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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY.DOCKET NO./TITLE	REQUEST ID
15/569,484	10/26/2017	Christian HANISCH	03101160US	145074

Acknowledgement of Change to Small Entity Status

The entity status change request below filed through Private PAIR on 08/06/2021 has been accepted.

CERTIFICATIONS:

Change of Entity Status:

X Applicant asserting small entity status. See 37 CFR 1.27.

NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

This portion must be completed by the signatory or signatories making the entity status change in accordance with 37 CFR 1.4(d)(4).

Signature:	/Michael E. Whitham/
Name:	Michael E. Whitham
Registration Number:	32635

Ascend v. Duesenfeld
PGR2025-00037
Ex. 2014-001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re

U.S. Patent 11,050,097 issued June 29, 2021

from

In re patent application of

HANISCH et al. Confirmation No. 9373

Application No. 15/569,484 Group Art Unit 3725

Filed October 26, 2017 Examiner Swiatocha

For METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

SMALL ENTITY STATUS

Sir:

The patentee, Duesenfeld GmbH, of U.S. Patent 11,050,097 is entitled to small entity status.

Please change the status of the patentee in the USPTO so that the patentee may pay small entity fees for the maintenance fees going forward.

Respectfully submitted,

/Michael E. Whitham/

Michael E. Whitham
Reg. No. 32,635

W&C IP
11491 Sunset Hills Road, Suite 340
Reston, VA 20190
Tel. (703) 787-9400
Fax. (703) 787-7557

Customer No.: 30743

Ascend Elements EX1002 Page 2

Duesenfeld Ex. 2014-002

Electronic Acknowledgement Receipt

EFS ID:	43447909
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Michael E. Whitham
Filer Authorized By:	
Attorney Docket Number:	03101160US
Receipt Date:	06-AUG-2021
Filing Date:	26-OCT-2017
Time Stamp:	14:40:06
Application Type:	U.S. National Stage under 35 USC 371

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Miscellaneous Incoming Letter	03101160US_8_6_21.pdf	15337 c9b5d34f6f175989c9a4f5d0efe7124b59c64b9f	no	1

Warnings:

Information:	
Total Files Size (in bytes):	15337

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111
If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.
National Stage of an International Application under 35 U.S.C. 371
If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.
New International Application Filed with the USPTO as a Receiving Office
If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	06/29/2021	11050097	03101160US	9373

30743 7590 06/09/2021

W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(application filed on or after May 29, 2000)

The Patent Term Adjustment is 344 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Data Management (ODM) at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

Christian HANISCH, Braunschweig, GERMANY;
DUESENFELD GMBH, Braunschweig, GERMANY;
Bastian WESTPHAL, Braunschweig, GERMANY;
Wolfgang HASELRIEDER, Braunschweig, GERMANY;
Martin SCHOENITZ, Braunschweig, GERMANY;

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Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (02-18)
Approved for use through 11/30/2020. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1					

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U.S.PATENT APPLICATION PUBLICATIONS Remove

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
Change(s) applied to document, /F.W./ 6/3/2021	1	20160049699	A1	2016-02-18	MITSUBISHI MATERIALS CORPORATION Hayashi et al.	

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FOREIGN PATENT DOCUMENTS Remove

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	106259062	CN	B	2015-09-09	UNIV TSINGHUA GRADUATE SCHOOL		☒
	2	4424825	DE	A1	1996-01-18	GABLER ING KONTOR LUEBECK		☒
	3	10-0665626	KR	B1	2007-01-09	REMETECH CO., LTD.		☒

EFS Web 2.1.18

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.D.S./

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	
First Named Inventor	
Art Unit	
Examiner Name	
Attorney Docket Number	

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	9780419	B2	2017-10-03	Hanisch, et al. ~~LION ENGINEERING GMBH~~	

Change(s) applied to document, /M.P.C./ 3/24/2021

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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20050241943	A1	2005-11-03	Kakuta et al. ~~KAWASAKI JUKOGYO KABUSHIKI KAISHA~~	

If you wish to add additional U.S. Published Application citation information please click the Add button. Add

FOREIGN PATENT DOCUMENTS Remove

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	2010/102377	WO	A1	2010-09-16	STEVENS, WAYNE, C.		
	2	10 2011 110 083	DE	A1	2013-02-14	TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA		
	3	10 2012 024 876	DE	A1	2014-06-26	ELECTROCYCLING GMBH; VOLKSWAGEN AKTIENGESELLSCHAFT		

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.P.C./

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

30743 7590 03/01/2021
W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

(Typed or printed name)
(Signature)
(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

TITLE OF INVENTION: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	06/01/2021

EXAMINER	ART UNIT	CLASS-SUBCLASS
SWIATOCHA, GREGORY D.	3799	241-024130

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 W&C IP
2
3

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: DUESENFELD GMBH

(B) RESIDENCE: (CITY and STATE OR COUNTRY): BRAUNSCHWEIG, DE

Please check the appropriate assignee category or categories (will not be printed on the patent) : ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☒ Issue Fee ☐ Publication Fee (if required) ☐ Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

☒ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☐ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature /Michael E. Whitham/ Date 2021-05-26

Typed or printed name Michael E. Whitham Registration No. 32635

Ascend Elements EX1002 Page 8

Duesenfeld Ex. 2014-008

Electronic Patent Application Fee Transmittal

Application Number:	15569484
Filing Date:	26-Oct-2017
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Michael E. Whitham/Kyle Comiskey
Attorney Docket Number:	03101160US

Filed as Large Entity

Filing Fees for U.S. National Stage under 35 USC 371

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
UTILITY APPL ISSUE FEE	1501	1	1200	1200

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension-of-Time:				
Miscellaneous:				
		Total in USD ($)		**1200**

Electronic Acknowledgement Receipt	
EFS ID:	42827374
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Michael E. Whitham/Kyle Comiskey
Filer Authorized By:	Michael E. Whitham
Attorney Docket Number:	03101160US
Receipt Date:	26-MAY-2021
Filing Date:	26-OCT-2017
Time Stamp:	15:17:04
Application Type:	U.S. National Stage under 35 USC 371

Payment information:

Submitted with Payment	yes
Payment Type	CARD
Payment was successfully received in RAM	$1200
RAM confirmation Number	E20215PF17161644
Deposit Account	
Authorized User	
The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Issue Fee Payment (PTO-85B)	03101160US_IFP.pdf	91873 8825bda3f6747d5bef671e12f8d884fc4a8166d1	no	1
Warnings:					
Information:					
2	Fee Worksheet (SB06)	fee-info.pdf	30761 d5e857e5c188dec0eb26302b70405a4c8c4ea2ce	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	122634		

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

30743 7590 04/23/2021

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11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

EXAMINER	
SWIATOCHA, GREGORY D.	

ART UNIT	PAPER NUMBER
3799	

MAIL DATE	DELIVERY MODE
04/23/2021	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Corrected ***Notice of Allowability***	**Application No.** 15/569,484	**Applicant(s)** HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	**Art Unit** 3799	**AIA (FITF) Status** Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to communications filed 19 April 2021.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 1-9 and 11-22 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information , please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☑ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   Certified copies:

   a) ☑ All b) ☐ Some *c) ☐ None of the:

   1. ☑ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)
2. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.
5. ☐ Examiner's Amendment/Comment
6. ☐ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3799

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (02-18)
Approved for use through 11/30/2020. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20160049699	A1	2016-02-18	MITSUBISHI MATERIALS CORPORATION	

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Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	106259062	CN	B	2015-09-09	UNIV TSINGHUA GRADUATE SCHOOL		☒
	2	4424825	DE	A1	1996-01-18	GABLER ING KONTOR LUEBECK		☒
	3	10-0665626	KR	B1	2007-01-09	REMETECH CO., LTD.		☒

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.D.S./

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

If you wish to add additional Foreign Patent Document citation information please click the Add button [Add]

NON-PATENT LITERATURE DOCUMENTS [Remove]

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1	DEHNERT ET AL: "Vacuum Distillation", ALLGEMEINE CHEMIE, 14.3, page 14, 1979	X
	2	WOLF ET AL: Allgemeine und Physikalische Chemie, CHEMIE, 1985	☒

If you wish to add additional non-patent literature document citation information please click the Add button [Add]

EXAMINER SIGNATURE

Examiner Signature	/GREGORY D SWIATOCHA/	Date Considered	04/22/2021

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

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Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2021-04-16
Name/Print	Michael E. Whitham	Registration Number	32635

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Application Number:	15569484
Filing Date:	26-Oct-2017
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Michael E. Whitham/Kyle Comiskey
Attorney Docket Number:	03101160US

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Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
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EFS ID:	42484161
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Michael E. Whitham/Kyle Comiskey
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Attorney Docket Number:	03101160US
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Warnings:					
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2	Foreign Reference	CN103259062B.PDF	519377 e9829085fd5da9e7af7d8aceefce2133a5289882	no	7
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DESCRIPTION CN103259062B

10 Abstract The invention discloses a method for recovering and regenerating graphene from a waste lithium ion battery, which is characterized in that it comprises the following steps: S1. Recovery of battery electrode active material: precharging or precharging a spent lithium ion battery using graphite as a negative electrode material After pre-discharge treatment, the cathode and anode coating is separated from the current collector and the outer packaging to obtain the cathode and anode blend materials; S2, the separation of the intercalated graphite electrode material: the cathode and anode blend materials are dried and crushed Afterwards, the active materials of the positive and negative electrodes are separated by physical methods to obtain intercalated graphite powder; S3, intercalated graphite is used to prepare graphene: the intercalated graphite powder is used as a raw material, and the redox method or ultrasonic peeling method is adopted Preparation of graphene. The invention can realize the industrial recovery of lithium ion batteries and the high value-added regeneration of graphite negative electrodes into graphene materials, has strong application prospects and feasibility, and can produce greater economic and social benefits.

22 Method for recycling and regenerating graphene from waste lithium ion batteries

23 Technical field

24 The present invention relates to the field of recycling and utilization of waste batteries, in particular to a method for recycling and regenerating graphene from waste lithium ion batteries.

26 Background technique

27 As energy and environmental issues become increasingly prominent, the development of new energy sources has become a major trend in energy strategy. And energy storage and conversion devices play a very important role in the development of new energy strategies. In particular, after nearly 20 years of development, lithium-ion batteries have become an indispensable secondary power source in people' s lives with their excellent performance, occupying a dominant position in the power sources of electronic consumer products such as mobile phones and computers. With the rapid development of power vehicles, power lithium-ion batteries have also ushered in huge development opportunities, and their usage and scale of use are constantly increasing. The huge amount of lithium-ion batteries used will inevitably face the problem of recycling. At present, the recycling of lithium-ion batteries mainly focuses on the recovery of positive active materials such as transition metals such as cobalt, iron, and manganese, and the recovery of positive and negative current collectors, copper and aluminum. For graphite commonly used as anode materials, due to its low cost, wide

Ascend Elements EX1002 Page 28

Duesenfeld Ex. 2014-028

range of graphite sources, and low added value of recycling, the recycling technology and industry for graphite are not yet complete. However, carbon materials play an important role in the national economy and national defense security, and are also important precursors of various other carbon materials. In particular, the graphite material in lithium-ion batteries has undergone pre-purification, and its purity is much higher than that of graphite in graphite ore. Therefore, if it is possible to develop low-cost graphite recovery technology in the process of recovering metal elements, it will The obtained graphite is further recycled, which will improve the overall improvement and diversification of the lithium-ion battery recycling industry and downstream applications. At present, there are also related reports on the recovery of graphite electrodes in lithium-ion batteries. For example, patent CN101154757A discloses a carbon anode recovery method, which uses organic solvents to separate the anode material from the copper foil, and then sinters the anode material at high temperature to remove the binder Obtain a carbonaceous material. Patent CN1758478A directly uses a high-temperature sintering method to decompose the binder to achieve the separation between the negative electrode material and the current collector. Patent CN102709621A uses the method of shaking and washing to separate the negative electrode material from the current collector, and uses high-temperature sintering to purify the current collector, reducing the concentration of impurities, and obtaining high-purity graphite materials. Although the above-mentioned patents have certain characteristics in their respective recycling technologies, there is no special treatment for the lithium-carbon compounds after charging and discharging. At the same time, the recycling method is to first take out the negative electrode and then recycle it. This is not in industrial applications. It is not feasible. The precise separation and recovery of different components in the development of integrated recycling of lithium-ion batteries will be an important issue.

58 Graphene is a free-state two-dimensional crystal. It has good electrical, mechanical, optical and other properties. It has shown broad application prospects in the field of chips, energy storage devices, and biomedicine. At present, the large-scale production of graphene is mainly through the oxidation-reduction method of graphite, the graphite is oxidized to obtain graphene oxide, and then the graphene is obtained through chemical reduction or thermal cleavage. The graphite anode in lithium-ion batteries is suitable for intercalation due to its high purity and interlayer structure. If it can be recycled and then applied to the preparation of graphene, it will greatly improve the diversification of its industry and additional economic benefits.

66 Summary of the invention

67 The technical problem to be solved by the present invention is to provide a method for recycling and regenerating graphene from waste lithium ion batteries that is suitable for industrialization, low cost, environmentally friendly and economical.

70 The present invention solves the above technical problems through the following technical means:

71 A method for recycling and regenerating graphene from waste lithium ion batteries includes the following steps:

73 S1. Recovery of battery electrode active material: pre-charge or pre-discharge the waste lithium-ion battery using graphite as the negative electrode material, and then separate the cathode and anode coating from the current collector and the outer packaging to obtain a cathode and anode blend material ;

76 S2. Separation of intercalated graphite electrode materials: After drying and crushing the cathode and anode blend materials obtained in S1, physical methods are used to separate the active materials of the positive and negative electrodes to obtain intercalated graphite powder;

79 S3. Preparation of graphene from intercalated graphite: the intercalated graphite powder is used as a raw

material, and graphene is prepared by a redox method or an ultrasonic peeling method.

81 Since the present invention belongs to the field of recycling and reuse of waste batteries, the invention can be made practical only when the cost of the entire recycling and regeneration process is low. Otherwise, even if the recycling of waste is solved, the recycling cost is high, and it is also impossible. Realize its value. Compared with the prior art, the present invention can realize the industrial recovery of lithium-ion batteries and the high value-added regeneration of graphite anodes into graphene materials. It has strong application prospects and feasibility, and can produce greater economic benefits and Social benefits are in line with the strategy of sustainable development and are conducive to the improvement and diversification of the recycling industry chain of used lithium-ion batteries. Moreover, the present invention first performs charging or discharging treatment on the lithium ion battery, which utilizes the intercalation of lithium ions during the charging and discharging process of graphite for lithium ion batteries, thereby causing a certain degree of expansion between the graphite layers, which is beneficial to graphite Graphene is prepared by oxidizing or exfoliating lithium intercalated graphite by strong ultrasonic, and the prepared graphene has more excellent properties.

93 Preferably, in the step S1, the waste lithium ion battery using graphite as the negative electrode material is pre-discharged to below 3V ; the step S3 includes the following steps: the intercalated graphite powder obtained in step S2 is combined with a strong oxidizing substance Mixed oxidation is used to prepare graphite oxide, and then the graphite oxide is expanded to obtain graphene by means of thermal expansion. This preferred solution uses the charge and discharge process to expand the graphite layers to a certain extent, and at the same time, it can eliminate the lithium ions between the graphite layers as much as possible. Therefore, on the one hand, the expansion of the layers can facilitate the oxidation of graphite, and on the other hand, it can ensure the preparation of The high purity of graphene products.

101 Preferably, in the step S1, the waste lithium ion battery using graphite as the negative electrode material is fully charged; the step S3 includes the following steps: the intercalated graphite powder obtained in the step S2 is subjected to ultrasonic peeling treatment in an organic solvent, Then, it is washed and dried to obtain graphene. Compared with the discharge process, lithium ions are intercalated between the graphite layers during the charging process, which increases the spacing between the graphite layers, which is more conducive to the preparation of graphene by ultrasonic peeling. Therefore, when the graphene is prepared by ultrasonic peeling, the precharge treatment can be relatively For pre-discharge treatment to obtain better results.

109 Preferably, the step of separating the cathode and anode coating from the current collector and the outer packaging in step S1 includes surface cleaning, mechanical cutting, cleaning and shaking, and filtering; the cleaning liquid used in the cleaning and shaking is liquid carbonate, ethanol , One or a mixture of two or more in acetone.

113 Preferably, the baking temperature used for drying the filter cake in the step S2 is 45-500° C, the pressure is less than 80K Pa, and the time is 30min-20h.

115 Preferably, the physical method described in step S2 is airflow separation or buoyancy separation.

116 The airflow separation method includes the following steps: the powder obtained by drying and pulverizing the filter cake is added from the middle of the separation tube, and at the same time, an airflow is introduced into the lower end of the separation tube. Under the combined action of gravity and airflow blowing force, the density The large positive electrode active material particles decrease, and the density of the intercalated graphite powder increases to achieve the purpose of separation.

121 The physical separation method includes the following steps: adding the powder obtained after drying and

Ascend Elements EX1002 Page 30

Duesenfeld Ex. 2014-030

pulverizing the filter cake to a specific solvent, and under the combined action of gravity and the buoyancy of the solvent, the particles of the positive electrode active material with high density decrease and the density is small The intercalated graphite powder rises to achieve the purpose of separation; the specific solvent is one or a mixture of two or more of water, acetone, concentrated sulfuric acid, and concentrated nitric acid

126 In industrial production, it is difficult to accurately separate the positive electrode sheet and the negative electrode sheet due to the cost control. Therefore, the existing methods all require the negative electrode sheet to be taken out in advance and then recycled. The above-mentioned preferred solution uses the physical separation method after overall mixing. Realize fast and accurate separation at low cost and improve recovery efficiency.

131 Preferably, the strong oxidizing substance is one or a mixture of two or more of potassium permanganate, sulfuric acid, nitric acid, perchloric acid, concentrated sulfuric acid, sodium nitrate, and potassium perchlorate; the blended oxidation includes the following steps : Mix the intercalated graphite powder obtained in step S2 with a strong oxidizing substance, stir and react at a low temperature of -10-10° C for 0.5-20 hours; then react at a medium temperature of 30-60° C for 0.5-5 Hours; finally, add deionized water and react at a high temperature of 90-100° C for 0.5-5 hours; after the reaction, the resulting solution is filtered and washed, and then the resulting graphite oxide is dried and ground.

138 The drying adopts atmospheric drying, vacuum drying or freeze drying.

139 Preferably, in step S3, expanding the graphite oxide to obtain graphene by means of thermal expansion includes the following steps: placing the graphite oxide in a vacuum tube furnace under a protective atmosphere, and controlling the pressure in the tube furnace to be 1Pa-101KPa, The temperature is 100° C-1000° C, the graphite oxide is heated to expand for 10s-12h, and exfoliated into graphene; the protective atmosphere is a mixed atmosphere of one or more of nitrogen, argon, helium, and hydrogen.

144 Preferably, the processing time of the ultrasonic peeling treatment in the step S3 is 10s-12h, the ultrasonic power is 20W-2kW, and the organic solvent is esters, ethers, alcohols, ketones, N-methylpyrrolidone, N , One or a mixture of two or more of N-dimethylformamide, the drying adopts freeze drying or vacuum drying.

148 detailed description

149 The present invention will be further described below in conjunction with preferred embodiments.

150 Example one

151 Discharge the recovered soft-packed lithium cobaltate/graphite battery to 0V , clean the surface of the battery with ethanol, cut it into several pieces with a mechanical cutting machine, and place them in dimethyl carbonate (DMC) by ultrasonic vibration filtration, and collect the The fluid, isolation film, tabs and packaging bags are removed to obtain the coating slurry; the liquid components are filtered again to obtain a filter cake, which is dried at 45° C and 1KPa pressure to obtain a powder of positive and negative active materials blend , And then pulverize the mixed powder of positive and negative active materials for 20 minutes. Then through the air separation tube, under the action of gravity and air blowing force, the higher density lithium cobalt oxide sinks and the lower density graphite floats to realize the separation of the two, and the intercalated graphite material is recovered;

160 Take 20g of the obtained intercalated graphite, add 10g sodium nitrate, 400mL concentrated sulfuric acid, stir in an ice water bath and slowly add 80g potassium permanganate, react at low temperature for 2 hours; increase the temperature to 35° C, continue the reaction for half an hour, add to the system Slowly add 700 mL of deionized water, and then react at a high temperature of 98° C for 2 hours, and the reaction is

Ascend Elements EX1002 Page 31

Duesenfeld Ex. 2014-031

complete. Add 100 mL of hydrogen peroxide to the solution, stir for 5 minutes, vacuum filter, pickled with 5% hydrochloric acid for 3 times, take the filter cake for centrifugal washing value neutral, and place the precipitate in a vacuum oven at 50° C for 48 hours. Take out, grind and smash. The obtained graphite oxide powder is placed in a vacuum tube furnace, the pressure is 100 Pa, and the temperature is increased to 300° C at a rate of 10° C/min, and maintained for 3 hours. The graphite oxide is pyrolyzed and the volume expands violently to obtain a graphene material.

170 Example two

171 Discharge the recovered soft-packed lithium cobaltate/graphite battery to 3V, clean the surface of the battery with ethanol, cut it into several pieces with a mechanical cutting machine, and place them in dimethyl carbonate (DMC) by ultrasonic vibration filtering, and collect the The fluid, isolation film, tabs and packaging bags are removed to obtain the coating slurry; the liquid components are filtered again to obtain a filter cake, which is dried at 200° C and 30KPa pressure to obtain a powder of positive and negative active materials blended , And then pulverize the mixed powder of positive and negative active materials for 10 minutes. Then through the air separation tube, under the action of gravity and air blowing force, the higher density lithium cobalt oxide sinks and the lower density graphite floats to realize the separation of the two, and the intercalated graphite material is recovered;

180 Take 20g of the obtained intercalated graphite, add 10g sodium nitrate, 400mL concentrated sulfuric acid, stir in an ice water bath and slowly add 80g potassium permanganate, react at low temperature for 0.5 hours; increase the temperature to 30° C, continue the reaction for 5 hours, and add to the system Slowly add 700 mL of deionized water, and then react at a high temperature of 98° C for 2 hours, and the reaction is complete. Add 100mL of hydrogen peroxide to the solution, stir for 5 minutes, vacuum filter, pickled with 5% hydrochloric acid for 3 times, take the filter cake for centrifugal washing, and place the precipitate in a 70° C atmospheric oven for 48 hours. Take out, grind and smash. The obtained graphite oxide powder is placed in a vacuum tube furnace, the pressure is 20 Pa, and the temperature is raised to 300° C at a rate of 10° C/min, and kept for 3 hours. The graphite oxide is pyrolyzed and the volume expands violently to obtain a graphene material.

190 Example three

191 Discharge the recovered soft-packed lithium cobaltate/graphite battery to 1.0V, clean the surface of the battery with ethanol, cut it into several pieces with a mechanical cutting machine, put them in acetone and filter with ultrasonic vibration, and remove the current collector, isolation membrane, The tabs and packaging bags are removed to obtain the coating slurry; the liquid components are filtered again to obtain the filter cake, which is dried at 500° C and 30KPa pressure to obtain the powder of the positive and negative active material blends, and then the positive and negative electrodes are crushed 30min for powder blended with extremely active materials. Then through the air separation tube, under the action of gravity and air blowing force, the higher density lithium cobalt oxide sinks and the lower density graphite floats to realize the separation of the two, and the intercalated graphite material is recovered;

200 Take 20g of the obtained intercalated graphite, add 10g sodium nitrate, 400mL concentrated sulfuric acid, stir in an ice water bath and slowly add 80g potassium permanganate, react at low temperature for 20 hours; increase the temperature to 60° C, continue the reaction for half an hour, add to the system Slowly add 700 mL of deionized water, and then react at a high temperature of 90° C for 5 hours, and the reaction is complete. Add 100mL of hydrogen peroxide to the solution, stir for 5 minutes, vacuum filter, pickled with 5% hydrochloric acid for 3 times, take the filter cake for centrifugal washing, and place the precipitate in a

70° C atmospheric oven for 48 hours. Take out, grind and smash. The obtained graphite oxide powder is placed in a vacuum tube furnace at a pressure of 20 Pa and a temperature of 1000° C., and placed for 10 seconds, the graphite oxide is pyrolyzed and the volume expands violently to obtain graphene material.

209 Example four

210 Discharge the recovered soft-packed lithium cobaltate/graphite battery to 0V, clean the surface of the battery with ethanol, cut it into several pieces with a mechanical cutting machine, and place them in dimethyl carbonate (DMC) by ultrasonic vibration filtration, and collect the The fluid, isolation film, tabs and packaging bags are removed to obtain the coating slurry; the liquid components are filtered again to obtain a filter cake, which is dried at 45° C and 1KPa pressure to obtain a powder of positive and negative active materials blend , And then pulverize the mixed powder of positive and negative active materials for 20 minutes. Then the powder material is placed in a beaker filled with water and shaken, the higher density lithium cobalt oxide sinks, and the lower density graphite floats to separate the two, and the intercalated graphite material is recovered;

219 Take 16g of the obtained intercalated graphite, add 8g sodium nitrate, 400mL concentrated sulfuric acid, stir in an ice water bath and slowly add 70g potassium permanganate, react at low temperature for 2 hours; increase the temperature to 35° C, continue the reaction for half an hour, add to the system Slowly add 700 mL of deionized water, and then react at a high temperature of 98° C for 2 hours, and the reaction is complete. Add 90 mL of hydrogen peroxide to the solution, stir for 5 minutes, vacuum filter, pickled with 5% hydrochloric acid for 3 times, take the filter cake for centrifugal washing, and place the precipitate in a 70° C atmospheric oven for 48 hours. Remove and grind. The obtained graphite oxide powder is placed in a vacuum tube furnace, the pressure is 20 Pa, and the temperature is raised to 300° C at a rate of 10° C/min, and kept for 3 hours. The graphite oxide is pyrolyzed and the volume expands violently to obtain a graphene material.

229 Example five

230 The process and conditions of this embodiment are the same as those of the fourth embodiment, except that the washing method of graphite oxide is changed to suction filtration and washing until there is no SO4<2-> in the filtrate, and the subsequent steps are the same.

233 Example Six

234 This embodiment has the same process and conditions as the fourth embodiment, except that the drying method of graphite oxide is changed to freeze drying.

236 Example Seven

237 Fully charge the recovered soft-packed lithium cobaltate/graphite battery, clean the surface of the battery with ethanol, cut it into several pieces with a mechanical cutting machine, and place them in dimethyl carbonate (DMC) by ultrasonic vibration filtering, and the current collector , Isolation film, tabs and packaging bags are removed to obtain the coating slurry; filter again to remove the liquid components to obtain a filter cake, which is dried at 45° C and 1KPa pressure to obtain a powder of positive and negative active material blends. Then pulverize the mixed powder of the positive and negative active materials for 20 minutes. Then through the air separation tube, under the action of gravity and air blowing force, the higher density lithium cobalt oxide sinks and the lower density graphite floats to separate the two, and the lithium intercalation graphite material is recovered;

246 The obtained lithium intercalated graphite was sonicated in a PC solvent for 30 minutes with an ultrasonic power of 100 W, and then the obtained material was filtered and cleaned with ethanol and deionized water,

Ascend Elements EX1002 Page 33

Duesenfeld Ex. 2014-033

collected and then freeze-dried to obtain a graphene material.

249 Example eight

250 This embodiment has the same process and conditions as the seventh embodiment, except that the ultrasonic solvent is changed to DMF, the ultrasonic time is 2 hours, the power is 200W, and the drying method is vacuum drying.

253 Example 9

254 The process and conditions of this embodiment and the fourth embodiment are basically the same. The only difference is that the low-temperature reaction time of this embodiment is 15 hours, and the graphene material can also be obtained.

257 The above content is a further detailed description of the present invention in conjunction with specific preferred embodiments, and it cannot be considered that the specific implementation of the present invention is limited to these descriptions. For those skilled in the art to which the present invention belongs, without departing from the concept of the present invention, several equivalent substitutions or obvious modifications can be made, and the same performance or use should be regarded as belonging to the protection scope of the present invention.

Ascend Elements EX1002 Page 34

Duesenfeld Ex. 2014-034

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权利要求书1页 说明书5页

(54)发明名称

一种废旧锂离子电池回收再生石墨烯的方法

(57)摘要

本发明公开了一种废旧锂离子电池回收再生石墨烯的方法，其特征在于，包括以下步骤：S1、电池电极活性物质的回收：将使用石墨作为负极材料的废旧锂离子电池进行预充电或预放电处理，之后将阴阳极涂层与集流体、外包装分离开来，得到阴阳极共混物料；S2、插层化石墨电极材料的分离：将所述阴阳极共混物料烘干并粉碎后，采用物理方法将正负电极活性物质分离得到插层化石墨粉料；S3、插层化石墨制备石墨烯：以所述插层化石墨粉料作为原料，采用氧化还原法或超声剥离法制备获得石墨烯。本发明可实现锂离子电池的产业化回收以及石墨负极的高附加值再生为石墨烯材料，具有较强的应用前景和可行性，可产生较大的经济效益和社会效益。

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1. 一种废旧锂离子电池回收再生石墨烯的方法，其特征在于，包括以下步骤：

S1、电池电极活性物质的回收：将使用石墨作为负极材料的废旧锂离子电池进行预放电至 3V 以下，之后将阴阳极涂层与集流体、外包装分离开来，得到阴阳极共混物料；

S2、插层化石墨电极材料的分离：将阴阳极共混物料烘干并粉碎后，采用气流分离法或浮力分离法将正负电极活性物质分离得到插层化石墨粉料；

S3、插层化石墨制备石墨烯：将步骤 S2 得到的插层化石墨粉料与强氧化性物质共混氧化，制备得到氧化石墨，再通过热膨胀方式将所述氧化石墨膨化制备得到石墨烯。

2. 根据权利要求 1 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述步骤 S1 中阴阳极涂层与集流体、外包装分离开来的步骤包括表面清洗、机械切割、清洗震荡以及过滤；

所述清洗震荡所使用的清洗液为液态碳酸酯、乙醇、丙酮中的一种或两种以上的混合物。

3. 根据权利要求 1 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述步骤 S2 中将所述阴阳极共混物料烘干所采用的烘烤温度为 45-500℃、压力小于 80KPa、时间为 30min-20h。

4. 根据权利要求 1 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述气流分离法包括以下步骤：将所述阴阳极共混物料烘干并粉碎后得到的粉料从分离管中间加入，同时在分离管下端通入气流，在重力与气流吹力的共同作用下，密度大的正极活性物质颗粒下降、密度小的插层化石墨粉料上升从而实现分离的目的。

5. 根据权利要求 1 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述浮力分离法包括以下步骤：将所述阴阳极共混物料烘干并粉碎后得到的粉料加入特定溶剂中，在重力与溶剂浮力的共同作用下，密度大的正极活性物质颗粒下降、密度小的插层化石墨粉料上升从而实现分离的目的；

所述特定溶剂为水、丙酮、浓硫酸、浓硝酸中的一种或两种以上的混合物。

6. 根据权利要求 1 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述强氧化性物质为高锰酸钾、硫酸、硝酸、高氯酸、硝酸钠、高氯酸钾中的一种或两种以上的混合物；

所述步骤 S3 中所述共混氧化包括以下步骤：将步骤 S2 得到的插层化石墨粉料与强氧化性物质混合，在 -10℃ -10℃的低温下搅拌反应 0.5-20 小时；然后在 30-60℃的中温下反应 0.5-5 小时；最后加入去离子水在 90-100℃的高温下反应 0.5-5 小时；反应结束后将所得溶液进行过滤、洗涤，然后将得到的氧化石墨进行干燥、研磨。

7. 根据权利要求 1 或 6 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述步骤 S3 中通过热膨胀方式将所述氧化石墨膨化制备得到石墨烯包括以下步骤：将氧化石墨在保护气氛下置于真空管式炉中，控制管式炉内压力为 1Pa-101KPa、温度为 100℃ -1000℃，使氧化石墨受热膨胀 10s-12h，剥离为石墨烯；

所述保护气氛为氮气、氩气、氦气、氢气中的一种或两种以上的混合气氛。

8. 根据权利要求 6 所述的废旧锂离子电池回收再生石墨烯的方法，其特征在于：

所述干燥采用常压干燥、真空干燥或冷冻干燥。

Ascend Elements EX1002 Page 36

Duesenfeld Ex. 2014-036

一种废旧锂离子电池回收再生石墨烯的方法

技术领域

[0001] 本发明涉及废旧电池的回收利用领域，尤其是涉及一种废旧锂离子电池回收再生石墨烯的方法。

背景技术

[0002] 随着能源与环境问题的日益凸显，开发新型能源已成为能源战略的一大趋势。而能量的储存与转化器件在发展新能源战略中具有十分重要的角色。特别是锂离子电池经过近 20 年的发展，以其优异的性能，已成为人们生活中不可或缺的二次电源，在电子消费产品如手机、电脑的电源中占据主导地位。随着动力汽车的快速发展，动力锂离子电池也迎来了巨大的发展契机，其使用量和使用规模都在不断的增加。巨大的锂离子电池使用量必然会面临循环利用的问题，目前对于锂离子电池的回收主要集中在正极活性物质如钴、铁、锰等过渡金属以及正负极集流体铜、铝的回收。对于负极材料常用的石墨，由于其成本较低、石墨来源广泛、回收附加值较低，针对石墨的回收技术与产业尚不完善。然而碳材料在国民经济和国防安全中扮演着重要的角色，同时也是各种其他碳质材料的重要前躯体。特别是锂离子电池中的石墨材料已经经过前期的纯化，其纯度要远高于石墨矿中的石墨纯度，因此，如果可以在回收金属元素的过程中同时开发低成本的石墨回收技术，同时将所得石墨进一步循环利用，将整体提升锂离子电池回收产业和下游应用的完善化和多元化。目前对于锂离子电池石墨电极的回收也有相关报道，如专利 CN101154757A 公开了一种碳负极回收方法，利用有机溶剂将负极材料与铜箔分离开来，进而对负极材料进行高温烧结，除去粘结剂得到碳质材料。专利 CN1758478A 直接利用高温烧结的方法将粘结剂分解，进而达到负极材料与集流体之间的分离。专利 CN102709621A 利用震荡水洗的方法将负极材料与集流体进行分离，并利用高温烧结对集流体进行纯化，降低了杂质的浓度，得到高纯度的石墨材料。上述专利虽然在各自的回收技术上都有一定的特点，但是对充放电过后的锂碳化合物并没有特殊处理，同时回收方法都是首先将负极片取出再进行回收，这在产业化应用中并不可行，开发锂离子电池一体化回收过程中对不同组分精确分离回收将是重要课题。

[0003] 石墨烯，是一种自由态二维晶体，它具有良好的电学、力学、光学等性质，在芯片领域、储能器件、生物医药等各领域都显示出广阔的应用前景。目前石墨烯的规模化生产主要是通过石墨的氧化还原方法，将石墨进行氧化得到氧化石墨烯，进而通过化学还原或热解理的方法得到石墨烯。而锂离子电池中的石墨负极由于其纯度较高、层间结构适宜插层，如能进行回收并进而应用到石墨烯的制备中，将大大提高其产业的多元化和附加经济效益。

发明内容

[0004] 本发明所要解决的技术问题是，提出一种适宜产业化，成本低廉，环保经济的废旧锂离子电池回收再生石墨烯的方法。

[0005] 本发明通过以下技术手段解决上述技术问题：

[0006] 一种废旧锂离子电池回收再生石墨烯的方法，包括以下步骤：

Ascend Elements EX1002 Page 37

Duesenfeld Ex. 2014-037

[0007] S1、电池电极活性物质的回收：将使用石墨作为负极材料的废旧锂离子电池进行预充电或预放电处理，之后将阴阳极涂层与集流体、外包装分离开来，得到阴阳极共混物料；

[0008] S2、插层化石墨电极材料的分离：将S1所得阴阳极共混物料烘干并粉碎后，采用物理方法将正负电极活性物质分离得到插层化石墨粉料；

[0009] S3、插层化石墨制备石墨烯：以所述插层化石墨粉料作为原料，采用氧化还原法或超声剥离法制备获得石墨烯。

[0010] 由于本发明属于废旧电池回收和再利用的领域，只有当整个回收和再生的过程成本较低时才能使得该发明具有实际意义，否则即使解决了废物的回收再生，但回收成本较高，同样无法实现其价值。而与现有技术相比，本发明可实现锂离子电池的产业化回收以及石墨负极的高附加值再生为石墨烯材料，具有较强的应用前景和可行性，可产生较大的经济效益和社会效益，符合可持续发展战略，有利于废旧锂离子电池回收产业链的完善化和多元化。而且，本发明首先对锂离子电池进行充电或放电处理，其利用锂离子电池用石墨在充放电过程中会存在锂离子的插层，从而使得石墨层间发生一定程度的扩张，有利于石墨的氧化或者通过强超声剥离锂插层石墨制备石墨烯，且制备得到的石墨烯具有更加优良的性质。

[0011] 优选地，所述步骤S1中将使用石墨作为负极材料的废旧锂离子电池预放电至3V以下；所述步骤S3包括以下步骤：将步骤S2得到的插层化石墨粉料与强氧化性物质共混氧化，制备得到氧化石墨，再通过热膨胀方式将所述氧化石墨膨化制备得到石墨烯。本优选方案利用充放电过程使石墨层间发生一定程度的扩张，同时又能够尽量的排除石墨层间的锂离子，因而一方面层间扩张能够有利于石墨的氧化，另一方面能保证制备的石墨烯产品的高纯度。

[0012] 优选地，所述步骤S1中将使用石墨作为负极材料的废旧锂离子电池充满电；所述步骤S3包括以下步骤：将步骤S2得到的插层化石墨粉料在有机溶剂中进行超声剥离处理，然后清洗、干燥得到石墨烯。相比于放电过程，充电过程中锂离子嵌入到石墨层间，增大了石墨层间距，其更加有利于利用超声剥离制备石墨烯，因而在采用超声剥离制备石墨烯时，预充电处理能够相对于预放电处理获得更优的效果。

[0013] 优选地，所述步骤S1中阴阳极涂层与集流体、外包装分离开来的步骤包括表面清洗、机械切割、清洗震荡以及过滤；所述清洗震荡所使用的清洗液为液态碳酸酯、乙醇、丙酮中的一种或两种以上的混合物。

[0014] 优选地，所述步骤S2中将所述滤饼烘干所采用的烘烤温度为45-500℃、压力小于80KPa、时间为30min-20h。

[0015] 优选地，所述步骤S2中所述的物理方法为气流分离法或浮力分离。

[0016] 所述气流分离法包括以下步骤：将所述滤饼烘干并粉碎后得到的粉料从分离管中间加入，同时在分离管下端通入气流，在重力与气流吹力的共同作用下，密度大的正极活性物质颗粒下降、密度小的插层化石墨粉料上升从而实现分离的目的。

[0017] 所述物理分离方法包括以下步骤：将所述滤饼烘干并粉碎后得到的粉料加入特定溶剂中，在重力与溶剂浮力的共同作用下，密度大的正极活性物质颗粒下降、密度小的插层化石墨粉料上升从而实现分离的目的；所述特定溶剂为水、丙酮、浓硫酸、浓硝酸中的一种

Ascend Elements EX1002 Page 38

Duesenfeld Ex. 2014-038

或两种以上的混合物

[0018] 在工业化生产中，由于成本的控制很难精确地将正极片和负极片完全分开，因此现有方法均需要预先将负极片取出再进行回收，而上述优选方案利用整体混合后的物理分离法能够低成本地实现快速准确的分离，提高了回收效率。

[0019] 优选地，所述强氧化性物质为高锰酸钾、硫酸、硝酸、高氯酸、浓硫酸、硝酸钠、高氯酸钾中的一种或两种以上的混合物；所述共混氧化包括以下步骤：将步骤 S2 得到的插层化石墨粉料与强氧化性物质混合，在 -10-10℃的低温下搅拌反应 0.5-20 小时；然后在 30-60℃的中温下反应 0.5-5 小时；最后加入去离子水在 90-100℃的高温下反应 0.5-5 小时；反应结束后将所得溶液进行过滤、洗涤，然后将得到的氧化石墨进行干燥、研磨。

[0020] 所述干燥采用常压干燥、真空干燥或冷冻干燥。

[0021] 优选地，所述步骤 S3 中通过热膨胀方式将所述氧化石墨膨化制备得到石墨烯包括以下步骤：将氧化石墨在保护气氛下置于真空管式炉中，控制管式炉内压力为 1Pa-101KPa、温度为 100℃ -1000℃，使氧化石墨受热膨胀 10s-12h，剥离为石墨烯；所述保护气氛为氮气、氩气、氦气、氢气中的一种或两种以上的混合气氛。

[0022] 优选地，所述步骤 S3 中超声剥离处理的处理时间为 10s-12h，超声功率为 20W-2kW，所述有机溶剂为酯类、醚类、醇类、酮类、N- 甲基吡咯烷酮、N,N- 二甲基甲酰胺中的一种或两种以上的混合物，所述干燥采用冷冻干燥或真空干燥。

具体实施方式

[0023] 下面结合优选的实施方式对本发明作进一步说明。

[0024] 实施例一

[0025] 将回收的软包钴酸锂 / 石墨电池放电至 0V，利用乙醇清洗电池表面，利用机械切割机将其切割为若干块，放入碳酸二甲酯 (DMC) 中超声振荡过滤，将其中的集流体、隔离膜、极耳以及包装袋去除得到涂层浆料；再次过滤去除其中的液态组分得到滤饼，在 45℃，1KPa 压力下烘干得到正负极活性物质共混的粉料，然后粉碎正负极活性物质共混的粉料 20min。之后通过气流分离管，在重力和气流吹力的作用下，密度较大的钴酸锂下沉、密度较小的石墨上浮从而实现两者的分离，回收得到插层化石墨材料；

[0026] 取所得插层化石墨 20g，加入 10g 硝酸钠，400mL 浓硫酸，在冰水浴中搅拌并缓慢加入 80g 高锰酸钾，低温反应 2 小时；升温至 35℃，继续反应半小时，向体系中缓慢加入 700mL 去离子水，而后在 98℃高温反应 2 小时，反应结束。向溶液中加入 100mL 双氧水，搅拌 5 分钟后进行真空抽滤，利用 5% 盐酸酸洗 3 次后，取滤饼进行离心洗涤值中性，将沉淀置于 50℃真空烘箱烘干 48 小时，取出研磨粉碎。将所得氧化石墨粉末置于真空管式炉中，压力 100Pa，以 10℃ /min 速率升温至 300℃，保持 3 小时，氧化石墨发生热解理，体积剧烈膨胀，得到石墨烯材料。

[0027] 实施例二

[0028] 将回收的软包钴酸锂 / 石墨电池放电至 3V，利用乙醇清洗电池表面，利用机械切割机将其切割为若干块，放入碳酸二甲酯 (DMC) 中超声振荡过滤，将其中的集流体、隔离膜、极耳以及包装袋去除得到涂层浆料；再次过滤去除其中的液态组分得到滤饼，在 200℃，30KPa 压力下烘干得到正负极活性物质共混的粉料，然后粉碎正负极活性物质共混

Ascend Elements EX1002 Page 39

Duesenfeld Ex. 2014-039

的粉料10min。之后通过气流分离管，在重力和气流吹力的作用下，密度较大的钴酸锂下沉、密度较小的石墨上浮从而实现两者的分离，回收得到插层化石墨材料；

[0029] 取所得插层化石墨20g，加入10g硝酸钠，400mL浓硫酸，在冰水浴中搅拌并缓慢加入80g高锰酸钾，低温反应0.5小时；升温至30℃，继续反应5小时，向体系中缓慢加入700mL去离子水，而后在98℃高温反应2小时，反应结束。向溶液中加入100mL双氧水，搅拌5分钟后进行真空抽滤，利用5%盐酸酸洗3次后，取滤饼进行离心洗涤值中性，将沉淀置于70℃常压烘箱中48小时，取出研磨粉碎。将所得氧化石墨粉末置于真空管式炉中，压力20Pa，以10℃/min速率升温至300℃，保持3小时，氧化石墨发生热解理，体积剧烈膨胀，得到石墨烯材料。

[0030] 实施例三

[0031] 将回收的软包钴酸锂/石墨电池放电至1.0V，利用乙醇清洗电池表面，利用机械切割机将其切割为若干块，放入丙酮中超声振荡过滤，将其中的集流体、隔离膜、极耳以及包装袋去除得到涂层浆料；再次过滤去除其中的液态组分得到滤饼，在500℃，30KPa压力下烘干得到正负极活性物质共混的粉料，然后粉碎正负极活性物质共混的粉料30min。之后通过气流分离管，在重力和气流吹力的作用下，密度较大的钴酸锂下沉、密度较小的石墨上浮从而实现两者的分离，回收得到插层化石墨材料；

[0032] 取所得插层化石墨20g，加入10g硝酸钠，400mL浓硫酸，在冰水浴中搅拌并缓慢加入80g高锰酸钾，低温反应20小时；升温至60℃，继续反应半小时，向体系中缓慢加入700mL去离子水，而后在90℃高温反应5小时，反应结束。向溶液中加入100mL双氧水，搅拌5分钟后进行真空抽滤，利用5%盐酸酸洗3次后，取滤饼进行离心洗涤值中性，将沉淀置于70℃常压烘箱中48小时，取出研磨粉碎。将所得氧化石墨粉末置于真空管式炉中，压力20Pa，温度为1000℃，放入10s，氧化石墨发生热解理，体积剧烈膨胀，得到石墨烯材料。

[0033] 实施例四

[0034] 将回收的软包钴酸锂/石墨电池放电至0V，利用乙醇清洗电池表面，利用机械切割机将其切割为若干块，放入碳酸二甲酯(DMC)中超声振荡过滤，将其中的集流体、隔离膜、极耳以及包装袋去除得到涂层浆料；再次过滤去除其中的液态组分得到滤饼，在45℃，1KPa压力下烘干得到正负极活性物质共混的粉料，然后粉碎正负极活性物质共混的粉料20min。然后将粉体材料置于装有水的烧杯中，加以震荡，密度较大的钴酸锂下沉、密度较小的石墨上浮从而实现两者的分离，回收得到插层化石墨材料；

[0035] 取所得插层化石墨16g，加入8g硝酸钠，400mL浓硫酸，在冰水浴中搅拌并缓慢加入70g高锰酸钾，低温反应2小时；升温至35℃，继续反应半小时，向体系中缓慢加入700mL去离子水，而后在98℃高温反应2小时，反应结束。向溶液中加入90mL双氧水，搅拌5分钟后进行真空抽滤，利用5%盐酸酸洗3次后，取滤饼进行离心洗涤值中性，将沉淀置于70℃常压烘箱中48小时，取出研磨粉碎。将所得氧化石墨粉末置于真空管式炉中，压力20Pa，以10℃/min速率升温至300℃，保持3小时，氧化石墨发生热解理，体积剧烈膨胀，得到石墨烯材料。

[0036] 实施例五

[0037] 本实施例与实施例四过程与条件相同，只是将氧化石墨的洗涤方式变为抽滤洗涤至滤液中无SO_4^{2-}，后续步骤相同。

Ascend Elements EX1002 Page 40

Duesenfeld Ex. 2014-040

[0038] 实施例六

[0039] 本实施例与实施例四过程与条件相同，只是将氧化石墨的烘干方式改为冷冻干燥。

[0040] 实施例七

[0041] 将回收的软包钴酸锂 / 石墨电池充满电，利用乙醇清洗电池表面，利用机械切割机将其切割为若干块，放入碳酸二甲酯 (DMC) 中超声振荡过滤，将其中的集流体、隔离膜、极耳以及包装袋去除得到涂层浆料；再次过滤去除其中的液态组分得到滤饼，在 45℃，1KPa 压力下烘干得到正负极活性物质共混的粉料，然后粉碎正负极活性物质共混的粉料 20min。之后通过气流分离管，在重力和气流吹力的作用下，密度较大的钴酸锂下沉、密度较小的石墨上浮从而实现两者的分离，回收得到锂插层石墨材料；

[0042] 将所得锂插层石墨在 PC 溶剂中超声 30 分钟，超声功率 100W，进而利用乙醇、去离子水过滤清洗所得材料，收集后进行冷冻干燥，得到石墨烯材料。

[0043] 实施例八

[0044] 本实施例与实施例七过程与条件相同，只是将超声溶剂变为 DMF，超声时间为 2 小时，功率 200W，干燥方式为真空干燥。

[0045] 实施例九

[0046] 本实施例与实施例四的过程与条件基本相同，区别仅在于，本实施例的低温反应时间为 15 小时，同样可得到石墨烯材料。

[0047] 以上内容是结合具体的优选实施方式对本发明所作的进一步详细说明，不能认定本发明的具体实施只局限于这些说明。对于本发明所属技术领域的技术人员来说，在不脱离本发明构思的前提下，还可以做出若干等同替代或明显变型，而且性能或用途相同，都应当视为属于本发明的保护范围。

Ascend Elements EX1002 Page 41

Duesenfeld Ex. 2014-041

Espacenet

DE4424825A1 Safe disposal of dry batteries and recovery of materials

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Safe disposal of dry batteries and recovery of materials

Abstract

Process for the disposal of spent, or partly spent, dry, metal-cased, batteries, especially those containing lithium, involves cooling the batteries before disintegration to such a low temperature that the partially freed components cannot react chemically or physically under normal conditions of temperature, pressure and the presence of oxygen. Further provision is made for the separation and recovery of materials which can be recycled. Batteries are cooled by direct contact with, pref., liquid nitrogen at -196 deg C. The gasified nitrogen is used in a pre-cooling chamber and also to provide an inert atmosphere in the disintegration section, including the materials separation chambers. A coarse separation is carried out in a flotation process followed by a further separation of individual materials, some of which will be used for recycling.

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(54) Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien

(57) Das Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, umfaßt ein Zerkleinern der Batterien durch beispielsweise eine Shreddereinrichtung. Um das Shreddern gefahrlos durchführen zu können, werden die Batterien vor dem Zerkleinern auf eine solche tiefe Temperatur abgekühlt, daß eine chemische Reaktion der vom Metallgehäuse wenigstens teilweise befreiten Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet. Danach kann dann eine Trennbehandlung der entstandenen Fraktionsanteile der Batterien mit herkömmlicher Technik erfolgen.

DE 44 24 825 A 1

Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen

Ascend Elements Ex 1002 - Page 43

BUNDESDRUCKEREI 11. 95 508 063/308

Duesenfeld Ex. 2014-043

Beschreibung

Die Erfindung betrifft ein Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien gemäß dem Oberbegriff des Patentanspruchs 1.

Das Problem bei der Entsorgung von Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, auch wenn sie entladen bzw. verbraucht sind, besteht darin, daß die Bestandteile der Batterien beim Zerkleinerungsvorgang, zum Beispiel durch Shreddern, bei Normalbedingungen in Luft noch chemisch und physikalisch heftig miteinander reagieren und dabei Knall- und Verbrennungsgeräusche verursachen, weshalb die Zerkleinerungsanlage mit umfangreichen Schutzmaßnahmen versehen sein muß, was mit einem erheblichen Material- und Kostenaufwand verbunden ist. Desweiteren werden dabei auch schädliche Gase, Dämpfe und feste Stoffe freigesetzt, die Mensch und Umwelt belasten und ebenfalls mit erheblichem Material- und Kostenaufwand einzukapseln und zu entsorgen sind.

Die Aufgabe der Erfindung besteht darin, ein verbessertes Verfahren der eingangs angeführten Art vorzuschlagen, das ein gefahrloses Öffnen bzw. Zerkleinern von gasdicht gekapselten Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, ermöglicht und eine einfache Weiterverarbeitung der zerkleinerten Batterien gestattet.

Die Lösung dieser Aufgabe geht von dem einleitend angeführten Verfahren aus und ist durch die Merkmale des Patentanspruchs 1 gekennzeichnet.

Durch die erfindungsgemäße Lösung ist ein gefahrloses Öffnen und Zerkleinern von zu entsorgenden Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, ermöglicht, denn das Restenergievermögen der Batterien ist durch deren Tiefkühlung vor dem Zerkleinern nicht oder im wesentlichen nicht mehr zu einer chemischen und physikalischen Reaktion in Luft unter normalen Druck- und Temperaturbedingungen fähig, wenn das schützende Metallgehäuse der Batterien geöffnet bzw. entfernt worden ist. Die Batterien können also ohne Gefahr in Luft bei Normalbedingungen mechanisch zerkleinert werden, zum Beispiel durch Shreddern, wobei keinerlei Knall- und Verbrennungsgeräusche unter Rauch- und/oder Flammenentwicklung mehr entstehen. Weiterhin entstehen während der Tiefkühlbehandlung nur geringfügig schädliche Gase und Dämpfe. Die sich an die vollständige Zerkleinerung der Batterien anschließende Trennbehandlung aller entstandenen Fraktionsanteile sorgt dafür, daß sich die langsam wieder auf etwa Raumtemperatur erwärmten, nun losen Batteriebestandteile ebenfalls nicht mehr oder nur noch in einem unbedeutenden Umfang unkontrolliert chemisch reagieren können. Die Trennbehandlung kann mit herkömmlichen Verfahren und Vorrichtungen durchgeführt werden, derart, daß wenigstens einige der Bestandteile der zerstörten Batterien als Recycle-Rohstoff einer Wiederverwendung zugeführt werden können und nur noch wenige, nach Möglichkeit keine Reststoffe auf eine Deponie gegeben werden müssen.

Eine bevorzugte Tiefkühltemperatur beträgt wenigstens etwa −190° Celcius und als Tiefkühlmittel wird vorzugsweise flüssiger Stickstoff oder auch Argon verwendet.

Falls bei einigen zu entsorgenden Batterien nach dem Tiefkühlen beim Shreddern noch eine geringe chemische Reaktion zu befürchten ist, kann aus dem beim

Tiefkühlen verwendeten Tiefkühlmittel ein gasförmiger Anteil abgezweigt und für eine chemisch inerte Atmosphäre beim Zerkleinern der tiefgekühlten Batterien verwendet werden. Hierdurch erfolgt das Shreddern in einer eine mögliche chemische Reaktion unterbindenden Atmosphäre.

Die Trennbehandlung der Fraktionsanteile der zerstörten Batterien wird vorzugsweise durch einen Flotationsvorgang eingeleitet, wobei das Flotationsgemisch einer Leichtfraktionsbehandlung und einer Schwerfraktionsbehandlung unterworfen wird, um eine Grobtrennung vorzunehmen.

Die Erfindung ist nachstehend anhand des anliegenden Ablaufdiagramms beispielsweise näher erläutert. In **Fig.** 1 ist ein erster Teil des Ablaufdiagramms gezeigt, während in **Fig.** 2 der zweite Teil des Ablaufdiagramms gezeigt ist.

Das vorgeschlagene Verfahren eignet sich zur Entsorgung von Feststoff-Batterien **1** und **2**, die zum Beispiel in Zylinderform und Quaderform im Handel erhältlich sind, und zwar insbesondere für solche Batterien, die Lithium enthalten. Lithiumbatterien wiederum enthalten je nach Zellentyp außer Lithium noch Mangandioxid, Schwefeldioxid, Thionylchlorid und Acetonnitril als elektrolytische Reaktoren. Derartige Batterien enthalten, auch wenn sie entladen oder fast entladen sind, noch eine relativ hohe Energiedichte, so daß eine chemische und physikalische Reaktion der genannten Substanzen stattfindet, wenn das schützende Metallgehäuse der Batterien bei Normalbedingungen in Luft geöffnet wird.

Gemäß dem vorgeschlagenen Verfahren nach den **Fig.** 1 und 2 werden die zu entsorgenden Batterien in einer Packstation **3** zu einer vorbestimmten Menge zusammengesetzt, welche dann in wenigstens einen Schleppkasten **4** eines Vorkühlraumes **5** gegeben wird. Die in die Schlepp- oder Transportkästen **4** eingefüllten Batterien durchlaufen dann mit Hilfe eines Transportsystems eine Vorkühlstrecke **6**, innerhalb der sie von Raumtemperatur auf eine Temperatur von etwa −30° bis −50° Celsius abgekühlt werden. Die Durchlaufdauer durch die Vorkühlstrecke **6** wird von der Temperatur und der Art des zugeführten Kühlgases und der Batteriemasse bestimmt und kann beispielsweise 15—30 Minuten betragen.

Der Vorkühlraum **5** wird mit einem Vorkühlgas beschickt, welches beispielsweise eine Temperatur von −80° bis −190° Celsius aufweisen kann. Vorzugsweise wird das Kühlgas aus einem Hauptkühlraum **7** abgezweigt, in welchem die vorgekühlten Batterien mittels der Kästen **4** eingeführt werden. Das Vorkühlgas wird mittels einer Pumpe **8** aus dem Hauptkühlraum **7** abgesaugt und über eine Leitung **9** in den Vorkühlraum **5** eingeleitet.

Die vorgekühlten Batterien in den Kästen **4** durchlaufen dann mittels eines Transportsystems für eine vorbestimmte Zeit den Hauptkühlraum und werden darin auf eine solche tiefe Temperatur abgekühlt, daß eine chemische und physikalische Reaktion der später vom Metallgehäuse wenigstens teilweise befreiten Batterie-Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet. Eine solche Tiefkühltemperatur beträgt wenigstens etwa −80° Celsius, vorzugsweise etwa −190° Celsius.

Ein geeignetes Kühlmittel ist flüssiger Stickstoff (LIN), mit dem die vorgekühlten Batterien in Kontakt gebracht werden. Eine solche Kontaktdauer kann bei-

Duesenfeld Ex. 2014-044

spielsweise 15 bis 30 Minuten oder mehr betragen, was davon abhängig ist, in welcher Form die vorgekühlten Batterien mit dem flüssigen Stickstoff in Kontakt kommen. Hierfür kann beispielsweise ein Eintauchvorgang in Frage kommen, so daß die Batterien in den flüssigen Stickstoff eingetaucht werden. Es kann aber auch eine Besprühung oder Begießen der vorgekühlten Batterien mit flüssigem Stickstoff in Frage kommen. Auch eine Mischung aller oder zweier Kontaktarten ist möglich. Der flüssige Stickstoff weist eine Temperatur von wenigstens −196° Celsius auf und wird mittels einer Pumpe **10** aus einem Vorratsbehälter **11** über eine Leitung **12** in den Hauptkühlraum **7** eingeleitet.

Während des Tiefkühlvorganges der Batterien in dem Hauptkühlraum **7** entsteht in diesem Raum aus dem flüssigen Stickstoff ein gasförmiger Anteil, der für verschiedene Weiterbehandlungsvorgänge der Batterien bzw. Batterieteile verwendet wird. Wie bereits angedeutet, wird ein gewisser Anteil für die Vorkühlung der Batterien in dem Vorkühlraum **5** verwendet.

Das nicht mehr benötigte Vorkühlgas in dem Vorkühlraum **5** wird aus diesem, gegebenenfalls zusammen mit Abluft, abgesaugt und für die Wiederverwendung im Hauptkühlraum **7** vorbereitet. Hierzu ist eine Rückgewinnungseinrichtung **13** vorgesehen, aus welcher der wieder flüssige Stickstoff über eine Leitung **14** dem Vorratsbehälter **11** zugeführt wird. Gegebenenfalls vorhandene Abluft wird aus der Einrichtung **13** ins Freie abgeleitet.

Die auf die vorbestimmte Tiefkühltemperatur abgekühlten Batterien werden dann in eine Zerkleinerungsvorrichtung **15** gegeben, wo sie zerkleinert werden. Diese Vorrichtung **15** kann beispielsweise aus einer mehrstufigen Shreddervorrichtung bestehen. Es können aber auch Sägeeinrichtungen oder andere Einrichtungen alternativ oder zusätzlich eingesetzt werden, um die tiefkalten Batterien zu ausreichend kleinstückigen Fraktionsanteilen zu zerstören. In der Vorrichtung **15** herrscht vorzugsweise ein Druck von 1 bar (10^5 Pa) und eine Temperatur von maximal etwa 20° Celsius. Im übrigen kann der Innenraum der Zerkleinerungsvorrichtung **15** mit Luft gefüllt sein.

Wenn es aus bestimmten Sicherheitsgründen erforderlich ist, anstelle von Luft in der Vorrichtung **15** ein inertes Gas zu verwenden, so kann dazu ebenfalls gasförmiger Stickstoff aus dem Hauptkühlraum **7** abgezweigt werden, der mittels einer Pumpe **16** über eine Leitung **17** in die Vorrichtung **15** eingeleitet wird.

Die Fraktionsanteile der zerkleinerten Batterien werden in Auffangbehältern **18** gesammelt und einem Bekken einer Flotationsstation **19** zugeführt, wo eine erste Trennungsbehandlung der Fraktionsanteile stattfindet. Zur Durchführung des Flotationsvorganges befindet sich in dem Becken der Flotationsstation **19** neben einem aufbereiteten Wasser auch ein alkalisches Lösungsmittel, zum Beispiel Natronlauge, welche zusammen mit dem Wasser eine Suspension ergibt. Weiterhin wird neben Luft auch ein inertes Gas eingeleitet, welches wiederum gasförmiger Stickstoff sein kann und vorzugsweise aus dem Hauptkühlraum **7** über eine Leitung **20** und eine Pumpe **21** entnommen wird. Dieses Gas kann ebenfalls eine tiefe Temperatur aufweisen, beispielsweise −180° Celsius.

Durch die Flotation erfolgt eine Grobtrennung der Fraktionsanteile voneinander, und zwar eine Trennung der schwereren Fraktionsanteile, wie zum Beispiel Eisenmetalle, Nichteisenmetalle, Glas- und Keramikanteile, von den leichteren Fraktionsanteilen, die insbesondere aus Kunststoff bestehen. Die für die Flotation insgesamt in die Station **19** eingeleiteten Gase, die während der Flotation ihre Aufgabe erfüllt haben, und die während der Flotation freigewordenen Gase, insbesondere organische Gase, werden über eine Leitung **22** abgeführt und einer Einrichtung **23** zum Unschädlichmachen dieser Abgase zugeführt. Dieser Einrichtung **23** können auch die nicht mehr benötigten Gase aus der Shreddervorrichtung **15** zugeführt werden, wie es mit der Leitung **24** angegeben ist.

Nachdem sich die schwereren von den leichten Fraktionsanteilen der zerstörten Batterien in der Flotationsstation abgesondert haben, ist es ohne weiteres möglich, die so grob getrennten Fraktionsanteile aus der Flotationsstation **19** zu entnehmen und weiteren Trennungsbehandlungen zu unterwerfen. Die verbrauchte Lösungssuspension wird ebenfalls aus der Station **19** abgeführt und einer Behandlung zum Unschädlichmachen und Wiederaufbereiten unterworfen, die jedoch nicht Gegenstand dieser Erfindung ist.

Die aus der Flotationsstation **19** entnommenen Leichtfraktionsanteile werden zunächst einer Trocknereinrichtung **25** zugeführt, wonach die getrockneten Anteile in einem Behältnis **26** einer Sichteinrichtung **27** zugeführt werden, wo nochmals, falls erforderlich, eine Trennung der Kunststoffteile vorgenommen wird. Die so gewonnenen Kunststoffteile können als Recyclerohstoff in der chemischen Industrie wiederverwertet werden.

Auch die Schwerfraktionsanteile aus der Flotationsstation **19** werden zunächst einer Trocknereinrichtung **29** zugeführt, um sie dann besser weiterbehandeln zu können. Nach dem Trocknen können die Schwerfraktionsanteile zur weiteren und feineren Zerkleinerung einer weiteren Shreddereinrichtung **30** zugeführt werden. Von dort gelangen sie dann in eine erste Abscheideeinrichtung **31**, wo mittels bekannter Technik die Eisenanteile abgeschieden werden. In einer dann folgenden zweiten Abscheideeinrichtung **32** werden die Nichteisenanteile, insbesondere Kupfer, abgeschieden. Die Eisenanteile und die Nichteisenanteile können dann als Recyclematerial als wiederverwertbare Rohstoffe zur Verfügung stehen und mittels der Verhüttungstechnik verarbeitet werden. Die nun noch verbleibenden Restanteile, wie insbesondere Glas, können deponiert oder beispielsweise in der Glasindustrie oder im Straßenbau wiederverwendet werden. Erforderlichenfalls können durch entsprechende Trenneinrichtungen auch die Restanteile nochmals getrennt werden, um zum Beispiel die Keramikanteile abzuscheiden und einer Wiederverwertung zuzuführen.

Bevorzugt wird als Tiefkühlmittel im Hauptkühlraum **7** flüssiger Stickstoff verwendet. Davon abgezweigter Stickstoff in Gasform wird bevorzugt zur Vorkühlung im Vorkühlraum **5** und für die anderen, vorstehend verwendeten Zwecke eingesetzt. Stattdessen kann jedoch auch ein anderes Gas verwendet werden, zum Beispiel Argon. Obwohl das vorstehende Verfahren hauptsächlich für Lithium enthaltende Feststoff-Batterien vorgesehen ist, können auch andere Feststoff-Batterien, beispielsweise Nickel-Hydrid-Batterien, wie vorstehend beschrieben, entsorgt werden.

Patentansprüche

1. Verfahren zum Entsorgen von entladenen und geladenen elektrische Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, die

aus einem Metallgehäuse und darin enthaltenen festen Inhaltsstoffen bestehen, gemäß dem die zu entsorgenden Batterien mechanisch zerkleinert werden, **dadurch gekennzeichnet,** daß die Batterien vor dem Zerkleinern auf eine solche tiefe Temperatur abgekühlt werden, daß eine chemische und physikalische Reaktion der vom Metallgehäuse wenigstens teilweise befreiten Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet und daß das nach dem Zerkleinern erhaltene Gemisch der Fraktionsanteile der zerstörten Batterien zur Gewinnung von wiederverwendbarem Ausgangsmaterial einer Trennbehandlung unterworfen wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Tiefkühlen der Batterien durch in berührungbringen derselben mit einem chemisch inerten Tiefkühlmittel erfolgt, dessen Temperatur wenigstens etwa – 196° Celsius beträgt.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß als Tiefkühlmittel flüssiger Stickstoff verwendet wird.

4. Verfahren nach Anspruch 2 oder 3, dadurch gekennzeichnet, daß aus dem beim Tiefkühlen verwendeten Tiefkühlmittel eine gasförmiger Anteil abgezweigt und zum Durchführen einer Vorkühlung der Batterien verwendet wird.

5. Verfahren nach Anspruch 1, 2, 3 oder 4, dadurch gekennzeichnet, daß das Zerkleinern der tiefgekühlten Batterien in einer chemisch inerten Atmosphäre durchgeführt wird.

6. Verfahren nach Anspruch 5, dadurch gekennzeichnet, daß aus dem beim Tiefkühlen verwendeten Tiefkühlmittel ein gasförmiger Anteil abgezweigt und für eine chemisch inerte Atmosphäre beim Zerkleinern der tiefgekühlten Batterien verwendet wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß das Gemisch der Fraktionsanteile der zerstörten Batterien einem Flotationsvorgang unterworfen wird, um eine Grobtrennung des Flotationsgemisches in Form eines Leichtfraktionsvorganges und eines Schwerfraktionsvorganges durchzuführen.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß der Flotationsvorgang wenigstens im wesentlichen in einer chemisch inerten Atmosphäre durchgeführt wird.

9. Verfahren nach Anspruch 7 oder 8, dadurch gekennzeichnet, daß sich an den Leichtfraktions- und den Schwerfraktionsvorgang jeweils weitere Trennvorgänge zur Gewinnung einzelner Materialsorten aus den Fraktionsanteilen der zerstörten Batterien anschließen und daß wenigstens einige der gewonnenen Materialsorten der Wiederverwendung zugeführt werden.

Hierzu 2 Seite(n) Zeichnungen

Nummer: DE 44 24 825 A1
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Offenlegungstag: 18. Januar 1996

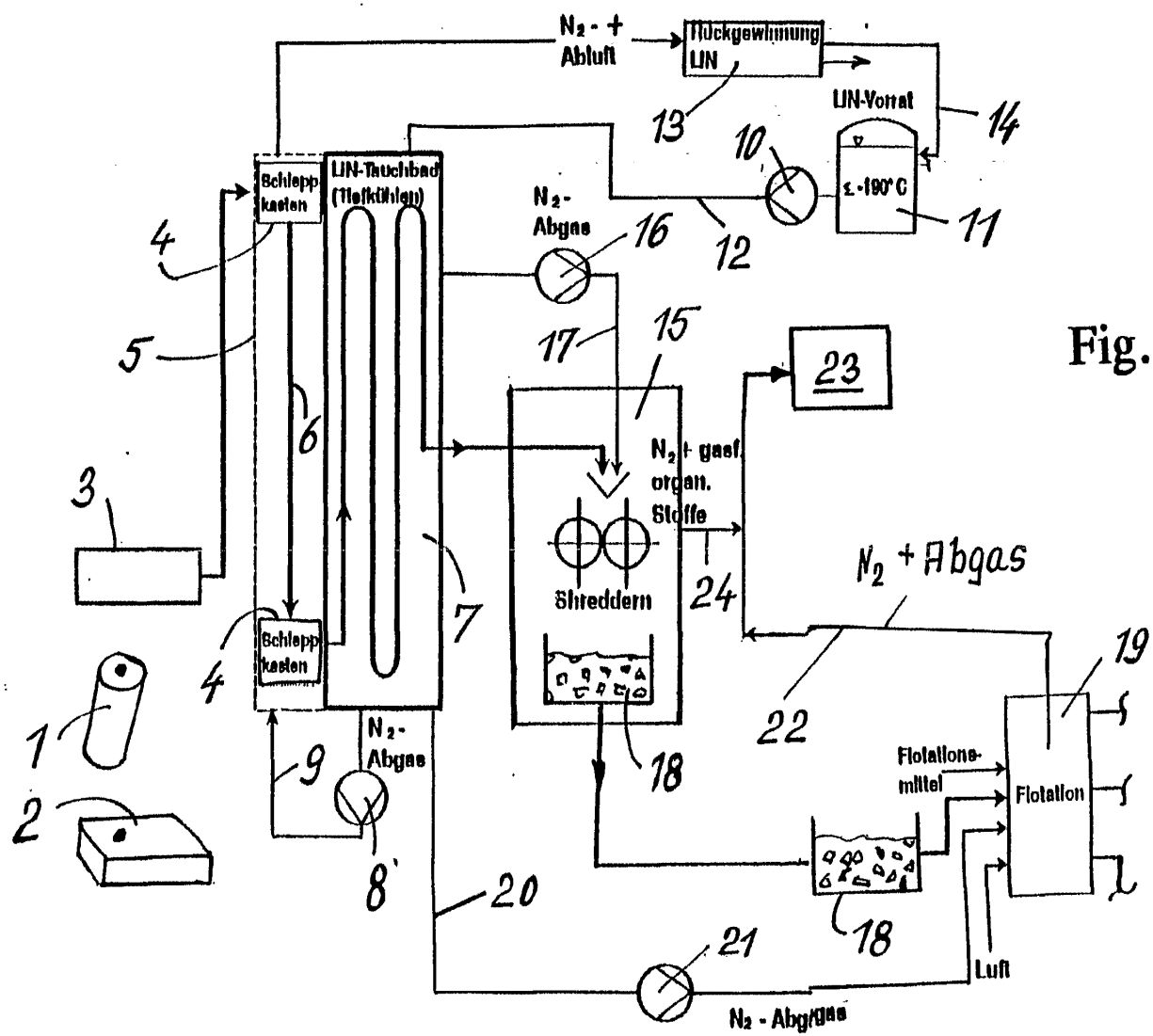

Fig. 1

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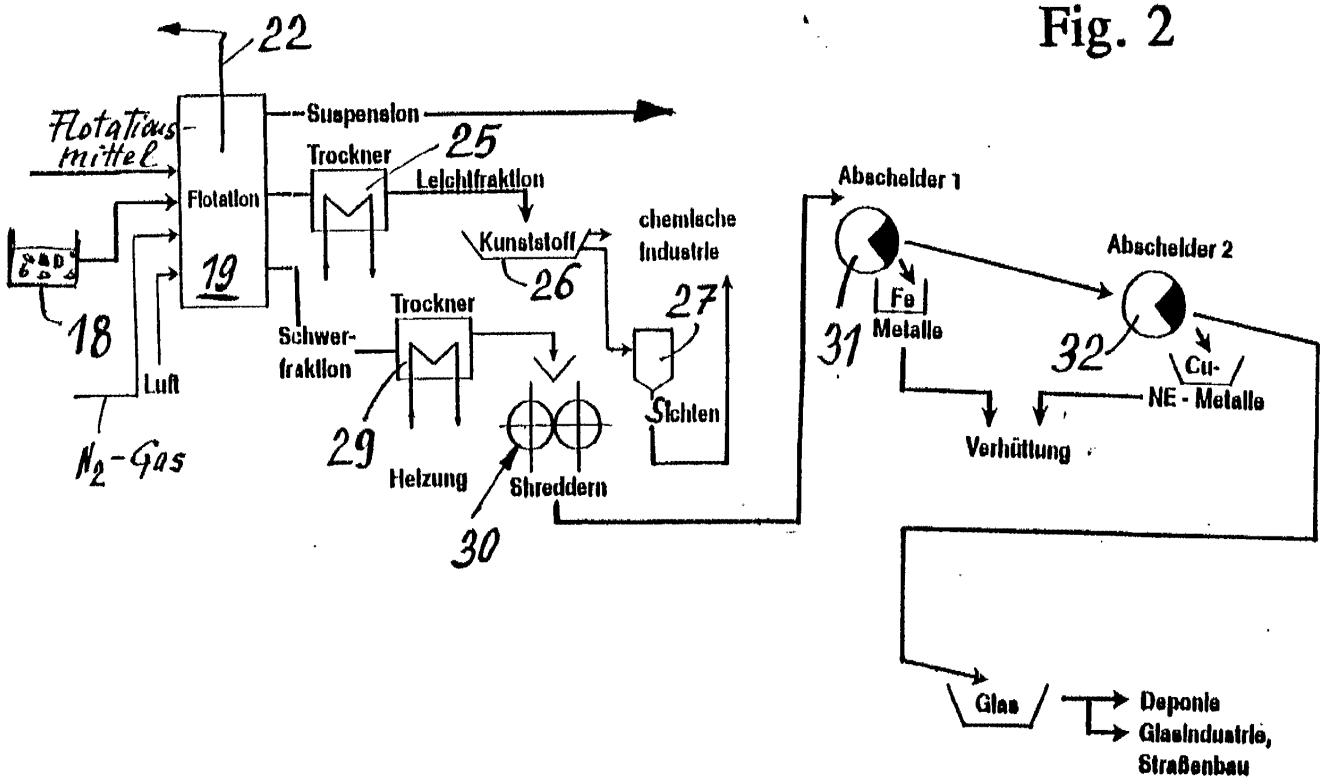
Fig. 2



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(54) 폐건전지의 코발트 건식 분리방법

(57) 요약

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것으로, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 구성됨으로써, 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있음은 물론, 폐리튬 이온 전지의 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2차 오염을 원천 방지할 수 있는 효과가 있다.

특허청구의 범위

청구항 1.

(a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와;

(b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와;

Ascend Elements EX1002 Page 49

Duesenfeld Ex. 2014-049

(c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와;

(d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법.

청구항 2.

제 1 항에 있어서,

상기 (a) 단계는,

상기 폐리튬 이온 전지의 파쇄 조각들이 100 내지 1,600㎟의 면적을 갖도록 파쇄되는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 3.

제 1 항에 있어서,

상기 (b) 단계는,

상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 4.

제 1 항에 있어서,

상기 (c) 단계는,

상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와,

상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와,

상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 5.

제 1 항에 있어서,

상기 (c) 단계는,

상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와,

상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Ascend Elements EX1002 Page 50

Duesenfeld Ex. 2014-050

명세서

발명의 상세한 설명

발명의 목적

발명이 속하는 기술 및 그 분야의 종래기술

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것이다.

리튬 이온 전지(1차 및 2차 전지 포함)는 고 에너지 밀도 및 경박 경량의 특성으로 인하여 소형 전기, 전자장비의 전원 등으로 폭넓게 활용되고 있다. 사용이 완료된 폐리튬 이온 전지는 환경 오염 및 자원 재활용의 측면에서 특정 방법에 의해 유가 물질이 수득되고 있다.

통상적으로, 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 수득하는 기술은 대한민국 특허등록번호 10-0448273(2004.09.01), 대한민국 특허등록번호 10-040742(2003.10.27) 및 대한민국 특허공개번호 특2003-0011737(2003.02.11) 등에 다양하게 개시되어 있으며, 리튬-코발트 산화물을 수득하는 기술은 대한민국 특허공개번호 특2003-0093583호(2003.12.11)에 개시되어 있다.

이러한 기술들은 습식 방법에 의해 고순도의 코발트(또는 코발트 산화물) 및 리튬-코발트 산화물을 수득할 수 있으나, 수득 공정이 매우 복잡하고 정밀 제어가 요구되기 때문에 다량의 폐리튬 이온 전지를 처리할 수 없음은 물론, 강산 또는 강알카리가 사용됨으로써 2차 오염을 유발할 수 있는 문제점이 있다.

발명이 이루고자 하는 기술적 과제

상기와 같은 문제점을 해소하기 위하여 창출된 본 발명의 목적은, 코발트 또는 코발트 산화물 수득 공정시 야기될 수 있는 2차 오염없이 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 건식 방법으로 수득할 수 있는 폐건전지의 코발트 건식 분리 방법을 제공함에 있다.

이러한 본 발명의 목적은, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법에 의해 달성될 수 있다.

바람직하게는, 본 발명의 상기 (a) 단계는 상기 폐리튬 이온 전지가 100 내지 1,600㎟의 면적을 갖도록 파쇄된다.

바람직하게는, 본 발명의 상기 (b) 단계는 상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거된다.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시(mesh)의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와, 상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와, 상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함한다.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와, 상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함한다.

발명의 구성

이하, 본 발명의 구성을 상세히 설명하면 다음과 같다.

(a) 단계 - 적어도 하나 이상의 폐리튬 이온 전지 파쇄

Ascend Elements EX1002 Page 51

Duesenfeld Ex. 2014-051

폐리튬 이온 전지는 파쇄기를 이용하여 폐리튬 이온 전지의 파쇄 조각들이100 내지 1,600㎟ 의 면적이 되도록 파쇄되는 것이 바람직하다. 이러한 방법은 철 재질인 케이스 부분과 내용물 부분이 파쇄물에 혼합된 상태로 수득된다.

다른 방법으로는 폐리튬 이온 전지의 철 재질인 케이스와 케이스에 내장된 내용물을 분리시키는 것으로 폐리튬 이온 전지의 중심 부분이나 양단부를 절단하여 케이스로부터 내용물을 분리한 다음, 내용물을 파쇄기를 이용하여 파쇄하는 것이다.

이러한 방법은 내용물만을 파쇄하기 때문에 전술된 방법(케이스가 포함되는 경우) 보다는 보다 적은 면적을 갖도록 파쇄하는 것이 바람직하다.

(b) 단계 - 파쇄물을 건조하여 전해질을 제거

파쇄물은 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거한다.

여기서, 건조 과정이 300℃ 이만의 온도에서 건조가 수행되는 경우에는 작업 건조 시간이 길어져서 작업성이 양호치 못하고, 550℃ 초과의 온도에서 건조가 수행되는 경우에는 파쇄물 중의 페이스트 및 유기물 등이 착화되어 오염 물질이 발생되는 문제점이 있다.

그리고, 건조 시간은 파쇄물의 양에 의해 결정된다.

(c) 단계 - 파쇄물 미세 분쇄 및 코발트 화합물 분리

건조가 완료된 파쇄물은 진동 밀 또는 볼 밀 등에 의해 미쇄 분쇄된다. 여기서, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 것이 매우 중요하다.

미세 분쇄시 코발트 화합물은 50 내지 100 메시의 입도를 갖는 분말로 존재하고, 양극 성분인 알루미늄과 음극 성분인 구리는 각기 박판으로 존재하게 되며, 니켈 및 철은 코발트 화합물 분말에 비하여 5 내지 15배의 입도를 갖게 된다.

그리고, 분쇄물(분말)은 공급되는 고압의 공기에 의해 전술된 코발트 화합물 분말만이 비산되고, 코발트 화합물 분말을 제외한 알루미늄 및 구리 박판은 밀 표면에 압착되어 있으며 코발트 화합물 분말보다 상대적으로 중량인 니켈과 철 성분은 밀에 잔류된다. 여기서, 공급되는 고압의 공기는 코발트 화합물 분말 만이 비산(공기 분급 방법)되도록 이에 상응하는 압력과 공기량을 갖는다.

비산된 코발트 화합물 분말은 덕트 등과 같은 폐쇄 경로를 통해 유동된 후 포집된다. 여기서, 비산된 코발트 화합물 분말 중에 불순물(특히, 알루미늄 또는 구리 박판)이 혼입될 수 있기 때문에 포집 전 단계에서 불순물 제거용 필터를 경유시켜 전술된 불순물을 제거한 후 순수한 코발트 화합물 분말을 수득하는 것이 바람직하다.

또한, 전술된 공기 분급 방법 이외에 간단한 코발트 화합물 분말을 수득하는 방법은 망체(網體)가 구비된 요동되는 스크린 장치의 상부에 미세 분쇄된 분말을 공급하는 것으로, 망체의 메시는 코발트 화합물 분말보다는 적어도 입도가 큰 것이 바람직하다. 따라서, 코발트 화합물 분말만이 스크린 장치의 하부로 자유 낙하되기 때문에 순수한 코발트 화합물을 수득할 수 있다.

(d) 단계 - 코발트 화합물 분말을 환원시켜 순순한 코발트 분말 수득

전술된 (c) 단계에서 수득된 코발트 화합물 분말 속에는 미량의 탄소 분말이 함유될 수 있기 때문에 우선적으로, 300℃ 이상의 고열로 가열함으로써 탄소를 제거한 후 환원처리하면 순수한 코발트 분말을 수득할 수 있다.

이상에서 설명된 본 발명은 전술된 실시예에 한정되어 설명되었지만, 이에 한정되지 않고 본 발명이 속하는 분야의 통상적인 기술분야에서 통상의 지식을 가진 자가 용이하게 실시할 수 있는 정도의 변형은 본 발명의 기술적 사상에 속하는 것임은 자명하다.

발명의 효과

Ascend Elements EX1002 Page 52

Duesenfeld Ex. 2014-052

이상의 구성을 갖는 본 발명은 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있는 효과가 있다. 또한, 이러한 구성을 갖는 본 발명은 폐리튬 이온 전지의 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2차 오염을 원천 방지할 수 있는 특징이 있다.

Ascend Elements EX1002 Page 53

Duesenfeld Ex. 2014-053

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발명의 명칭

폐건전지의 코발트 건식 분리방법

Title of Invention

Dry-method of separating Co from battery scarp

요약

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것으로, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 피쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미서 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 구성됨으로써, 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있음은 물론, 폐리튬 이온 전지의 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2차 오염을 원천 방지할 수 있는 효과가 있다.

폐건전지, 건전지 재생, 코발트, 건식 분리

Abstract

The present invention relates to the cobalt dry separation method of the waste battery and has the effect that the step of crushing the wasted lithium ion battery of (a) at least one, the step of drying and removing the electrolyte (b) crushed material, the grinding the crushed material from, the step of separating the cobalt compound from the fine pulverization article using the particle size difference, and the step of returning (d) cobalt compound and obtaining the pure cobalt are comprised. In that way the pure cobalt the large amount can be obtained from the wasted lithium ion battery. Also the operation procedure is not dangerous and the secondary pollution due to this can be at the source prevented because the strong acid or the strong alkali is not used in the recycle process of the wasted lithium ion battery. The grinding the crushed material from (c) electrolyte is removed.

The waste battery, the reclamation of battery, cobalt, the dry separation .

청구의 범위

청구 **1**항:

Scope of Claims

Claim 1:

Ascend Elements EX1002 Page 54

Duesenfeld Ex. 2014-054

(a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법.

The cobalt dry separation method of the waste battery the step of crushing the wasted lithium ion battery of (a) at least one, the step of drying and removing the electrolyte (b) crushed material, the grinding the crushed material from, the step of separating the cobalt compound from the fine pulverization article using the particle size difference, and the step of returning (d) cobalt compound and obtaining the pure cobalt are included, and the grinding the crushed material from (c) electrolyte is removed.

청구 2항:

제 1 항에 있어서, 상기 (a) 단계는, 상기 폐리튬 이온 전지의 파쇄 조각들이 100 내지 1,600㎟의 면적을 갖도록 파쇄되는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Claim 2:

As for claim 1, the cobalt dry separation method of the waste battery in which (a) step is crushed so that the smash pieces of the wasted lithium ion battery have the area of 100 through 1,600㎟.

청구 3항:

제 1 항에 있어서, 상기 (b) 단계는, 상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Claim 3:

As for claim 1, the cobalt dry separation method of the waste battery which hots air-dry the crushed material to 300 through 550℃ and in which (b) step utterly removes the electrolyte.

청구 4항:

제 1 항에 있어서, 상기 (c) 단계는, 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와, 상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와, 상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Claim 4:

As for claim 1, the cobalt dry separation method of the waste battery which comprises (c) - 3 step wherein (c) step obtains the depositing the pure cobalt compound powder passing the impurity removing filter the cobalt compound powder through and removes the impurity and that is sprayed to (c) - 2 step which disperses, and the above the cobalt compound powder supplying (c) - 1 step which it minutely pulverizes the crushed material using the vibrating mill or the ball mill ; and it pulverizes so that it becomes the powder in which the cobalt compound has the particle size of 50 through 100 mesh., and the air of the high pressure in the fine pulverization article.

청구 5항:

제 1 항에 있어서, 상기 (c) 단계는, 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와, 상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Claim 5:

As for claim 1, the cobalt dry separation method of the waste battery wherein (c) step comprises (c) - 5 step that obtains the cobalt compound powder by the selection by (c) - 4 step which it pulverizes so that it becomes the powder, and the free fall the fine pulverization article is provided at the upper part of the screen device for being shaken having the cobalt compound is the particle size of 50 through 100 mesh the crushed material is minutely pulverized using the vibrating mill or the ball mill.

배경기술

Background Art

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것이다.

The present invention relates to the cobalt dry separation method of the waste battery.

리튬 이온 전지(1차 및 2차 전지 포함)는 고 에너지 밀도 및 경박 경량의 특성으로 인하여 소형 전기, 전자장비의 전원 등으로 폭넓게 활용되고 있다. 사용이 완료된 폐리튬 이온 전지는 환경오염 및 자원 재활용의 측면에서 특정 방법에 의해 유가 물질이 수득되고 있다.

Lithium ion battery (the first and the secondary battery inclusion) are widely used as the small size electricity, the power source of the electronic apparatus etc. due to the property of the frivolity light weight and high energy density. The valuable materials are w

Ascend Elements EX1002 Page 55

Duesenfeld Ex. 2014-055

asted lithium ion battery, in which the use is completed is obtained in the side of the resource reuse and enviro nmental contamination with the specific method.

통상적으로, 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 수득하는 기술은 대한민국 특허등록번호 10-0448273(2004.09.01), 대한민국 특허등록번호 10-040742(2003.10.27) 및 대한민국 특허공개번호 특2003-0011737(2003.02.11) 등에 다양하게 게시되어 있으며, 리튬-코발트 산화물을 수득하는 기술은 대한민국 특허공개번호 특2003-0093583호(2003.12.11)에 게시되어 있다.

Generally, cobalt or the technology obtaining the cobalt oxide is variously disclosed from the wasted lithium ion battery in KR10-0448273 B (2004.09.01), KR10-040742 B (2003.10.27) and KR2003-0011737 A (2003.02.11) etc. and the technology obtaining the lithium - cobalt oxide is disclosed in KR2003-0093583 A (2003.12.11).

이러한 기술들은 습식 방법에 의하 고순도의 코발트(또는 코발트 산화물) 및 리튬-코발트 산화물을 수득할 수 있으나, 수득 공정이 메우 복잡하고 정밀 제어기 요구되기 때문에 다량의 폐리튬 이온 전지를 처리할 수 없음은 물론, 강산 또는 강알카리가 사용됨으로써 2차 오염을 유발할 수 있는 문제점이 있다.

There is a problem that such technologies can obtain the cobalt (or, the cobalt oxide) and lithium - cobalt oxide of the high purity with the wet method. But a large amount of wasted lithium ion battery cannot be processed because the acquisition process is very complicated and the precision control is required. Also the secondary pollution can be induced since the strong acid or the high-density-alkali is used.

발명의 내용

Summary of Invention

발명의 효과

Effects of the Invention

이상의 구성을 갖는 본 발명은 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있는 효과가 있다. 또한, 이러한 구성을 갖는 본 발명은 폐리튬 이온 전지으 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2치 오염을 원천 방지할 수 있는 특징이 있다.

It has the configuration described in the above. And the present invention has the effect that the pure cobalt the large amount can be obtained from the wasted lithium ion battery. Moreover, it has the characteristic which the operation procedure is not dangerous and can prevent at the source the secondary pollution due to this because the invention having such configuration does not use the strong acid or the strong alkali in the recycle process of the wasted lithium ion battery.

기술적 과제

Technical Task

상기와 같은 문제점을 해소하기 위하여 창출된 본 발명의 목적은, 코발트 또는 코발트 산화물 수득 공정시 야기될 수 있는 2차 오염없이 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 건식 방법으로 수득할 수 있는 폐건전지의 코발트 건식 분리 방법을 제공함에 있다.

To solve the problem, it is created. And an object of the present invention is to provide the cobalt dry separation method of cobalt from the wasted lithium ion battery without cobalt or the secondary pollution which can be caused in the cobalt oxide acquisition process or the waste battery obtaining the cobalt oxide by the dry process.

이러한 본 발명의 목적은, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법에 의해 달성될 수 있다.

The step that the object of such invention crushes the wasted lithium ion battery of (a) at least one, the step of drying and removing the electrolyte (b) crushed material, the grinding the crushed material from, the step of separating the cobalt compound from the fine pulverization article using the particle size difference, and the step of returning (d) cobalt compound and obtaining the pure cobalt are included and it can be achieved by the cobalt dry separation method of the waste battery. The grinding the crushed material from (c) electrolyte is removed.

바람직하게는, 본 발명의 상기 (a) 단계는 상기 폐리튬 이온 전지가 100 내지 1,600㎟의 면적을 갖도록 파쇄된다.

Preferably, (a) step of the invention is crushed so that the wasted lithium ion battery has the area of 100 through 1,600㎟.

바람직하게는, 본 발명의 상기 (b) 단계는 상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거된다.

Preferably, (b) step of the invention hots air-dry the crushed material to 300 through 550℃ and it is utterly the electrolyte removed.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시(mesh)의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와, 상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와, 상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함한다.

Preferably, (c) step of the invention minutely pulverizes the crushed material using the vibrating mill or the ball mill. (c) - 1 step pulverized, (c) - 2 step supplying the air of the high pressure to the fine pulverization article and disperses the cobalt compound powder, and (c) - 3 step passing the impurity removing filter the above-mentioned sprayed cobalt compound powder through and removes the impurity and the depositing obtains the pure cobalt compound powder are included so that it becomes the powder in which the cobalt compound has the particle size of 50 through 100 mesh.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와, 상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함한다.

Preferably, (c) step of the invention minutely pulverizes the crushed material using the vibrating mill or the ball mill. (c) - 4 step pulverized, and (c) - 5 step providing the fine pulverization article at the upper part of the screen device for being shaken and the selection obtains the cobalt compound powder with the free fall are included so that it becomes the powder in which the cobalt compound has the particle size of 50 through 100 mesh.

발명의 구성 및 작용

Structure & Operation of the Invention

이하, 본 발명의 구성을 상세히 설명하면 다음과 같다.

Hereinafter, the configuration of the invention is made to be equal to as described in detail, the next.

(a) 단계 - 적어도 하나 이상의 폐리튬 이온 전지 파쇄

The wasted lithium ion battery fracturing of (a) step - at least one.

폐리튬 이온 전지는 파쇄기를 이용하여 폐리튬 이온 전지의 파쇄 조각들이 100 내지 1,600㎟ 의 면적이 되도록 파쇄되는 것이 바람직하다. 이러한 방법은 철 재질인 케이스 부분과 내용물 부분이 파쇄물에 혼합된 상태로 수득된다.

It is the be desirable to be crushed so that the area of the smash piece lice 100 of the wasted lithium ion battery through 1,600㎟ becomes as to the wasted lithium ion battery, using the crusher. It is obtained by the case part in which such method is the iron material and the state where the content part is mixed in the crushed material.

다른 방법으로는 폐리튬 이온 전지의 철 재질인 케이스와 케이스에 내장된 내용물을 분리시키는 것으로 폐리튬 이온 전지의 중심 부분이나 양단부를 절단하여 케이스로부터 내용물을 분리한 다음, 내용물을 파쇄기를 이용하여 파쇄하는 것이다.

After the case which is the iron material of the wasted lithium ion battery and the content built in case are separated by the dissimilar method the center part or both end parts of the wasted lithium ion battery is cut and the content is separated from case the content is crushed using the crusher.

이러한 방법은 내용물만을 파쇄하기 때문에 전술된 방법(케이스가 포함되는 경우) 보다는 보다 적은 면적을 갖도록 파쇄하는 것이 바람직하다.

It is the be desirable to crush in order to have the area which is less than the method (in that case, case is included) in which such method is aforementioned because it crushes only the content.

(b) 단계 - 파쇄물을 건조하여 전해질을 제거

The one the electrolyte (b) step - crushed material is dried.

파쇄물은 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거한다.

It hots air-dry at 300 through 550℃ and the crushed material utterly removes the electrolyte.

여기서, 건조 과정이 300℃ 이만의 온도에서 건조가 수행되는 경우에는 작업 건조 시간이 길어져서 작업성이 양호치 못하고, 550℃ 초과의 온도에서 건조가 수행되는 경우에는 파쇄물 중의

Here, there is a problem that the operation drying time gets longer in case the drying of the drying procedure is performed at a temperature of 300℃ this much and th

페이스트 및 유기물 등이 착화되어 오염 물질이 발생되는 문제점이 있다.

e workability is unable to Yang HoHa and it becomes paste among the crushed material and organic compound etc. with ignition in case the drying is performed at a temperature of 550℃ excess and the contaminant is produced.

그리고, 건조 시간은 파쇄물의 양에 의해 결정된다.

And the drying time is determined by amount of the crushed material.

(c) 단계 - 파쇄물 미세 분쇄 및 코발트 화합물 분리

(c) step - crushed material grinding and cobalt compound separation .

건조가 완료된 파쇄물은 진동 밀 또는 볼 밀 등에 의해 미세 분쇄된다. 여기서, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 것이 매우 중요하다.

The crushed material in which the drying is finished is pulverized into the U.S. concatenation with the vibrating mill or the ball mill etc. Here, it is very important to pulverize so that it becomes the powder in which the cobalt compound has the particle size of 50 through 100 mesh.

미세 분쇄시 코발트 화합물은 50 내지 100 메시의 입도를 갖는 분말로 존재하고, 양극 성분인 알루미늄과 음극 성분인 구리는 각기 박판으로 존재하게 되며, 니켈 및 철은 코발트 화합물 분말에 비하여 5 내지 15배의 입도를 갖게 된다.

The cobalt compound in the grinding has the aluminum which exists as the powder having the particle size of 50 through 100 mesh and is the anode component and nickel and iron is the particle size of the cobalt compound powder 5 through 15 times the copper which is the cathode component respectively exists as the thin plate.

그리고, 분쇄물(분말)은 공급되는 고압의 공기에 의해 전술된 코발트 화합물 분말만이 비산되고, 코발트 화합물 분말을 제외한 알루미늄 및 구리 박판은 밀 표면에 압착되어 있으며 코발트 화합물 분말보다 상대적으로 중량인 니켈과 철 성분은 밀에 잔류된다. 여기서, 공급되는 고압의 공기는 코발트 화합물 분말만이 비산(공기 분급 방법)되도록 이에 상응하는 압력과 공기량을 갖는다.

And it is sprayed with the air of the high pressure in which the crushed material (powder) is supplied only the aforementioned cobalt compound powder and while aluminum except for the cobalt compound powder and copper foil are compressed in the mill surface the relatively nickel and the Fe component which is the weight are left behind in the mill than the cobalt compound powder. Here, the air of the supplied high pressure has the pressure which thus corresponds so that it becomes only the cobalt compound powder with the flying (air classification method) and the air volume.

비산된 코발트 화합물 분말은 덕트 등과 같은 폐쇄 경로를 통해 유동된 후 포집된다. 여기서, 비산된 코발트 화합물 분말 중에 불순물(특히, 알루미늄 또는 구리 박판)이 혼입될 수 있기 때문에 포집 전 단계에서 불순물 제거용 필터를 경유시켜 전술된 불순물을 제거한 후 순수한 코발트 화합물 분말을 수득하는 것이 바람직하다.

After the sprayed cobalt compound powder is flown through the closed contour including the duct etc. it is sampled. Here, it is be desirable to obtain the pure cobalt compound powder after the impurity which passes through the impurity removing filter in the depositing pre-phase because the impurity (especially, aluminum or the copper foil) can be mixed among the sprayed cobalt compound powder and is aforementioned is removed.

또한, 전술된 공기 분급 방법 이외에 간단한 코발트 화합물 분말을 수득하는 방법은 망체(網體)가 구비된 요동되는 스크린 장치의 상부에 미세 분쇄된 분말을 공급하는 것으로, 망체의 메시는 코발트 화합물 분말보다는 적어도 입도가 큰 것이 바람직하다. 따라서, 코발트 화합물 분말만이 스크린 장치의 하부로 자유 낙하되기 때문에 순수한 코발트 화합물을 수득할 수 있다.

Moreover, the powder which is minutely crushed is supplied to the upper portion of the screen device for being shaken in which as to the method, for obtaining the simple cobalt compound powder besides the aforementioned air classification method the net body is equipped. It is the be desirable that the mesh of the net body at least particle size is greater than the cobalt compound powder. Therefore, because it free drops to the lower part of the screen device only the cobalt compound powder the pure cobalt compound can be obtained.

(d) 단계 - 코발트 화합물 분말을 환원시켜 순순한 코발트 분말 수득

The obedient cobalt powder acquisition returning (d) step - cobalt compound powder

전술된 (c) 단계에서 수득된 코발트 화합물 분말 속에는 미량의 탄소 분말이 함유될 수 있기 때문에 우선적으로, 300℃ 이상의

If it does reduction and processes after carbon is removed by preferentially heating with superfine more

Ascend Elements EX1002 - Page 58

Duesenfeld Ex. 2014-058

고열로 가열함으로써 탄소를 제거한 후 환원처리하면 순수한 코발트 분말을 수득할 수 있다.

than 300℃ because the carbon-powder of the trace amount can be contained in the cobalt compound powder obtained in aforementioned (c) step the pure cobalt powder can be obtained.

이상에서 설명된 본 발명은 전술된 실시예에 한정되어 설명되었지만, 이에 한정되지 않고 본 발명이 속하는 분야의 통상적인 기술분야에서 통상의 지식을 가진 자가 용이하게 실시할 수 있는 정도의 변형은 본 발명의 기술적 사상에 속하는 것임은 자명하다.

In the above, it was restricted to the embodiment in which the explained invention was aforementioned and the embodiment was explained. However it is obvious that the deformation of the extent which a person skilled in the art easily can perform in the normal technical field of the field in which restricted the invention belongs belongs to the technical mapping of the invention.

Disclaimer

본 문서는 특허 및 과학기술문헌 전용의 첨단 자동번역 시스템을 이용해 생성되었습니다. 따라서 부분적으로 오역의 가능성이 있으며, 본 문서를 자격을 갖춘 전문 번역가에 의한 번역물을 대신하는 것으로 이용되어서는 안 됩니다. 시스템 및 네트워크의 특성때문에 발생한 오역과 부분 누락, 데이터의 불일치등에 대하여 본원은 법적인 책임을 지지 않습니다. 본 문서는 당사의 사전 동의 없이 권한이 없는 일반 대중을 위해 DB 및 시스템에 저장되어 재생, 복사, 배포될 수 없음을 알려드립니다.
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UNITED STATES DEPARTMENT OF COMMERCE
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NOTICE OF ALLOWANCE AND FEE(S) DUE

30743 7590 03/01/2021

W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

EXAMINER	
SWIATOCHA, GREGORY D.	
ART UNIT	PAPER NUMBER
3799	

DATE MAILED: 03/01/2021

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

TITLE OF INVENTION: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	06/01/2021

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.

Ascend Elements EX1002 Page 60

Duesenfeld Ex. 2014-060

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

30743 7590 03/01/2021

W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

(Typed or printed name)
(Signature)
(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

TITLE OF INVENTION: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	06/01/2021

EXAMINER	ART UNIT	CLASS-SUBCLASS
SWIATOCHA, GREGORY D.	3799	241-024130

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

❑ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

❑ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1_____
2_____
3_____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE (B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent) : ❑ Individual ❑ Corporation or other private group entity ❑ Government

4a. Fees submitted: ❑Issue Fee ❑Publication Fee (if required) ❑Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

❑ Electronic Payment via EFS-Web ❑ Enclosed check ❑ Non-electronic payment by credit card (Attach form PTO-2038)

❑ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

❑ Applicant certifying micro entity status. See 37 CFR 1.29

❑ Applicant asserting small entity status. See 37 CFR 1.27

❑ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____ Date _____

Typed or printed name _____ Registration No. _____

Ascend Elements EX1002 Page 61

Duesenfeld Ex. 2014-061

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

30743 7590 03/01/2021

W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

EXAMINER	
SWIATOCHA, GREGORY D.	
ART UNIT	**PAPER NUMBER**
3799	

DATE MAILED: 03/01/2021

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

Ascend Elements EX1002 Page 62

Duesenfeld Ex. 2014-062

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application [illegible] or a patent issued thereon.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Ascend Elements EX1002 Page 63

Duesenfeld Ex. 2014-063

Notice of Allowability	Application No. 15/569,484	Applicant(s) HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	Art Unit 3799	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to communications filed 26 June 2020.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 1-9 and 11-22 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information , please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☑ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   Certified copies:

   a) ☑ All b) ☐ Some *c) ☐ None of the:

   1. ☑ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)
2. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.
5. ☐ Examiner's Amendment/Comment
6. ☑ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3799

DETAILED ACTION

Notice of Pre-AIA or AIA Status

1) The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

2) The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Continued Examination Under 37 CFR 1.114

3) A request for continued examination under 37 CFR 1.114, including the fee set forth in 37 CFR 1.17(e), was filed in this application after final rejection. Since this application is eligible for continued examination under 37 CFR 1.114, and the fee set forth in 37 CFR 1.17(e) has been timely paid, the finality of the previous Office action has been withdrawn pursuant to 37 CFR 1.114. Applicant's submission filed on 4 June 2020 has been entered.

Information Disclosure Statement

4) The information disclosure statement (IDS) submitted is in compliance with the provisions of 37 CFR 1.97. Accordingly, the information disclosure statement is being considered by the examiner.

Response to Arguments

5) Applicant's arguments, filed 4 June 2020, with respect to Claims 1 and 7 have been fully considered and are persuasive. The rejection of Claims 1 and 7 has been withdrawn.

Allowable Subject Matter

6) Claims 1-9 and 11-22 are allowed.

7) The following is an examiner's statement of reasons for allowance:

8) Applicant's arguments, filed 4 June 2020, with respect to Claims 1 and 7 have been fully considered and are persuasive. The rejection of Claims 1 and 7 has been withdrawn and the claims are allowed.

9) Regarding Claims 4, 11, and 20, it is the opinion of the Examiner that the art of record neither anticipates nor renders obvious wherein "a pump liquid of the jet pump has at least one of a pH value of at least 8, and contains a substance that precipitates fluoride" or "wherein the pump liquid is conducted within a circuit and the pump liquid contains a substance that reacts with hydrogen fluoride" in combination with the rest of the claimed limitations set forth in the dependent claim. Searching by the Examiner yielded prior art as cited below:

10) Stevens discloses the removal of an electrolyte including lithium from the recycled battery using a vacuum system, scrubber, and an atomizer using a 50% concentration of sodium hydroxide in water, and this discloses a liquid with a pH of at least 9. Sodium hydroxide is also known to react with hydrogen fluoride to precipitate fluoride. However, the electrolyte disclosed by Stevens is lithium perchlorate in propylene carbonate and dimethoxyethane. While, Tanii et al. (U.S. 6,524,737) discloses the recycling of lithium batteries containing lithium phosphohexafluoride as the electrolyte, and thus the electrolyte of Stevens could be replaced with lithium phosphohexafluoride, the fluoride compounds are treated in a wastewater tank instead of with a jet pump. Further, while the use of jet pumps containing alkaline liquids such as sodium hydroxide is known from Eckardt et al. (U.S. 2014/0003568) such a use is not disclosed for the recycling of a lithium electrolyte battery.

11) Therefore, Stevens, Tanii, and Eckardt alone, or in combination with any other prior art, do not anticipate or render obvious the claimed invention.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled “Comments on Statement of Reasons for Allowance.”

Conclusion

12) Any inquiry concerning this communication or earlier communications from the examiner should be directed to GREGORY D. SWIATOCHA whose telephone number is (571)272-7022. The examiner can normally be reached on Monday-Friday (9-5).

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, **Tom Sweet** can be reached at **571-272-4761**. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3799

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3799

CPC - Searched*		
Symbol	**Date**	**Examiner**
Y02W30/84	09/04/2019	G.S.
Y02P10/212	09/04/2019	G.S.
B02C23/08, 10	09/04/2019	G.S.
B09B3/008, 0058	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102	09/04/2019	G.S.
H01M10/54	02/25/2021	G.S.
H01M6/52	02/25/2021	G.S.

CPC Combination Sets - Searched*		
Symbol	**Date**	**Examiner**

US Classification - Searched*			
Class	**Subclass**	**Date**	**Examiner**

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3725	

Duesenfeld Ex. 2014-068

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3799

Search Notes		
Search Notes	**Date**	**Examiner**
EAST Inventor search, Assignee search, CPC search, Text search, Combination class../text search	09/04/2019	G.S.
Espacenet search	09/04/2019	G.S.
IP.com search	09/04/2019	G.S.
NPL Search	09/04/2019	G.S.
Y02W30/84 (See search history printout)	09/04/2019	G.S.
Y02P10/212 (See search history printout)	09/04/2019	G.S.
B02C23/08, 10 (See search history printout)	09/04/2019	G.S.
B09B3/008, 0058 (See search history printout)	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102 (See search history printout)	09/04/2019	G.S.
Updated Search	03/04/2020	G.S.
Updated Search	02/25/2021	G.S.
Interference Search	02/25/2021	G.S.

Interference Search			
US Class/CPC Symbol	**US Subclass/CPC Group**	**Date**	**Examiner**
	Text search	02/25/2021	G.S.
Y02W	30/84	02/25/2021	G.S.
Y02P	10/212	02/25/2021	G.S.
B02C	23/08,10	02/25/2021	G.S.
B09B	3/008,0058	02/25/2021	G.S.
B01D	53/05, 002; 11/00; 2250/102	02/25/2021	G.S.
H01M	10/54; 6/52	02/25/2021	G.S.

/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3725	

Index of Claims	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3799

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

CLAIMS

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIM		DATE								
Final	Original	09/05/2019	03/04/2020	02/25/2021						
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3	3	✓	✓	=						
11	4	O	=	=						
4	5	✓	✓	=						
7	6	✓	✓	=						
12	7	✓	✓	=						
13	8	✓	✓	=						
14	9	✓	✓	=						
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16	13	✓	✓	=						
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21	20	O	=	=						
9	21		✓	=						
10	22			=						

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3799

CPC							
Symbol						**Type**	**Version**
H01M	/	10	/	54		F	2013-01-01
C22B	/	1	/	005		I	2013-01-01
H01M	/	6	/	52		I	2013-01-01
B02C	/	21	/	00		I	2013-01-01
B02C	/	23	/	10		I	2013-01-01
B02C	/	23	/	20		I	2013-01-01
B09B	/	3	/	0058		I	2013-01-01
Y02W	/	30	/	84		A	2015-05-01
Y02P	/	10	/	20		A	2015-11-01
B01D	/	53	/	002		A	2013-01-01
B01D	/	53	/	04		A	2013-01-01
B01D	/	2253	/	102		A	2013-01-01

CPC Combination Sets								
Symbol					**Type**	**Set**	**Ranking**	**Version**
	/		/					

NONE		Total Claims Allowed:	
(Assistant Examiner)	(Date)	21	
/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3799	25 February 2021	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	1

U.S. Patent and Trademark Office — Part of Paper No.: 20210225

Duesenfeld Ex. 2014-071

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3799

INTERNATIONAL CLASSIFICATION				
CLAIMED				
H01M10/54	/	10	/	54
B09B3/00	/	3	/	00
B01D53/00	/	53	/	00

NON-CLAIMED				
	/		/	

US ORIGINAL CLASSIFICATION	
CLASS	**SUBCLASS**

CROSS REFERENCES(S)						
CLASS	**SUBCLASS (ONE SUBCLASS PER BLOCK)**					

NONE		**Total Claims Allowed:**	
(Assistant Examiner)	(Date)	21	
/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3799	25 February 2021	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	1

U.S. Patent and Trademark Office — Part of Paper No.: 20210225

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3799

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIMS

Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original
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NONE		**Total Claims Allowed:**	
(Assistant Examiner)	(Date)	21	
/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3799	25 February 2021	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	1

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (02-18)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

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Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
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Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	2005197149	JP	A	2005-07-21	TOYOTA MOTOR CORP		X

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Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.D.S./

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

	1		

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EXAMINER SIGNATURE

Examiner Signature	/GREGORY D SWIATOCHA/	Date Considered	02/24/2021

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

☒ That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2020-06-26
Name/Print	Michael E. Whitham	Registration Number	32635

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Ascend Elements EX1002 Page 77

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Duesenfeld Ex. 2014-077

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L2	5,543	(H01M10/54 or H01M6/52).cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/25 22:40
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S3	3	duesenfeld.as.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 10:43
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S20	198	AU-2017347388-$.did. CA-3040793-$.did. US-20180301769-$.did. EP-3312922-$.did. CA-2983768-$.did. EP-3312922-$.did. DE-102016120046-$.did. WO-2018073101-$.did. EP-3529841-$.did. WO-2016174156-$.did. EP-3289627-$.did. DE-102015207843-$.did. DE-102015207843-$.did. CN-109845008-$.did. KR-20190084985-$.did. CN-107636875-$.did. WO-2019149698-$.did. EP-2446497-$.did. WO-2010149611-$.did. EP-2446497-$.did. DE-102013011470-$.did. DE-102018102026-$.did. DE-102009027179-$.did. EP-3517641-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 13:26

		DE-102013011470-$.did. EP-2742559-$.did. WO-2013023640-$.did. DE-102012024876-$.did. DE-4118001-$.did. EP-2742559-$.did. DE-102011110083-$.did. DE-4424825-$.did. DE-102011110083-$.did. DE-102015206153-$.did. AT-407753-$.did. IN-285315-$.did. US-20100162619-$.did. CA-2710719-$.did. AU-2007340905-$.did. US-9512376-$.did. DE-19547151-$.did. US-6789757-$.did. US-20030038197-$.did. CA-2391865-$.did. AU-2007340905-$.did. WO-2002023651-$.did. US-20170283725-$.did. EP-1269554-$.did. CA-2409218-$.did. WO-2019015892-$.did. EP-1269554-$.did. CA-2730336-$.did. US-20040028585-$.did. EP-2741357-$.did. CA-2710719-$.did. CA-3031475-$.did. TW-201728531-$.did. CH-678131-$.did. AU-2013201163-$.did. US-6461766-$.did. US-7192564-$.did. DE-102008058444-$.did. JP-H06338353-$.did. DE-19518643-$.did. JP-3240814-$.did. WO-2001091210-$.did. EP-1293003-$.did. EP-1215023-$.did. EP-1652253-$.did. WO-2005015668-$.did. WO-2008074352-$.did. EP-2376597-$.did. JP-3495707-$.did. WO-2008081408-$.did. EP-673075-$.did. WO-2018218358-$.did. CN-106684487-$.did. DE-102011082187-$.did. DE-202013010308-$.did. WO-2008081407-$.did. EP-2106435-$.did. CA-3043947-$.did. JP-H06338352-$.did. CN-106684487-$.did. WO-1994007277-$.did. WO-2008081409-$.did. WO-2008081410-$.did. US-5352270-$.did. EP-673075-$.did. EP-1333523-$.did. JP-3935594-$.did. WO-2008119525-$.did. US-5679477-$.did. US-5612150-$.did. JP-H11167936-$.did. US-20150080630-$.did. DE-10062947-$.did. DE-10045241-$.did. EP-3063811-$.did. US-9450277-$.did. JP-3079849-$.did. JP-2013229326-$.did. EP-1333522-$.did. US-20170012328-$.did. RO-106045-$.did. DE-10021383-$.did. EP-1333523-$.did. WO-2015063007-$.did. US-9620828-$.did. US-8067107-$.did. CN-105144464-$.did. US-6223449-$.did. US-20030180604-$.did. DE-102008055508-$.did. JP-6124001-$.did.				

		DE-19809400-$.did. US-20050244704-$.did. DE-10228471-$.did. US-6524737-$.did. DE-19547151-$.did. US-7833646-$.did. US-20170207504-$.did.				
S21	5,959	y02W30/84.cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:19
S22	1,437	(y02p10/212.cpc. b02c21/$.cpc. (b02c23/08,10).cpc. b09b3/008.cpc. (b01d53/02,002).cpc. b01d11/00.cpc. b09b3/0058.cpc. b01d2253/102.cpc.) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:31
S23	3	wo-2010102377-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S24	3	de-102011110083-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S25	2	de-102012024876-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S26	2	wo-2010149611-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S27	1	kr-20060101683-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO;	OR	ON	2019/09/04 14:37

			JPO; DERWENT; IBM_TDB			
S28	0	wo-2106174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S29	2	wo-2016174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S30	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S31	2	cn-101394015-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S32	3	de-102015207843-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S33	3	de-602005001937-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S34	3	cn-102496752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO;	OR	ON	2019/09/04 14:37

			DERWENT; IBM_TDB			
S35	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S38	352	S22 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S39	2,006	S21 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S40	3	de-19954998-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 19:41
S41	2	de-29803442-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 19:41
S42	1	(S38 S39) AND (liquid WITH gas WITH jet WITH (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:19
S44	484	(liquid WITH gas WITH jet WITH (ejector pump)) WITH vacuum	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO;	OR	ON	2019/09/04 20:21

			DERWENT; IBM_TDB			
S45	8	(liquid WITH gas WITH jet WITH (ejector pump) WITH vacuum) SAME pH	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:21
S46	21	(jet ADJ (ejector pump)) WITH ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:26
S47	169	(S38 S39) AND ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:27
S48	3	WO-2013140039-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:31
S49	3	(S38 S39) AND (jet ADJ (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:39
S50	8	(jet ADJ (ejector pump)) WITH fluoride	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:41
S51	1	(jet ADJ (ejector pump)) WITH (fluoride fluorine) WITH (carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO;	OR	ON	2019/09/04 20:42

			DERWENT; IBM_TDB			
S53	174	(S38 S39) AND (electrolyte WITH (remov$4 weight))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 21:02
S54	28	(S38 S39) AND (electrolyte WITH (remov$4 vaporiz$3) WITH (dri$2 dry$3))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 21:06
S55	5,959	y02W30/84.cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34
S56	658	S55 and lithium and (dry$3 dri$2)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34
S61	11	(venturi adj pump) SAME (fluorine fluoride carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 10:54
S62	12	"9780419" "20050241943" "7713396"	US-PGPUB; USPAT; USOCR	OR	ON	2019/09/05 11:07
S63	3	wo-2010102377-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S64	3	de-102011110083-$.did.	US-PGPUB; USPAT;	OR	ON	2019/09/05 11:07

			USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB			
S65	2	de-102012024876-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S66	2	wo-2010149611-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S67	1	kr-20060101683-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S68	0	wo-2106174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S69	2	wo-2016174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S70	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S71	2	cn-101394015-$.did.	US-PGPUB; USPAT; USOCR;	OR	ON	2019/09/05 11:07

			FPRS; EPO; JPO; DERWENT; IBM_TDB			
S72	3	de-102015207843-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S73	3	de-602005001937-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S74	3	cn-102496752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S75	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S76	27	S63 S64 S65 S66 S67 S68 S69 S70 S71 S72 S73 S74 S75	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S77	160	CA-2983768-$.did. US-20190260101-$.did. AU-2017347388-$.did. WO-2016174156-$.did. EP-3289627-$.did. CA-3040793-$.did. DE-102015207843-$.did. DE-102015207843-$.did. EP-3312922-$.did. EP-3312922-$.did. DE-102016120046-$.did. WO-2018073101-$.did. EP-3529841-$.did. CN-109845008-$.did. KR-20190084985-$.did. CN-107636875-$.did. WO-2019149698-$.did. DE-102013011470-	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:08

		$.did. DE-102018102026-$.did. EP-2446497-$.did. EP-3517641-$.did. WO-2010149611-$.did. EP-2446497-$.did. DE-102012024876-$.did. EP-2742559-$.did. WO-2013023640-$.did. DE-102013011470-$.did. EP-2742559-$.did. DE-102009027179-$.did. DE-102011110083-$.did. WO-2008074352-$.did. DE-4424825-$.did. DE-102011110083-$.did. DE-102015206153-$.did. WO-2019015892-$.did. TW-201728531-$.did. JP-H06338353-$.did. US-5612150-$.did. EP-673075-$.did. US-5679477-$.did. JP-2013229326-$.did. JP-3240814-$.did. DE-202013010308-$.did. CN-106684487-$.did. JP-3495707-$.did. DE-102011082187-$.did. EP-673075-$.did. CN-105144464-$.did. JP-3935594-$.did. US-3748186-$.did. US-8067107-$.did. US-20050244704-$.did. CN-106684487-$.did. WO-2018218358-$.did. EP-2741357-$.did. CN-105021024-$.did. CA-3043947-$.did. JP-6124001-$.did. JP-H11167936-$.did. DE-19924707-$.did. WO-2002023651-$.did. CA-2391865-$.did. EP-1269554-$.did. KR-20190078653-$.did. CN-108306050-$.did. DE-102017212266-$.did. WO-2005015668-$.did. EP-1652253-$.did. CA-2730336-$.did. JP-H06338352-$.did. EP-1056146-$.did. US-20040028585-$.did. EP-1269554-$.did. CN-105594056-$.did. US-6524737-$.did. KR-20150135304-$.did. US-7192564-$.did. DE-102015010955-$.did. CN-105594056-$.did. WO-2014155784-$.did. DE-102018100158-$.did. US-9937481-$.did. US-7198865-$.did. EP-1041659-$.did. CN-109777957-$.did. CN-107910610-$.did. EP-3063811-$.did. WO-1994007277-$.did. JP-3079849-$.did. TW-I543431-$.did. US-20150080630-$.did. CA-2311792-$.did. CN-109449525-$.did. DE-19518643-$.did. US-5352270-$.did. TW-201724628-$.did. DE-4118001-$.did. DE-202014007301-$.did. DE-202013006631-$.did. US-9450277-$.did. DE-202013006632-$.did. WO-2015063007-$.did. US-20170012328-$.did. AT-407753-$.did. JP-2016207463-$.did. RU-2201018-$.did.				

S78	156	US-20170170529-$.did. KR-100300829-$.did. JP-2016207464-$.did. US-9620828-$.did. WO-2003061056-$.did. US-6511639-$.did. DE-19532626-$.did. EP-1472756-$.did. DE-102013111853-$.did. US-20070292746-$.did. JP-2017084707-$.did. KR-19980064595-$.did. CN-1442928-$.did. EP-1333523-$.did. DE-102013105676-$.did. AU-2003205087-$.did. US-7858216-$.did. US-20180198115-$.did. EP-1472756-$.did. CA-2309773-$.did. EP-1333522-$.did. US-9843077-$.did. US-20170207504-$.did. WO-2017000157-$.did. KR-20070090851-$.did. US-20160049699-$.did. US-20030186110-$.did. EP-3063811-$.did. DE-10049097-$.did. DE-102012109032-$.did. JP-2001332237-$.did. EP-1333523-$.did. US-9912022-$.did. WO-2017073686-$.did. KR-20030039376-$.did. JP-6245412-$.did. DE-19547151-$.did. JP-4187804-$.did. JP-2017084706-$.did. US-20030180604-$.did. DE-10049097-$.did. WO-2019150403-$.did. JP-5050845-$.did. JP-2000223123-$.did. JP-H0785889-$.did. CN-104412426-$.did. US-20130122398-$.did. WO-2015046211-$.did. KR-100997349-$.did. US-7833646-$.did. CN-109478473-$.did. US-20040096746-$.did. US-20180198179-$.did. DE-102007012693-$.did. JP-2014222582-$.did. US-9728807-$.did. US-20150188137-$.did. EP-854530-$.did. CN-107910610-$.did. DE-102014226241-$.did. JP-2019008980-$.did. CN-1442928-$.did. JP-2012204131-$.did. US-6789757-$.did. US-20030038197-$.did. JP-2016152226-$.did. JP-5894688-$.did. WO-1998044580-$.did. EP-1330299-$.did. CN-107403930-$.did. US-5972531-$.did. EP-854530-$.did. DE-102013012250-$.did. US-8999009-$.did. DE-102010020738-$.did. KR-20160064104-$.did. US-6986967-$.did. WO-2002028500-$.did. US-6461766-$.did. JP-2014160575-$.did. JP-2015179666-$.did. EP-2894702-$.did. US-20130232772-$.did. JP-2013146680-$.did. EP-972313-$.did. US-7910242-$.did. CA-2285275-$.did. CA-2418527-	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:08

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S81	500	S77 S78 S79 S80	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:08
S82	204	(vacuum WITH scrub$4) SAME (fluorine fluoride carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:14
S83	204	(vacuum WITH scrub$4) SAME (fluorine fluoride carbonate)S82 and (battery batteries)	US-PGPUB; USPAT; USOCR;	OR	ON	2019/09/05 11:14

			FPRS; EPO; JPO; DERWENT; IBM_TDB			
S84	9	S82 and (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:15
S85	111	(vacuum WITH scrub$4) SAME (fluorine fluoride)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:28
S86	4	(vacuum WITH scrub$4) SAME ((fluorine fluoride) AND carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:28
S87	2	wo-2013023640-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 16:16
S88	106	((sodium adj hydroxide) NaOH) WITH ((jet adj pump) ejector)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 13:48
S89	23	(US-20190260101-$ or US-20180301769-$ or US-20180205122-$ or US-20030186110-$ or US-20070134546-$ or US-20160251740-$ or US-20160043450-$ or US-20140290438-$ or US-20100230518-$ or US-20040113588-$ or US-20030180604-$ or US-20140003568-$).did. or (US-9780419-$ or US-6524737-$ or US-5632863-$).did. or (DE-4424825-$ or CA-2311792-$).did.	US-PGPUB; USPAT; FPRS; DERWENT	OR	ON	2019/09/07 13:50

		or (WO-2010102377-$ or DE-102011110083-$ or WO-2013140039-$ or CN-102496752-$ or CN-101183740-$ or EP-1333522-$).did.				
S91	8	S89 and (fluor$3)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 13:50
S92	1	(carbonate WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:24
S93	0	(base WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:24
S94	0	((basic alkalin$3) WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S95	18	((bas$2 alkalin$3) WITH ((jet adj pump) ejector) SAME (fluorine fluoride))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S96	3	(carbonate WITH ((jet adj pump) ejector) SAME (fluorine fluoride))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S97	2	"20070134546"	US-PGPUB; USPAT; USOCR	OR	ON	2019/09/08 12:20

S99	2	wo-2013023640-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/08 14:28
S100	1	"8210456".pn.	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:39
S101	214	"8210456" "1587623" "3393876" "3456886" "3493183" "3777994" "3892563" "4026477" "4384683" "4775107" "5377920" "5575907" "5632863" "5707015" "9780419" "20050241943"	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:48
S102	2	"20140290438"	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:49
S104	24	(US-20190260101-$ or US-20180301769-$ or US-20180205122-$ or US-20030186110-$ or US-20070134546-$ or US-20160251740-$ or US-20160043450-$ or US-20140290438-$ or US-20100230518-$ or US-20040113588-$ or US-20030180604-$ or US-20140003568-$).did. or (US-9780419-$ or US-6524737-$ or US-5632863-$ or US-8210456-$).did. or (DE-4424825-$ or CA-2311792-$).did. or (WO-2010102377-$ or DE-102011110083-$ or WO-2013140039-$ or CN-102496752-$ or CN-101183740-$ or EP-1333522-$).did.	US-PGPUB; USPAT; FPRS; DERWENT	OR	ON	2020/03/04 20:53
S105	21	(S100 S101 S102 S104) AND (dry$3 SAME (vacuum pressure))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/03/04 20:53
S106	957	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/24 21:46

		((dry$3 dried inactivat$3) SAME (vacuum Mpa hPa kPa)))				
S107	571	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND ((dry$3 dried inactivat$3) WITH (vacuum Mpa hPa kPa)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/24 21:47
S108	210	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND (batter$3 electrode) AND ((dry$3 dried inactivat$3) WITH (vacuum Mpa hPa kPa)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/24 21:51
S109	44	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND (batter$3 electrode) AND ((dry$3 dried inactivat$3) WITH (vacuum pressure) WITH (Mpa hPa kPa)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/24 22:02
S112	294	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND (batter$3 electrode) AND ((dry$3 dried inactivat$3) WITH (vacuum pressure)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/25 11:33
S113	275	(H01M10/54 or H01M6/52 or B02C21/00 or B02C23/10 or B02C23/20 or B09B3/0058 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002 or B01D53/04 or B01D2253/102).cpc. AND ((shred$4 crush$4 comminut$4 grind$3) AND (batter$3 electrode) AND ((dry$3 dried inactivat$3) WITH (vacuum negative)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/25 11:33

S119	360	S112 S113	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/25 14:58
S121	360	S119 and ((shred$4 crush$4 comminut$4 grind$3) AND ((dry$3 dried inactivat$3) WITH (vacuum pressure negative)))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2021/02/25 15:13

EAST Search History (Interference)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	6	((shred$4 crush$4 comminut$4 grind$3) AND inactiv$5 AND (dry$3 drie$2 oven) AND (pressure vacuum negative) and (battery batteries electrode)).clm.	US-PGPUB; USPAT	OR	ON	2021/02/25 22:12
L3	23	((shred$4 crush$4 comminut$4 grind$3) AND (dry$3 drie$2 oven) AND (pressure vacuum negative) and (battery batteries electrode)).clm. AND (H01M10/54 or H01M6/52 or B02C21/00 or B02C23/08,10 or B02C23/20 or B09B3/0058,008 or C22B1/005 or Y02P10/20 or Y02W30/84 or B01D53/002,05 b01d11/00 or B01D53/04 or B01D2253/102 Y02P10/212).cpc.	US-PGPUB; USPAT	OR	ON	2021/02/25 22:43

2/25/2021 10:48:06 PM
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

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	1	2005197149	JP	A	2005-07-21	TOYOTA MOTOR CORP		X

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

	1		

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Examiner Signature		Date Considered	

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

☒ That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2020-06-26
Name/Print	Michael E. Whitham	Registration Number	32635

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Espacenet

Bibliographic data: JP2005197149 (A) — 2005-07-21

LITHIUM BATTERY PROCESSING METHOD

Beschreibt ein Erwärmen auf 85°C unter 100 hPa.

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Classification: - international: ***C01D15/02; C01F7/42; C01F7/50; C22B21/00; C22B26/10; C22B7/00; H01M10/54;*** (IPC1-7): C01D15/02; C01F7/42; C01F7/50; C22B21/00; C22B26/10; C22B7/00; H01M10/54

- cooperative: Y02P10/212 (EP); Y02W30/84 (EP)

Application number: JP20040003786 20040109 Global Dossier

Priority number(s): JP20040003786 20040109

Abstract of JP2005197149 (A)

PROBLEM TO BE SOLVED: To provide a processing method of a lithium battery having a fluorine based electrolyte in which the electrolyte is effectively converted into a morphology of good handling nature. ;SOLUTION: The processing method comprises a hydrolysis process (step 240) in which the treating material containing lithium-transition metal-containing complex oxide (positive electrode active material etc.) and aluminum member (positive electrode current collector etc.) is treated in a high temperature high humidity atmosphere. In this process, lithium hydroxide is generated from the compound oxide and a hydroxide of aluminum is generated from the aluminum member. The aluminum hydroxide originated from the hydroxide is reacted with fluorine-containing substance (for example, PF<SB>5</SB>) obtained by heating and removing the electrolyte (for example, LiPF<SB>6</SB>) from the lithium battery (step 282), and a substance with good handling nature (for example, AlF<SB>3</SB>and AlPO<SB>4</SB>) is generated (step 284). ;COPYRIGHT: (C)2005,JPO&NCIPI

Duesenfeld Ex. 2014-106

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CLAIMS JP2005197149

1.
A method for treating a lithium battery comprising a positive electrode active material substantially composed of a composite oxide containing lithium and one or more transition metal elements, an electrolyte containing fluorine as a constituent element, and an aluminum member. H2O is supplied to a material to be treated, which is derived from the lithium battery and includes the positive electrode active material and the aluminum member, to generate lithium hydroxide from the positive electrode active material and to oxidize aluminum that constitutes the aluminum member. A method for treating a lithium battery, comprising: a step of producing lithium hydroxide or the like to generate a substance; and a step of reacting a fluorine-containing substance derived from the electrolyte with aluminum hydroxide derived from the hydroxide.

2.
The step of removing the electrolyte from the lithium battery is performed prior to the step of producing lithium hydroxide or the like, and the fluorine-containing substance derived from the removed electrolyte is reacted with aluminum hydroxide. Method.

3.
The method according to claim 1 or 2, wherein the step of reacting the fluorine-containing substance with aluminum hydroxide includes a treatment of bringing a treatment liquid containing aluminum hydroxide into contact with a gas containing a thermal decomposition product of the electrolyte.

Ascend Elements EX1002 Page 107

Duesenfeld Ex. 2014-107

4.
The method according to any one of claims 1 to 3, wherein the electrolyte comprises lithium hexafluorophosphate.

5.
A step of eluting water with hydroxides of lithium hydroxide and aluminum produced in the step of producing lithium hydroxide or the like to separate from the insoluble matter of the material to be treated, and aluminum hydroxide from the solution obtained in the step of separating. The method according to any one of claims 1 to 4, further comprising a collecting step.

6.
The method according to claim 5, wherein the collecting step includes a process of adjusting the pH of the solution obtained in the separating step to precipitate aluminum hydroxide from the solution.

7.
The method further comprising: a step of supplying carbon dioxide gas and / or carbonated water to the lithium solution obtained through the separation step to precipitate lithium carbonate; and a step of separating the precipitated lithium carbonate from the solution. Or the method according to 6.

8.
The method according to claim 1, wherein the aluminum member is a positive electrode current collector, and the material to be treated includes a positive electrode having the positive electrode active material on the positive electrode current collector.

9.
The lithium battery includes a wound-type electrode body having a sheet-shaped positive electrode and a sheet-shaped negative electrode, and at least one of the positive electrode and the negative electrode includes an aluminum current collector and includes the wound-type electrode body. The method according to any one of claims 1 to 8, wherein the material to be treated is subjected to the step of producing lithium hydroxide or the like.

10.
The method according to any one of claims 1 to 9, wherein the lithium battery is heated to

Ascend Elements EX1002 Page 108

Duesenfeld Ex. 2014-108

remove the electrolytic solution, and a part or all of the battery constituent member remaining after the heating is subjected to the lithium hydroxide etc. producing step. .

11.

10. The lithium battery is heated to remove the electrolyte and organic matter, and a part or all of the battery constituent members remaining after the heating is subjected to the lithium hydroxide etc. generating step. Method.

12.

The step of producing lithium hydroxide or the like includes a step of producing a hydroxide of at least one transition metal element among the one or two or more transition metal elements together with lithium hydroxide from the positive electrode active material. 11. The method according to any one of 11.

13.

A step of eluting at least one transition metal element among the one or more transition metal elements contained in the insoluble matter of the material to be treated with an acid, and a neutralizing agent added to the solution to elute into the acid The method according to any one of claims 1 to 12, further comprising: a step of depositing a hydroxide of the transition metal element.

14.

The method includes a step of heating the lithium battery to volatilize and remove a volatile material, and recover the volatile matter. Before the volatile matter recovery step, a step of previously removing water in an apparatus used for the recovery is performed. The method according to any one of claims 1 to 13.

Ascend Elements EX1002 Page 109

Duesenfeld Ex. 2014-109

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DESCRIPTION JP2005197149

PROBLEM TO BE SOLVED: To provide a method for treating a lithium battery provided with a fluorine-based electrolyte, which is capable of efficiently converting the electrolyte into a form having good handleability. SOLUTION: The treatment method of the present invention is a hydrolysis step of treating a material to be treated including a lithium / transition metal-containing composite oxide (positive electrode active material etc.) and an aluminum member (positive electrode current collector etc.) in a high temperature and high humidity atmosphere. (Step 240). In this step, lithium hydroxide is produced from the composite oxide and aluminum hydroxide is produced from the aluminum member. Aluminum hydroxide derived from the hydroxide is reacted with a fluorine-containing substance (for example, PF) obtained by heating and removing an electrolyte (for example, LiPF) from a lithium battery (step 282), and a substance having good handleability (AlF and AiPO), for example (step 284). [Selection diagram]

Lithium battery treatment method

[0001]

The present invention relates to a technique for treating a lithium battery, and particularly to a technique for efficiently treating a lithium battery including an electrolyte containing fluorine as a constituent element.

[0002]

Various methods have been proposed for recovering valuables from waste lithium batteries.

Ascend Elements EX1002 Page 110

Duesenfeld Ex. 2014-110

For example, Patent Document 1 describes a technique for recovering and regenerating a lithium component and a transition metal component from a composite oxide of lithium and a transition metal element (lithium cobalt oxide or the like). In this document, the battery positive electrode waste material is dissolved in nitric acid, and this is filtered to separate into a filtrate and a residue containing lithium nitrate and a nitrate of a transition metal element, and the filtrate is subjected to various treatments to give a transition metal component. And lithium components are respectively recovered and regenerated. Patent documents 2-12 are mentioned as other prior art documents.

[0003]
JP 11-54159 A JP 10-255861 A JP 10-255862 A JP 11-6020 A JP 2000-15216 A JP 2003-27151 A US Pat. No. 5,888,463 JP-A-8-41554, JP-A-8-134556, JP-A-2000-119763, JP-A-11-167936, JP-A-2000-106221

[0004]

By the way, most of lithium batteries use lithium salts (LiPF6, LiBF4, LiAsF6, etc.) containing fluorine as a constituent element as an electrolyte (supporting salt).

A treatment process of a lithium battery including such a fluorine-based electrolyte (for example, a process of recovering a valuable material) may involve a step of treating the electrolyte. For example, a treatment process of a lithium battery containing LiPF6 may produce a highly active fluorine-containing compound such as phosphorus fluoride. By providing the step of converting the phosphorus fluoride or the like into a compound having good handleability (stable), the fluorine-based electrolyte can be appropriately treated (discarded, reused, etc.).

[0005]

Therefore, the present invention provides a method for treating a lithium battery including a fluorine-based electrolyte, which is capable of efficiently converting the electrolyte into a form having good handleability (eg, suitable for disposal). With the goal. Another related object is to provide a method for recovering valuables from lithium batteries in a highly useful (eg, reusable) form and / or more efficiently.

Ascend Elements EX1002 Page 111

Duesenfeld Ex. 2014-111

[0006]

The present inventor has noticed that aluminum members are often used as constituent elements of lithium batteries. Then, the hydroxide of aluminum can be used to convert the fluorine-based electrolyte into a form having good handleability, and the aluminum hydroxide can be oxidized from the aluminum member (for example, the positive electrode current collector) in the treatment process of the lithium battery. The present invention has been completed by finding that the product can be efficiently produced.

[0007]

One invention provided by this application is a positive electrode active material substantially composed of a composite oxide containing lithium and one or more transition metal elements, an electrolyte containing fluorine as a constituent element, and aluminum. And a method for treating a lithium battery (typically a lithium-ion secondary battery) including a member. The method is to supply H 2 O to a material to be treated derived from the lithium battery, the material to be treated including the positive electrode active material and the aluminum member to generate lithium hydroxide from the positive electrode active material and the aluminum member. From the step of producing aluminum hydroxide (lithium hydroxide etc. producing step). Further, the method includes a step of reacting a fluorine-containing substance derived from the electrolyte with aluminum hydroxide derived from the hydroxide.

[0008]

Here, the "aluminum member" means that aluminum (aluminum or an alloy mainly containing aluminum is a main constituent material). same as below.) Refers to a battery constituent member. An electrolyte containing fluorine as a constituent element (hereinafter, also referred to as “fluorine-based electrolyte”). Examples of () include LiPF6, LiBF4, LiAsF6, LiCF3SO3, LiC4F9SO3, LiN (CF3SO2) 2, LiC (CF3SO2) 3 and the like. The present invention can be applied to a lithium battery including one or more of these. Particularly, it is preferably applied to a lithium battery including an electrolyte containing lithium hexafluorophosphate (LiPF6). The “fluorine-containing substance derived from a fluorine-based electrolyte” means the fluorine-based electrolyte itself (LiPF6, etc.) and a fluorine compound (PF5, HF) produced by decomposition (eg, thermal decomposition or hydrolysis) of the electrolyte. Etc.) and is meant to include.

Ascend Elements EX1002 Page 112

Duesenfeld Ex. 2014-112

[0009]

A preferable example of the composite oxide is a composite oxide containing lithium and nickel. Here, the "composite oxide containing lithium and nickel" means an oxide containing lithium and nickel as constituent metal elements, and at least one other metal element other than lithium and nickel (that is, lithium and nickel). It is also meant to include complex oxides containing transition metal elements other than the above and / or typical metal elements). Such a complex oxide can be represented by, for example, the general formula LiNi1-xAxO2. A in the formula corresponds to the above-mentioned "at least one other metal element". Further, x in the formula is a value satisfying $0 \leqq x < 0.5$, preferably $0.1 < x < 0.3$. Other preferable examples of the composite oxide include a composite oxide containing lithium and cobalt (including a composite oxide containing at least one metal element other than lithium and cobalt) and a composite oxide containing lithium and manganese. Products (including those containing at least one metal element other than lithium and manganese).) Is mentioned.

[0010]

It is possible to convert a fluorine-containing substance derived from a fluorine-based electrolyte into a stable compound by utilizing aluminum hydroxide. For example, phosphorus fluoride (for example, PF5) generated by decomposition of lithium hexafluorophosphate (LiPF6) is reacted with aluminum hydroxide (typically, aluminum hydroxide represented by the composition formula Al (OH) 3). By doing so, aluminum trifluoride (AlF3), aluminum phosphate (AlPO4), or the like can be generated. All of these reaction products are stable compounds. Therefore, it is relatively easy to handle. It is also a substance suitable for disposal (stable waste). As such, aluminum hydroxide can function as a fixing agent for converting the fluorine-based electrolyte into a stable compound.

[0011]

In the step of producing lithium hydroxide or the like, the composite oxide contained in the material to be treated (hereinafter, also referred to as "lithium / transition metal-containing composite oxide" or simply "composite oxide").) Is reacted with H2O to produce lithium hydroxide. The presence of such lithium hydroxide exposes the aluminum member to alkaline conditions, which promotes the reaction of producing aluminum hydroxide (corrosion of the

Ascend Elements EX1002 Page 113

Duesenfeld Ex. 2014-113

aluminum member). Therefore, the aluminum hydroxide can be efficiently generated from the aluminum member. Then, aluminum hydroxide derived from the hydroxide (aluminum hydroxide produced in the production step such as lithium hydroxide and aluminum hydroxide obtained by subjecting the hydroxide produced in the step to an appropriate treatment Meaning of inclusion.) Can be used to efficiently convert the fluorine-based electrolyte into a stable compound. As described above, according to the above treatment method, the aluminum hydroxide generated from the constituent member (aluminum member) of the lithium battery is used in the treatment process of the lithium battery to fix the fluorine-containing substance derived from the fluorine-based electrolyte. be able to. Therefore, it is not necessary to bring in a fixing agent such as calcium hydroxide (Ca (OH) 2) from the outside of the treatment process for use. Therefore, according to the above treatment method, the fluorine-based electrolyte can be efficiently converted into a stable compound. Further, the aluminum member which is a constituent material of the lithium battery can be effectively utilized as a treating agent (fixing agent) for the fluorine-based electrolyte.

[0012]

In a preferred embodiment of the method disclosed herein, a step of removing the electrolyte from the lithium battery is performed prior to the step of producing lithium hydroxide or the like. The fluorine-containing substance derived from the removed electrolyte is reacted with aluminum hydroxide. As described above, according to the aspect in which the fluorine-containing substance derived from the electrolyte removed in advance and the aluminum hydroxide are reacted, the electrolyte can be more efficiently converted into a stable compound, and the handleability of the member to be treated is improved. At least one of the effects can be realized. As a method of removing the electrolyte from the lithium battery, a method of heating the lithium battery to volatilize (pyrolysis) the electrolyte contained in the lithium battery and remove it can be preferably adopted. In addition, a lithium battery is extracted with a non-aqueous organic solvent or the like to have an electrolytic solution (generally, an electrolyte is dissolved in an organic solvent.) May be removed to remove the electrolyte from the lithium battery. As another method for removing the electrolyte from the lithium battery, as shown in JP-A-8-22846, the battery is immersed in an alkaline aqueous solution or an acidic aqueous solution, and the aqueous solution is submerged from a safety valve opened to form an electrolytic solution. There is a method of dissolving and removing.

[0013]

In another preferred embodiment of the method disclosed herein, the step of reacting the

Ascend Elements EX1002 Page 114

Duesenfeld Ex. 2014-114

fluorine-containing substance with aluminum hydroxide comprises a treatment liquid containing aluminum hydroxide (typically Al (OH) 3 is used as an aqueous solvent. (Dissolved and / or dispersed) is contacted with a gas containing a thermal decomposition product of the electrolyte. According to this aspect, the thermal decomposition product of the electrolyte (including the fluorine-containing substance) and aluminum hydroxide can be efficiently reacted. The gas containing the thermal decomposition product of the electrolyte (pyrolysis gas) may be generated by heating a lithium battery provided with the electrolyte, or may be generated by separately heating (thermally decomposing) the electrolyte previously removed from the lithium battery. It may be a thing. In the case of a lithium battery provided with an electrolyte containing lithium hexafluorophosphate, the thermal decomposition products that can be contained in the thermal decomposition gas include phosphorus fluoride (for example, PF5) and hydrogen fluoride (HF).

[0014]

Another preferred embodiment of the method disclosed herein may include a step of eluting lithium hydroxide and aluminum hydroxide generated in the step of producing lithium hydroxide or the like into water to separate them from insoluble matter of the material to be treated. it can. Further, a step of recovering aluminum hydroxide from the solution obtained by the separating step can be included. In the step of producing lithium hydroxide or the like, H 2 O is caused to act on the lithium / transition metal-containing composite oxide contained in the material to be treated to generate lithium hydroxide. Lithium hydroxide is water soluble. The aluminum hydroxide produced from the aluminum member in the lithium hydroxide production step is eluted with water together with lithium hydroxide as, for example, Al (OH) 3, Li [Al (OH) 4 (H2O)]. obtain. On the other hand, the lithium / transition metal-containing composite oxide before the production step such as lithium hydroxide and the transition metal compound (typically transition metal hydroxide) that can be produced together with lithium hydroxide in the step are mainly substantially It is insoluble in water. Therefore, the hydroxide of lithium hydroxide and aluminum can be separated and taken out from the insoluble matter (including the transition metal component) of the material to be treated by means such as elution of the material to be treated with H2O with water. it can. For example, the treated material (positive electrode active material) after the treatment is dispersed in water and then filtered to obtain a liquid phase containing the insoluble matter of the treated material and a lithium component and an aluminum component (lithium hydroxide and aluminum water). A solution in which an oxide is dissolved in water) can be easily separated. The step of generating hydroxides of lithium and aluminum from the material to be treated and the step of separating the lithium component and the aluminum component from the insoluble matter of the material to be treated may be simultaneously performed. The lithium and aluminum hydroxide generated from the material to be treated may be eluted together or sequentially. For example, first, lithium hydroxide may be

Ascend Elements EX1002 Page 115

Duesenfeld Ex. 2014-115

eluted, and then aluminum hydroxide may be eluted (separated from insoluble matter).

[0015]

When aluminum hydroxide is recovered from the solution separated from the insoluble matter of the material to be treated in this way, a method including a treatment of adjusting the pH of the solution to precipitate aluminum hydroxide can be preferably adopted. For example, aqueous ammonia is added to the above solution containing a lithium component and an aluminum component to precipitate gelatinous aluminum hydroxide. By centrifuging this, a supernatant solution containing lithium hydroxide (lithium solution) and gelatinous aluminum hydroxide can be separated and recovered. By reacting the aluminum hydroxide thus recovered with the above-mentioned fluorine-containing substance, the aluminum hydroxide derived from the battery constituent material (aluminum member) can be efficiently used as a fixing agent. For example, by contacting a treatment liquid in which the recovered aluminum hydroxide is dissolved and / or dispersed in an aqueous solvent (typically water) with a gas containing a thermal decomposition product of a fluorine-based electrolyte (PF5 or the like), The thermal decomposition product can be efficiently converted into a stable compound (stable waste). As another method for recovering aluminum hydroxide from the above solution, the pH of the solution is adjusted as shown in Item 0025 of JP-A-11-54159 and Item 0041 of JP-A-11-97076. Examples of the method include precipitation and separation / collection.

[0016]

A preferred embodiment of the method disclosed herein includes the step of precipitating lithium carbonate from the lithium solution obtained through the step of separating the insoluble matter of the material to be treated as described above. For example, lithium carbonate can be deposited by supplying carbon dioxide gas (CO2) and / or carbonated water (H2CO3) to the lithium solution. It is preferable to further include a step of separating the precipitated lithium carbonate from the solution. As the lithium solution, it is preferable to use a solution obtained by further separating the aluminum component (aluminum hydroxide) from the solution separated from the insoluble matter of the material to be treated. Thereby, lithium carbonate with higher purity can be obtained.

[0017]

Ascend Elements EX1002 Page 116

Duesenfeld Ex. 2014-116

According to this aspect, the lithium component (lithium ion) contained in the lithium solution can be recovered as a solid lithium compound (lithium carbonate). The lithium component thus recovered as a solid is convenient for reuse. Carbon dioxide or carbonated water (more preferably carbon dioxide) may be used to precipitate lithium carbonate from the lithium solution. By precipitating lithium carbonate using a material containing no alkali metal component in this way, lithium carbonate containing less alkali metal elements other than lithium can be obtained. For example, as compared with the case where sodium carbonate is used instead of carbon dioxide gas or carbonated water, the amount of sodium component mixed in the obtained lithium carbonate can be significantly reduced. It is generally difficult to separate the sodium component once mixed from the lithium component, but according to the above-described embodiment, the purity is high by a simple operation (particularly, the alkali metal element other than lithium and the transition metal component are less mixed). Lithium carbonate can be obtained. Thus, high-purity lithium carbonate is excellent in reusability for various uses. For example, it is suitable as a material for a lithium battery (raw material for producing a positive electrode active material, etc.).

[0018]

In a typical aspect of this treatment method, in the step of producing lithium hydroxide or the like, a hydroxide of at least one transition metal element among the one or two or more transition metal elements together with lithium hydroxide from the positive electrode active material. (For example, in the case of a positive electrode active material substantially composed of a lithium-nickel-containing composite oxide, nickel hydroxide (Ni (OH) 2) is mainly produced. In general, transition metal hydroxides are substantially water-insoluble, and their solubility in water is significantly lower than that of at least lithium hydroxide. Therefore, when the lithium hydroxide is eluted with water from the insoluble matter of the treated material treated with H2O, the transition metal hydroxide remains as an insoluble matter. By utilizing such a difference in solubility in water, lithium hydroxide (lithium component) and transition metal hydroxide (transition metal component) can be easily separated.

[0019]

The reaction of the lithium-transition metal-containing composite oxide with H2O to form lithium hydroxide and transition metal hydroxide can be regarded as a hydrolysis reaction of the composite oxide. The lithium-nickel-containing composite oxide is more susceptible to the above hydrolysis (lower in hydrolysis resistance) than the composite oxide of lithium and another transition metal element (for example, lithium-cobalt-containing composite oxide). Therefore, a

Ascend Elements EX1002 Page 117

Duesenfeld Ex. 2014-117

positive electrode active material substantially composed of a lithium-nickel-containing composite oxide or a lithium battery using such a positive electrode active material is particularly suitable as an application target of the treatment method of the present invention. An example of such a lithium-nickel-containing composite oxide is one represented by the general formula LiNiO2. Further, as another example, a compound represented by the general formula LiNi1-xCoxO2 can be given. Here, 0 <x <0.5, preferably 0.1 <x <0.3. It may be a composite oxide containing Al, Mn, Cr, Fe or V instead of Co. Still another example is a compound represented by the general formula LiNi1-yzCoyAlzO2. Here, 0 ≦ y <0.5 (preferably 0 <y <0.2), 0 <z <0.5 (preferably 0 <z <0.1), and 0 <(y + z) <0.5 (preferably 0.1 <(y + z) <0.3). A type selected from the group consisting of Mn, Cr, Fe, V, Mg, Ti, Zr, Nb, Mo, W, Cu, Zn, Ga, In, Sn, La and Ce instead of Al or together with Al. Alternatively, it may be a composite oxide having two or more kinds of metal elements.

[0020]

In the step of producing lithium hydroxide or the like, H2O is preferably supplied under the condition that the positive electrode active material is brought into contact with a vapor phase containing water vapor (H2O) and oxygen (O2) and / or carbon dioxide (CO2). It is further preferable to supply H2O under the condition of being brought into contact with a gas phase containing H2O, O2 and CO2. Thereby, the reaction (typically the above hydrolysis reaction) in which lithium hydroxide and / or transition metal hydroxide is generated from the lithium / transition metal-containing composite oxide can be efficiently progressed. Further, in order to further promote such reaction, it is also effective to supply H2O under heating and / or pressurizing conditions. It is preferable to carry out the step of producing lithium hydroxide or the like under such conditions from the viewpoint of accelerating the reaction of producing aluminum hydroxide from the aluminum member.

[0021]

The aluminum member included in the member to be treated may be a battery constituent member such as a positive electrode and / or negative electrode current collector, an electrode lead plate, an electrode terminal, a container, a safety valve, or the like. In a preferred embodiment, the material to be treated includes a positive electrode current collector made of aluminum. In general, since the positive electrode current collector has a shape having a relatively large surface area, a reaction (corrosion of the positive electrode current collector) that produces aluminum hydroxide from the positive electrode current collector (aluminum member) can be efficiently progressed. The material to be treated preferably has a lithium / transition

Ascend Elements EX1002 Page 118

Duesenfeld Ex. 2014-118

metal-containing composite oxide (positive electrode active material) on an aluminum member (preferably a positive electrode current collector). According to this aspect, the surface (in the vicinity) of the aluminum member can be efficiently brought into a highly alkaline state (preferably pH 9 or higher, more preferably pH 11 or higher) by the lithium hydroxide generated from the composite oxide. This can further promote the reaction of producing aluminum hydroxide from the aluminum member.

[0022]

In a preferred aspect of the method disclosed herein, the material to be treated includes a sheet-shaped aluminum member (preferably a positive electrode current collector). An aluminum member having such a shape (for example, an aluminum foil) is preferable because it has a large surface area and can efficiently produce aluminum hydroxide (corrosion of the aluminum member). A sheet-shaped positive electrode (typically, a sheet-shaped positive electrode current collector having a positive electrode current collector attached to its surface) and a sheet-shaped negative electrode (typically, a sheet-shaped negative electrode current collector surface) A lithium battery including an electrode body having a structure in which a negative electrode current collector is attached to the electrode body) is an example of a lithium battery suitable as an application target of the method disclosed herein. Examples of the electrode body having such a structure include a so-called laminated electrode body and a wound electrode body.

[0023]

The method of the present invention is particularly preferably applied to a lithium battery including a wound electrode body having a sheet-shaped positive electrode and a sheet-shaped negative electrode. The material to be treated including the wound electrode body may be subjected to the step of producing lithium hydroxide or the like. It is preferable that at least one of the positive electrode and the negative electrode constituting the wound electrode body is provided with a current collector made of aluminum, and at least the positive electrode current collector is preferably made of aluminum. In this case, in the step of producing lithium hydroxide or the like, the positive electrode current collector (aluminum member) forms aluminum hydroxide and loses its regularity (corrodes and dissolves), so that it adheres to the positive electrode current collector. The positive electrode material that had been used comes off. Therefore, the operation of separating the positive electrode material from the positive electrode current collector can be simplified. For example, the operation of separating (peeling) the positive electrode material from the positive electrode current collector can be omitted. This is advantageous in recovering various valuable materials derived from the positive electrode

Ascend Elements EX1002 Page 119

Duesenfeld Ex. 2014-119

material, as described later.

[0024]

The above effect of facilitating the separation of the positive electrode current collector and the positive electrode material by generating aluminum hydroxide from the aluminum positive electrode current collector (aluminum member) is the same as the above-mentioned effect. The same effect can be exhibited in a mode not used for the treatment (immobilization) of the fluorine-based electrolyte. Therefore, the invention disclosed in this specification includes a positive electrode active material substantially composed of a composite oxide containing lithium and one or more transition metal elements and a positive electrode current collector made of aluminum. A method for treating a lithium battery, comprising: supplying H2O to a material to be treated including a positive electrode having a structure in which the positive electrode active material is attached to the positive electrode current collector to generate lithium hydroxide from the positive electrode active material. And a method for treating a lithium battery including a step of generating aluminum hydroxide from the positive electrode current collector. This treatment method further includes a step of eluting lithium hydroxide (preferably lithium hydroxide and aluminum hydroxide) generated in the above-mentioned lithium hydroxide production step into water to separate it from the insoluble matter of the member to be treated. be able to.

[0025]

The material to be treated containing the positive electrode active material and the aluminum member is preferably subjected to the step of producing lithium hydroxide etc. in a state where other battery constituent materials are removed (separated) as much as possible. This makes it possible to obtain a valuable material with better reusability. For example, it is possible to obtain lithium carbonate in which a transition metal element, another alkali metal element or the like (different metal element) is less mixed (high quality). Alternatively, a more useful transition metal compound (transition metal hydroxide) can be obtained. However, the separation of the material to be treated that is used in the production process such as lithium hydroxide, the cost and labor for performing such separation, and the magnitude of the effect produced by it (reusability of the valuable resources recovered (quality) And the like), so that they are properly balanced.

[0026]

Ascend Elements EX1002 Page 120

Duesenfeld Ex. 2014-120

For example, many common lithium batteries include a positive electrode having a structure in which a positive electrode material containing a positive electrode active material is attached to an aluminum foil (sheet-shaped aluminum member) as a positive electrode current collector. This positive electrode material typically contains a binder such as polyvinylidene fluoride and polytetrafluoroethylene in addition to the positive electrode active material. Further, it may contain a conductive material such as carbon black. The positive electrode in which the positive electrode material containing these is attached to the positive electrode current collector (aluminum member) can be used as it is as a material to be treated (provided in the step of producing lithium hydroxide or the like). Further, the positive electrode from which at least a part (preferably substantially all) of the binder contained in the positive electrode material has been removed by the heat treatment described below may be used as the material to be treated. An electrode body (rolled type electrode body, laminated type electrode body) having a configuration in which a negative electrode having a configuration in which a negative electrode material containing a negative electrode active material is attached to the surface of a sheet-shaped negative electrode current collector (for example, copper foil) and the positive electrode are laminated. In a lithium battery including a body, etc., an electrode body including both the positive electrode and the negative electrode can be used as a material to be treated. A carbon material (carbon black or the like) capable of inserting and releasing lithium ions is generally used as the negative electrode active material of a lithium battery. In the step of producing lithium hydroxide or the like, lithium hydroxide can be produced also from lithium inserted in the negative electrode active material. By eluting this lithium hydroxide with water and separating it from the insoluble matter, the lithium component in the negative electrode active material can be easily taken out. Therefore, when the material to be treated that is used in the production step such as lithium hydroxide contains a positive electrode material and a negative electrode material, lithium hydroxide is produced from the lithium components contained in both of these electrode materials, and this is treated with water. Can be eluted and collected.

[0027]

On the other hand, an electrolytic solution is an example of a battery constituent material that is preferably separated from the material to be treated in advance. Generally, an electrolytic solution has a structure in which an electrolyte (supporting salt) is dissolved in an organic solvent. The material to be treated is preferably separated from at least one of the organic solvent and the electrolyte that make up the electrolytic solution, and more preferably separated from both the organic solvent and the electrolyte. As a result, the constituent materials of the electrolyte (organic solvent, etc.) can be effectively reused, the electrolyte can be more efficiently converted into stable waste, and the handleability of the material to be treated is improved. At least one effect is obtained. The separation of the electrolytic solution and the material to be

Ascend Elements EX1002 Page 121

Duesenfeld Ex. 2014-121

treated can be suitably performed by heating the lithium battery to remove the electrolytic solution (organic solvent and electrolyte). A part or all of the battery constituent member remaining after the heating may be used as a material to be treated in a process for producing lithium hydroxide or the like. Further, as another method for separating the electrolytic solution and the material to be treated, a method of extracting the electrolytic solution by extracting the lithium battery with a non-aqueous organic solvent or the like can be mentioned.

[0028]

In another preferred embodiment, the lithium battery to be treated is heated to remove the volatile material. Here, the "volatile material" refers to a material that can be removed by being heated into a gas state (regardless of types such as evaporation, sublimation, and thermal decomposition) by heating. Examples of such a volatile material include one or more kinds of organic solvents, electrolytes (lithium salts, etc.), separators, binders, insulating materials, exterior coatings, etc. that make up the electrolytic solution. It is preferable to remove substantially all of the constituent materials, but the effect of the present invention can be exhibited by removing at least a part of each constituent material. It is preferable to remove most of the electrolyte and organic substances (organic solvent, separator, binder, etc.) contained in the lithium battery, and it is more preferable to remove substantially all of them. The heating of the lithium battery is preferably performed in the order in which the heating temperature gradually increases. A part or all of the battery constituent member remaining after the heating can be subjected to a process for producing lithium hydroxide or the like as a material to be treated. When the present invention is applied to a lithium battery including a wound type electrode body or a laminated type electrode body having a current collector (positive electrode current collector) made of aluminum, the electrode body is used as another battery constituent material (for example, The material separated from the battery container) can be preferably used as the material to be treated. This separation can be easily performed by, for example, cutting the battery container and taking out the electrode body.

[0029]

The step of heating the lithium battery to remove the volatile materials (electrolyte and organic matter) is preferably performed under reduced pressure. In this case, the volatile material (organic solvent or the like) can be volatilized at a lower temperature than that under normal pressure by utilizing the boiling point decrease due to the reduced pressure. As a result, for example, it is possible to suppress the alteration (eg, thermal decomposition) of the organic solvent contained in the electrolytic solution and obtain a recovered product with good

Ascend Elements EX1002 Page 122

Duesenfeld Ex. 2014-122

reusability. Further, since the volatilization rate can be increased by reducing the pressure, the time of heating can be shortened, and the effect of obtaining a recovered material with less deterioration (good reusability) can be obtained. By heating under reduced pressure in this way, the oxidation of the metal member that constitutes the lithium battery can be suppressed. This is advantageous in recovering and reusing the metal member.

[0030]

The invention disclosed herein includes a step of heating the lithium battery to volatilize and remove a volatile material, and recover the volatile matter, and a device for use in the recovery before the volatile matter recovery step. A method for treating a lithium battery, which includes a step of removing water in advance, is included. As a result, it is possible to prevent water from being mixed into the collected material. Therefore, a more useful (for example, highly pure) collected product can be obtained. The volatile matter recovery process may be divided into a plurality of processes at different heating temperatures of the lithium battery. It is preferable to carry out the plurality of steps in the order in which the heating temperature of the lithium battery increases stepwise. The step of removing the water may be performed before any of these sections, or may be performed before all the sections. It is preferable that the lithium battery to be treated is almost completely discharged in advance. As a result, at least one of the following effects can be obtained: improvement in handling and workability during processing, improvement in working environment, downsizing and simplification of the processing apparatus.

[0031]

The treatment method disclosed herein can further include a step of eluting an acid with at least one transition metal element of the one or more transition metal elements contained in the insoluble matter of the material to be treated. . Further, a step of adding a neutralizing agent to the solution to precipitate a hydroxide of a transition metal element contained in the solution can be included. It is preferable to separate (for example, filter) the solution obtained in the acid elution step from the insoluble matter of the material to be treated, and add the neutralizing agent to the separated solution. The positive electrode active material treated by this method is sometimes referred to as lithium, the first transition metal element, and at least one other metal element (hereinafter, referred to as "other metal element").) And can be included. Suitable examples of the first transition metal element include Ni, Co and Mn. The other metal element is a metal element different from the first transition metal element, and is Co, Al, Mn, Cr, Fe, V, Mg, Ti, Zr, Nb, Mo, W, Cu, Zn. , Ga, In, Sn, La and Ce may be one or more metal elements selected from

Ascend Elements EX1002 Page 123

Duesenfeld Ex. 2014-123

the group consisting of. In such a case, the step of eluting the first transition metal element and the at least one other metal element contained in the insoluble matter of the material to be treated with an acid, and the solution obtained in the acid elution step A neutralizing agent is added to (preferably separated from the insoluble matter of the material to be treated) to precipitate the hydroxide of the at least one other metal element together with the hydroxide of the first transition metal element from the solution. You should let me.

[0032]

At this time, with respect to the solution obtained in the acid elution step, the ratio (molar ratio, mass ratio, etc.) of the contents of the first transition metal element and the other metal element is grasped, and the ratio is out of the predetermined range. If so, the amount ratio is adjusted to fall within the predetermined range. For example, when the first transition metal element is nickel, the other element may be one kind or two or more kinds selected from Co, Al, Mg, Mn, Cr and the like. The ratio of the contents of these metal elements is grasped (for example, by composition analysis), and the amount ratio is adjusted to fall within a predetermined range by a method such as compensating for a component lacking the specified amount. Then, it is preferable to precipitate (preferably coprecipitate) the hydroxide of the other metal element together with the hydroxide of the first transition metal element from the solution. In particular, when the first transition metal element is nickel and the other metal element is cobalt, a state suitable for a raw material for a positive electrode active material is obtained by coprecipitating nickel hydroxide and cobalt hydroxide. In this way, a recovered product in which nickel and cobalt are fused is obtained. In general, cobalt hydroxide precipitated from a solution tends to be colloidal, but it can be easily separated from the liquid phase by coprecipitation with nickel hydroxide.

[0033]

Ammonia water is preferably used as the neutralizing agent. This ammonia water may be used alone or in combination with another neutralizing agent (alkali metal hydroxide, etc.). When aqueous ammonia is used as the neutralizing agent, the precipitation step is performed in the following manner. In the solution containing the transition metal ion and the first transition metal element and the other metal element, the ion of the first transition metal element (first (Transition metal ion) concentration), the pH of the solution, and the concentration of ammonium ion (NH4 <+>) are preferably controlled so that at least one item falls within a predetermined range. In the deposition step, it is preferable to control two or more of these items to fall within a predetermined range, and it is particularly preferable to control all three items to fall within a

Ascend Elements EX1002 Page 124

Duesenfeld Ex. 2014-124

predetermined range. As a result, a precipitate (mainly a transition metal hydroxide) having at least one of the average particle diameter, the specific surface area, and the half-value width of the X-ray diffraction peak in the desired range can be obtained. It is preferable that two or more of these characteristic values are in a desired range, and it is particularly preferable that all three characteristic values are in a desired range. Such a precipitate is suitable as a raw material for producing a lithium / transition metal-containing composite oxide used as a positive electrode active material of a lithium battery. A lithium battery including a lithium / transition metal-containing composite oxide produced using such a deposit as a positive electrode active material can have excellent charge characteristics, discharge characteristics, and the like. Further, the lithium / transition metal-containing composite oxide produced using the precipitate can have good storage stability. The average particle diameter can be measured by, for example, a particle diameter measuring device using a laser diffraction / scattering method. The specific surface area can be measured by, for example, a specific surface area measuring device using the BET method. The full width at half maximum can be obtained, for example, from an X-ray diffraction intensity curve obtained by using a general powder X-ray diffractometer.

[0034]

The present invention provides a positive electrode active material substantially composed of a composite oxide in which the main (first) transition metal element of the lithium / transition metal-containing composite oxide is nickel (that is, a lithium / nickel-containing composite oxide). It is particularly preferably applied to a lithium battery provided with. A preferred example of such a composite oxide is a lithium-nickel-containing composite oxide in which the first transition metal element is nickel and cobalt is included as the other metal element (subcomponent). In a lithium battery including such a positive electrode active material, the advantages of adopting the treatment method of the present invention can be exhibited particularly well.

[0035]

Another invention disclosed herein relates to a positive electrode active material for a lithium battery (typically, for a lithium ion secondary battery). The positive electrode active material is mainly composed of a lithium / transition metal-containing composite oxide produced by mixing lithium hydroxide and a transition metal hydroxide at a predetermined ratio and firing the mixture. Here, as the lithium hydroxide, lithium hydroxide obtained by firing lithium carbonate recovered from a lithium battery by any of the above-mentioned treatment methods and reacting the fired product with water (for example, lithium hydroxide). A monohydrate ($LiOH.H_2O$) can be used. The lithium carbonate obtained by the above treatment method can be highly pure. In

Ascend Elements EX1002 Page 125

Duesenfeld Ex. 2014-125

particular, the amount of an alkali metal element (sodium or the like) other than lithium mixed in can be small. Therefore, lithium hydroxide obtained (prepared) from such lithium carbonate can be suitably used as a raw material for producing a positive electrode active material of a lithium battery. Further, as the transition metal hydroxide, the transition metal hydroxide recovered from the lithium battery by any of the methods described above can be preferably used.

[0036]

Another invention disclosed herein is a method for producing a positive electrode active material for a lithium battery using a lithium battery using a positive electrode active material mainly composed of a lithium / transition metal-containing composite oxide (a positive electrode for a lithium battery). Active material manufacturing method). The method includes a step of producing a transition metal hydroxide raw material and a lithium hydroxide raw material for producing a positive electrode active material, and a step of mixing these raw materials at a predetermined ratio and firing. Here, as the lithium hydroxide raw material, the following steps: a step of hydrolyzing a positive electrode active material contained in a material to be treated derived from a lithium battery as a starting material to generate lithium hydroxide; Step of eluting in water to separate insoluble matter from the material to be treated; Step of supplying carbon dioxide (CO2) gas and / or carbonated water (H2CO3) to the obtained lithium solution to precipitate lithium carbonate from the solution; What was obtained through the step of separating lithium carbonate from the solution; the step of firing the lithium carbonate and further reacting with water can be used. In addition, as the transition metal hydroxide raw material, the following steps: a step of eluting the transition metal component in the insoluble matter with an acid; a step of adding a neutralizing agent to the acid solution to precipitate the transition metal; What was obtained after that can be used. According to such a manufacturing method, the lithium component and the transition metal component used in the lithium battery as a starting material (mainly contained in the positive electrode active material) are recovered, and they are mixed and fired. Positive electrode active material for lithium battery (The composition may be substantially the same as or different from that of the positive electrode active material of the lithium battery as a starting material.) Can be played. In the firing step, it is preferable to heat the mixture of the transition metal hydroxide raw material and the lithium hydroxide raw material stepwise. The obtained positive electrode active material for lithium batteries (lithium / transition metal-containing composite oxide) is suitable as a constituent material of a lithium battery after being subjected to a treatment for making the particle size uniform, a treatment for removing adsorbed moisture, etc., if necessary. Can be reused.

[0037]

Ascend Elements EX1002 Page 126

Duesenfeld Ex. 2014-126

The invention disclosed by this specification includes a lithium battery constructed using the positive electrode active material for a lithium battery described above. As a typical example of such a lithium battery, a positive electrode having a positive electrode material containing a positive electrode active material is provided on a positive electrode current collector, and a negative electrode material having a negative electrode active material is provided on a negative electrode current collector. A negative electrode and an electrolytic solution disposed between the positive electrode material and the negative electrode material, wherein the positive electrode active material is used (reused) as the positive electrode active material contained in the positive electrode material. The ones that are built by Such a lithium battery can be suitably manufactured by the following method, for example. That is, lithium hydroxide and transition metal hydroxide are mixed in a predetermined composition ratio. The mixture is fired to form a lithium / transition metal-containing composite oxide. A step of attaching the composite oxide to a current collector to produce a positive electrode can be provided. A step of accommodating the positive electrode together with an electrolytic solution and a negative electrode arranged via the electrolytic solution in a battery container to construct a lithium battery can be provided. As the lithium hydroxide, it is possible to use lithium hydroxide produced by firing lithium carbonate recovered from a lithium battery by any of the above-mentioned treatment methods and then reacting it with water. Moreover, as the transition metal hydroxide, the transition metal hydroxide recovered from the lithium battery by any of the above-mentioned treatment methods can be used.

[0038]

Hereinafter, specific examples of the present invention will be described, but the present invention is not intended to be limited to those shown in the examples. Further, technical matters other than the contents particularly referred to in the present specification and necessary for implementing the present invention can be grasped as design matters of a person skilled in the art based on the conventional technology. The present invention can be implemented based on the technical contents disclosed in this specification and the common general technical knowledge in the field.

[0039]

<Example: Treatment method of lithium-ion secondary battery> First, the configuration of a lithium battery (here, a used vehicle lithium-ion secondary battery) treated in this example will be described. FIG. 3 is a schematic cross-sectional view showing the lithium-ion secondary battery

Ascend Elements EX1002 Page 127

Duesenfeld Ex. 2014-127

according to the present embodiment, and FIG. 4 is an explanatory view in which a part thereof is enlarged. As shown in these figures, the secondary battery 1 has a wound electrode body in which a pair of electrode sheets (a positive electrode sheet 12 and a negative electrode sheet 14) are flatly wound via two separator sheets 16. 10 and a flat rectangular parallelepiped shape that houses the electrode body 10 (also referred to as a rectangular shape or a flat shape).), And a positive electrode terminal 30 and a negative electrode terminal 40 connected to both axial ends of the electrode body 10.

[0040]

As shown in FIG. 4, the positive electrode sheet 12 constituting the electrode body 10 includes a long positive electrode current collector 122, and a positive electrode material layer 124 provided by adhering positive electrode materials in layers on both surfaces thereof. Equipped with. In addition, the negative electrode sheet 14 includes a long negative electrode current collector 142 and a negative electrode material layer 144 provided by depositing a negative electrode material in layers on both surfaces thereof. The electrode body 10 has a configuration in which these sheets are laminated in this order on the positive electrode sheet 12, the separator sheet 16, the negative electrode sheet 14, and the separator sheet 16, and the laminated body is wound in the longitudinal direction. A separator sheet 16 insulates the stacked positive electrode sheet 12 and negative electrode sheet 14 from each other. As shown in FIG. 3, the positive electrode terminal 30 is connected to the positive electrode 12 at one axial end of the wound electrode body 10, and the negative electrode terminal 40 is connected to the negative electrode 14 at the other axial end.

[0041]

The positive electrode material layer 124 of the lithium ion secondary battery 1 is a positive electrode that is substantially composed of a lithium-nickel-containing composite oxide in which the first transition metal element is nickel and cobalt is included as another metal element. Active material is the main component. Such a positive electrode active material can be represented by the general formula LiNi1-xCoxO2 (0 <x <0.5, preferably 0.1 <x <0.3). In the secondary battery 1 according to the present embodiment, the lithium-nickel-containing composite oxide (that is, the composite oxide represented by LiNi0.8Co0.2O2) in which x in the above general formula is about 0.2 is used as the positive electrode active material. Used for. The positive electrode material layer 124 further contains carbon black (CB) and polytetrafluoroethylene (PTFE). The content ratio thereof is such that the mass ratio of positive electrode active material: CB: PTFE becomes about 85: 10: 5. On the other hand, the negative electrode material layer (graphite

Ascend Elements EX1002 Page 128

Duesenfeld Ex. 2014-128

layer) 144 contains carbon black (CB) as a negative electrode active material as a main component and polytetrafluoroethylene (PTFE) as a binder. The content ratio of them is, for example, a ratio at which the mass ratio of CB: PTFE becomes about 90:10.

[0042]

The positive electrode current collector 122 configuring the secondary battery 1 is a member made of aluminum (aluminum member). The positive electrode terminal 30 is also made of aluminum. The thickness of the aluminum member (aluminum foil) forming the positive electrode current collector 122 is preferably about 50 μm or less (for example, 2 to 50 μm), and more preferably about 20 μm or less (for example, 5 to 20 μm). .. According to the positive electrode current collector 122 having such a shape, the reaction of producing aluminum hydroxide from the positive electrode current collector 122 can be efficiently progressed in the step of producing lithium hydroxide or the like described later. In the secondary battery 1 according to this example, an aluminum foil having a thickness of about 15 μm is used as the positive electrode current collector 122. On the other hand, the negative electrode current collector 142 and the negative electrode terminal 40 are made of copper. Although not particularly limited, the thickness of the copper member (copper foil) forming the negative electrode current collector 142 is, for example, preferably about 2 to 50 μm, and more preferably about 5 to 20 μm. The separator sheet 16 is a porous sheet made of polyolefin (here, polypropylene).

[0043]

The battery container 20 is made of aluminum, and includes a bottomed cylindrical main body (battery case) 22 and a lid body (battery lid) 24 that seals the upper end opening of the main body 22. The wound electrode body 10 is housed in the container 20. The positive electrode terminal 30 and the negative electrode terminal 40 penetrate the lid body 24 and extend to the outside of the container 20. These terminals 30 and 40 are fixed to the lid by nuts 32 and 42. The negative electrode terminal 40 and the lid 24 are separated by an insulator 26. The lid 24 has a liquid injection port 27 used for injecting an electrolytic solution and the like. The liquid injection port 27 is in a sealed state during normal use of the secondary battery 1. Further, the lid 24 is provided with a safety valve 28. The safety valve 28 is configured to automatically connect the inside and the outside of the container 20 to release the pressure when the internal pressure of the container 20 exceeds a predetermined set value and becomes high.

[0044]

Ascend Elements EX1002 Page 129

Duesenfeld Ex. 2014-129

The electrode body 10 is impregnated with an electrolytic solution (not shown). As the organic solvent constituting this electrolytic solution, γ -butyrolactone, ethylene carbonate, propylene carbonate, dimethyl carbonate, diethyl carbonate, ethylmethyl carbonate, 1,2-dimethoxyethane, 1,2-diethoxyethane, tetrahydrofuran, 1, One or more selected from the group consisting of 3-dioxolane and the like can be used. The secondary battery 1 according to this example uses a mixed solvent of dimethyl carbonate (DMC) and ethylmethyl carbonate (EMC) in a ratio of 7: 3 (mass ratio). Further, as the electrolyte (supporting salt) that constitutes this electrolytic solution, one or more selected from various lithium salts having fluorine as a constituent element can be used. For example, one or more selected from the group consisting of LiPF6, LiBF4, LiAsF6, LiCF3SO3, LiC4F9SO3, LiN (CF3SO2) 2, LiC (CF3SO2) 3 and the like can be used. In the secondary battery 1 according to this example, lithium hexafluorophosphate (LiPF6) is used as the electrolyte. Its concentration is about 1 mol / liter.

[0045]

Next, a procedure for treating the lithium-ion secondary battery 1 having the above structure will be described. FIG. 1 is a flowchart showing the outline of the procedure.

[0046]

First, a used lithium-ion secondary battery 1 is prepared (step 210), and a process of heating the lithium-ion secondary battery 1 to recover volatile components such as an electrolyte and an organic substance is performed (step 220). As shown in FIGS. 1 and 2, this step 220 includes a step 222 of removing (recovering) an organic solvent, a step 224 of removing (recovering) an electrolyte, and a step 226 of removing (recovering) a separator. And step 228 of removing (recovering) the adhesive. In FIG. 1, some of the steps included in step 220 are omitted.

[0047]

FIG. 5 schematically shows an example of a schematic configuration of an apparatus used for collecting such volatile components. In FIG. 5, the reduced pressure heating furnace 60 includes a main body 61 that divides a processing chamber 64. An induction heating coil 62 is provided on the wall surface of the main body 61. The temperature inside the processing chamber 64 can

Ascend Elements EX1002 Page 130

Duesenfeld Ex. 2014-130

be controlled in an arbitrary pattern by the temperature controller 63 connected to the coil 62. A vacuum pump 65 is connected to the main body 61 so that the internal pressure of the processing chamber 64 can be arbitrarily controlled. A primary cooling device 67 is provided below the processing chamber 64. The gas in the processing chamber 64 (for example, the organic solvent volatilized from the lithium ion secondary battery) is introduced into the primary cooling device 67 via the in-furnace gas conduit 66. An induction heating coil 68 is provided on the wall surface of the primary cooling device 67, and the degree of cooling in the primary cooling device 67 can be adjusted. A secondary cooling device 71 is connected downstream of the primary cooling device 67 via an out-of-furnace gas conduit 70. The collected matter that is cooled by the secondary cooling device 71 and condensed (liquefied or solidified) is collected in the collector 74. The remaining gas is discharged to the outside via the exhaust gas purifier 72.

[0048]

1 hPa

Using the apparatus having such a configuration, for example, the organic solvent is volatilized and removed from the secondary battery 1 and the volatile matter is recovered (step 222) as follows. That is, the used lithium-ion secondary battery 1 opened in advance is housed in the processing chamber 64. The method of opening the secondary battery 1 may be appropriately selected from a method of forming a through hole in the container 20, a method of unsealing the liquid injection port 27, a method of operating the safety valve 28, and the like. The vacuum pump 65 is operated to reduce the pressure inside the processing chamber 64. The degree of this reduced pressure is not particularly limited. Usually, it is suitable to reduce the pressure to at least 80 kPa or less (typically 0.1 to 80 kPa). It is more preferable to reduce the pressure to 50 kPa or less (typically 0.1 to 50 kPa). In this embodiment, the pressure inside the processing chamber 64 is reduced to 1 to 10 kPa. While maintaining such a reduced pressure state, the induction heating coil 62 is energized to raise the temperature in the processing chamber 64 (temperature in the furnace). Thereby, the secondary battery 1 is heated. The heating temperature at this time is the boiling point of the organic solvent of the electrolytic solution used in the secondary battery 1 (or the boiling point of each component when two or more kinds of organic solvents are included, depending on the pressure in the processing chamber 64). It can be around their azeotropic point). Alternatively, it is also preferable to heat to a temperature higher than the boiling point of the organic solvent within a range in which the battery constituent materials other than the organic solvent do not substantially volatilize. Thereby, the organic solvent can be efficiently volatilized from the secondary battery 1. The boiling point of DMC at atmospheric pressure is about 90 °C, and the boiling point of EMC is about 107 °C.

[0049]

Ascend Elements EX1002 Page 131

Duesenfeld Ex. 2014-131

In this embodiment, as shown in the temperature chart shown in FIG. 6, the temperature inside the furnace is gradually raised from room temperature and maintained at about 85 ° C. for 30 minutes (at the stage indicated by arrow a in the figure). As a result, DMC of the organic solvent volatilizes (gasifies) preferentially. The generated gas is introduced into the secondary cooling device 71 through the in-furnace gas conduit 66, the primary cooling device 67, and the out-of-furnace gas conduit 70. The gas is cooled by this secondary cooling device 71 to condense (liquefy) the organic solvent and collect it in the collector 74. Next, the temperature in the furnace is further raised and maintained at 102 ° C. for 30 minutes (step shown by arrow b in the figure). As a result, the remaining organic solvent (mainly EMC) is volatilized (gasified) and liquefied and recovered by the secondary cooling device 71.

[0050]

As described above, in this embodiment, the organic solvent (mixed solvent) containing two components having different boiling points is volatilized by heating in two steps. That is, after the majority of DMC having a relatively low boiling point is volatilized in the above step a, the heating temperature is further raised to volatilize the remaining organic solvent (mainly EMC). As described above, the heating temperature is changed stepwise and the low-boiling solvent is sequentially extracted from the high-boiling solvent, whereby the organic solvent can be efficiently volatilized and recovered while suppressing alteration (decomposition, etc.) due to heating. The vapor pressure of DMC at 24 ° C is about 2.4 kPa, and the vapor pressure of EMC at 25 ° C is about 3.3 kPa. Therefore, the DMC and EMC volatilized in the above steps a to b are typically recovered as a mixed solvent. As will be described later, the recovered mixed solvent has a small content ratio of impurities (such as a thermal decomposition product of DMC and / or EMC) and has good reusability. For example, it can be used (reused) as an electrolytic solution for a lithium ion secondary battery.

[0051]

Next, the battery constituent material remaining after the above heating is further heated to thermally decompose the electrolyte (LiPF6 in this case), and the gas containing the thermally decomposed material (decomposed gas) is removed (step 224). That is, for example, as in the temperature chart shown in FIG. 6, the furnace temperature is maintained in a temperature range equal to or higher than the thermal decomposition temperature of LiPF6 (about 160 ° C.) (step indicated by arrow c in the figure). The heating temperature at this time can be in the range of, for example, about 160 to 200 ° C, and is preferably in the range of about 180 to 200 ° C

Ascend Elements EX1002 Page 132

Duesenfeld Ex. 2014-132

(about 200 ° C in this embodiment). As a result, LiPF6 is thermally decomposed, and mainly lithium fluoride (LiF) and phosphorus fluoride (PF5) are generated. The decomposed gas (exhaust gas) containing phosphorus fluoride is further processed by the steps described below. The heating temperature for performing the step c is not limited to the above temperature, and may be any temperature range in which the electrolyte can be volatilized at a practical rate to be removed from the secondary battery 1. For example, it can be carried out in a temperature range equal to or higher than the volatilization (pyrolysis) temperature of the electrolyte. It is preferable to set the temperature in the range of volatilization (pyrolysis) to the temperature + 50 ° C. If the degree of pressure reduction is about the same, it is preferable that the heating temperature in the step c is set to 80 ° C. or more higher than the temperatures in the steps a and b.

[0052]

The separator is volatilized (mainly pyrolyzed) from the battery constituent material remaining after step 224 shown in FIG. 2 to be removed, and the volatile matter is recovered (step 226). That is, as in the temperature chart shown in FIG. 6, the temperature inside the furnace is maintained at about 300 ° C. (step shown by arrow d in the figure). As a result, the separator 16 (porous polypropylene sheet) forming the electrode body 10 shown in FIG. 3 is thermally decomposed to generate a gas such as a lower hydrocarbon. The gas can be recovered in the primary cooling device 67 and / or the secondary cooling device 71 as a liquid (for example, a viscous liquid mainly composed of a mixture of plural kinds of hydrocarbons) or a solid. The recovered material can be effectively reused as fuel or the like. Further, the binder constituting the electrode material is volatilized (mainly thermally decomposed) to be removed from the battery constituent material remaining after the heating, and the volatile matter is recovered (step 228). That is, as in the temperature chart shown in FIG. 6, the temperature inside the furnace is maintained at about 400 ° C. (step indicated by arrow e in the figure). As a result, the binder (polytetrafluoroethylene) forming the electrode material is thermally decomposed to generate a gas such as a lower hydrocarbon or a fluoride. The generated gas can be collected (collected) as a liquid or a solid in one or more of the primary cooling device 67, the secondary cooling device 71, and the exhaust gas purifier 72.

[0053]

The heating temperature for carrying out the steps d and e is not limited to the above temperature, and the separator and the binder are volatilized (pyrolysis etc.) at a practical rate to remove the secondary battery 1 from the secondary battery 1. Any temperature range that can

Ascend Elements EX1002 Page 133

Duesenfeld Ex. 2014-133

be removed may be used. From the viewpoint of shortening the time required to remove the separator and the binder (organic matter) and / or improving the removal rate of the binder, the heating temperature for performing the d step and / or the e step is set to be high. Preferably. However, the temperature is preferably such that the metal member constituting this battery is not melted (for example, in the case of aluminum, it is about 570 ° C or lower, more preferably about 550 ° C or lower).

[0054]

In the above process (step 220), the heating temperature is raised stepwise to recover the volatile matter at each stage. In this way, by sequentially extracting the constituent materials of the secondary battery from the material having a low volatilization temperature to the material having a high volatilization temperature, a recovered material (valued material) having good reusability can be obtained. In addition, it is preferable to first remove (remove) the organic solvent from the battery in this manner, because the internal resistance of the battery is increased and the handleability of the battery in the subsequent steps is improved. Further, by extracting the organic solvent (step 222) under reduced pressure, (1). Prevents alteration of organic solvent due to excessive heating (2). Shorten the time required to remove the organic solvent, (3). At least one of the effects of saving energy for heating can be obtained.

[0055]

The electrolyte (decomposition gas thereof) removed in step 224 described above can be recovered as a stable compound by the following process (phosphorus fluoride stabilization process). That is, the decomposition gas (exhaust gas) containing phosphorus fluoride generated by the decomposition of the electrolyte passes through the primary cooling device 67 and the secondary cooling device 71 shown in FIG. 5, and is connected to the exhaust gas purifier 72 downstream of the vacuum pump 65. (Step 282). A treatment liquid 722 containing aluminum hydroxide is stored in the exhaust gas purifier 72. As described later, the aluminum hydroxide contained (dissolved and / or dispersed) in the treatment liquid 722 is derived from the positive electrode current collector 122 as the aluminum member in the lithium battery treatment process of the present embodiment. The Al (OH) 3 produced and recovered can be used.

[0056]

Ascend Elements EX1002 Page 134

Duesenfeld Ex. 2014-134

The exhaust gas is bubbled into the treatment liquid 722 from an exhaust gas supply port 724 provided near the bottom of the exhaust gas purifier 72. As a result, aluminum hydroxide (Al (OH) 3) in the treatment liquid 722 reacts with phosphorus fluoride in the exhaust gas (step 284) to produce aluminum fluoride (AlF3) and aluminum phosphate (AlPO4). Specifically, phosphorus fluoride reacts with water to be separated into hydrogen fluoride and phosphoric acid to form a solution, which rapidly reacts with aluminum hydroxide dispersed and / or dissolved in the solution to cause AlF3 and AlPO4. Changes to. Here, AlF3 is sparingly soluble, and its solubility is 0.5 g / 100 g. AlPO4 is insoluble, and its solubility product (Ksp) is 1.3×10^{-20}. Thus, since AlF3 and AlPO4 are sparingly soluble or insoluble, these products can be easily recovered as a solid by filtering if necessary (step 286). In this way, by using aluminum hydroxide as a purifying agent for phosphorus fluoride gas, it is possible to collect (collect) the fluorine component and phosphorus component contained in LiPF6 as the electrolyte in a form with good handleability. For example, since AlF3 and AlPO4 are not harmful designated substances, they can be appropriately disposed of as stable waste (step 288). Although FIG. 5 shows a configuration in which two exhaust gas purifiers 72 are provided in series for the purpose of increasing the processing efficiency of the electrolyte (the decomposition gas thereof), the number of the exhaust gas purifiers 72 may be one. .. Alternatively, two or more exhaust gas purifiers 72 may be provided in series or in parallel. Further, when introducing the exhaust gas into the treatment liquid 722, it is preferable to bubble the exhaust gas in the form of fine bubbles to increase the contact area between the exhaust gas and the treatment liquid to enhance the reaction efficiency.

[0057]

Through the above process (step 220), an organic matter-removed battery from which the electrolyte and organic matter are removed is obtained (step 230). This organic substance-removed battery (heating residue of battery constituent material) is a container 20 including a container body (battery case) and a lid, a positive electrode sheet 12 including a positive electrode current collector and a positive electrode active material, and a negative electrode current collector. And a negative electrode sheet 14 containing a negative electrode active material. The positive electrode sheet 12 and the negative electrode sheet 14 form a wound electrode body 10. In step 232 shown in FIG. 1, the battery 230 from which the organic solvent has been removed is divided by, for example, cutting off (cutting) the lid portion 24 of the container 20 (step 232) and the wound electrode body 10 is taken out (Step 236). The container 20 (battery case 22 and lid 24) separated from the electrode body 10 can be recovered (step 233) and reused for various purposes as an aluminum material.

[0058]

Ascend Elements EX1002 Page 135

Duesenfeld Ex. 2014-135

In the wound electrode body 10 separated from the other battery constituent materials in this manner, the separator sheet 16 is already lost (volatilized), so that the separator sheet 16 is interposed between the positive electrode sheet 12 and the negative electrode sheet 14. There are many voids. Step 240 shown in FIG. 1 is a step in which the electrode body 10 is used as a material to be treated and is subjected to a hydrolysis treatment (supplying H 2 O or the like). A schematic configuration example of the apparatus used in this step is schematically shown in FIG.

[0059]

The hydrolysis treatment device 80 includes a sealed pressure vessel 81 that divides the treatment chamber 90. The pressure vessel 81 includes a reaction tank 812 and a lid 814 and is made of stainless steel. The pressure vessel 81 includes a steam supply means 88 and an oxygen gas supply means 89. The steam supply unit 88 includes a steam generator 882 and a steam supply pipe 884 extending from the generator 882 and connected to the container 81. Further, the oxygen gas supply unit 89 includes an oxygen gas supply source 892 and an oxygen gas supply pipe 894 extending from the generation source 892 and connected to the container 81. The steam supply pipe 884 and the oxygen gas supply pipe 894 are arranged so as to penetrate the lid 814 and supply steam and oxygen gas to the processing chamber 90. A heater 92 is provided below the reaction tank 812 of the pressure vessel 81. Water (here, pure water) 92 is stored in the reaction tank 812 to a position higher than the heater 92. Further, the pressure vessel 81 is provided with pressure control means 91 for adjusting the pressure in the processing chamber 90. The pressure control means 91 includes a pressure control valve 912 and an exhaust port 914. Further, the pressure vessel 81 is provided with a temperature sensor (thermometer) 84 for detecting the internal temperature of the processing chamber 90 and a temperature adjusting mechanism (not shown).

[0060]

Inside the reaction tank 812, a breathable partition plate 83 is provided at a position slightly higher than the liquid surface of the water 92. The electrode body 10 (material to be processed) to be processed is arranged on the partition plate 83. The water 92 is heated to a predetermined temperature (here, about 80 ° C.) by the heater 82. This promotes the generation of water vapor. The inside of the processing chamber 90 is filled with steam and oxygen at high temperature (about 80 in this case) by steam generated from the water 92 and steam and oxygen

Ascend Elements EX1002 Page 136

Duesenfeld Ex. 2014-136

gas introduced from the steam supply means 88 and the oxygen gas supply means 89, respectively, if necessary. ° C) The atmosphere is maintained. Further, the inside of the processing chamber 90 is maintained at a predetermined tank internal pressure by the pressure control means 91. The hydrolysis treatment device 80 is configured to include a carbon dioxide gas supply device and a carbon dioxide gas supply pipe in place of the oxygen gas supply device and the oxygen supply pipe, or in addition to the oxygen gas supply device and the oxygen supply pipe. be able to.

[0061]

By exposing the electrode body 10 to the atmosphere in the processing chamber 90 (supplying H2O to the electrode body 10), the positive electrode active material contained in the electrode body 10 is hydrolyzed, and lithium hydroxide and transition metal hydroxide are added. This produces substances (mainly nickel hydroxide). In addition, lithium hydroxide may be generated from lithium inserted in the negative electrode active material included in the electrode body 10. At this time, it is considered that, for example, a phenomenon represented by the following reaction formula occurs in the pressure vessel.

[0062]

$$3LiNiO2+2H2O\rightarrow 3NiOOH+Li2O+LiOH \cdots (1)$$

$$2NiOOH+H2O\rightarrow 2Ni(OH)2+1/2O2 \cdots (2)$$

$$Li2O+H2O\rightarrow 2LiOH \cdots (3)\ C/Li+H2O\rightarrow C+LiOH+1/2H2 \cdots (4)$$

[0063]

The above reaction formulas (1) to (3) are reactions relating to the positive electrode active material.

Reaction formula (1) produces nickel oxyhydroxide (NiOOH). This nickel oxyhydroxide is decomposed into nickel hydroxide (Ni (OH) 2) according to the reaction formula (2). On the other hand, the lithium oxide (Li2O) generated by the reaction formula (1) becomes lithium hydroxide (LiOH) according to the reaction formula (3). This reaction similarly occurs during the reaction in the presence of carbon dioxide gas and water vapor (production of Li2O and LiOH), but finally the produced lithium hydroxide (LiOH) reacts with carbon dioxide gas. To produce lithium

Ascend Elements EX1002 Page 137

Duesenfeld Ex. 2014-137

carbonate (Li2CO3). The oxygen gas generated by the reaction formula (2) escapes into the gas phase, and eventually lithium hydroxide and nickel hydroxide are produced from lithium nickelate by these reaction formulas. The cobalt component contained in the positive electrode active material can similarly produce cobalt hydroxide.

[0064]

The above reaction formula (4) is a reaction relating to lithium inserted in the negative electrode active material. Further, "C / Li" in the formula represents that this lithium is in a state of being inserted into the carbon material (here, carbon black). In the reaction formula (4), lithium in the negative electrode active material generates lithium hydroxide (LiOH) and hydrogen gas is generated. This hydrogen gas is stabilized by generating water by the oxygen gas with which the processing chamber 90 is filled.

[0065]

The surface of the positive electrode current collector that constitutes the electrode body 10 is in a highly alkaline state (typically pH 11 or higher) due to lithium hydroxide or the like generated from the positive electrode active material by such hydrolysis. Thus, the positive electrode current collector exposed to the highly alkaline wet condition is gradually corroded from its surface. That is, aluminum hydroxide such as Al (OH) 3, Li [Al (OH) 4 (H2O) 2] is generated from aluminum constituting the positive electrode current collector. This corrosion (reaction that produces hydroxide) starts from the outside (outer surface, end face, etc.) of the wound electrode body 10 and gradually progresses to the central portion of the electrode body 10. Here, as described above, the electrode body 10 having a large amount of voids is advantageous from the viewpoint of promoting the progress of corrosion to the central portion of the electrode body 10. In order to promote the progress of such corrosion, it is also effective to divide the electrode body 10 into several parts (for example, divide it into two or more parts on a plane orthogonal to the winding axis) in advance. In the step of cutting the battery container 20 (step 232), the electrode body 10 may be cut together with the container 20.

[0066]

Conditions such as treatment temperature, treatment pressure, treatment time, water vapor concentration and oxygen concentration in the atmosphere can be appropriately adjusted

Ascend Elements EX1002 Page 138

Duesenfeld Ex. 2014-138

depending on the progress of the hydrolysis reaction and / or the corrosion reaction of the positive electrode current collector. By treating under heating, the hydrolysis reaction and / or the corrosion reaction can be promoted. For example, it can be treated at 40 to 120 ° C, preferably 50 to 100 ° C, more preferably 60 to 90 ° C. In addition, the treatment under pressure can accelerate the hydrolysis reaction and / or the corrosion reaction. However, excessively increasing the processing pressure tends to increase the size of the processing apparatus 80 (processing container 81 and the like). In consideration of these factors, the preferable processing pressure is about 0.1 to 0.5 MPa (about 1 to 5 atm), and the more preferable pressure is about 0.1 to 0.2 MPa (about 1 to 2 atm).

[0067]

The inside of the processing chamber 90 is preferably an atmosphere having a high water vapor concentration, and it is more preferable to maintain the inside of the processing chamber 90 at a substantially saturated water vapor pressure. The processing conditions may be adjusted so that water circulates in the processing chamber 90. For example, the water 92 stored at the bottom of the reaction tank 812 evaporates, the water vapor contacts the electrode body (material to be treated) 10 and the like to be condensed, and further propagates through the electrode body 10 and passes through the partition plate 83 to pass the water 92. To return to. Thereby, the lithium hydroxide and / or the aluminum hydroxide generated from the electrode body 10 can be dissolved (extracted) in the water 92. The oxygen partial pressure in the atmospheric pressure in the processing chamber 90 is preferably 50% or more (typically 50 to 90%, more preferably 50 to 75%). Further, the atmosphere in the processing chamber 90 may be an atmospheric atmosphere containing water vapor (preferably saturated with water vapor). Oxygen and / or carbon dioxide gas may be supplied to the air atmosphere to increase the oxygen concentration and / or carbon dioxide concentration. Although not particularly limited, the supply rate of oxygen gas may be, for example, 10 to 1000 ml / min (preferably 100 to 500 ml / min). Further, the supply amount of water vapor can be set to, for example, 1 to 20 g / min (preferably 5 to 10 g / min).

[0068]

The timing of ending the hydrolysis step (step 240) can be determined by looking at the degree of progress of the hydrolysis reaction and / or the corrosion reaction. Usually, it is preferable to continue the hydrolysis step until the corrosion of the positive electrode current collector (production of aluminum hydroxide) is almost completed. As a result, aluminum constituting the positive electrode current collector becomes an aluminum hydroxide such as Al (OH) 3, Li [Al

Ascend Elements EX1002 Page 139

Duesenfeld Ex. 2014-139

(OH) 4 (H2O) 2] (preferably dissolved and / or dispersed in water). The positive electrode collector has substantially disappeared. As a result, the positive electrode material attached to the positive electrode current collector 122 loses the support and falls off, so that the operation of separating (eg, peeling) the positive electrode material from the positive electrode current collector 122 becomes unnecessary. Under preferable processing conditions, corrosion of the positive electrode current collector can be completed (substantially disappearing of the positive electrode current collector) within a period of about one month. Both the oxidizing atmosphere and the high temperature (here, about 80 ° C.) high humidity atmosphere in the processing chamber 90 accelerate the corrosion of the positive electrode current collector 122 and shorten the time required for the hydrolysis step. Is advantageous for.

[0069]

The material to be treated that has been subjected to the hydrolysis step 240 in this manner is in a state containing a negative electrode current collector (copper foil), lithium hydroxide, aluminum hydroxide, transition metal hydroxide, carbon black (graphite), and the like. Has become. Of these, the negative electrode current collector (copper foil) remains (mainly in a metallic state) while maintaining its original shape. This negative electrode current collector can be directly recovered (step 243) and reused as a copper material. Alternatively, the negative electrode current collector may be collected during the hydrolysis step 240.

[0070]

In a preferred embodiment, during the hydrolysis step, lithium hydroxide and aluminum hydroxide produced in the step are eluted with water. Further, after the completion of the hydrolysis step 240, the residue may be washed with water and the hydroxides of lithium hydroxide and aluminum may be eluted from the residue with water. On the other hand, the solubility of nickel hydroxide (Ni (OH) 2) and cobalt hydroxide (Co (OH) 2) is about 7.5 × 10 <-4> g / 100g (H2O) and about 8-22 × 10 <-4> g / 100g (H2O). These transition metal components (nickel hydroxide and cobalt hydroxide) do not substantially elute in water. Further, carbon black is substantially insoluble in water. Therefore, the material to be hydrolyzed is eluted (extracted, washed with water, etc.) with water, and the liquid phase and insoluble matter are separated by filtration or the like (step 244) to obtain lithium hydroxide and aluminum hydroxide. A filtrate (solution) containing is obtained (step 245). In addition, an insoluble matter (residue) containing a transition metal hydroxide such as nickel hydroxide and carbon black is obtained by this separation (step 260).

Ascend Elements EX1002 Page 140

Duesenfeld Ex. 2014-140

[0071]

The solution obtained by the separation in step 244 contains lithium hydroxide and aluminum hydroxide (Al (OH) 3, Li [Al (OH) 4 (H2O) 2] etc.). Aluminum hydroxide is recovered from this solution (steps 246-248). That is, by adding aqueous ammonia (NH4OH) to the solution (step 246), gelatinous Al (OH) 3 is produced from the aluminum hydroxide in the solution. When this is centrifuged (step 247), a supernatant liquid (lithium solution) containing lithium hydroxide and gelatinous Al (OH) 3 are separated into layers. Thereby, aluminum hydroxide (Al (OH) 3) can be easily separated (collected) from the solution (step 258). The recovered Al (OH) 3 can be circulated and utilized in step 282 shown in FIG. 1 as an effective component (fixing agent for the decomposition gas of the electrolyte) of the treatment liquid 722 provided in the exhaust gas purifier 72 shown in FIG. .. According to this embodiment, such a recycling system can be constructed.

[0072]

Further, the supernatant in step 247 is separated from aluminum hydroxide to obtain a lithium solution (step 250). The process of recovering the lithium component as a solid from this lithium solution will be described. That is, this solution is once heated to release ammonia. Then, when carbon dioxide gas (CO2) is supplied to the solution, lithium ions in the liquid react with CO2 to produce lithium carbonate (Li2CO3) (step 252). At this time, the lithium solution obtained in Step 247 may be concentrated and used within a range in which lithium hydroxide does not precipitate. Since lithium carbonate has a lower solubility in water than lithium hydroxide, it precipitates out of solution and precipitates. The solid lithium carbonate can be recovered by filtering the precipitate (precipitate) from the liquid phase and drying.

[0073]

Here, as a method of supplying carbon dioxide gas to the lithium solution, a method of bubbling CO 2 gas into the solution or the like can be adopted. In order to react the lithium solution (lithium ion) and CO2 more efficiently, the lithium solution is sprayed in an atmosphere containing CO2, which is reacted under a pressure atmosphere containing CO2 (for example, from atmospheric pressure to about 1 MPa). , Etc. can be appropriately adopted. It is preferable that the carbon dioxide concentration in the atmosphere is high. For example, the reaction is preferably performed in an atmosphere having a CO2 concentration of about 50 to 100 vol%

Ascend Elements EX1002 Page 141

Duesenfeld Ex. 2014-141

(more preferably about 80 to 100 vol%). It is particularly preferable to react under an atmosphere consisting essentially of CO2 gas. The solubility of lithium carbonate tends to increase as the concentration of CO2 dissolved in the reaction solution increases. Therefore, for example, after reacting lithium ions and CO2 under atmospheric pressure or pressurized atmosphere (for example, atmospheric pressure to approximately 1 MPa or less) with a CO2 concentration of approximately 100%, the amount of CO2 dissolved in the reaction solution is reduced. It is preferable to carry out a treatment for removing CO2 from the liquid. When the CO2 concentration of the solution decreases due to this decarboxylation treatment, the solubility of lithium carbonate decreases. As a result, a part of the lithium carbonate dissolved in the reaction solution before the decarboxylation treatment is precipitated (recrystallized), which can be easily separated and recovered from the liquid phase. The decarboxylation treatment is carried out, for example, by stirring the reaction solution in an atmospheric atmosphere of normal pressure or reduced pressure, bubbling air or an inert gas (nitrogen gas, etc.) into the reaction solution in the atmosphere. be able to. Also, the solubility of lithium carbonate tends to decrease slightly as the temperature rises (about 0.72 g / 100 g (H2O) at 100 ° C). Therefore, by increasing the temperature of the reaction liquid (for example, heating to about 40 to 80 ° C.), the solubility of lithium carbonate can be decreased and the amount of precipitation can be increased. By such a method, the recovery rate of the lithium component (lithium carbonate) from the lithium solution can be further improved.

[0074]

By heating the lithium carbonate (LiCO3) thus recovered, the lithium carbonate can be decomposed into Li2O and CO2. Usually, it is appropriate that the heating temperature at this time is approximately 1500 ° C or higher. By supplying water vapor (H2O) to this Li2O, LiOH.H2O can be generated (step 254). This lithium hydroxide monohydrate (LiOH.H2O) can be effectively used, for example, as a raw material (lithium hydroxide material) for producing a lithium / transition metal-containing composite oxide used as a positive electrode active material of a lithium battery. it can.

[0075]

The treatment of the insoluble matter filtered out by the filtration in step 244 will be described. First, an acid (sulfuric acid (H2SO4) in this embodiment) was used for this insoluble matter.) Is added to elute the metal component (step 262). This is filtered (step 264) and separated into an acid solution containing ions of the metal component (here, mainly nickel and cobalt) and an insoluble component (steps 265 and 266). The insoluble matter separated here is mainly a

Ascend Elements EX1002 Page 142

Duesenfeld Ex. 2014-142

carbon material (carbon black) used as a conductive material of a positive electrode material and a negative electrode active material. The recovered carbon material can be effectively used as, for example, a snow melting material, a soil improving material, or the like.

[0076]

On the other hand, the filtrate obtained in step 264 is an acid solution containing nickel ions and cobalt ions (here, a sulfuric acid solution). In the following steps, using this acid solution, a positive electrode active material in which cobalt was added to a lithium-nickel composite oxide (typically, LiNi1-xCoxO2 (0 <x <0.5, preferably 0.1 A transition metal hydroxide material useful as a raw material for producing a positive electrode active material represented by <x <0.3) is synthesized. Here, a case of synthesizing (preparing) a raw material for producing a positive electrode active material represented by LiNi0.8Co0.2O2 will be described. The concentrations of nickel ions and cobalt ions contained in the sulfuric acid solution are measured by a conventional method. When the ratio of the concentrations (contents) of these ions deviates from the preset target value (here, the molar ratio of Ni: Co in terms of metal is 0.8: 0.2) by a predetermined amount or more. Externally supplements (adds to the solution) the missing ionic species. Thereby, the amount ratio of both ions contained in the sulfuric acid solution is adjusted to be within a predetermined range. Since only the shortage is compensated in this way, the amount of transition metal component (e.g. cobalt) newly used can be saved. In addition, if it is possible to predict (understand) the amount ratio of nickel ions and cobalt ions contained in the sulfuric acid solution obtained in step 264 from previous processing experience and preliminary experiments, the operation of measuring the concentration of these ions should be performed. It can be omitted.

[0077]

A neutralizer is added to a sulfuric acid solution containing nickel ions and cobalt ions in a predetermined amount ratio. In this example, aqueous ammonia (NH4OH) and sodium hydroxide (NaOH) were used as neutralizing agents. The addition of the neutralizing agent maintains the concentration of nickel ions in the solution in the range of 50 to 500 ppm, the pH of the solution in the range of 8 to 13, and the concentration of ammonium ions in the range of 5 to 15 g / liter. I went while controlling to maintain. At this time, nickel hydroxide having a relatively large amount of precipitation was smoothly precipitated (coprecipitated) while incorporating cobalt hydroxide. This precipitate is a fusion of nickel and cobalt in a state suitable as a raw material for a lithium-transition metal-containing composite oxide for a positive electrode active material. The obtained precipitate was recovered from the solution by filtration or the like and dried to obtain

Ascend Elements EX1002 Page 143

Duesenfeld Ex. 2014-143

a transition metal hydroxide material (step 267). The average particle size of this transition metal hydroxide material was in the range of 3 to 20 μm. The specific surface area was in the range of 3 to 20 m <2> / g. The full width at half maximum of the X-ray diffraction peak was in the range of 0.2 to 14 deg. A positive electrode active material produced using such a transition metal hydroxide material can constitute a lithium ion secondary battery having excellent battery performance (for example, discharge capacity).

[0078]

As described above, according to the present embodiment, the lithium-nickel constituting the positive electrode active material is formed by performing the hydrolysis treatment that maintains the material to be treated containing the positive electrode active material in a high temperature, high humidity, and oxidizing atmosphere. Lithium hydroxide can be produced from the composite oxide. This lithium hydroxide exposes the positive electrode current collector to a highly alkaline state, and realizes an environment in which aluminum forming the positive electrode current collector easily forms a hydroxide. As described above, according to the present embodiment, the hydroxide of aluminum (Al (OH)) is removed from the positive electrode current collector (aluminum foil) by an extremely simple method of leaving the electrode body as the material to be treated under predetermined conditions. 3, Li [Al (OH) 4 (H2O) 2] and the like) can be efficiently generated. The treatment method of this example (particularly, the step of decomposing water) can be carried out with a simple device configuration. Moreover, it is also excellent in the ability to process a large amount of material at once. When corrosion of the positive electrode current collector (generation of aluminum hydroxide) progresses, the positive electrode current collector gradually dissolves and loses its regularity. As a result, the positive electrode material attached to the positive electrode current collector falls off. That is, since the positive electrode material can be naturally dropped from the positive electrode current collector in this way, the step of separating (peeling) the positive electrode material from the positive electrode current collector is not necessary. Therefore, according to this embodiment, the processing steps of the lithium battery can be simplified. In addition, it is easy to collect the negative electrode current collector.

[0079]

The produced aluminum hydroxide (Al (OH) 3, Li [Al (OH) 4 (H2O) 2] and the like) can be eluted with water together with lithium hydroxide and separated from the insoluble matter of the material to be treated. Further, gelatinous Al (OH) 3 can be recovered by separating from the lithium solution (lithium hydroxide solution). The aluminum hydroxide thus recovered can be

Ascend Elements EX1002 Page 144

Duesenfeld Ex. 2014-144

effectively utilized as a fixing agent for the decomposition gas of the fluorine electrolyte. Lithium carbonate can be recovered from the lithium solution after separating Al (OH) 3. Further, using the lithium carbonate, a lithium hydroxide material suitable for use in manufacturing a positive electrode active material (lithium / transition metal-containing composite oxide) and the like can be obtained. On the other hand, from the transition metal component contained in the insoluble matter of the material to be treated, it is possible to obtain a transition metal hydroxide material suitable for applications such as production of a positive electrode active material (lithium / transition metal-containing composite oxide). As described above, according to this embodiment, various materials contained in the lithium ion secondary battery can be recovered in a highly useful form. In addition, the collected materials can be effectively reused.

[0080]

<Reference Example 1: Method for producing positive electrode active material> The transition metal hydroxide materials obtained in the above-described examples include nickel and cobalt mixed (fused) in an appropriate state. It is suitable as a raw material for production. For example, this transition metal hydroxide material and a lithium hydroxide material (LiOH.H2O, etc.) are mixed at a predetermined ratio and baked under appropriate conditions to obtain a lithium / nickel-containing composite oxide containing cobalt. It is possible to obtain a positive electrode active material (for example, a positive electrode active material represented by LiNi0.8Co0.2O2) substantially composed of the composite oxide containing. Here, as the lithium hydroxide material used together with the transition metal hydroxide material, the material obtained in the above-mentioned example (step 254) can be preferably used. Since such lithium hydroxide (monohydrate) has a high purity and especially contains a small amount of different alkali metal elements such as sodium, a separate purification step (for example, separation of lithium ions from sodium ions using a thin film separation device) is performed. It is possible to directly use it for the production of the positive electrode active material without performing the step of

[0081]

<Reference Example 2: Method for producing lithium-ion secondary battery> An example of producing a lithium-ion secondary battery by using (reusing) the positive electrode active material obtained in Reference Example 1 will be briefly described. For example, in order to manufacture the lithium-ion secondary battery 1 having the structure shown in FIG. 3, this positive electrode active material is dispersed in a suitable solvent together with carbon black (CB) and polytetrafluoroethylene (PTFE) powder to form a positive electrode material. Prepare the paste. As the solvent, water, N-methylpyrrolidone or the like can be used. The paste is applied

Ascend Elements EX1002 Page 145

Duesenfeld Ex. 2014-145

to a positive electrode current collector (aluminum foil or the like) to volatilize the solvent. In this way, a positive electrode sheet in which the positive electrode material layers are provided on both surfaces of the positive electrode current collector is produced. The positive electrode material paste can be applied using a comma coater, a die coater, or the like. On the other hand, carbon black (CB) and polytetrafluoroethylene (PTFE) powder are dispersed in a suitable solvent to prepare a negative electrode material paste. This is applied to a negative electrode current collector (copper foil or the like) and the solvent is volatilized to produce a negative electrode sheet in which negative electrode material layers are provided on both surfaces of the negative electrode current collector. These electrode sheets are laminated via a separator sheet (porous polypropylene sheet or the like). The laminated body is wound to produce an electrode body. The positive electrode terminal and the negative electrode terminal are connected to both ends of the electrode body in the axial direction by welding or the like. This is stored together with the electrolytic solution in a battery container made of aluminum or the like. As the electrolytic solution, a solution obtained by dissolving about 1 mol / liter of LiPF6 in a 7: 3 (mass ratio) mixed solvent of dimethyl carbonate (DMC) and ethylmethyl carbonate (EMC) can be used. As the DMC-EMC mixed solvent, the one recovered in step 222 (see FIG. 2) of the above-described example can be used. In this way, the lithium ion secondary battery can be constructed (regenerated).

[0082]

Specific examples of the present invention have been described above in detail, but these are merely examples and do not limit the scope of the claims. The technology described in the claims includes various modifications and changes of the specific examples illustrated above. Further, the technical elements described in the present specification or the drawings exert technical utility alone or in various combinations, and are not limited to the combinations described in the claims at the time of filing. Further, the technique illustrated in the present specification or the drawings achieves a plurality of purposes at the same time, and achieving one of the purposes has technical utility.

[0083]

It is a flow chart which shows an outline of a procedure which collects a valuable thing from a lithium ion secondary battery. It is a flowchart which shows the outline of the procedure of recovering a volatile component from a lithium battery. It is sectional drawing which shows the structure of a lithium ion secondary battery typically. It is explanatory drawing which expands and shows the electrode body of a lithium ion secondary battery. It is a schematic diagram which

Ascend Elements EX1002 Page 146

Duesenfeld Ex. 2014-146

shows the schematic structural example of the apparatus used for collection | recovery of a volatile component. It is explanatory drawing which shows the transition of the temperature in a furnace. It is a schematic diagram which shows the schematic structural example of the apparatus which makes H2O act on an electrode material.

Explanation of symbols

[0084]

1: Lithium ion secondary battery (lithium battery) 10: Winding type electrode body (material to be treated) 122: Positive electrode current collector (aluminum member) 124: Positive electrode material layer 142: Negative electrode current collector 144: Negative electrode material Layer 16: Separator sheet 20: Battery container (aluminum member) 60: Decompression heating furnace 72: Exhaust gas purifier 722: Treatment liquid 80: Hydrolysis treatment device 81: Pressure vessel 88: Water vapor supply means 89: Oxygen gas supply means

Ascend Elements EX1002 Page 147

Duesenfeld Ex. 2014-147

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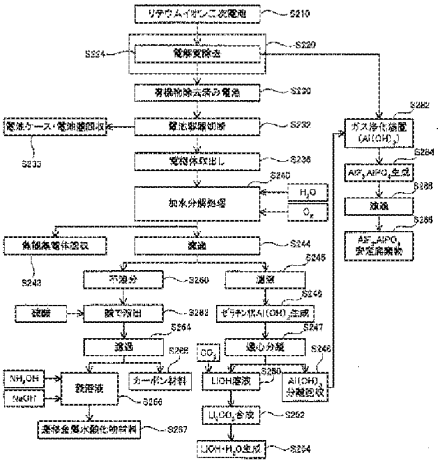
(54)【発明の名称】リチウム電池処理方法

(57)【要約】
【課題】　フッ素系電解質を備えるリチウム電池の処理方法であって、該電解質を取扱性のよい形態に効率よく変換することのできる処理方法を提供する。
【解決手段】　本発明の処理方法は、リチウム・遷移金属含有複合酸化物（正極活物質等）およびアルミニウム部材（正極集電体等）を含む被処理材を高温多湿雰囲気で処理する加水分解工程（ステップ２４０）を備える。この工程では、上記複合酸化物から水酸化リチウムを、上記アルミニウム部材からアルミニウムの水酸化物を生じさせる。その水酸化物に由来する水酸化アルミニウムを、リチウム電池から電解質（例えば$LiPF_6$）を加熱除去して得られた含フッ素物質（例えばPF_5）と反応させて（ステップ２８２）、取扱性のよい物質（例えばAlF_3および$AiPO_4$）を生じさせる（ステップ２８４）。
【選択図】　図１



【特許請求の範囲】
【請求項1】
リチウムと一種または二種以上の遷移金属元素とを含む複合酸化物から実質的に構成される正極活物質と、フッ素を構成元素として含む電解質と、アルミニウム部材とを備えるリチウム電池を処理する方法であって、
前記リチウム電池に由来し前記正極活物質および前記アルミニウム部材を含む被処理材にH_2Oを供給して該正極活物質から水酸化リチウムを生じさせるとともに該アルミニウム部材を構成するアルミニウムの水酸化物を生じさせる水酸化リチウム等生成工程と、
前記電解質に由来する含フッ素物質と前記水酸化物に由来する水酸化アルミニウムとを反応させる工程と、を包含するリチウム電池処理方法。
【請求項2】
前記水酸化リチウム等生成工程に先立って該リチウム電池から前記電解質を除去する工程を実施し、その除去された電解質に由来する前記含フッ素物質と水酸化アルミニウムとを反応させる請求項１に記載の方法。
【請求項3】
前記含フッ素物質と水酸化アルミニウムとを反応させる工程は、水酸化アルミニウムを含む処理液に前記電解質の熱分解物を含むガスを接触させる処理を含む請求項１または２に記載の方法。
【請求項4】
前記電解質はヘキサフルオロリン酸リチウムを含む請求項１から３のいずれか一項に記載の方法。
【請求項5】
前記水酸化リチウム等生成工程で生じた水酸化リチウムおよびアルミニウムの水酸化物を水に溶出させて該被処理材の不溶分から分離する工程と、
その分離工程により得られた溶液から水酸化アルミニウムを回収する工程と、
をさらに備える請求項１から４のいずれか一項に記載の方法。
【請求項6】
前記回収工程は、前記分離工程により得られた溶液のｐＨを調整して該溶液から水酸化アルミニウムを析出させる処理を含む請求項５に記載の方法。
【請求項7】
前記分離工程を経て得られたリチウム溶液に炭酸ガスおよび／または炭酸水を供給して炭酸リチウムを析出させる工程と、
該析出した炭酸リチウムを該溶液から分離する工程とをさらに包含する請求項５または６に記載の方法。
【請求項8】
前記アルミニウム部材は正極集電体であり、前記被処理材は該正極集電体上に前記正極活物質を有する正極を含む請求項１から７のいずれか一項に記載の方法。
【請求項9】
前記リチウム電池はシート状の正極およびシート状の負極を有する捲回型電極体を備え、該正極および該負極の少なくも一方はアルミニウム製の集電体を備え、該捲回型電極体を含む前記被処理材を前記水酸化リチウム等生成工程に供する請求項１から８のいずれか一項に記載の方法。
【請求項10】
前記リチウム電池を加熱して電解液を除去し、その加熱後に残った電池構成部材の一部または全部を前記水酸化リチウム等生成工程に供する請求項１から９のいずれか一項に記載の方法。
【請求項11】
前記リチウム電池を加熱して電解質および有機物を除去し、その加熱後に残った電池構成部材の一部または全部を前記水酸化リチウム等生成工程に供する請求項１から９のいず

れか一項に記載の方法。
【請求項12】
前記水酸化リチウム等生成工程は、前記正極活物質から水酸化リチウムとともに前記一種または二種以上の遷移金属元素のうち少なくとも一種の遷移金属元素の水酸化物を生じさせる処理を含む請求項１から１１のいずれか一項に記載の方法。
【請求項13】
前記被処理材の不溶分に含まれる前記一種または二種以上の遷移金属元素のうち少なくとも一種の遷移金属元素を酸に溶出させる工程と、
その溶液に中和剤を添加して前記酸に溶出させた遷移金属元素の水酸化物を析出させる工程とをさらに備える請求項１から１２のいずれか一項に記載の方法。
【請求項14】
前記リチウム電池を加熱して揮発性材料を揮発除去し、その揮発物を回収する工程を備え、該揮発物回収工程の前に、該回収に使用する装置内の水分をあらかじめ除去する工程を含む請求項１から１３のいずれか一項に記載の方法。
【発明の詳細な説明】
【技術分野】
【0001】
本発明は、リチウム電池を処理する技術に関し、特に、フッ素を構成元素として含む電解質を備えるリチウム電池を効率よく処理する技術に関する。
【背景技術】
【0002】
廃リチウム電池から有価物を回収することに関する種々の方法が提案されている。例えば特許文献１には、リチウムと遷移金属元素との複合酸化物（コバルト酸リチウム等）からリチウム成分および遷移金属成分を回収および再生する技術が記載されている。この文献では、上記電池正極廃材を硝酸で溶解し、これを沪過して硝酸リチウムおよび遷移金属元素の硝酸塩を含有する沪液と残渣とに分離し、この沪液に種々の処理を施して遷移金属成分およびリチウム成分をそれぞれ回収し、再生している。他の先行技術文献として特許文献２～１２が挙げられる。
【0003】
【特許文献1】特開平１１－５４１５９号公報
【特許文献2】特開平１０－２５５８６１号公報
【特許文献3】特開平１０－２５５８６２号公報
【特許文献4】特開平１１－６０２０号公報
【特許文献5】特開２０００－１５２１６号公報
【特許文献6】特開２００３－２７１５１号公報
【特許文献7】米国特許第５８８８４６３号明細書
【特許文献8】特開平８－４１５５４号公報
【特許文献9】特開平８－１３４５５６号公報
【特許文献10】特開２０００－１１９７６３号公報
【特許文献11】特開平１１－１６７９３６号公報
【特許文献12】特開２０００－１０６２２１号公報
【発明の開示】
【発明が解決しようとする課題】
【0004】
ところで、リチウム電池の多くのものは、電解質（支持塩）としてフッ素を構成元素とするリチウム塩（$LiPF_6$，$LiBF_4$，$LiAsF_6$等）を用いている。かかるフッ素系電解質を備えるリチウム電池の処理プロセス（例えば有価物を回収するプロセス）は、該電解質を処理するための工程を伴うことがある。例えば、$LiPF_6$を含むリチウム電池の処理プロセスでは、フッ化リンのような活性の高い含フッ素化合物が生成し得る。このフッ化リン等を取扱性のよい（安定な）化合物に変換する工程を設けることにより、フッ素系電解質を適切に処理（廃棄、再利用等）することができる。

【0005】
そこで本発明は、フッ素系電解質を備えるリチウム電池の処理方法であって、該電解質を取扱性のよい（例えば廃棄に適した）形態に効率よく変換することのできるリチウム電池処理方法を提供することを目的とする。関連する他の目的は、リチウム電池から有価物を、有用性の高い（例えば再利用しやすい）形態で、かつ／または、より効率よく回収する方法を提供することである。
【課題を解決するための手段】
【0006】
本発明者は、リチウム電池の構成要素としてアルミニウム製の部材がよく用いられている点に着目した。そして、アルミニウムの水酸化物を利用してフッ素系電解質を取扱性のよい形態に変換し得ること、および、リチウム電池の処理プロセス内において上記アルミニウム部材（例えば正極集電体）からアルミニウムの水酸化物を効率よく生成し得ることを見出して本発明を完成した。
【0007】
この出願により提供される一つの発明は、リチウムと一種または二種以上の遷移金属元素とを含む複合酸化物から実質的に構成される正極活物質と、フッ素を構成元素として含む電解質と、アルミニウム部材とを備えるリチウム電池（典型的にはリチウムイオン二次電池）を処理する方法に関する。その方法は、該リチウム電池に由来する被処理材であって前記正極活物質および前記アルミニウム部材を含む被処理材にH_2Oを供給して該正極活物質から水酸化リチウムを生じさせるとともに該アルミニウム部材からアルミニウムの水酸化物を生じさせる工程（水酸化リチウム等生成工程）を含む。さらに、前記電解質に由来する含フッ素物質と前記水酸化物に由来する水酸化アルミニウムとを反応させる工程を含む。
【0008】
ここで「アルミニウム部材」とは、アルミニウム製（アルミニウムまたはアルミニウムを主体とする合金を主構成材料とすることをいう。以下同じ。）の電池構成部材をいう。また、フッ素を構成元素として含む電解質（以下、「フッ素系電解質」ともいう。）の例としては、$LiPF_6$，$LiBF_4$，$LiAsF_6$，$LiCF_3SO_3$，$LiC_4F_9SO_3$，$LiN(CF_3SO_2)_2$，$LiC(CF_3SO_2)_3$等が挙げられる。本発明は、これらのうち一種または二種以上を備えるリチウム電池に対して適用され得る。特に、ヘキサフルオロリン酸リチウム（$LiPF_6$）を含む電解質を備えるリチウム電池に対して好ましく適用される。なお、上記「フッ素系電解質に由来する含フッ素物質」とは、フッ素系電解質それ自体（$LiPF_6$等）と、該電解質の分解（例えば熱分解、加水分解）等により生成したフッ素化合物（PF_5，HF等）とを包含する意味である。
【0009】
上記複合酸化物の好適例としては、リチウムとニッケルとを含む複合酸化物が挙げられる。ここで、「リチウムとニッケルとを含む複合酸化物」とは、リチウムとニッケルとを構成金属元素とする酸化物の他、リチウムおよびニッケル以外に他の少なくとも一種の金属元素（すなわち、リチウムおよびニッケル以外の遷移金属元素および／または典型金属元素）を含む複合酸化物をも包含する意味である。そのような複合酸化物は、例えば一般式$LiNi_{1-x}A_xO_2$で表すことができる。式中のAは、上記「他の少なくとも一種の金属元素」に対応する。また、式中のxは、$0 \leq x < 0.5$、好ましくは$0.1 < x < 0.3$を満たす値である。上記複合酸化物の他の好適例としては、リチウムとコバルトとを含む複合酸化物（リチウムおよびコバルト以外の少なくとも一種の金属元素を含有するものを包含する。）、リチウムとマンガンとを含む複合酸化物（リチウムおよびマンガン以外の少なくとも一種の金属元素を含有するものを包含する。）が挙げられる。
【0010】
アルミニウムの水酸化物を利用して、フッ素系電解質に由来する含フッ素物質を安定な化合物に変換することが可能である。例えば、ヘキサフルオロリン酸リチウム（$LiPF_6$）の分解により生じたフッ化リン（例えばPF_5）を水酸化アルミニウム（典型的には

、組成式Ａｌ（ＯＨ）$_3$で表されるアルミニウム水酸化物）と反応させることにより、三フッ化アルミニウム（AlF_3）、リン酸アルミニウム（$AlPO_4$）等を生じさせることができる。これらの反応生成物はいずれも安定な化合物である。したがって取り扱いが比較的容易である。また、廃棄するのに適した物質（安定廃棄物）である。このように、水酸化アルミニウムは、フッ素系電解質を安定な化合物に変換するための固定剤として機能し得る。

【0011】

上記水酸化リチウム等生成工程では、被処理材に含まれる複合酸化物（以下、「リチウム・遷移金属含有複合酸化物」あるいは単に「複合酸化物」ともいう。）にH_2Oを作用させて水酸化リチウムを生じさせる。かかる水酸化リチウムの存在によりアルミニウム部材がアルカリ性条件に曝されることとなり、これによりアルミニウムの水酸化物を生成する反応（アルミニウム部材の腐蝕）が促進される。したがって、該アルミニウム部材からアルミニウムの水酸化物を効率よく生じさせることができる。そして、その水酸化物に由来する水酸化アルミニウム（水酸化リチウム等生成工程で生じた水酸化アルミニウムと、該工程で生じた水酸化物に適当な処理を施して得られた水酸化アルミニウムとを包含する意味である。）を利用してフッ素系電解質を安定化合物に効率よく変換することができる。

このように、上記処理方法によると、リチウム電池の処理プロセス内で該リチウム電池の構成部材（アルミニウム部材）から生じるアルミニウム水酸化物を利用して、フッ素系電解質に由来する含フッ素物質を固定することができる。このため、該処理プロセスの外部から水酸化カルシウム（Ｃａ（ＯＨ）$_2$）等の固定剤を持ち込んで使用する必要がない。したがって、上記処理方法によるとフッ素系電解質を効率よく安定化合物に変換することができる。また、リチウム電池の構成材料であるアルミニウム部材を、フッ素系電解質の処理剤（固定剤）として有効に活用することができる。

【0012】

ここで開示される方法の好ましい態様では、前記水酸化リチウム等生成工程に先立って該リチウム電池から前記電解質を除去する工程を実施する。その除去された電解質に由来する含フッ素物質と水酸化アルミニウムとを反応させる。このように、あらかじめ除去した電解質に由来する含フッ素物質と水酸化アルミニウムとを反応させる態様によると、該電解質をより効率よく安定な化合物に変換することができる、被処理部材の取扱性が向上する、のうち少なくとも一つの効果を実現し得る。

リチウム電池から電解質を除去する方法としては、リチウム電池を加熱して該リチウム電池に含まれる電解質を揮発（熱分解）させて除去する方法を好ましく採用することができる。また、リチウム電池を非水系有機溶媒等により抽出して電解液（通常、電解質が有機溶媒に溶解した構成を有する。）を抜き取る方法により、該リチウム電池から電解質を除去してもよい。リチウム電池から電解質を除去する他の方法としては、特開平８－２２８４６号公報に示されるようにアルカリ性水溶液または酸性水溶液中に電池を浸漬し、開口させた安全弁から浸水した前記水溶液によって、電解液を溶解させて除去する方法が挙げられる。

【0013】

ここで開示される方法の他の好ましい態様では、前記含フッ素物質と水酸化アルミニウムとを反応させる工程が、水酸化アルミニウムを含む処理液（典型的には、Ａｌ（ＯＨ）$_3$が水系溶媒に溶解および／または分散したもの）に前記電解質の熱分解物を含むガスを接触させる処理を含む。かかる態様によると、電解質の熱分解物（含フッ素物質を含む）と水酸化アルミニウムとを効率よく反応させることができる。

上記電解質の熱分解物を含むガス（熱分解ガス）は、電解質を備えるリチウム電池を加熱して生じたものでもよく、あらかじめリチウム電池から除去した電解質を別途加熱して（熱分解させて）生じたものでもよい。ヘキサフルオロリン酸リチウムを含む電解質を備えるリチウム電池の場合、この熱分解ガスに含まれ得る熱分解物としては、フッ化リン（例えばPF_5）、フッ化水素（ＨＦ）等が挙げられる。

Ascend Elements EX1002　Page 152

Duesenfeld Ex. 2014-152

【0014】
ここで開示される方法の他の好ましい態様は、前記水酸化リチウム等生成工程で生じた水酸化リチウムおよびアルミニウム水酸化物を水に溶出させて被処理材の不溶分から分離する工程を含むことができる。また、その分離工程により得られた溶液から水酸化アルミニウムを回収する工程を含むことができる。
上記水酸化リチウム等生成工程では、被処理材に含まれるリチウム・遷移金属含有複合酸化物にH_2Oを作用させて水酸化リチウムを生じさせる。水酸化リチウムは水溶性である。また、この水酸化リチウム等生成工程においてアルミニウム部材から生じたアルミニウム水酸化物は、例えば$Al(OH)_3$，$Li[Al(OH)_4(H_2O)]$等として、水酸化リチウムとともに水によって溶出され得る。
一方、上記水酸化リチウム等生成工程前のリチウム・遷移金属含有複合酸化物や、該工程において水酸化リチウムとともに生じ得る遷移金属化合物（典型的には遷移金属水酸化物）は、主として実質的に非水溶性である。したがって、H_2Oで処理した被処理材を水で溶出する等の手段によって、水酸化リチウムおよびアルミニウムの水酸化物を、被処理材の不溶分（遷移金属成分を含む）から分離して取り出すことができる。例えば、上記処理後の被処理材（正極活物質）を水に分散させた後に沪過することにより、被処理材の不溶分と、リチウム成分およびアルミニウム成分を含む液相（水酸化リチウムおよびアルミニウム水酸化物が水に溶解した溶液）とを容易に分離することができる。
なお、被処理材からリチウムおよびアルミニウムの水酸化物を生じさせる工程と、被処理材の不溶分からリチウム成分およびアルミニウム成分を分離する工程とを同時進行的に行ってもよい。また、被処理材から生じたリチウムとアルミニウムの水酸化物とは、一緒に溶出してもよく、順次に溶出してもよい。例えば、まず水酸化リチウムを溶出した後に、アルミニウムの水酸化物を溶出（不溶分から分離）してもよい。
【0015】
このようにして被処理材の不溶分から分離した溶液から水酸化アルミニウムを回収する際には、該溶液のｐＨを調整して水酸化アルミニウムを析出させる処理を含む方法を好ましく採用することができる。例えば、リチウム成分およびアルミニウム成分を含む上記溶液にアンモニア水を加えてゼラチン状の水酸化アルミニウムを析出させる。これを遠心して、水酸化リチウムを含む上澄み溶液（リチウム溶液）とゼラチン状の水酸化アルミニウムとを分離・回収することができる。このようにして回収した水酸化アルミニウムを上記含フッ素物質と反応させることにより、電池構成材料（アルミニウム部材）に由来する水酸化アルミニウムを固定剤として効率よく利用することができる。例えば、回収した水酸化アルミニウムを水系溶媒（典型的には水）に溶解および／または分散させた処理液に、フッ素系電解質の熱分解物（PF_5等）を含むガスを接触させることにより、該熱分解物を安定な化合物（安定廃棄物）に効率よく変換することができる。なお、上記溶液から水酸化アルミニウムを回収する他の方法としては、特開平１１－５４１５９号公報の第００２５項および特開平１１－９７０７６号公報の第００４１項に示されるように溶液のｐＨを調整することにより沈殿させ分離回収する方法等を例示することができる。
【0016】
ここで開示される方法の好ましい態様は、上述のように被処理材の不溶分から分離する工程を経て得られたリチウム溶液から炭酸リチウムを析出させる工程を含む。例えば、該リチウム溶液に炭酸ガス（CO_2）および／または炭酸水（H_2CO_3）を供給することにより炭酸リチウムを析出させることができる。析出した炭酸リチウムを溶液から分離する工程をさらに含むことが好ましい。リチウム溶液としては、被処理材の不溶分から分離した溶液から、さらにアルミニウム成分（水酸化アルミニウム）を分離して得られた溶液を用いることが好ましい。これにより、より純度の高い炭酸リチウムを得ることができる。
【0017】
かかる態様によると、リチウム溶液に含まれるリチウム成分（リチウムイオン）を、固体状のリチウム化合物（炭酸リチウム）として回収することができる。このように固体状

として回収されたリチウム成分は再利用するのに都合がよい。リチウム溶液から炭酸リチウムを析出させるには、炭酸ガスまたは炭酸水（より好ましくは炭酸ガス）を用いるとよい。このようにアルカリ金属成分を含まない材料を用いて炭酸リチウムを析出させることにより、リチウム以外のアルカリ金属元素の混入が少ない炭酸リチウムを得ることができる。例えば、炭酸ガスまたは炭酸水に代えて炭酸ナトリウムを用いた場合と比較すると、得られた炭酸リチウムに混入したナトリウム成分の量を顕著に少なくすることができる。いったん混入したナトリウム成分をリチウム成分から分離することは一般に困難であるところ、上記態様によると、簡単な操作によって純度の高い（特に、リチウム以外のアルカリ金属元素や遷移金属成分等の混入が少ない）炭酸リチウムを得ることができる。このように高純度の炭酸リチウムは各種用途への再利用性に優れている。例えば、リチウム電池用の材料（正極活物質製造用の原料等）として好適である。

【0018】

この処理方法の典型的な態様では、前記水酸化リチウム等生成工程で、前記正極活物質から水酸化リチウムとともに前記一種または二種以上の遷移金属元素のうち少なくとも一種の遷移金属元素の水酸化物（例えば、リチウム・ニッケル含有複合酸化物から実質的に構成される正極活物質であれば主として水酸化ニッケル（$Ni(OH)_2$））を生じさせる。一般に遷移金属水酸化物は実質的に非水溶性であり、少なくとも水酸化リチウムに比べて水に対する溶解度が著しく低い。したがって、H_2Oで処理した被処理材の不溶分から水酸化リチウムを水で溶出する際、遷移金属水酸化物は不溶分として残る。このような水に対する溶解性の違いを利用して、水酸化リチウム（リチウム成分）と遷移金属水酸化物（遷移金属成分）とを容易に分離することができる。

【0019】

リチウム・遷移金属含有複合酸化物がH_2Oと接触して水酸化リチウムおよび遷移金属水酸化物を生じる反応は、該複合酸化物の加水分解反応としてとらえることができる。リチウム・ニッケル含有複合酸化物は、リチウムと他の遷移金属元素との複合酸化物（例えばリチウム・コバルト含有複合酸化物）に比べて上記加水分解を受けやすい（耐加水分解性が低い）。したがって、リチウム・ニッケル含有複合酸化物から実質的に構成される正極活物質またはそのような正極活物質を用いたリチウム電池は、本発明の処理方法の適用対象として特に適している。そのようなリチウム・ニッケル含有複合酸化物の一例としては、一般式$LiNiO_2$で表されるものが挙げられる。また、他の例として、一般式$LiNi_{1-x}Co_xO_2$で表されるものが挙げられる。ここで、$0<x<0.5$であり、好ましくは$0.1<x<0.3$である。Coに代えてAl，Mn，Cr，FeまたはVを有する複合酸化物であってもよい。さらに他の例としては、一般式$LiNi_{1-y-z}Co_yAl_zO_2$で表されるものが挙げられる。ここで、$0 \leqq y<0.5$（好ましくは$0<y<0.2$）であり、$0<z<0.5$（好ましくは$0<z<0.1$）であり、かつ$0<(y+z)<0.5$（好ましくは$0.1<(y+z)<0.3$）である。Alに代えて、あるいはAlとともに、Mn，Cr，Fe，V，Mg，Ti，Zr，Nb，Mo，W，Cu，Zn，Ga，In，Sn，LaおよびCeからなる群から選択される一種または二種以上の金属元素を有する複合酸化物であってもよい。

【0020】

上記水酸化リチウム等生成工程では、前記正極活物質を水蒸気（H_2O）と、酸素（O_2）および／または炭酸ガス（CO_2）を含む気相に接触させた条件でH_2Oを供給することが好ましい。H_2O，O_2およびCO_2を含む気相に接触させた条件でH_2Oを供給することがさらに好ましい。これにより、リチウム・遷移金属含有複合酸化物から水酸化リチウムおよび／または遷移金属水酸化物が生じる反応（典型的には上記加水分解反応）を効率よく進行させることができる。また、かかる反応をさらに促進するには、加熱および／または加圧条件下でH_2Oを供給することも有効である。かかる条件の下で水酸化リチウム等生成工程を実施することは、アルミニウム部材からアルミニウムの水酸化物を生じる反応を促進するという観点からも好ましい。

【0021】

Ascend Elements EX1002　Page 154

Duesenfeld Ex. 2014-154

上記被処理部材に含まれるアルミニウム製部材は、例えば正極および／または負極の集電体、電極リード板、電極端子、容器、安全弁等の電池構成部材であり得る。好ましい態様では、被処理材がアルミニウム製の正極集電体を含む。一般に正極集電体は比較的表面積の広い形状を有するので、該正極集電体（アルミニウム部材）からアルミニウム水酸化物を生じる反応（正極集電体の腐蝕）を効率よく進行させることができる。被処理材は、アルミニウム部材（好ましくは正極集電体）上にリチウム・遷移金属含有複合酸化物（正極活物質）を有することが好ましい。かかる態様によると、該複合酸化物から生じた水酸化リチウムによってアルミニウム部材の表面（近傍）を効率よく高アルカリ状態（好ましくはｐＨ９以上、より好ましくはｐＨ１１以上）とすることができる。このことによって、アルミニウム部材からアルミニウムの水酸化物を生じる反応がさらに促進され得る。

【0022】

ここで開示される方法の好ましい態様では、上記被処理材がシート状のアルミニウム部材（好ましくは正極集電体）を含む。このような形状のアルミニウム部材（例えばアルミニウム箔）は、表面積が広いことから、アルミニウムの水酸化物の生成（アルミニウム部材の腐蝕）を効率よく進行させることができるので好ましい。シート状の正極（典型的には、シート状の正極集電体の表面に正極集電体が付着したもの）と、シート状の負極（典型的には、シート状の負極集電体の表面に負極集電体が付着したもの）とが重ね合わされた構成の電極体を備えるリチウム電池は、ここで開示される方法の適用対象として好適なリチウム電池の一例である。かかる構成の電極体としては、いわゆる積層型電極体、捲回型電極体等が挙げられる。

【0023】

本発明の方法は、シート状の正極およびシート状の負極を有する捲回型電極体を備えたリチウム電池に対して特に好ましく適用される。その捲回型電極体を含む被処理材を前記水酸化リチウム等生成工程に供するとよい。捲回型電極体を構成する正極および負極の少なくも一方はアルミニウム製の集電体を備えることが好ましく、少なくとも正極集電体がアルミニウム製であることが好ましい。この場合には、水酸化リチウム等生成工程において、正極集電体（アルミニウム部材）がアルミニウム水酸化物を生じて定形性を失う（腐蝕して溶解する）ことにより、正極集電体に付着していた正電極材が脱落する。したがって正極集電体から正電極材を分別する操作を簡略化することができる。例えば、正極集電体から正電極材を分離（剥離）する操作を省略することができる。このことは、後述するように、正電極材に由来する各種有価物を回収する上で有利である。

【0024】

なお、アルミニウム製の正極集電体（アルミニウム部材）からアルミニウム水酸化物を生じさせることにより正極集電体と正電極材との分別を容易にするという上記効果は、生成したアルミニウム水酸化物をフッ素系電解質の処理（固定化）に用いない態様においても同様に発揮され得る。したがって、この明細書により開示される発明には、リチウムと一種または二種以上の遷移金属元素とを含む複合酸化物から実質的に構成される正極活物質とアルミニウム製の正極集電体とを備えるリチウム電池を処理する方法であって、前記正極活物質が前記正極集電体に付着している構成の正極を含む被処理材にH_2Oを供給して前記正極活物質から水酸化リチウムを生じさせるとともに前記正極集電体からアルミニウムの水酸化物を生じさせる工程を含むリチウム電池処理方法が含まれる。この処理方法は、さらに、上記水酸化リチウム等生成工程で生じた水酸化リチウム（好ましくは、水酸化リチウムおよびアルミニウム水酸化物）を水に溶出させて被処理部材の不溶分から分離する工程を含むことができる。

【0025】

正極活物質およびアルミニウム部材を含む被処理材は、他の電池構成材料をなるべく取り除いた（分別した）状態で水酸化リチウム等生成工程に供することが好ましい。このことによって、より再利用性のよい有価物を得ることができる。例えば、遷移金属元素や他のアルカリ金属元素等（異種金属元素）の混入がより少ない（品質のよい）炭酸リチウムが得られる。あるいは、より有用な遷移金属化合物（遷移金属水酸化物）が得られる。も

っとも、水酸化リチウム等生成工程に供する被処理材の分別は、かかる分別を行うためのコストや手間等と、それにより奏される効果の大きさ（回収される有価物の再利用性（品質）等）とを勘案して、それらが適切なバランスとなるように行えばよい。

【0026】

例えば、一般的なリチウム電池の多くは、正極集電体としてのアルミニウム箔（シート状のアルミニウム部材）に正極活物質を含む正電極材が付着した構成の正極を備える。この正電極材は、典型的には正極活物質に加えてポリフッ化ビニリデン、ポリテトラフルオロエチレン等の結着剤（バインダ）を含有する。さらにカーボンブラック等の導電材を含有することもある。これらを含む正電極材が正極集電体（アルミニウム部材）に付着している正極を、そのまま被処理材として用いる（水酸化リチウム等生成工程に供する）ことができる。また、後述する加熱処理等によって正電極材に含まれる結着剤の少なくとも一部（好ましくは実質的に全部）を除去した正極を被処理材として用いてもよい。シート状の負極集電体（例えば銅箔）の表面に負極活物質を含む負電極材が付着した構成の負極と上記正極とを積層した構成の電極体（捲回型電極体、積層型電極体等）を備えるリチウム電池にあっては、そのような正極および負極の双方を含む状態の電極体を被処理材として用いることができる。

なお、一般にリチウム電池の負極活物質としては、リチウムイオンを挿入・脱離し得るカーボン材料（カーボンブラック等）が用いられる。上記水酸化リチウム等生成工程では、負極活物質に挿入されているリチウムからも水酸化リチウムが生成し得る。この水酸化リチウムを水で溶出して不溶分と分離することにより、負極活物質中のリチウム成分を容易に取り出すことができる。したがって、水酸化リチウム等生成工程に供される被処理材が正電極材と負電極材とを含む場合には、これら両電極材に含まれるリチウム成分から水酸化リチウムを生じさせ、これを水で溶出して回収することができる。

【0027】

一方、あらかじめ被処理材と分別しておくことが好ましい電池構成材料の一例としては電解液が挙げられる。一般に電解液は、有機溶媒に電解質（支持塩）が溶解した構成を有する。被処理材は、電解液を構成する有機溶媒および電解質の少なくとも一方と分別しておくことが好ましく、有機溶媒および電解質の双方と分別しておくことがより好ましい。このことによって、電解質の構成材料（有機溶媒等）を有効に再利用することができる、電解質をより効率よく安定廃棄物に変換することができる、被処理材の取扱性が向上する、のうち少なくとも一つの効果が得られる。この電解液と被処理材との分別は、リチウム電池を加熱して電解液（有機溶媒および電解質）を除去することにより好適に行うことができる。その加熱後に残った電池構成部材の一部または全部を被処理材として水酸化リチウム等生成工程に供するとよい。また、電解液と被処理材とを分別する他の方法としては、リチウム電池を非水系有機溶媒等により抽出して電解液を抜き取る方法が挙げられる。

【0028】

他の一つの好ましい態様では、処理対象たるリチウム電池を加熱して揮発性材料を除去する処理を行う。ここで「揮発性材料」とは、加熱により気体状（蒸発、昇華、熱分解等の種類を問わない）となって除去し得る材料をいう。そのような揮発性材料としては、電解液を構成する有機溶媒、電解質（リチウム塩等）、セパレータ、結着剤、絶縁材、外装塗料等の一種または二種以上が挙げられる。各構成材料は、実質的に全部を除去することが好ましいが、少なくとも一部を除去することによっても本発明の効果が発揮され得る。リチウム電池に含まれる電解質および有機物（有機溶媒、セパレータ、結着剤等）の大部分を除去することが好ましく、実質的に全部を除去することがより好ましい。リチウム電池の加熱は、加熱温度が段階的に高くなる順序で実施することが好ましい。

その加熱後に残った電池構成部材の一部または全部を被処理材として水酸化リチウム等生成工程に供することができる。本発明をアルミニウム製の集電体（正極集電体）を有する捲回型電極体または積層型電極体を備えたリチウム電池に適用する場合には、該電極体を他の電池構成材料（例えば電池容器）から分別したものを被処理材として好ましく用いることができる。この分別は、例えば、電池容器を切断して電極体を取り出す等の操作に

よって容易に実施することができる。

【0029】

リチウム電池を加熱して揮発性材料（電解質および有機物）を除去する工程は、減圧下で行うことが好ましい。この場合には、減圧による沸点降下を利用して、常圧の場合よりも低温で揮発性材料（有機溶媒等）を揮発させることができる。これにより、例えば電解液に含まれる有機溶媒の変質（熱分解等）を抑制して、再利用性のよい回収物を得ることができる。また、減圧により揮発速度を高めることができるので、加熱下におかれる時間が短くなり、より変質の少ない（再利用性のよい）回収物を得るという効果が得られる。このように減圧下で加熱することにより、リチウム電池を構成する金属部材の酸化が抑制され得る。このことは金属部材を回収・再利用する上で有利である。

【0030】

ここで開示される発明には、前記リチウム電池を加熱して揮発性材料を揮発除去し、その揮発物を回収する工程を備え、該揮発物回収工程の前に該回収に使用する装置内の水分をあらかじめ除去する工程を行うリチウム電池処理方法が含まれる。これにより、回収物に水分が混入することを防止することができる。したがって、より有用な（例えば純度の高い）回収物を得ることができる。前記揮発物回収工程は、リチウム電池の加熱温度の異なる複数の工程に区分され得る。それら複数の工程は、リチウム電池の加熱温度が段階的に高くなる順序で実施することが好ましい。前記水分を除去する工程は、これら各区分のうち任意の区分の前に行ってもよく、全ての区分の前に行ってもよい。

なお、処理対象たるリチウム電池は、事前にほぼ完全に放電させておくことが好ましい。このことによって、処理時の取扱性や作業性の向上、作業環境の改善、処理装置の小型化・簡略化等のうち少なくとも一つの効果が得られる。

【0031】

ここで開示される処理方法は、さらに前記被処理材の不溶分に含まれる前記一種または二種以上の遷移金属元素のうち少なくとも一種の遷移金属元素を酸に溶出させる工程を包含することができる。また、その溶液に中和剤を添加して、該溶液に含まれる遷移金属元素の水酸化物を析出させる工程を包含することができる。上記酸溶出工程で得られた溶液を被処理材の不溶分から分離し（例えば沪別し）、その分離した溶液に中和剤を添加することが好ましい。

この方法で処理する正極活物質は、リチウムと、第一の遷移金属元素と、他の少なくとも一種の金属元素（以下、「他の金属元素」ということもある。）とを含むものであり得る。上記第一遷移金属元素の好適例としては、Ｎｉ，ＣｏおよびＭｎが挙げられる。また、上記他の金属元素は、第一遷移金属元素とは異なる金属元素であって、Ｃｏ，Ａｌ，Ｍｎ，Ｃｒ，Ｆｅ，Ｖ，Ｍｇ，Ｔｉ，Ｚｒ，Ｎｂ，Ｍｏ，Ｗ，Ｃｕ，Ｚｎ，Ｇａ，Ｉｎ，Ｓｎ，ＬａおよびＣｅからなる群から選択される一種または二種以上の金属元素であり得る。このような場合には、前記被処理材の不溶分に含まれる前記第一遷移金属元素および前記他の少なくとも一種の金属元素を酸に溶出させる工程を行い、その酸溶出工程で得られた溶液（好ましくは被処理材の不溶分から分離したもの）に中和剤を添加して、前記第一遷移金属元素の水酸化物とともに前記他の少なくとも一種の金属元素の水酸化物を該溶液から析出させるとよい。

【0032】

このとき、前記酸溶出工程で得られた溶液につき、前記第一遷移金属元素および前記他の金属元素の含有量の比（モル比、質量比等）を把握し、その比が所定範囲から外れている場合には該所定範囲内の量比に調整する。例えば、前記第一遷移金属元素がニッケルである場合、前記他の元素はＣｏ，Ａｌ，Ｍｇ，Ｍｎ，Ｃｒ等から選択される一種または二種以上であり得る。これらの金属元素の含有量の比を把握し（例えば組成分析等により）、規定量よりも不足している成分を補う等の手法によって所定範囲内の量比となるように調整する。その後、当該溶液から前記第一遷移金属元素の水酸化物とともに前記他の金属元素の水酸化物を析出（好ましくは共沈）させるとよい。特に、前記第一遷移金属元素がニッケルであり、前記他の金属元素がコバルトである場合には、水酸化ニッケルと水酸化

コバルトとを共沈させることにより、正極活物質用原料として適した状態でニッケルとコバルトとが融合した回収物が得られる。また、一般に溶液から析出された水酸化コバルトはコロイド状となりやすいところ、水酸化ニッケルと共沈させることにより液相と容易に分離することができる。

【0033】

前記中和剤としてはアンモニア水を用いることが好ましい。このアンモニア水は単独で使用してもよく、他の中和剤（アルカリ金属水酸化物等）と併用してもよい。中和剤としてアンモニア水を用いる場合、前記析出工程は、遷移金属イオンの濃度（第一遷移金属元素および他の金属元素を含有する溶液にあっては、第一遷移金属元素のイオン（第一遷移金属イオン）の濃度）、溶液のｐＨ、アンモニウムイオン（NH_4^+）の濃度、のうち少なくとも一つの項目が所定範囲となるように管理しつつ行うことが好ましい。上記析出工程では、これらの項目のうち二以上の項目が所定範囲となるように管理することが好ましく、三つの項目の全てが所定範囲内となるように管理することが特に好ましい。これにより、平均粒径、比表面積、Ｘ線回折ピークの半価幅、のうち少なくとも一つの特性値が所望の範囲にある析出物（主として遷移金属水酸化物）が得られる。これらの特性値のうち二つ以上が所望の範囲にあることが好ましく、三つの特性値の全てが所望の範囲にあることが特に好ましい。このような析出物は、リチウム電池の正極活物質として用いられるリチウム・遷移金属含有複合酸化物を製造するための原料として好適である。かかる析出物を用いて製造したリチウム・遷移金属含有複合酸化物を正極活物質として備えるリチウム電池は、充電特性、放電特性等に優れたものとなり得る。また、該析出物を用いて製造したリチウム・遷移金属含有複合酸化物は、保存性の良好なものとなり得る。なお、上記平均粒径は、例えばレーザ回折・散乱法を利用した粒径測定装置等により測定することができる。上記比表面積は、例えばＢＥＴ法を利用した比表面積測定装置等により測定することができる。上記半価幅は、例えば一般的な粉末Ｘ線回折装置を用いて得られたＸ線回折強度曲線から得ることができる。

【0034】

本発明は、リチウム・遷移金属含有複合酸化物の主たる（第一の）遷移金属元素がニッケルである複合酸化物（すなわち、リチウム・ニッケル含有複合酸化物）から実質的に構成される正極活物質を備えるリチウム電池に対して特に好ましく適用される。かかる複合酸化物の好適例としては、第一遷移金属元素がニッケルであり、他の金属元素（副成分）としてコバルトを含むリチウム・ニッケル含有複合酸化物が挙げられる。このような正極活物質を備えるリチウム電池においては、本発明の処理方法を採用することによる長所が特によく発揮され得る。

【0035】

ここで開示される他の発明は、リチウム電池用（典型的には、リチウムイオン二次電池用）の正極活物質に関する。その正極活物質は、水酸化リチウムと遷移金属水酸化物とを所定の割合で混合し、その混合物を焼成して生成させたリチウム・遷移金属含有複合酸化物を主体とする。ここで、水酸化リチウムとしては、上述したいずれかの処理方法によってリチウム電池から回収された炭酸リチウムを焼成し、その焼成物を水と反応させて得られた水酸化リチウム（例えば、水酸化リチウム一水塩（$LiOH \cdot H_2O$））を用いることができる。上記処理方法により得られた炭酸リチウムは純度の高いものとなり得る。特に、リチウム以外のアルカリ金属元素（ナトリウム等）の混入量の少ないものとすることができる。したがって、そのような炭酸リチウムから得られた（調製された）水酸化リチウムは、リチウム電池の正極活物質製造用の原料として好適に用いることができる。また、遷移金属水酸化物としては、上述したいずれかの方法によってリチウム電池から回収された遷移金属水酸化物を好ましく用いることができる。

【0036】

ここで開示される他の発明は、リチウム・遷移金属含有複合酸化物を主体とする正極活物質を用いたリチウム電池を出発原料としてリチウム電池用の正極活物質を製造する方法（リチウム電池用正極活物質の製造方法）に関する。その方法は、正極活物質製造用の遷

移金属水酸化物原料および水酸化リチウム原料を製造する工程と、それらの原料を所定の割合で混合して焼成する工程とを包含する。ここで、水酸化リチウム原料としては、以下の工程：出発原料としてのリチウム電池に由来する被処理材に含まれる正極活物質を加水分解して水酸化リチウムを生じさせる工程；その水酸化リチウムを水に溶出させて被処理材の不溶分から分離する工程；得られたリチウム溶液に炭酸（CO_2）ガスおよび／または炭酸水（H_2CO_3）を供給して該溶液から炭酸リチウムを析出させる工程；析出した炭酸リチウムを溶液と分離する工程；その炭酸リチウムを焼成し、さらに水と反応させる工程；を経て得られたものを用いることができる。また、遷移金属水酸化物原料としては、以下の工程：上記不溶分中の遷移金属成分を酸に溶出させる工程；その酸溶液に中和剤を添加して前記遷移金属を析出させる工程；を経て得られたものを用いることができる。

かかる製造方法によると、出発原料としてのリチウム電池に用いられている（主としてその正極活物質に含まれている）リチウム成分および遷移金属成分をそれぞれ回収し、それらを混合して焼成することによって、リチウム電池用の正極活物質（出発原料としてのリチウム電池の正極活物質と実質的に同一組成でもよく、異なる組成でもよい。）を再生することができる。

なお、上記焼成工程では、遷移金属水酸化物原料および水酸化リチウム原料の混合物を段階的に加熱することが好ましい。得られたリチウム電池用正極活物質（リチウム・遷移金属含有複合酸化物）は、必要に応じて粒度を揃える処理、吸着水分を除去する処理等を施した後に、リチウム電池の構成材料として好適に再利用することができる。

【0037】

この明細書により開示される発明には、上述したリチウム電池用の正極活物質を用いて構築されているリチウム電池が含まれる。そのようなリチウム電池の典型例としては、正極活物質を含む正電極材が正極集電体上に設けられている正極と、負極活物質を含む負電極材が負極集電体上に設けられている負極と、その正電極材と負電極材の間に配置されている電解液とを備え、ここで、正電極材に含まれる正極活物質として上記正極活物質を利用（再利用）して構築されているものが挙げられる。

このようなリチウム電池は、例えば以下の方法により好適に製造することができる。すなわち、水酸化リチウムと遷移金属水酸化物とを所定の組成比で混合する。その混合物を焼成してリチウム・遷移金属含有複合酸化物を生じさせる。その複合酸化物を集電体に付着させて正極を作製する工程を備えることができる。その正極を、電解液およびその電解液を介して配置された負極とともに電池容器に収容してリチウム電池を構築する工程を備えることができる。水酸化リチウムとしては、上述したいずれかの処理方法によってリチウム電池から回収された炭酸リチウムを焼成した後に水と反応させて生成させた水酸化リチウムを用いることができる。また、遷移金属水酸化物としては、上述したいずれかの処理方法によってリチウム電池から回収された遷移金属水酸化物を用いることができる。

【発明を実施するための最良の形態】

【0038】

以下、本発明に関する具体的実施例を説明するが、本発明をかかる実施例に示すものに限定することを意図したものではない。また、本明細書において特に言及している内容以外の技術的事項であって本発明の実施に必要な事項は、従来技術に基づく当業者の設計事項として把握され得る。本発明は、本明細書に開示されている技術内容と当該分野における技術常識とに基づいて実施することができる。

【0039】

＜実施例：リチウムイオン二次電池の処理方法＞

まず、この実施例で処理するリチウム電池（ここでは、使用済みの車両用リチウムイオン二次電池）の構成を説明する。

図３は、本実施例に係るリチウムイオン二次電池を示す模式的断面図であり、図４はその一部を拡大した説明図である。これらの図に示されるように、二次電池１は、一対の電極シート（正極シート１２および負極シート１４）が二枚のセパレータシート１６を介して偏平状に捲回された捲回型電極体１０と、電極体１０を収容する偏平な直方体状（角型

または平型ともいう。）の電池容器２０と、電極体１０の軸方向両端部にそれぞれ接続された正極端子３０および負極端子４０とを備える。

【0040】

図４に示すように、電極体１０を構成する正極シート１２は、長尺状の正極集電体１２２と、その両面に正電極材を層状に付着させて設けられた正電極材層１２４とを備える。また、負極シート１４は、長尺状の負極集電体１４２と、その両面に負電極材を層状に付着させて設けられた負電極材層１４４とを備える。電極体１０は、これらのシートを正極シート１２、セパレータシート１６、負極シート１４、セパレータシート１６の順に積層し、この積層体を長尺方向に捲回した構成を有する。積層された正極シート１２と負極シート１４との間はセパレータシート１６によって絶縁されている。図３に示すように、この捲回型電極体１０の軸方向の一端では正極端子３０が正極１２に接続され、軸方向の他端では負極端子４０が負極１４に接続されている。

【0041】

このリチウムイオン二次電池１の正電極材層１２４は、第一遷移金属元素がニッケルであって、他の金属元素としてコバルトを含有するリチウム・ニッケル含有複合酸化物から実質的に構成される正極活物質を主成分とする。かかる正極活物質は、一般式$LiNi_{1-x}Co_xO_2$（$0<x<0.5$、好ましくは$0.1<x<0.3$）で表すことができる。本実施例に係る二次電池１では、上記一般式におけるｘが約０．２であるリチウム・ニッケル含有複合酸化物（すなわち、$LiNi_{0.8}Co_{0.2}O_2$で表される複合酸化物）を正極活物質に用いている。正電極材層１２４は、カーボンブラック（ＣＢ）およびポリテトラフルオロエチレン（ＰＴＦＥ）をさらに含有する。それらの含有割合は、例えば正極活物質：ＣＢ：ＰＴＦＥの質量比が凡そ８５：１０：５となる割合である。一方、負電極材層（グラファイト層）１４４は、負極活物質としてのカーボンブラック（ＣＢ）を主成分とし、結着剤としてのポリテトラフルオロエチレン（ＰＴＦＥ）を含有する。それらの含有割合は、例えばＣＢ：ＰＴＦＥの質量比が凡そ９０：１０となる割合である。

【0042】

二次電池１を構成する正極集電体１２２はアルミニウム製の部材（アルミニウム部材）である。正極端子３０もアルミニウム製である。正極集電体１２２を構成するアルミニウム部材（アルミニウム箔）の厚さは凡そ５０μｍ以下（例えば２～５０μｍ）程度であることが好ましく、凡そ２０μｍ以下（例えば５～２０μｍ）程度であることがより好ましい。このような形状の正極集電体１２２によると、後述する水酸化リチウム等生成工程において、正極集電体１２２からアルミニウム水酸化物を生じる反応を効率よく進行させることができる。本実施例に係る二次電池１では、正極集電体１２２として厚さ約１５μｍのアルミニウム箔を用いている。一方、負極集電体１４２および負極端子４０は銅製である。特に限定するものではないが、負極集電体１４２を構成する銅部材（銅箔）の厚さは例えば凡そ２～５０μｍ程度であることが好ましく、凡そ５～２０μｍ程度であることがより好ましい。また、セパレータシート１６はポリオレフィン製（ここではポリプロピレン製）の多孔質シートである。

【0043】

電池容器２０はアルミニウム製であって、有底筒状の本体（電池ケース）２２と、本体２２の上端開口部を封止する蓋体（電池蓋）２４とを備える。この容器２０に捲回型電極体１０が収容されている。正極端子３０および負極端子４０は、蓋体２４を貫通して容器２０の外方に延びている。これらの端子３０，４０はナット３２，４２により蓋体に固定されている。負極端子４０と蓋体２４は絶縁体２６により隔てられている。蓋体２４は、電解液の注入等に用いられる注液口２７を有する。この注液口２７は、二次電池１の通常の使用時には封止された状態にある。また、蓋体２４には安全弁２８が設けられている。この安全弁２８は、容器２０の内部圧力が所定の設定値を超えて高くなると容器２０の内外を自動的に連通させて圧力を解放するように構成されている。

【0044】

電極体１０には図示しない電解液が含浸されている。この電解液を構成する有機溶媒と

しては、γ－ブチロラクトン、エチレンカーボネート、プロピレンカーボネート、ジメチルカーボネート、ジエチルカーボネート、エチルメチルカーボネート、１，２－ジメトキシエタン、１，２－ジエトキシエタン、テトラヒドロフラン、１，３－ジオキソラン等からなる群から選択された一種または二種以上を用いることができる。本実施例に係る二次電池１ではジメチルカーボネート（ＤＭＣ）とエチルメチルカーボネート（ＥＭＣ）との７：３（質量比）混合溶媒を用いている。また、この電解液を構成する電解質（支持塩）としては、フッ素を構成元素とする各種リチウム塩から選択される一種または二種以上を用いることができる。例えば、$LiPF_6$，$LiBF_4$，$LiAsF_6$，$LiCF_3SO_3$，$LiC_4F_9SO_3$，$LiN(CF_3SO_2)_2$，$LiC(CF_3SO_2)_3$等からなる群から選択される一種または二種以上を用いることができる。本実施例に係る二次電池１では、電解質としてヘキサフルオロリン酸リチウム（$LiPF_6$）を用いている。その濃度は約１mol/リットルである。

【0045】

次に、上記構成のリチウムイオン二次電池１を処理する手順につき説明する。図１は、その手順の概略を示すフローチャートである。

【0046】

まず、使用済みのリチウムイオン二次電池１を用意し（ステップ２１０）、これを加熱して電解質および有機物等の揮発性成分を回収する処理を行う（ステップ２２０）。図１および図２に示すように、このステップ２２０は、有機溶媒を除去（回収）するステップ２２２と、電解質を除去（回収）するステップ２２４と、セパレータを除去（回収）するステップ２２６と、結着剤を除去（回収）するステップ２２８とを含む。図１では、ステップ２２０に含まれる上記ステップのうち一部の図示を省略している。

【0047】

かかる揮発性成分の回収に用いる装置の概略構成例を図５に模式的に示す。図５において、減圧加熱炉６０は、処理室６４を区画する本体６１を備える。本体６１の壁面には誘導加熱コイル６２が設けられている。処理室６４内の温度は、コイル６２に接続された温度制御器６３によって任意のパターンに制御することができる。本体６１には真空ポンプ６５が接続されており、処理室６４の内部圧力を任意に制御できるようになっている。

処理室６４の下方には一次冷却装置６７が設けられている。処理室６４内の気体（例えばリチウムイオン二次電池から揮発した有機溶媒）は炉内ガス導管６６を介して一次冷却装置６７に導入される。一次冷却装置６７の壁面には誘導加熱コイル６８が設けられており、この一次冷却装置６７における冷却の程度を調節することができるようになっている。一次冷却装置６７の下流には、炉外ガス導管７０を介して二次冷却装置７１が連結されている。この二次冷却装置７１で冷却されて凝縮（液体化または固体化）した回収物はコレクタ７４に集められる。残りの気体は排ガス浄化器７２を介して外部に排出される。

【0048】

このような構成の装置を用いて、例えば以下のようにして二次電池１から有機溶媒を揮発除去し、その揮発物を回収する（ステップ２２２）。すなわち、あらかじめ開口した使用済みリチウムイオン二次電池１を処理室６４に収容する。二次電池１を開口する方法は、容器２０に貫通孔をあける方法、注液口２７の封止を解く方法、安全弁２８を作動させる方法等から適宜選択すればよい。真空ポンプ６５を稼動させて処理室６４内を減圧する。この減圧の程度は特に限定されない。通常は少なくとも８０kPa以下（典型的には０．１～８０kPa）に減圧することが適当である。５０kPa以下（典型的には０．１～５０kPa）に減圧することがより好ましい。本実施例では処理室６４内を１～１０kPaに減圧している。

かかる減圧状態を維持しつつ、誘導加熱コイル６２に通電して処理室６４内の温度（炉内温度）を上昇させる。これにより二次電池１を加熱する。このときの加熱温度は、処理室６４内の圧力に応じて、二次電池１に用いられている電解液の有機溶媒の沸点（二種以上の有機溶媒を含む場合には各成分の沸点またはそれらの共沸点）付近とすることができる。あるいは、上記有機溶媒以外の電池構成材料が実質的に揮発しない範囲で、該有機溶

Duesenfeld Ex. 2014-161

媒の沸点を上回る温度に加熱することも好適である。これにより二次電池１から有機溶媒を効率よく揮発させることができる。なお、常圧におけるＤＭＣの沸点は約９０℃であり、ＥＭＣの沸点は約１０７℃である。

【0049】

本実施例では、図６に示す温度チャートのように、炉内温度を常温から徐々に上昇させ、約８５℃に３０分間維持する（図中に矢印ａで示す段階）。これにより上記有機溶媒のうちＤＭＣが優先的に揮発（ガス化）する。発生したガスは、炉内ガス導管６６、一次冷却装置６７および炉外ガス導管７０を経て二次冷却装置７１に導入される。この二次冷却装置７１でガスを冷却して有機溶媒を凝結（液化）させ、コレクタ７４に回収する。次いで、炉内温度をさらに上昇させ、１０２℃に３０分間維持する（図中に矢印ｂで示す段階）。これにより、残存する有機溶媒（主としてＥＭＣ）を揮発（ガス化）させて二次冷却装置７１で液化・回収する。

【0050】

このように本実施例では、沸点の異なる二成分を含む有機溶媒（混合溶媒）を、二段階の加熱によって揮発させる。すなわち、上記ａの段階で相対的に低沸点のＤＭＣの大部分を揮発させた後に、加熱温度をさらに上昇させて残りの有機溶媒（主としてＥＭＣ）を揮発させる。このように加熱温度を段階的に変化させて低沸点溶媒から高沸点溶媒へと順に抜き取ることにより、加熱による変質（分解等）を抑制しつつ有機溶媒を効率よく揮発・回収することができる。

なお、２４℃におけるＤＭＣの蒸気圧は約２．４kPaであり、２５℃におけるＥＭＣの蒸気圧は約３．３kPaである。このため、上記ａ～ｂ段階で揮発したＤＭＣおよびＥＭＣは、典型的には混合溶媒として回収される。後述するように、この回収された混合溶媒は、不純物（ＤＭＣおよび／またはＥＭＣの熱分解物等）の含有割合が少なく、再利用性のよいものである。例えば、リチウムイオン二次電池用の電解液として使用（再利用）することができる。

【0051】

次いで、上記加熱後に残った電池構成材料をさらに加熱して電解質（ここでは$LiPF_6$）を熱分解させ、その熱分解物を含むガス（分解ガス）を除去する（ステップ２２４）。すなわち、例えば図６に示す温度チャートのように、炉内温度を$LiPF_6$の熱分解温度（約１６０℃）以上の温度域に維持する（図中に矢印ｃで示す段階）。このときの加熱温度は、例えば約１６０～２００℃の範囲とすることができ、約１８０～２００℃の範囲とすることが好ましい（本実施例では約２００℃）。これにより$LiPF_6$が熱分解して、主にフッ化リチウム（ＬｉＦ）とフッ化リン（PF_5）が生じる。このフッ化リンを含む分解ガス（排ガス）は、後述する工程によってさらに処理される。なお、このｃの段階を行う際の加熱温度は上記温度に限定されるものではなく、実用的な速度で電解質を揮発させて二次電池１から除去し得る温度域であればよい。例えば、電解質の揮発（熱分解）温度と同等以上の温度域で行うことができる。揮発（熱分解）温度～該温度＋５０℃の範囲とすることが好ましい。減圧度が同程度であれば、ｃ工程における加熱温度は上記ａおよびｂの段階の温度よりも８０℃以上高く設定することが好ましい。

【0052】

図２に示すステップ２２４の後に残った電池構成材料からセパレータを揮発（主として熱分解）させて除去し、その揮発物を回収する（ステップ２２６）。すなわち、図６に示す温度チャートのように、炉内温度を約３００℃に維持する（図中に矢印ｄで示す段階）。これにより、図３に示す電極体１０を構成するセパレータ１６（多孔質ポリプロピレンシート）が熱分解して低級炭化水素等のガスを生じる。そのガスは、一次冷却装置６７および／または二次冷却装置７１にて液状（例えば、複数種類の炭化水素の混合物を主体とする粘稠な液体状）または固体状として回収することができる。得られた回収物は、燃料等として有効に再利用することができる。

さらに、上記加熱後に残った電池構成材料から、電極材を構成する結着剤を揮発（主として熱分解）させて除去し、その揮発物を回収する（ステップ２２８）。すなわち、図６

に示す温度チャートのように、炉内温度を約４００℃に維持する（図中に矢印ｅで示す段階）。これにより、電極材を構成する結着剤（ポリテトラフルオロエチレン）が熱分解して、低級炭化水素やフッ化物等のガスを生じる。生じたガスは、一次冷却装置６７、二次冷却装置７１および排ガス浄化器７２のうち一または二以上の箇所で液体状または固体状として回収（捕集）することができる。

【0053】

なお、上記ｄおよびｅの段階を行う際の加熱温度は上記温度に限定されるものではなく、それぞれセパレータおよび結着剤を実用的な速度で揮発（熱分解等）させて二次電池１から除去し得る温度域であればよい。セパレータおよび結着剤（有機物）の除去に要する時間の短縮および／または結着剤の除去率の向上という観点からは、このｄ段階および／またはｅ段階を行う際の加熱温度を高めに設定することが好ましい。ただし、この電池を構成する金属部材が溶融しない程度の温度（例えば、アルミニウムの場合には凡そ５７０℃以下、より好ましくは凡そ５５０℃以下）とすることが好ましい。

【0054】

以上の工程（ステップ２２０）では、加熱温度を段階的に上昇させて各段階毎に揮発物を回収する。このように二次電池からその構成材料を揮発温度の低い材料から高い材料へと順次抜き取ることにより、再利用性のよい回収物（有価物）を得ることができる。なお、このように電池からまず有機溶媒を抜き取る（除去する）ことは、電池の内部抵抗が高まること等から、以後の工程における電池の取扱性が向上するので好ましい。また、この有機溶媒の抜き取り（ステップ２２２）を減圧下で行うことにより、(1).過度の加熱による有機溶媒の変質を防ぐ、(2).有機溶媒の除去に要する時間を短縮する、(3).加熱のためのエネルギーを節約する、のうち少なくとも一つの効果を得ることができる。

【0055】

上述したステップ２２４で除去された電解質（その分解ガス）は、以下の工程（フッ化リン安定化工程）により、安定的な化合物として回収することができる。

すなわち、電解質の分解により生じたフッ化リンを含む分解ガス（排ガス）は、図５に示す一次冷却装置６７および二次冷却装置７１を経て、真空ポンプ６５の下流に接続された排ガス浄化器７２に導入される（ステップ２８２）。この排ガス浄化器７２の器内には、水酸化アルミニウムを含む処理液７２２が貯留されている。後述するように、この処理液７２２に含まれる（溶解および／または分散している）水酸化アルミニウムとしては、本実施例のリチウム電池処理プロセス内において、アルミニウム部材としての正極集電体１２２に由来して生成し、回収された$Al(OH)_3$を使用することができる。

【0056】

排ガス浄化器７２の底部付近に設けられた排ガス供給口７２４から処理液７２２中に排ガスをバブリングする。これにより、処理液７２２中の水酸化アルミニウム（$Al(OH)_3$）が排ガス中のフッ化リンと反応し（ステップ２８４）、フッ化アルミニウム（AlF_3）およびリン酸アルミニウム（$AlPO_4$）を生じる。詳しくは、フッ化リンが水と反応してフッ化水素およびリン酸に分離していったん溶液となり、これが液中に分散および／または溶解している水酸化アルミニウムと速やかに反応してAlF_3と$AlPO_4$に変化する。ここで、AlF_3は難溶性であり、その溶解度は０．５ｇ／１００ｇである。また、$AlPO_4$は不溶性であり、その溶解度積（K_{sp}）は1.3×10^{-20}である。このように、AlF_3，$AlPO_4$は難溶性または不溶性であるため、必要に応じて沪過することにより（ステップ２８６）、これらの生成物を固形物として容易に回収することができる。このように、水酸化アルミニウムをフッ化リンガスの浄化剤として利用することにより、電解質としての$LiPF_6$に含まれるフッ素成分およびリン成分を取扱性のよい形態で回収（捕集）することができる。例えば、AlF_3および$AlPO_4$はいずれも有害指定物質ではないことから、安定廃棄物として適切に処分することができる（ステップ２８８）。

なお、図５には電解質（その分解ガス）の処理効率を高める等の目的から二つの排ガス浄化器７２を直列に設けた構成を示しているが、排ガス浄化器７２の数は一つでもよい。

あるいは、二以上の排ガス浄化器７２を直列または並列に設けた構成としてもよい。また、処理液７２２中に排ガスを導入する際には、該排ガスを細泡状でバブリングすることにより、排ガスと処理液との接触面積を多くして反応効率を高めることが好ましい。

【0057】

上記工程（ステップ２２０）を経て、電解質および有機物が除去された有機物除去済み電池を得る（ステップ２３０）。この有機物除去済み電池（電池構成材料の加熱残分）は、容器本体（電池ケース）および蓋体を含む容器２０と、正極集電体および正極活物質を含む正極シート１２と、負極集電体および負極活物質とを含む負極シート１４とを包含する。正極シート１２と負極シート１４とは捲回型電極体１０を構成している。図１に示すステップ２３２では、この有機溶媒除去済み電池２３０を、例えば容器２０の蓋部２４を切り落とす（切断する）等の方法によって分割し（ステップ２３２）、捲回型電極体１０を取り出す（ステップ２３６）。電極体１０から分別した容器２０（電池ケース２２および蓋体２４）は、回収し（ステップ２３３）、アルミニウム材料として各種用途に再利用することができる。

【0058】

このようにして他の電池構成材料から分離された捲回型電極体１０は、すでにセパレータシート１６が失われている（揮発している）ことにより、正極シート１２と負極シート１４との間に空隙の多い状態となっている。図１に示すステップ２４０は、この電極体１０を被処理材として、これに加水分解処理を施す（H_2O等を供給する）工程である。この工程に用いる装置の概略構成例を図７に模式的に示す。

【0059】

加水分解処理装置８０は、処理室９０を区画する密閉型圧力容器８１を備える。この圧力容器８１は反応槽８１２および蓋体８１４を含み、ステンレス鋼を用いて構成されている。圧力容器８１は、水蒸気供給手段８８および酸素ガス供給手段８９を備える。水蒸気供給手段８８は、水蒸気発生器８８２と、該発生器８８２から延びて容器８１に接続された水蒸気供給管８８４とを備える。また、酸素ガス供給手段８９は、酸素ガス供給源８９２と、該発生源８９２から延びて容器８１に接続された酸素ガス供給管８９４とを備える。水蒸気供給管８８４および酸素ガス供給管８９４は、それぞれ蓋体８１４を貫通して処理室９０に水蒸気および酸素ガスを供給可能に配設されている。圧力容器８１の反応槽８１２の下部にはヒータ９２が設けられている。反応槽８１２には、このヒータ９２よりも高い位置まで水（ここでは純水）９２が貯留されている。また、圧力容器８１には処理室９０内の圧力を調整する圧力制御手段９１を備える。この圧力制御手段９１には圧力制御弁９１２および排気口９１４が含まれる。さらに、圧力容器８１には、処理室９０の内部温度を検出する温度センサ（温度計）８４および図示しない温度調節機構が設けられている。

【0060】

反応槽８１２の内部には、水９２の液面よりもやや高い位置に、通気性の仕切板８３が設けられている。この仕切板８３の上に処理対象たる電極体１０（被処理材）が配置されている。水９２はヒータ８２によって所定温度（ここでは約８０℃）に加温されている。これにより水蒸気の発生を促進している。処理室９０内は、水９２から生じる水蒸気と、必要に応じて水蒸気供給手段８８および酸素ガス供給手段８９からそれぞれ導入される水蒸気および酸素ガスによって、水蒸気および酸素が充満した高温（ここでは約８０℃）雰囲気に維持されている。また、処理室９０内は圧力制御手段９１によって所定の槽内圧力に維持されている。

なお、この加水分解処理装置８０は、上記酸素ガス供給装置および酸素供給管に代えて、あるいは酸素ガス供給装置および酸素供給管に加えて、炭酸ガス供給装置および炭酸ガス供給管を備える構成とすることができる。

【0061】

かかる処理室９０内の雰囲気に電極体１０を曝す（電極体１０にH_2Oを供給する）ことにより、この電極体１０に含まれる正極活物質が加水分解して、水酸化リチウムおよび

遷移金属水酸化物（主として水酸化ニッケル）を生じる。また、電極体１０に含まれる負極活物質に挿入されたリチウムからも水酸化リチウムが生じ得る。このとき圧力容器内では、例えば以下の反応式により表される現象等が起こっているものと考えられる。

【0062】

$$3LiNiO_2 + 2H_2O \rightarrow 3NiOOH + Li_2O + LiOH \quad \cdots (1)$$

$$2NiOOH + H_2O \rightarrow 2Ni(OH)_2 + 1/2O_2 \quad \cdots (2)$$

$$Li_2O + H_2O \rightarrow 2LiOH \quad \cdots (3)$$

$$C/Li + H_2O \rightarrow C + LiOH + 1/2H_2 \quad \cdots (4)$$

【0063】

上記反応式（１）～（３）は正極活物質に関連する反応である。反応式（１）によりオキシ水酸化ニッケル（ＮｉＯＯＨ）が生じる。このオキシ水酸化ニッケルは、反応式（２）により分解して水酸化ニッケル（$Ni(OH)_2$）となる。一方、反応式（１）で生成した酸化リチウム（Li_2O）は、反応式（３）により水酸化リチウム（ＬｉＯＨ）となる。なお、この反応は炭酸ガスと水蒸気の存在する雰囲気下において反応途中では同様に起こっている（Li_2O，ＬｉＯＨの生成）が、最終的には生成水酸化リチウム（ＬｉＯＨ）と炭酸ガスとが反応して炭酸リチウム（Li_2CO_3）を生成する。

反応式（２）で生成した酸素ガスは気相中に逃げ、結局これらの反応式によりニッケル酸リチウムから水酸化リチウムおよび水酸化ニッケルが生じることになる。正極活物質に含まれるコバルト成分も同様にして水酸化コバルトを生じ得る。

【0064】

また、上記反応式（４）は、負極活物質に挿入されているリチウムに関連する反応である。また、式中の「Ｃ／Ｌｉ」は、このリチウムがカーボン材料（ここではカーボンブラック）に挿入された状態にあることを表している。反応式（４）では、負極活物質中のリチウムが水酸化リチウム（ＬｉＯＨ）を生成するとともに水素ガスが発生する。この水素ガスは、処理室９０に充填されている酸素ガスにより水を生じて安定化する。

【0065】

かかる加水分解によって正極活物質から生じた水酸化リチウム等により、電極体１０を構成する正極集電体の表面が高アルカリ状態（典型的にはｐＨ１１以上）となる。このように高アルカリの湿潤状態に曝された正極集電体は、その表面から徐々に腐蝕する。すなわち、正極集電体を構成するアルミニウムから、$Al(OH)_3$，$Li[Al(OH)_4(H_2O)_2]$等のアルミニウム水酸化物が生じる。この腐蝕（水酸化物を生じる反応）は、捲回型電極体１０の外部（外表面、端面等）から始まり、次第に電極体１０の中央部へと進行する。ここで、上述のように電極体１０が空隙の多い状態になっていることは、電極体１０の中央部への腐蝕の進行を促進するという観点から有利である。かかる腐蝕の進行を促進するために、あらかじめ電極体１０をいくつかに分割（例えば、捲回軸に直交する平面で二つまたはそれ以上の部分に分割）しておくことも有効である。電池容器２０を切断する工程（ステップ２３２）において、該容器２０とともに電極体１０を切断してもよい。

【0066】

処理温度、処理圧力、処理時間、雰囲気中の水蒸気濃度および酸素濃度等の条件は、加水分解反応および／または正極集電体の腐蝕反応の進行状況に応じて適宜調整することができる。加熱下で処理することにより、加水分解反応および／または腐蝕反応を促進することができる。例えば４０～１２０℃で処理することができ、好ましくは５０～１００℃、より好ましくは６０～９０℃である。また、加圧下で処理することにより、加水分解反応および／または腐蝕反応を促進することができる。もっとも、処理圧力を過度に高くすることは、処理装置８０（処理容器８１等）の大型化を招きやすい。これらの兼ね合いから、好ましい処理圧力は凡そ０．１～０．５MPa（約１～５atm）程度であり、より好ましい圧力は凡そ０．１～０．２MPa（約１～２atm）程度である。

【0067】

処理室９０内は水蒸気濃度の高い雰囲気とすることが好ましく、処理室９０内をほぼ飽

和水蒸気圧に維持することがより好ましい。処理室９０内で水が循環するように処理条件を調節するとよい。例えば、反応槽８１２の底部に貯留された水９２が蒸発し、その水蒸気が電極体（被処理材）１０等に接触して凝結し、さらに電極体１０を伝い仕切板８３を通って水９２に戻るようにする。これにより、電極体１０から生成した水酸化リチウムおよび／またはアルミニウム水酸化物を水９２中に溶かし出す（抽出する）ことができる。処理室９０内の雰囲気圧力に占める酸素分圧は５０%以上（典型的には５０～９０%、より好ましくは５０～７５%）とすることが好ましい。また、処理室９０内の雰囲気を、水蒸気を含む（好ましくは水蒸気の飽和した）大気雰囲気としてもよい。かかる大気雰囲気に酸素および／または炭酸ガスを供給して酸素濃度および／または炭酸ガス濃度を高めた雰囲気としてもよい。特に限定するものではないが、酸素ガスの供給量は例えば１０～１０００ｍｌ／分（好ましくは１００～５００ｍｌ／分）とすることができる。また、水蒸気の供給量は例えば１～２０ｇ／分（好ましくは5～１０ｇ／分）とすることができる。

【0068】

加水分解工程（ステップ２４０）を終了する時期は、加水分解反応および／または腐蝕反応の進行の程度をみて決めることができる。通常は、正極集電体の腐蝕（アルミニウム水酸化物の生成）がほぼ完了するまで加水分解工程を継続することが好ましい。これにより、正極集電体を構成するアルミニウムが$Al(OH)_3$，$Li[Al(OH)_4(H_2O)_2]$等のアルミニウム水酸化物となって（好ましくは水に溶解および／または分散して）正極集電体が実質的に消失している。これにより、正極集電体１２２に付着していた正電極材は支持体を失って脱落するので、正極集電体１２２から正電極材を分離（例えば剥離）する操作が不要となる。

好ましい処理条件では、約１ヶ月以内の期間で正極集電体の腐蝕を完了する（正極集電体を実質的に消失させる）ことができる。処理室９０内が酸化性雰囲気であること、高温（ここでは約８０℃）多湿雰囲気であることは、いずれも正極集電体１２２の腐蝕を促進して加水分解工程にかかる時間を短縮するために有利である。

【0069】

このようにして加水分解工程２４０を施した被処理材は、負極集電体（銅箔）、水酸化リチウム、アルミニウムの水酸化物、遷移金属水酸化物、カーボンブラック（グラファイト）等を含む状態となっている。これらのうち負極集電体（銅箔）は、当初の形状を維持したまま（主として金属状態で）残留している。この負極集電体はそのまま回収し（ステップ２４３）、鋼材料として再利用することができる。あるいは、加水分解工程２４０の途中で負極集電体を回収してもよい。

【0070】

好ましい態様では、上記加水分解工程の間に、該工程で生じた水酸化リチウム、アルミニウムの水酸化物が水で溶出されている。また、加水分解工程２４０の終了後に残留物を水洗して、該残留物から水酸化リチウムおよびアルミニウムの水酸化物を水で溶出してもよい。一方、水酸化ニッケル（$Ni(OH)_2$）および水酸化コバルト（$Co(OH)_2$）の溶解度は、２５℃の水に対してそれぞれ約7.5×10^{-4}ｇ／１００ｇ（H_2O）および約$8\sim22\times10^{-4}$ｇ／１００ｇ（H_2O）である。これらの遷移金属成分（水酸化ニッケルおよび水酸化コバルト）は実質的に水に溶出しない。また、カーボンブラックは実質的に水不溶性である。したがって、加水分解処理された被処理材を水で溶出（抽出、水洗等）し、沪過等によって液相と不溶分とを分離することにより（ステップ２４４）、水酸化リチウムおよびアルミニウムの水酸化物を含む沪液（溶液）を得る（ステップ２４５）。また、この分離によって水酸化ニッケル等の遷移金属水酸化物およびカーボンブラックを含む不溶分（残渣）を得る（ステップ２６０）。

【0071】

ステップ２４４の分離により得られた溶液は、水酸化リチウムおよびアルミニウム水酸化物（$Al(OH)_3$，$Li[Al(OH)_4(H_2O)_2]$等）を含む。この溶液から水酸化アルミニウムを回収する（ステップ２４６～２４８）。すなわち、該溶液にアンモニア水（NH_4OH）を添加することにより（ステップ２４６）、該溶液中のアルミニウ

ム水酸化物からゼラチン状のＡｌ（ＯＨ）$_3$を生じさせる。これを遠心分離機にかけると（ステップ２４７）、水酸化リチウムを含む上澄み液（リチウム溶液）とゼラチン状のＡｌ（ＯＨ）$_3$とに層分離する。これにより、上記溶液から水酸化アルミニウム（Ａｌ（ＯＨ）$_3$）を容易に分離（回収）することができる（ステップ２５８）。回収したＡｌ（ＯＨ）$_3$は、図１に示すステップ２８２において、図５に示す排ガス浄化器７２に備えられる処理液７２２の有効成分（電解質の分解ガスの固定剤）として循環利用することができる。本実施例によると、このような循環型リサイクルシステムを構築することができる。

【0072】

また、上記ステップ２４７における上澄み液を水酸化アルミニウムと分離してリチウム溶液を得る（ステップ２５０）。このリチウム溶液からリチウム成分を固体状として回収する工程につき説明する。すなわち、この溶液をいったん加熱してアンモニアを放出させる。その後、該溶液に炭酸ガス（CO_2）を供給すると、液中のリチウムイオンがCO_2と反応して炭酸リチウム（Li_2CO_3）を生じる（ステップ２５２）。このとき、ステップ２４７で得られたリチウム溶液を、水酸化リチウムが析出しない程度の範囲で濃縮して用いてもよい。炭酸リチウムは水酸化リチウムに比べて水に対する溶解度が低いので、溶液から析出して沈殿を生じる。その沈殿（析出物）を液相と沪別して乾燥させることにより、固体状の炭酸リチウムを回収することができる。

【0073】

ここで、リチウム溶液に炭酸ガスを供給する方法としては、溶液にCO_2ガスをバブリングする方法等を採用することができる。リチウム溶液（リチウムイオン）とCO_2とをより効率よく反応させるために、CO_2を含む加圧（例えば、常圧～凡そ１MPaの）雰囲気下で反応させる、CO_2を含む雰囲気中にリチウム溶液を噴霧する、等の手段を適宜採用することができる。雰囲気中の炭酸ガス濃度は高いほうが好ましい。例えばCO_2濃度が凡そ５０～１００vol%（より好ましくは凡そ８０～１００vol%）の雰囲気中で反応させることが好ましい。実質的にCO_2ガスからなる雰囲気下で反応させることが特に好ましい。

なお、反応液に溶存するCO_2の濃度が高くなると炭酸リチウムの溶解度が上昇する傾向にある。そこで、例えばCO_2濃度がほぼ１００%の常圧または加圧（例えば、常圧～凡そ１MPa以下の）雰囲気下でリチウムイオンとCO_2とを反応させた後、反応液に溶存するCO_2の量を低下させる（液中からCO_2を追い出す）処理を行うことが好ましい。この脱炭酸処理によって溶液のCO_2濃度が低下すると、炭酸リチウムの溶解度が低下する。その結果、該脱炭酸処理前には反応液に溶解していた炭酸リチウムの一部が析出（再結晶）するので、これを液相から容易に分離回収することができる。上記脱炭酸処理は、例えば、反応液を常圧または減圧の大気雰囲気中で撹拌する、その雰囲気中で反応液に空気または不活性ガス（窒素ガス等）をバブリングする、等の手段により実施することができる。また、炭酸リチウムの溶解度は温度の上昇とともにやや低下する傾向にある（１００℃では約０．７２ｇ／１００ｇ（H_2O））。したがって、反応液の温度を高める（例えば約４０～８０℃に加温する）ことにより、炭酸リチウムの溶解度を低下させて析出量を増すことができる。このような手法によって、リチウム溶液からのリチウム成分（炭酸リチウム）の回収率をさらに向上させることができる。

【0074】

このようにして回収した炭酸リチウム（$LiCO_3$）を加熱することにより、炭酸リチウムをLi_2OとCO_2に分解させることができる。通常、このときの加熱温度は凡そ１５００℃以上とすることが適当である。このLi_2Oに水蒸気（H_2O）を供給することにより$LiOH \cdot H_2O$を生成することができる（ステップ２５４）。この水酸化リチウム一水塩（$LiOH \cdot H_2O$）は、例えば、リチウム電池の正極活物質等として用いられるリチウム・遷移金属含有複合酸化物の製造原料（水酸化リチウム材料）として有効に利用することができる。

【0075】

上記ステップ２４４の沪過により沪別された不溶分の処理につき説明する。

まず、この不溶分に酸（本実施例では硫酸（H_2SO_4）を用いた。）を加えて金属成分を溶出する（ステップ２６２）。これを沪過して（ステップ２６４）該金属成分（ここでは主としてニッケルおよびコバルト）のイオンを含む酸溶液と不溶分とに分別する（ステップ２６５，２６６）。ここで分別された不溶分は、主に正電極材の導電材および負極活物質として用いられていたカーボン材料（カーボンブラック）である。回収されたカーボン材料は、例えば融雪材、土壌改良材等として有効に利用することができる。

【0076】

一方、ステップ２６４で得られた沪液は、ニッケルイオンおよびコバルトイオンを含有する酸溶液（ここでは硫酸溶液）である。以下のステップでは、この酸溶液を用いて、リチウム－ニッケル複合酸化物にコバルトが添加された正極活物質（典型的には、$LiNi_{1-x}Co_xO_2$（$0<x<0.5$、好ましくは$0.1<x<0.3$）で表される正極活物質）を製造するための原料等として有用な遷移金属水酸化物材料を合成する。ここでは、$LiNi_{0.8}Co_{0.2}O_2$で表される正極活物質を製造するための原料を合成（調製）する場合につき説明する。

上記硫酸溶液に含まれるニッケルイオンおよびコバルトイオンの濃度を常法により測定する。そして、これらのイオンの濃度（含有量）の比が、あらかじめ設定した目標値（ここでは、金属換算でＮｉ：Ｃｏのモル比が０．８：０．２）から所定以上外れている場合には、足りないイオン種を外部から補充（溶液中に添加）する。これにより硫酸溶液に含まれる両イオンの量比が所定範囲内となるように調整する。このように不足分のみを補うので、新たに使用する遷移金属成分（例えばコバルト）の量を節約することができる。なお、以前の処理経験や予備実験等からステップ２６４で得られた硫酸溶液に含まれるニッケルイオンおよびコバルトイオンの量比を予測（把握）できる場合には、これらのイオンの濃度を測定する操作を省略することができる。

【0077】

ニッケルイオンおよびコバルトイオンを所定の量比で含有する硫酸溶液に中和剤を添加する。本実施例では、中和剤としてアンモニア水（NH_4OH）および水酸化ナトリウム（ＮａＯＨ）を使用した。中和剤の添加は、溶液中のニッケルイオンの濃度を５０～５００ppmの範囲に維持し、溶液のｐＨを８～１３の範囲に維持し、かつアンモニウムイオンの濃度を５～１５ｇ／リットルの範囲に維持するように制御しつつ行った。このとき、相対的に析出量の多い水酸化ニッケルが、水酸化コバルトを取り込みつつ順調に沈殿（共沈）した。この沈殿物は、正極活物質用のリチウム・遷移金属含有複合酸化物の原料として適した状態でニッケルとコバルトとが融合したものである。得られた沈殿物を沪別等により溶液から回収し、乾燥させて、遷移金属水酸化物材料を得た（ステップ２６７）。この遷移金属水酸化物材料の平均粒径は３～２０μmの範囲にあった。また、比表面積は３～２０m^2／ｇの範囲にあった。Ｘ線回折ピークの半価幅は０．２～１４degの範囲にあった。このような遷移金属水酸化物材料を用いて製造した正極活物質は、電池性能（例えば放電容量）に優れたリチウムイオン二次電池を構成し得る。

【0078】

以上説明したように、本実施例によると、正極活物質を含む被処理材を高温、多湿、酸化性雰囲気に維持する加水分解処理を実施することによって、該正極活物質を構成するリチウム・ニッケル複合酸化物から水酸化リチウムを生じさせることができる。この水酸化リチウムによって正極集電体が高アルカリ状態に曝されることとなり、該正極集電体を構成するアルミニウムが水酸化物を生成しやすい環境が実現される。このように、本実施例によると、被処理材としての電極体を所定条件下に放置するという極めて簡便な方法によって、正極集電体（アルミニウム箔）からアルミニウムの水酸化物（$Al(OH)_3$，$Li[Al(OH)_4(H_2O)_2]$等）を効率よく生じさせることができる。本実施例の処理方法（特に上水分解工程）は、簡単な装置構成によって実施することができる。また、大量の被処理材を一度に処理する能力にも優れている。正極集電体の腐蝕（アルミニウム水酸化物の生成）が進行すると、正極集電体は次第に溶解して定形性を失う。これにより正極集電体に付着していた正電極材が脱落する。すなわち、このように、正極集電体か

ら正電極材を自然に脱落させることができるので、正極集電体から正電極材を分離（剥離）する工程が不要となる。したがって、本実施例によるとリチウム電池の処理工程を簡略化することができる。また、負極集電体の回収も容易である。

【0079】

生成したアルミニウム水酸化物（Ａｌ（ＯＨ）$_3$，Ｌｉ［Ａｌ（ＯＨ）$_4$（H_2O）$_2$］等）は、水酸化リチウムとともに水で溶出して被処理材の不溶分から分離することができる。さらに、リチウム溶液（水酸化リチウム溶液）から分離してゼラチン状のＡｌ（ＯＨ）$_3$を回収することができる。このようにして回収された水酸化アルミニウムは、フッ素電解質の分解ガスの固定剤として有効に活用することができる。Ａｌ（ＯＨ）$_3$を分離した後のリチウム溶液からは炭酸リチウムを回収することができる。さらに、その炭酸リチウムを利用して、正極活物質（リチウム・遷移金属含有複合酸化物）の製造等の用途に適した水酸化リチウム材料を得ることができる。一方、被処理材の不溶分に含まれる遷移金属成分からは、正極活物質（リチウム・遷移金属含有複合酸化物）の製造等の用途に適した遷移金属水酸化物材料を得ることができる。このように、本実施例によると、リチウムイオン二次電池に含まれる各種材料を有用性の高い形態で回収することができる。また、それらの回収物を有効に再利用することができる。

【0080】

＜参考例１：正極活物質の製造方法＞

上記実施例により得られた遷移金属水酸化物材料は、ニッケルとコバルトとが適切な状態に混在（融合）していること等から、正極活物質製造用の原料として好適である。例えば、この遷移金属水酸化物材料と水酸化リチウム材料（ＬｉＯＨ・H_2O等）とを所定の割合で混合し、適切な条件で焼成することにより、リチウム・ニッケル含有複合酸化物であってコバルトを含む複合酸化物から実質的に構成される正極活物質（例えば$LiNi_{0.8}Co_{0.2}O_2$で表される正極活物質）を得ることができる。ここで、遷移金属水酸化物材料とともに用いる水酸化リチウム材料としては、上記実施例（ステップ２５４）により得られたものを好ましく用いることができる。かかる水酸化リチウム（一水塩）は高純度であり、特にナトリウム等の異種アルカリ金属元素の含有量が少ないことから、別途精製工程（例えば、薄膜分離装置を用いてナトリウムイオンからリチウムイオンを分離する工程等）を行うことなく、そのまま正極活物質の製造等に利用することが可能である。

【0081】

＜参考例２：リチウムイオン二次電池の製造方法＞

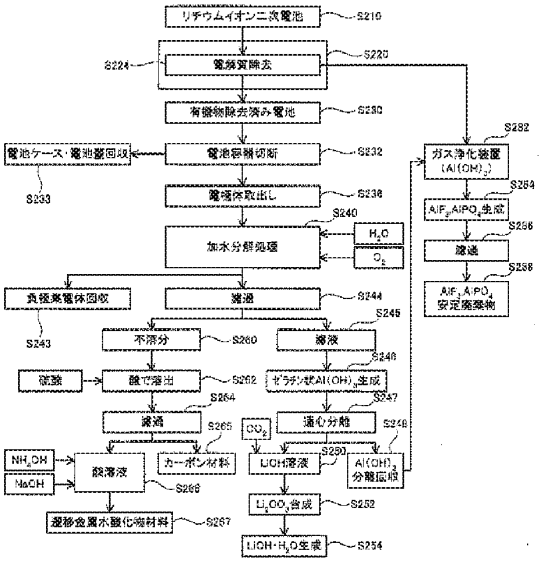
参考例１により得られた正極活物質を利用（再利用）してリチウムイオン二次電池を製造する例につき簡単に説明する。例えば、図３に示す構成のリチウムイオン二次電池１を製造するには、この正極活物質をカーボンブラック（ＣＢ）およびポリテトラフルオロエチレン（ＰＴＦＥ）粉末とともに適当な溶媒に分散させて正電極材ペーストを調製する。溶媒としては、水、Ｎ－メチルピロリドン等を用いることができる。このペーストを正極集電体（アルミニウム箔等）に塗布して溶媒を揮発させる。このようにして、正極集電体の両面に正電極材層が設けられた正極シートを作製する。正電極材ペーストの塗布は、コンマコーター、ダイコーター等を用いて行うことができる。一方、カーボンブラック（ＣＢ）およびポリテトラフルオロエチレン（ＰＴＦＥ）粉末を適当な溶媒に分散させて負電極材ペーストを調製する。これを負極集電体（銅箔等）に塗布して溶媒を揮発させることにより、負極集電体の両面に負電極材層が設けられた負極シートを作製する。セパレータシート（多孔質ポリプロピレンシート等）を介してこれらの電極シートを積層する。その積層体を捲回して電極体を作製する。電極体の軸方向両端に正極端子および負極端子を溶接等により接続する。これを電解液とともにアルミニウム製等の電池容器に収容する。電解液としては、ジメチルカーボネート（ＤＭＣ）とエチルメチルカーボネート（ＥＭＣ）との７：３（質量比）混合溶媒に約１㏖/リットルの$LiPF_6$を溶解させたもの等を用いることができる。このＤＭＣ－ＥＭＣ混合溶媒としては、上記実施例のステップ２２２（図２参照）により回収したもの等を使用可能である。このようにしてリチウムイオン二次電池を構築（再生）することができる。

【0082】
以上、本発明の具体例を詳細に説明したが、これらは例示にすぎず、特許請求の範囲を限定するものではない。特許請求の範囲に記載の技術には、以上に例示した具体例を様々に変形、変更したものが含まれる。
また、本明細書または図面に説明した技術要素は、単独であるいは各種の組み合わせによって技術的有用性を発揮するものであり、出願時請求項記載の組み合わせに限定されるものではない。また、本明細書または図面に例示した技術は複数目的を同時に達成するものであり、そのうちの一つの目的を達成すること自体で技術的有用性を持つものである。
【図面の簡単な説明】
【0083】
【図1】リチウムイオン二次電池から有価物を回収する手順の概略を示すフローチャートである。
【図2】リチウム電池から揮発性成分を回収する手順の概略を示すフローチャートである。
【図3】リチウムイオン二次電池の構成を模式的に示す断面図である。
【図4】リチウムイオン二次電池の電極体を拡大して示す説明図である。
【図5】揮発性成分の回収に用いる装置の概略構成例を示す模式図である。
【図6】炉内温度の推移を示す説明図である。
【図7】電極材にH_2Oを作用させる装置の概略構成例を示す模式図である。
【符号の説明】
【0084】
１：リチウムイオン二次電池（リチウム電池）
１０：捲回型電極体（被処理材）
１２２：正極集電体（アルミニウム部材）
１２４：正電極材層
１４２：負極集電体
１４４：負電極材層
１６：セパレータシート
２０：電池容器（アルミニウム部材）
６０：減圧加熱炉
７２：排ガス浄化器
７２２：処理液
８０：加水分解処理装置
８１：圧力容器
８８：水蒸気供給手段
８９：酸素ガス供給手段

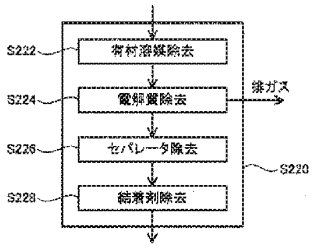
Ascend Elements EX1002　Page 170

Duesenfeld Ex. 2014-170

【図1】


リチウムイオン二次電池
S210
電解質除去
S220
S224
有機物除去済み電池
S230
電池ケース・電池蓋回収
S233
電池容器切断
S232
電極体取出し
S236
加水分解処理
S240
H_2O
O_2
負極集電体回収
S243
濾過
S244
不溶分
S260
濾液
S245
硫酸
酸で溶出
S262
ゼラチン状$Al(OH)_3$生成
S246
濾過
S264
S265
CO_2
遠心分離
S247
S248
NH_4OH
NaOH
酸溶液
S266
カーボン材料
LiOH溶液
S250
$Al(OH)_3$分離回収
Li_2CO_3合成
S252
遷移金属水酸化物材料
S267
LiOH・H_2O生成
S254
ガス浄化装置($Al(OH)_3$)
S282
AlF_3AlPO_4生成
S284
濾過
S286
AlF_3AlPO_4安定廃棄物
S288

【図2】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有材溶媒除去
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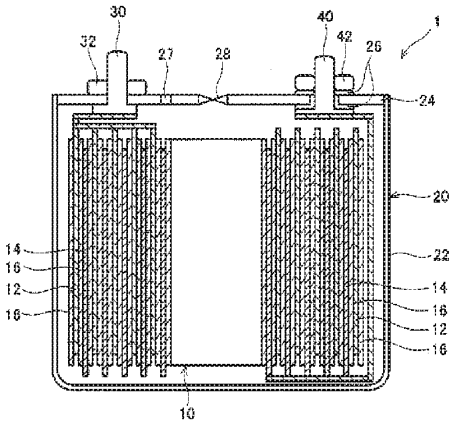
S220

【図3】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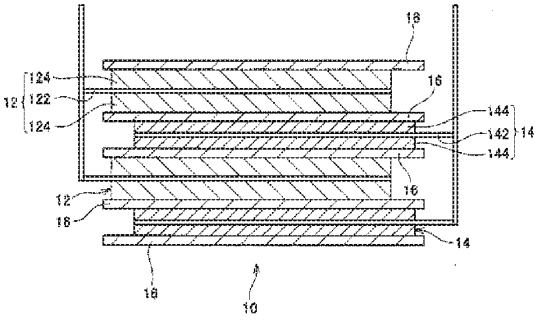
【図4】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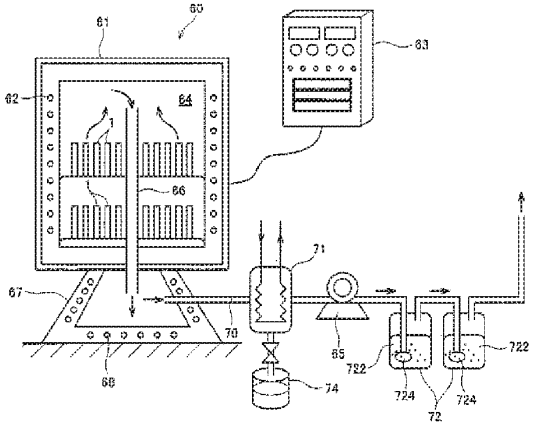
【図5】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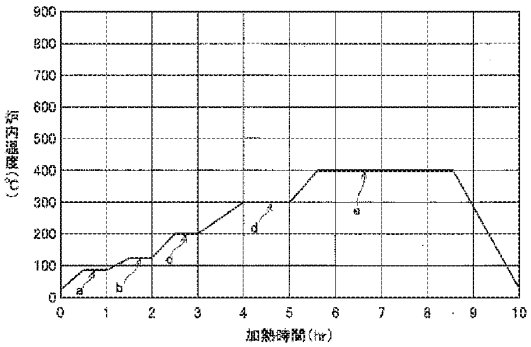
【図6】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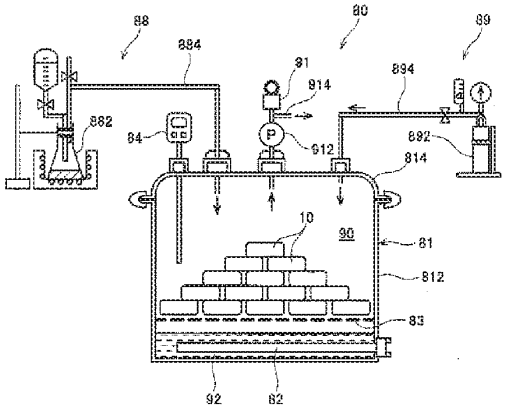
【図7】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(51)Int.Cl.[7]	FI	テーマコード（参考）
Ｃ２２Ｂ　21/00	Ｃ２２Ｂ　21/00	
Ｃ２２Ｂ　26/10	Ｃ２２Ｂ　26/10	

Ｆターム(参考)　4K001 AA02　AA07　AA19　AA34　BA22　DB07　DB22　DB23
　　5H031 AA02　BB09　EE03　RR02

Electronic Acknowledgement Receipt

EFS ID:	39839679
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Michael E. Whitham/Kyle Comiskey
Filer Authorized By:	Michael E. Whitham
Attorney Docket Number:	03101160US
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Filing Date:	26-OCT-2017
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Application Type:	U.S. National Stage under 35 USC 371

Payment information:

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Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Information Disclosure Statement (IDS) Form (SB08)	03101160US_IDS4.pdf	1034297 3436506200b780575aaef3ce7ffa796a60ce1cc9	no	4

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Ascend Elements EX1002 Page 174

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National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Electronic Patent Application Fee Transmittal

Application Number:	15569484
Filing Date:	26-Oct-2017
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Timothy Bryce Miller
Attorney Docket Number:	03101160US

Filed as Large Entity

Filing Fees for U.S. National Stage under 35 USC 371

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
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Miscellaneous-Filing:				
Petition:				
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Post-Allowance-and-Post-Issuance:				

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Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
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If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.
National Stage of an International Application under 35 U.S.C. 371
If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.
New International Application Filed with the USPTO as a Receiving Office
If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Doc code: RCEX
Doc description: Request for Continued Examination (RCE)

PTO/SB/30EFS (02-18)
Approved for use through 11/30/2020. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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REQUEST FOR CONTINUED EXAMINATION(RCE)TRANSMITTAL
(Submitted Only via EFS-Web)

Application Number	15569484	Filing Date	2017-10-26	Docket Number (if applicable)	03101160US	Art Unit	3725
First Named Inventor	HANISCH			Examiner Name	Swiatocha		

This is a Request for Continued Examination (RCE) under 37 CFR 1.114 of the above-identified application.
Request for Continued Examination (RCE) practice under 37 CFR 1.114 does not apply to any utility or plant application filed prior to June 8, 1995, or to any design application. The Instruction Sheet for this form is located at WWW.USPTO.GOV

SUBMISSION REQUIRED UNDER 37 CFR 1.114

Note: If the RCE is proper, any previously filed unentered amendments and amendments enclosed with the RCE will be entered in the order in which they were filed unless applicant instructs otherwise. If applicant does not wish to have any previously filed unentered amendment(s) entered, applicant must request non-entry of such amendment(s).

☐ Previously submitted. If a final Office action is outstanding, any amendments filed after the final Office action may be considered as a submission even if this box is not checked.

- ☐ Consider the arguments in the Appeal Brief or Reply Brief previously filed on _____
- ☐ Other _____

☒ Enclosed

- ☒ Amendment/Reply
- ☐ Information Disclosure Statement (IDS)
- ☐ Affidavit(s)/ Declaration(s)
- ☐ Other _____

MISCELLANEOUS

☐ Suspension of action on the above-identified application is requested under 37 CFR 1.103(c) for a period of months _____
(Period of suspension shall not exceed 3 months; Fee under 37 CFR 1.17(i) required)

☐ Other _____

FEES

☒ **The RCE fee under 37 CFR 1.17(e) is required by 37 CFR 1.114 when the RCE is filed.**
The Director is hereby authorized to charge any underpayment of fees, or credit any overpayments, to Deposit Account No 502041

SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT REQUIRED

☒ Patent Practitioner Signature

Applicant Signature

PTO/SB/30EFS (02-18)
Approved for use through 11/30/2020. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Signature of Registered U.S. Patent Practitioner			
Signature	/T. Bryce Miller/	Date (YYYY-MM-DD)	2020-06-04
Name	T. Bryce Miller	Registration Number	72545

This collection of information is required by 37 CFR 1.114. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of

HANISCH et al.

Application No. 15/569,484

Filed October 26, 2017

Confirmation No. 9373

Group Art Unit 3725

Examiner Swiatocha

For METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

AMENDMENT UNDER 37 C.F.R. § 1.114

Sir:

This response is concurrently filed with an RCE.

In response to the Office Action mailed March 5, 2020, Applicant respectfully requests entry of the following Amendment and reconsideration.

Amendments to the Claims: begin on page 2.

Remarks: begin on page 7.

Ascend Elements EX1002 Page 183

Duesenfeld Ex. 2014-183

Listing of the Claims:

The following is a complete listing of all the claims in the application, with an indication of the status of each:

1. (currently amended) A method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained;

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained, wherein the inactivating step is performed during or after the comminuting step; and

(c) filling a transport container with the inactivated comminuted material;

wherein the inactivating step is performed by drying the comminuted material, and

wherein the drying occurs at a maximum pressure of 300 hPa.

2. (previously presented) The method according to claim 1, wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

3. (previously presented) The method according to claim 1, wherein the drying occurs under vacuum.

4. (previously presented) A method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained;

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained; and

(c) filling a transport container with the inactivated comminuted material;

wherein the inactivating step is performed by drying the comminuted material,

wherein the drying occurs under vacuum,

wherein the vacuum is created by means of a jet pump, and

wherein a pump liquid of the jet pump has at least one of

a pH value of at least 8, and

a substance that precipitates fluoride.

Ascend Elements EX1002 Page 184

Duesenfeld Ex. 2014-184

5. (previously presented) The method according to claim 1, further comprising the steps of:

removing the comminuted material from the transport container; and

one or more of

detaching hard parts,

separating active material from a carrier, such that an active material fraction and a carrier fraction are obtained, and

separately packing the active material fraction and carrier fraction in respective further containers.

6. (previously presented) The method according to claim 1, wherein the drying of the comminuted material is only completed if one or more of the following conditions is met:

no flammable or explosive gas mixture is formable above the comminuted material in the transport container, and

the comminuted material is so dry that no flammable or explosive gas mixture is able to emerge in the transport container.

7. (currently amended) A battery processing installation for treatment of used batteries, comprising:

(a) a comminution unit configured to comminute the batteries such that comminuted material is obtained;

(b) an inactivation device comprising a drying device configured to inactivate the comminuted material, wherein the inactivation device is configured to perform the inactivating step during or after the comminuting step of the comminution unit;

(c) a filling device configured to fill a transport container with the inactivated comminuted material; and

(d) a vacuum installation connected to the drying device and configured to generate a vacuum in the drying device.

8. (previously presented) The battery processing installation according to claim 7, wherein the drying device is configured to dry the comminuted material until an electrolyte content in the

Ascend Elements EX1002 Page 185

Duesenfeld Ex. 2014-185

comminuted material is so low that an electrochemical reaction is impossible.

9. (previously presented) The battery processing installation according to claim 7, wherein the comminution unit and the drying device are arranged in a container.

10. (canceled)

11. (previously presented) A battery processing installation for treatment of used batteries, comprising:

(a) a comminution unit configured to comminute the batteries such that comminuted material is obtained;

(b) an inactivation device configured to inactivate the comminuted material, wherein the inactivation device comprises a drying device;

(c) a filling device configured to fill a transport container with the inactivated comminuted material;

(d) a vacuum installation that is connected to the drying device and configured to generate a vacuum in the drying device;

wherein

the vacuum installation has a jet pump with a pump liquid,

the pump liquid is conducted within a circuit, and

the pump liquid contains a substance that reacts with hydrogen fluoride.

12. (previously presented) The battery processing installation according to claim 7 further comprising:

a hard metal detachment device and/or a light fraction detachment device;

a separation device configured to separate active material from a carrier such that an active material fraction and a carrier fraction occur; and

a second filling device configured to separately fill the active material fraction and the carrier fraction in respective containers.

Ascend Elements EX1002 Page 186

Duesenfeld Ex. 2014-186

13. (previously presented) The battery processing installation of claim 12 wherein the carrier fraction are aluminum and copper foils.

14. (previously presented) The battery processing installation of claim 12, wherein the separation device is an air jet sieving device.

15. (previously presented) The battery processing installation of claim 12 wherein the separation device is a second comminution device.

16. (previously presented) The battery processing installation of claim 7 wherein the batteries are lithium batteries.

17. (previously presented) The method according to claim 1 wherein the batteries are lithium batteries.

18. (previously presented) The method according to claim 5 wherein separation of the active material from the carrier is performed by air jet sieving.

19. (previously presented) The method according to claim 5 wherein separation of the active material from the carrier is performed using a second comminution stage.

20. (previously presented) A method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained,

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained, and

(c) filling a transport container with the inactivated comminuted material,

wherein the inactivating step is performed by drying the comminuted material,

wherein the drying occurs under vacuum,

wherein the vacuum is created by means of a jet pump, and

wherein a pump liquid of the jet pump has a pH value of at least 9.

Ascend Elements EX1002 Page 187

Duesenfeld Ex. 2014-187

21. (previously presented) The method according to claim 1 wherein drying occurs at a temperature of less than 80°C.

22. (new) The method according to claim 1 further comprising a step of sealing the transport container so as to be dust-tight after the step of filling with the inactivated comminuted material.

Ascend Elements EX1002 Page 188

Duesenfeld Ex. 2014-188

REMARKS

Claims 1 and 7 are amended. New claim 22 is added. Applicant respectfully requests reconsideration of the claims above in view of the remarks presented below.

35 U.S.C. 103

Claims 1, 3, 5, 7, 12, 13, 16, and 17 are rejected under 35 U.S.C. 103 as being unpatentable over Stevens (US 8,210,456) in view of Kellermann (DE 4424825) and Zhang (CN 102496752). This rejection is respectfully traversed. While some of the following remarks may refer only to claim 1 for convenience of discussion, the remarks are equally made with respect to independent claim 7 based on similar claim recitations and to the dependent claims which inherit the same limitations.

Stevens describes a method of recycling sealed batteries involving heating as well as drawing air from a pre-shredder, shredder, oven conveyer, and a sieve using a vacuum source (see, e.g., column 2 lines 54-60).

Kellermann discloses inactivating batteries by deep freezing and then shredding them in a frozen state (cf. figure 1). Freezing requires a lot of energy. The shredded material undergoes a floating procedure (Flotation 19) that removes the organic compounds from the flotation liquid.

The examiner's rejection is premised on a supposed suitability of "replac[ing] the inactivation and drying process of Stevens with the process of Zhang" (Action, paragraph bridging pgs. 6-7). Respectfully, one of ordinary skill in the art would not attempt or be able to combine the processes of these references.

Zhang is narrowly tailored to "recycling the used lithium ion battery electrolyte" (see EPO translation posted to PAIR by the examiner, first sentence of the Description). More specifically, Zhang is all about recovering "lithium hexafluorophosphate". According to Zhang, "lithium ion battery electrolyte is mainly a solution formed by dissolving lithium hexafluorophosphate in various organic solvents" (see EPO translation, pg. 1 final paragraph). Zhang is relied upon specifically for rejecting claim 1's recitation of "inactivating the comminuted material… wherein the inactivating step is performed by drying the comminuted material, and wherein the drying occurs at a maximum pressure of 300 hPa." The claim language expressly requires "**material**" which is "**comminuted**". The inactivation/drying is applied to

Ascend Elements EX1002 Page 189

Duesenfeld Ex. 2014-189

material, more specifically **"comminuted material."** These words are consequential not least because they require **solid pieces of battery** to be involved in the claimed process. **Zhang does not apply heating inactivation to solid materials; Zhang's inactivation/drying process is applied to a *liquid* solution, not solids**.

The translation of Zhang makes clear that both the heating and vacuum application cited by the examiner are employed specifically for "**distillation**" of electrolyte (see in particular the final paragraph of page 2 of the EPO translation). One of ordinary skill in the art would recognize that "distillation" is a process performed on liquid solutions, not on solid materials. In the context of Zhang's disclosure, this distinction becomes even more apparent. It is clear Zhang is applying the heating and vacuum application only to the "electrolyte" already removed from the battery, not to any solid pieces of battery.

It would not be obvious to one of ordinary skill in the art to encounter a distillation process as in Zhang and assume the distillation's heat and vacuum settings would be directly applicable to comminuted (pulverized) solid battery material. Such a dramatic shift in precisely what is being heated and to what a vacuum is being applied (a liquid solution versus pulverized solid pieces of battery) would not be obvious to try either.

Notwithstanding the previous distinctions, Zhang also leads one of ordinary skill in the art away from the *order* of steps recited in claim 1. Zhang discloses a step of "crushing," but it is literally the very last step of Zhang's procedure. In Zhang's Figure 1 flowchart, "crushing" is block 11, following only by "finished product" 12. "Distillation" on the other hand, which involves the heating and vacuum application cited by the examiner, is performed much earlier in Zhang's process, namely at block 3. This order of steps in Zhang, namely distilling with heat first and crushing afterwards, leads one of ordinary skill directly away from the order required by amended claim 1 herein. Claim 1 recites "inactivating the comminuted material…". Logically, to inactivate comminuted material, you must comminute either before or during the inactivation, but in no event can you wait to comminute until after the inactivation. While the previously recited claim language already impliedly asserted this sequence, amended claim 1 further specifies that "the inactivating step is performed during or after the comminuting step" to emphasize it further.

Ascend Elements EX1002 Page 190

Duesenfeld Ex. 2014-190

Even without the feature in claim 1 described in the preceding paragraph, the three documents cited by the examiner to reject claims 1 and 7 have incompatible teachings:

Stevens, Zhang, and Kellerman are three different approaches that are incompatible with one another. Zhang teaches to be careful with the battery when removing the electrolyte ("open the battery and carefully remove the electrolyte", see page 2 of the EPO translation; "carefully take out the electrolyte and put it into the tank" see page 4 of the EPO translation). This careful opening and removal is obviously an opposite strategy to that of Stevens and Kellerman, in which the batteries are shredded first.

Kellerman's invention is to use low temperatures to inactivate the electrolyte and to shred the frozen battery (claim 1 of Kellerman). If the skilled person wanted to combine the teachings of Kellerman and Zhang, he/she would have to ignore this significant and central feature of Kellerman's invention. However, ignoring specifically this feature of Kellerman is only justified by impermissible hindsight bias.

Stevens' invention is to shred the batteries and to heat them afterwards so that the volatile component evaporate (col. 5, lines 9-15). Heating the shredded feedstock is an integral part of Stevens' invention, as it is in all claims. To pursue the combination proposed in the Action, the skilled person would have to go against this feature of Stevens' disclosure.

Zhang, as already discussed, is focused on removing liquid electrolyte from "opened" but uncrushed/unpulverized batteries and then performed a distillation procedure on the liquid solution. There is no clear way to integrate such a distillation procedure into either approach of Stevens and Kellerman.

In sum, the differences in approach to recycling batteries is simply too disparate and varied among the three references to suggest their combination to one of ordinary skill in the art. Even were a combination attempted, which features to keep and which to eliminate from each reference is not self-apparent. To do so and arrive at the claimed subject matter must require hindsight bias based on knowledge of the claimed invention herein, which is not permitted under 35 U.S.C. 103.

New claim 22 recites an additional step of "sealing the transport container so as to be dust-tight after the step of filling with the inactivated comminuted material" (see, e.g., page 12 of the specification). None of the cited art describes this feature. Zhang, Kellerman, and Stevens all describe open systems. However, it is one aspect of the claimed invention to inactivate the

Ascend Elements EX1002 Page 191

Duesenfeld Ex. 2014-191

shredded batteries and to transport them to a central recycling facility. This requires closed transport containers.

Claims 2, 6, and 8 are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Sloop (US 2003/0186110). This rejection is respectfully traversed.

Sloop uses yet another method to remove the electrolyte from batteries, i.e. using supercritical CO2. Supercritical CO2 requires a pressure of more than 73,75 bar (1060 psi). Thus Sloop's teaching contradicts claim 1's recitation of "inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa". Sloop thus cannot cure the deficiencies of Stevens, Zhang, and Kellermann discussed above. As claims 2, 6, and 8 all depend from claim 1, they are new and nonobvious over any combination of the cited references for at least the same reasons as claim 1.

Claim 9 is rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Meador (US 5,632,863). This rejection is respectfully traversed.

Meador discloses a battery reclamation system and method which centers around pyrolysis, in particular decomposition temperatures between 350° and 650° F (see, e.g., Abstract). "Drying" and "drying device" as described in the disclosure at, e.g., page 4 of the specification, is distinguished from Meador's furnace 17. Accordingly, Meador fails to even have a "drying device" much less a "comminution unit and…drying device [that] are arranged in a container" as recited in claim 9.

Claims 14 and 18 have been rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Hanisch et al., herein "Hanisch," (WO 2013/023640), to which the examiner references using corresponding U.S. Patent No. 9,780,419. This rejection is respectfully traversed.

Like Meador, Hanisch involves heating to extremely high decomposition temperatures. In Hanisch's case, the desired temperatures are 350 to 800°C. A combination of Stevens, Kellermann, Zhang, and Hanisch fails to arrive at the subject matter of the claims as amended herein. Claims 14 and 18 are new and nonobvious for at least the same reasons discussed above in respect of the independent claims from which they depend.

Ascend Elements EX1002 Page 192

Duesenfeld Ex. 2014-192

Claims 15 and 19 have been rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Sloop (US 2016/0043450), herein "Sloop2". This rejection is respectfully traversed. Sloop2 describes a method of battery recycling which does not include or suggest ""inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa" as recited in claim 1 or "a vacuum installation connected to the drying device and configured to generate a vacuum in the drying device" as recited in claim 7. Claims 15 and 19, which depend from these independent claims, are new and nonobvious on at least this basis.

Allowable Subject Matter

Applicant acknowledges with appreciation indication of claims 4, 11, and 20 as being allowed.

Conclusion

In view of the foregoing, it is respectfully requested that the application be reconsidered; that claims 1-3, 5-9, 12-19, and 21-22 be allowed in addition to claims 4, 11, and 20; and that the application be passed to issue.

Although the present communication may include alterations to the application or claims, or characterizations of claim scope or referenced art, Applicant is not conceding in this application that previously pending claims are not patentable over the cited references. Rather, any alterations or characterizations are being made to facilitate expeditious prosecution of the application. Applicant reserves the right to pursue at a later time any previously pending or other broader or narrower claims that capture any subject matter supported by the present disclosure, including subject matter found to be specifically disclaimed herein or by any prior prosecution.

Accordingly, reviewers of this or any parent, child or related prosecution history shall not reasonably infer that Applicant has made any disclaimers or disavowals of any subject matter supported by the present application.

Should the Examiner find the application to be other than in condition for allowance, the Examiner is requested to contact the undersigned at the local telephone number listed below to discuss any other changes deemed necessary in a telephonic, WebEx, or personal interview.

A provisional petition is hereby made for any extension of time necessary for the

Ascend Elements EX1002 Page 193

Duesenfeld Ex. 2014-193

continued pendency during the life of this application. Please charge any fees for such provisional petition and any deficiencies in fees and credit any overpayment of fees to Attorney's Deposit Account No. 50-2041.

Respectfully submitted,

/T. Bryce Miller/

T. Bryce Miller
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Ascend Elements EX1002 Page 194

Duesenfeld Ex. 2014-194

Electronic Patent Application Fee Transmittal

Application Number:	15569484
Filing Date:	26-Oct-2017
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Timothy Bryce Miller
Attorney Docket Number:	03101160US

Filed as Large Entity

Filing Fees for U.S. National Stage under 35 USC 371

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Miscellaneous:				
RCE- 1ST REQUEST	1801	1	1300	1300
		Total in USD ($)		**1300**

Electronic Acknowledgement Receipt	
EFS ID:	39631751
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Timothy Bryce Miller
Filer Authorized By:	
Attorney Docket Number:	03101160US
Receipt Date:	04-JUN-2020
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Time Stamp:	17:53:48
Application Type:	U.S. National Stage under 35 USC 371

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Payment Type	CARD
Payment was successfully received in RAM	$1300
RAM confirmation Number	E202064H55291437
Deposit Account	
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The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Request for Continued Examination (RCE)	2020-06-04_03101160US_RCE.pdf	1349895 ee8d7ab29379e6ce5b76c80693a1d9ec0e2e4498	no	3
Warnings:					
Information:					
2		2020-06-04_03101160US_res1114.pdf	60241 e3b4f54551ca8a08616477d739a2502f856647ca	yes	12
	Multipart Description/PDF files in .zip description				
	Document Description		**Start**	**End**	
	Amendment Submitted/Entered with Filing of CPA/RCE		1	1	
	Claims		2	6	
	Applicant Arguments/Remarks Made in an Amendment		7	12	
Warnings:					
Information:					
3	Fee Worksheet (SB06)	fee-info.pdf	30478 8314a5e8359b7e97340b7ea1108144d313725b9f	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	1440614		

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PTO/SB/06 (09-11)
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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 15/569,484	Filing Date 10/26/2017	☐ To be Mailed

ENTITY: ☑ LARGE ☐ SMALL ☐ MICRO

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA		RATE ($)	FEE ($)
☐ BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A		N/A	
☐ SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A		N/A	
☐ EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A		N/A	
TOTAL CLAIMS (37 CFR 1.16(i))	minus 20 =	*		x $80 =	
INDEPENDENT CLAIMS (37 CFR 1.16(h))	minus 3 =	*		x $420 =	
☐ APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).				
☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))					
* If the difference in column 1 is less than zero, enter "0" in column 2.				TOTAL	

APPLICATION AS AMENDED - PART II

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT	06/04/2020	CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	* 21	Minus	** 20	= 1		x $100 =	100
	Independent (37 CFR 1.16(h))	* 5	Minus	*** 5	= 0		x $460 =	0
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	100

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	*	Minus	**	=		x $0 =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=		x $0 =	
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.	LIE
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".	/POLIN ANG/
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".	
The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.	

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/569,484	10/26/2017	Christian HANISCH	03101160US	9373

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EXAMINER	
SWIATOCHA, GREGORY D.	

ART UNIT	PAPER NUMBER
3725	

MAIL DATE	DELIVERY MODE
03/05/2020	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Office Action Summary	Application No. 15/569,484	Applicant(s) HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	Art Unit 3725	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☑ Responsive to communication(s) filed on 10 January 2020.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☑ This action is **FINAL.** 2b) ☐ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5) ☑ Claim(s) 1-9 and 11-21 is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6) ☑ Claim(s) 4,11 and 20 is/are allowed.

7) ☑ Claim(s) 1-3,5-9,12-19 and 21 is/are rejected.

8) ☐ Claim(s) _____ is/are objected to.

9) ☐ Claim(s) _____ are subject to restriction and/or election requirement

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov.**

Application Papers

10)☐ The specification is objected to by the Examiner.

11)☑ The drawing(s) filed on 10 January 2020 is/are: a)☐ accepted or b)☑ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☑ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☑ All b)☐ Some** c)☐ None of the:

1.☑ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☑ Notice of References Cited (PTO-892)

2) ☑ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date _____.

4) ☐ Other: _____.

DETAILED ACTION

Notice of Pre-AIA or AIA Status

1) The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

2) This application currently names joint inventors. In considering patentability of the claims the examiner presumes that the subject matter of the various claims was commonly owned as of the effective filing date of the claimed invention(s) absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and effective filing dates of each claim that was not commonly owned as of the effective filing date of the later invention in order for the examiner to consider the applicability of 35 U.S.C. 102(b)(2)(C) for any potential 35 U.S.C. 102(a)(2) prior art against the later invention.

Response to Arguments

3) Applicant's response filed 10 January 2020 has overcome the objections to the drawings, specification, and claims, and rejections under 35 U.S.C. 112. Accordingly, the objections to the drawings, specification, and claims, and rejections under 35 U.S.C. 112 have been withdrawn.

4) Applicant's Remarks filed 10 January 2020 regarding the interpretation of the terms, "an inactivation device," "a drying device," "a filling device," "a second filling device," and "separation device" have been considered and are persuasive.

5) Applicant's arguments with respect to claims 1 and 7 have been considered but are moot because the arguments do not apply to the current combination of references being used in the current rejection. Applicant's amendment necessitated the new grounds of rejection of this Office Action.

Information Disclosure Statement

6) The information disclosure statement (IDS) is in compliance with the provisions of 37 CFR 1.97. Accordingly, the information disclosure statement is being considered by the examiner.

Claim Interpretation

7) The following is a quotation of 35 U.S.C. 112(f):

> (f) Element in Claim for a Combination. – An element in a claim for a combination may be expressed as a means or step for performing a specified function without the recital of structure, material, or acts in support thereof, and such claim shall be construed to cover the corresponding structure, material, or acts described in the specification and equivalents thereof.

The following is a quotation of pre-AIA 35 U.S.C. 112, sixth paragraph:

> An element in a claim for a combination may be expressed as a means or step for performing a specified function without the recital of structure, material, or acts in support thereof, and such claim shall be construed to cover the corresponding structure, material, or acts described in the specification and equivalents thereof.

8) The claims in this application are given their broadest reasonable interpretation using the plain meaning of the claim language in light of the specification as it would be understood by one of ordinary skill in the art. The broadest reasonable interpretation of a claim element (also commonly referred to as a claim limitation) is limited by the description in the specification when 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is invoked.

As explained in MPEP § 2181, subsection I, claim limitations that meet the following three-prong test will be interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph:

(A) the claim limitation uses the term "means" or "step" or a term used as a substitute for "means" that is a generic placeholder (also called a nonce term or a non-structural term having no specific structural meaning) for performing the claimed function;

(B) the term "means" or "step" or the generic placeholder is modified by functional language, typically, but not always linked by the transition word "for" (e.g., "means for") or another linking word or phrase, such as "configured to" or "so that"; and

(C) the term "means" or "step" or the generic placeholder is not modified by sufficient structure, material, or acts for performing the claimed function.

Use of the word "means" (or "step") in a claim with functional language creates a rebuttable presumption that the claim limitation is to be treated in accordance with 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph. The presumption that the claim limitation is interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is rebutted when the claim limitation recites sufficient structure, material, or acts to entirely perform the recited function.

Absence of the word "means" (or "step") in a claim creates a rebuttable presumption that the claim limitation is not to be treated in accordance with 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph. The presumption that the claim limitation is not interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is rebutted when the claim limitation recites function without reciting sufficient structure, material or acts to entirely perform the recited function.

Claim limitations in this application that use the word "means" (or "step") are being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, except as otherwise indicated in an Office action. Conversely, claim limitations in this application that do not use the word "means" (or "step") are not being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, except as otherwise indicated in an Office action.

9) This application includes one or more claim limitations that do not use the word "means," but are nonetheless being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, because the claim limitation(s) uses a generic placeholder that is coupled with functional language without reciting sufficient structure to perform the recited function and the generic placeholder is not preceded by a structural modifier. Such claim limitation(s) is/are: **"a hard metal detachment device and/or light fraction detachment device" (Claim 12, line 3).**

Because this/these claim limitation(s) is/are being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, it/they is/are being interpreted to cover the corresponding structure

described in the specification as performing the claimed function, and equivalents thereof. **The following appears to be the corresponding structure as disclosed:**

Hard metal detachment device - magnet or other separator

Light fraction detachment device - zigzag separator or air separator

10) If applicant does not intend to have this/these limitation(s) interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, applicant may: (1) amend the claim limitation(s) to avoid it/them being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph (e.g., by reciting sufficient structure to perform the claimed function); or (2) present a sufficient showing that the claim limitation(s) recite(s) sufficient structure to perform the claimed function so as to avoid it/them being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph.

Claim Rejections - 35 USC § 103

11) The following is a quotation of 35 U.S.C. 103 which forms the basis for all obviousness rejections set forth in this Office action:

> A patent for a claimed invention may not be obtained, notwithstanding that the claimed invention is not identically disclosed as set forth in section 102, if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a person having ordinary skill in the art to which the claimed invention pertains. Patentability shall not be negated by the manner in which the invention was made.

12) The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103 are summarized as follows:

1. Determining the scope and contents of the prior art.

2. Ascertaining the differences between the prior art and the claims at issue.

3. Resolving the level of ordinary skill in the pertinent art.

4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

13) **Claims 1, 3, 5, 7, 12, 13, 16, and 17 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens (U.S. 8,210,456) in view of Kellermann (DE 4424825) and Zhang (CN 102496752).**

14) Regarding Claim 1, Stevens discloses a method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained (Lithium batteries are shredded: Col 5, L16-17),

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained (Oven conveyor Fig. 3, #20 & Dried material: Col 1, L37 & VOCs: Col 5, L15);

wherein the inactivating step is performed by drying the comminuted material (Dried material: Col 1, L37). Stevens is not explicit to filling a transport container with the inactivated comminuted material. **Kellermann** teaches a method for the disposal of lithium batteries including filling a transport container with the inactivated comminuted material (The fractional fractions of the crushed batteries are collected in collection vessels: Page 2) in order to further process the batteries. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to collect the inactivated comminuted material of Stevens in a transport container, as taught by Kellermann, in order to further process the batteries. While Stevens discloses an inactivation step and drying step using an oven conveyor, Stevens does not disclose an inactivation and drying step wherein the drying occurs at a maximum pressure of 300 hPa.

However, **Zhang** teaches an alternative method of inactivating material in lithium ion batteries including a drying process, where the drying occurs at a maximum pressure of 300 hPa (Dry room & Vacuum degree 0 to 20 kPa: Page 2) in order to recycle the electrolyte, protect the electrolyte from hydrolysis reactions and oxidation reactions, and prevent decomposition side reactions of lithium hexafluorophosphate. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the inactivation and drying process of

Stevens with the process of Zhang, as doing so would protect the electrolyte from hydrolysis reactions and oxidation reactions and prevent the decomposition of the lithium hexafluorophosphate since Lithium hexafluorophosphate is a heat-sensitive material, which is easy to decompose at high temperatures, and it is very active and easily reacts with air and moisture (Zhang: Page 2).

15) Regarding Claim 3, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Zhang further teaches wherein the drying occurs under vacuum Vacuum: Page 2).

16) Regarding Claim 5, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses one or more of detaching hard parts (magnetic component separated by the magnetic wheel: Col 5, L35), separating active material from a carrier, such that an active material fraction and a carrier fraction are obtained (The specific gravity separator separates out nonferrous fluff encapsulated with this magnetic component and also separates the steel, magnetic alloys, aluminum, and copper from each other: Col 5, L38-41). Kellermann further teaches removing the comminuted material from the transport container (The fractional fractions of the crushed batteries are collected in collection vessels and sent to a pool of a flotation station where a first separation treatment of fractions takes place: Page 2. Examiner notes that the comminuted material would be removed from the collection vessels in order to be put into the separation pool) and separately packing the active material fraction and carrier fraction in containers (Dried parts are fed in a container: Page 3 & The iron component are deposited: Page 3 & The non-ferrous components are deposited: Page 3).

17) Regarding Claim 7, Stevens discloses (a) a comminution unit configured to comminute the batteries such that comminuted material is obtained (Shredder: Fig. 3, #14);

(b) an inactivation device comprising a drying device configured to inactivate the comminuted material (Oven conveyor Fig. 3, #20 & Dried material: Col 1, L37 & VOCs: Col 5, L15). Stevens is not explicit to a filling device configured to fill a transport container with the inactivated comminuted material.

Kellermann teaches a device for the disposal of lithium batteries including a filling device configured to fill a transport container with the inactivated comminuted material (Collection vessels: Fig. 1, #18. Examiner notes that the filling vessels are located along the process path or within the shredder device and therefor would include a filling device or frame where the collection vessels would be filled) in order to further process the batteries. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to collect the inactivated comminuted material of Stevens in a transport container with a filling device, as taught by Kellermann, in order to further process the batteries. While Stevens discloses an inactivation device and drying device using an oven conveyor, Stevens does not disclose a vacuum installation connected to the drying device configured to generate a vacuum in the drying device.

However, **Zhang** teaches an alternative method of inactivating material in lithium ion batteries including a drying device (Dry room: Page 2) and a vaccum installation connected to the drying device configured to generate a vacuum in the drying device (Dry room & Vacuum degree 0 to 20 kPa: Page 2) in order to recycle the electrolyte, protect the electrolyte from hydrolysis reactions and oxidation reactions, and prevent decomposition side reactions of lithium hexafluorophosphate. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the inactivation and drying process of Stevens with the process of Zhang, as doing so would protect the electrolyte from hydrolysis reactions and oxidation reactions and prevent the decomposition of the lithium hexafluorophosphate since Lithium hexafluorophosphate is a heat-sensitive material, which is easy to decompose at high temperatures, and it is very active and easily reacts with air and moisture (Zhang: Page 2).

18) Regarding Claim 12, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses a hard metal detachment device (Magnetic wheel: Fig. 3, #52) and/or a light fraction detachment device,
a separation device configured to separate active material from a carrier such that an active material fraction and a carrier fraction occur (Specific gravity separator: Fig. 3, #68). Kellermann further teaches a second filling device configured to separately fill the active material fraction and the carrier fraction in respective containers (Dried parts are fed in a container: Page 3 & The iron component are deposited: Page 3 & The non-ferrous components are deposited: Page 3. Examiner notes that depositing of the material would occur with a filling device.).

19) Regarding Claim 13, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Steven further discloses wherein the carrier fraction are aluminum and copper foils (Aluminum and copper: Col 5, L36-37).

20) Regarding Claim 16, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses wherein the batteries are lithium batteries (Lithium battery: Col 5, L5).

21) Regarding Claim 17, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses wherein the batteries are lithium batteries (Lithium battery: Col 5, L5).

22) Regarding Claim 21, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Zhang further teaches wherein drying occurs at a temperature of less than 80°C (20 and 120 degrees: Page 2. Examiner notes that the range of 20-120 degrees has substantial overlap with less than 80 degrees. Further, the range 20-120 degrees achieves the same function of less than 80°C. Examiner notes that Page 4 of Applicant's specification discloses that the advantage of low temperatures is that the formation of hydrogen fluoride is hindered. Page 2 of Zhnag

discloses that high vacuum and reduced pressure distillation process which takes place between 20 and 120 degrees prevent the decomposition side reactions of lithium hexafluorophosphate, which would break down into hydrogen fluoride).

23) **Claims 2, 6, and 8 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Sloop (U.S. 2003/0186110).**

24) Regarding Claim 2, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried material (Col 5, L19), but is not explicit to wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

Sloop teaches the recycling of lithium batteries including wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible (Dry, non-flammable, minimally reactive form: Par. 0042) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens, or Stevens in view of Kellermann and Zhang, would be such that an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

25) Regarding Claim 6, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried material (Col 5, L19), but is not explicit to wherein the drying of the comminuted material is only completed if one or more of the following conditions is met: no flammable or explosive gas mixture is formed above the comminuted material in the transport container, and the comminuted material is so dry that no flammable or explosive gas mixture is able to emerge in the transport container.

Sloop teaches the recycling of lithium batteries including wherein the drying of the comminuted material is only completed if one or more of the following conditions is met: no flammable or explosive gas mixture is formable above the comminuted material in the transport container, and the comminuted material is so dry that no flammable or explosive gas mixture is able to emerge in the transport container (Dry, non-flammable, minimally reactive form: Par. 0042. Examiner notes that if the dried material in non-flammable and minimally reactive, no flammable or explosive gas would emerge) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens, or Stevens in view of Kellermann and Zhang, would be such that no flammable or explosive gas mixture can form above the comminuted material in the transport container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

26) Regarding Claim 8, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried material (Col 5, L19), but is not explicit to wherein the drying device is configured to dry the comminuted material until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

Sloop teaches the recycling of lithium batteries including wherein the drying device is configured to dry the comminuted material until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible (After the electrolyte is removed . . . Dry, non-flammable, minimally reactive form: Par. 0042) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens, or Stevens in view of Kellermann and Zhang,

would be such that an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

27) **Claim 9 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Meador (U.S. 5,632,863).**

28) Regarding Claim 9, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. The combination of Steven, Kellerman, and Zhang do not teach wherein the comminution device and the drying device are arranged in a container.

Meador teaches a battery recycling process wherein the comminution device (27) and the drying device (17) are arranged in a container (See frame of device & Col 4, L36-53) in order to prevent oxygen from entering the device which could cause an explosion hazard. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to arrange the comminution device and drying device of Stevens, or Stevens in view of Kellermann and Zhang, in a container, as taught by Meador, in order to prevent oxygen from entering the device (Meador: Col 4, L52).

29) **Claims 14 and 18 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhang, and Hanisch et al., hereinafter Hanisch, (WO 2013/023640). Note that citations for Hanisch are taken from corresponding U.S. Patent No. 9,780,419.**

30) Regarding Claim 14, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. The combination of Stevens, Kellerman, and Zhang, do not teach wherein the separation device is an air jet sieving device.

Hanisch teaches a separation device for an active material and a carrier wherein the separation device is an air jet sieving device (Air jet sieve: Fig. 1, #50) in order to separate materials with a high degree of purity. Therefore, it would have been obvious to one of ordinary skill in the art before the effective

filing date of the claimed invention to replace the specific gravity separator of Stevens with the air jet sieve of Hanisch in order to separate materials with a high degree of purity (Hanisch: Col 2, L6).

31) Regarding Claim 18, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. The combination of Stevens, Kellerman, and Zhnag do not teach wherein separation of the active material from the carrier is performed by air jet sieving.

Hanisch teaches a separation device for an active material and a carrier wherein separation of the active material from the carrier is performed by air jet sieving (Air jet sieve: Fig. 1, #50) in order to separate materials with a high degree of purity. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the air jet sieve of Hanisch in order to separate materials with a high degree of purity (Hanisch: Col 2, L6).

32) **Claims 15 and 19 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann, Zhnag, and Sloop (U.S. 2016/0043450).**

33) Regarding Claim 15, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein the separation device is a second comminution device.

Sloop teaches a device for the recycling of lithium batteries including a comminuting stage (14), drying stage (20), and separating stage (22, 24) wherein the separation device is a second comminution device (Grinding step: Par. 0019) in order to improve yield in subsequent sieving. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the grinder of Sloop in order to improve yield in subsequent sieving (Sloop: Par. 0019).

34) Regarding Claim 19, the combination of Stevens, Kellermann, and Zhang teach all elements of the claimed invention as stated above. The combination of Stevens, Kellerman, and Zhang do not teach

wherein separation of the active material from the carrier is performed using a second comminution stage.

Sloop teaches a device for the recycling of lithium batteries including a comminuting stage (14), drying stage (20), and separating stage (22, 24) wherein separation of the active material from the carrier (24) is performed using a second comminution stage (Grinding step: Par. 0019) in order to improve yield in subsequent sieving. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the grinder of Sloop in order to improve yield in subsequent sieving (Sloop: Par. 0019).

Allowable Subject Matter

35) Claims 4, 11, and 20 are allowed.

36) The following is a statement of reasons for the indication of allowable subject matter:

37) Regarding Claims 4, 11, and 20, it is the opinion of the Examiner that the art of record neither anticipates nor renders obvious wherein "*a pump liquid of the jet pump has at least one of a pH value of at least 8, and contains a substance that precipitates fluoride" or "wherein the pump liquid is conducted within a circuit and the pump liquid contains a substance that reacts with hydrogen fluoride*" in combination with the rest of the claimed limitations set forth in the dependent claim. Searching by the Examiner yielded prior art as cited below:

38) Stevens discloses the removal of an electrolyte including lithium from the recycled battery using a vacuum system, scrubber, and an atomizer using a 50% concentration of sodium hydroxide in water, and this discloses a liquid with a pH of at least 9. Sodium hydroxide is also known to react with hydrogen fluoride to precipitate fluoride. However, the electrolyte disclosed by Stevens is lithium perchlorate in propylene carbonate and dimethoxyethane. While, **Tanii et al. (U.S. 6,524,737)** discloses the recycling of lithium batteries containing lithium phosphohexafluoride as the electrolyte, and thus the electrolyte of Stevens could be replaced with lithium phosphohexafluoride, the fluoride compounds

are treated in a wastewater tank instead of with a jet pump. Further, while the use of jet pumps containing alkaline liquids such as sodium hydroxide is known from **Eckardt et al. (U.S. 2014/0003568)** such a use is not disclosed for the recycling of a lithium electrolyte battery.

39) Therefore, Stevens, Tanii, and Eckardt alone, or in combination with any other prior art, do not anticipate or render obvious the claimed invention.

Conclusion

40) Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

41) Any inquiry concerning this communication or earlier communications from the examiner should be directed to GREGORY D. SWIATOCHA whose telephone number is (571)272-7022. The examiner can normally be reached on Monday-Friday (9-5).

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Shelley Self can be reached on (571) 272-4524. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3725

Notice of References Cited	Application/Control No. 15/569,484	Applicant(s)/Patent Under Reexamination HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	Art Unit 3725	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	CPC Classification	US Classification
	A					
	B					
	C					
	D					
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FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	CPC Classification
	N					
	O					
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	Machine translation of CN102496752, 4 Pages. (Year: 2012)
	V	
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

Index of Claims	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3725

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

CLAIMS

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIM		DATE								
Final	Original	09/05/2019	03/04/2020							
	1	✓	✓							
	2	✓	✓							
	3	✓	✓							
	4	O	=							
	5	✓	✓							
	6	✓	✓							
	7	✓	✓							
	8	✓	✓							
	9	✓	✓							
	10	-	-							
	11	O	=							
	12	✓	✓							
	13	✓	✓							
	14	✓	✓							
	15	✓	✓							
	16	✓	✓							
	17	✓	✓							
	18	✓	✓							
	19	✓	✓							
	20	O	=							
	21		✓							

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3725

CPC - Searched*		
Symbol	**Date**	**Examiner**
Y02W30/84	09/04/2019	G.S.
Y02P10/212	09/04/2019	G.S.
B02C23/08, 10	09/04/2019	G.S.
B09B3/008, 0058	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102	09/04/2019	G.S.

CPC Combination Sets - Searched*		
Symbol	**Date**	**Examiner**

US Classification - Searched*			
Class	**Subclass**	**Date**	**Examiner**

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3725	

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3725

Search Notes		
Search Notes	**Date**	**Examiner**
EAST Inventor search, Assignee search, CPC search, Text search, Combination class../text search	09/04/2019	G.S.
Espacenet search	09/04/2019	G.S.
IP.com search	09/04/2019	G.S.
NPL Search	09/04/2019	G.S.
Y02W30/84 (See search history printout)	09/04/2019	G.S.
Y02P10/212 (See search history printout)	09/04/2019	G.S.
B02C23/08, 10 (See search history printout)	09/04/2019	G.S.
B09B3/008, 0058 (See search history printout)	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102 (See search history printout)	09/04/2019	G.S.
Updated Search	03/04/2020	G.S.

Interference Search			
US Class/CPC Symbol	**US Subclass/CPC Group**	**Date**	**Examiner**

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3725

Duesenfeld Ex. 2014-221

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (02-18)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

U.S.PATENTS Remove

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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20140290438	A1	2014-10-02	C. HANISCH; W. HASELRIEDER; A. KWADE	

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	1	2010/102337	WO	A1	2010-09-16	W. STEVENS		
	2	103959553	CN	A	2014-07-30	UNIV. BRAUNSCHWEIG TECH	Equivalent to WO2010/102337	
	3	102496752	CN	A	2012-06-13	CHINA NAT OFFSHORE OIL CORP ET AL.		X

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ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.D.S./

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

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Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
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EXAMINER SIGNATURE

Examiner Signature	/GREGORY D SWIATOCHA/	Date Considered	03/04/2020

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

☒ That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2019-09-12
Name/Print	Michael E. Whitham	Registration Number	32635

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

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Ascend Elements EX1002 Page 225

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Duesenfeld Ex. 2014-225

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L3	1	"8210456".pn.	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:39
L4	214	"8210456" "1587623" "3393876" "3456886" "3493183" "3777994" "3892563" "4026477" "4384683" "4775107" "5377920" "5575907" "5632863" "5707015" "9780419" "20050241943"	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:48
L5	2	"20140290438"	US-PGPUB; USPAT; USOCR	OR	ON	2020/03/04 20:49
L7	24	(US-20190260101-$ or US-20180301769-$ or US-20180205122-$ or US-20030186110-$ or US-20070134546-$ or US-20160251740-$ or US-20160043450-$ or US-20140290438-$ or US-20100230518-$ or US-20040113588-$ or US-20030180604-$ or US-20140003568-$).did. or (US-9780419-$ or US-6524737-$ or US-5632863-$ or US-8210456-$).did. or (DE-4424825-$ or CA-2311792-$).did. or (WO-2010102377-$ or DE-102011110083-$ or WO-2013140039-$ or CN-102496752-$ or CN-101183740-$ or EP-1333522-$).did.	US-PGPUB; USPAT; FPRS; DERWENT	OR	ON	2020/03/04 20:53
L8	21	(l3 l4 l5 l7) AND (dry$3 SAME (vacuum pressure))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/03/04 20:53
S1	9	((("HANISCH") near3 ("Christian")) OR (("WESTPHAL") near3 ("Bastian")) OR (("HASELRIEDER") near3 ("Wolfgang")) OR (("SCHOENITZ") near3 ("Martin"))).INV.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2019/09/04 10:38
S3	3	duesenfeld.as.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 10:43
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S11	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 11:04
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			EPO; JPO; DERWENT; IBM_TDB			
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S16	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 12:57
S17	160	CA-2983768-$.did. US-20190260101-$.did. AU-2017347388-$.did. WO-2016174156-$.did. EP-3289627-$.did. CA-3040793-$.did. DE-102015207843-$.did. DE-102015207843-$.did. EP-3312922-$.did. EP-3312922-$.did. DE-102016120046-$.did. WO-2018073101-$.did. EP-3529841-$.did. CN-109845008-$.did. KR-20190084985-$.did. CN-107636875-$.did. WO-2019149698-$.did. DE-102013011470-$.did. DE-102018102026-$.did. EP-2446497-$.did. EP-3517641-$.did. WO-2010149611-$.did. EP-2446497-$.did. DE-102012024876-$.did. EP-2742559-$.did. WO-2013023640-$.did. DE-102013011470-$.did. EP-2742559-$.did. DE-102009027179-$.did. DE-102011110083-$.did. WO-2008074352-$.did. DE-4424825-$.did. DE-102011110083-$.did. DE-102015206153-$.did. WO-2019015892-$.did. TW-201728531-$.did. JP-H06338353-$.did. US-5612150-$.did. EP-673075-$.did. US-5679477-$.did. JP-2013229326-$.did. JP-3240814-$.did. DE-202013010308-$.did. CN-106684487-$.did. JP-3495707-$.did. DE-102011082187-$.did. EP-673075-$.did. CN-105144464-$.did. JP-3935594-$.did. US-3748186-$.did. US-8067107-$.did. US-20050244704-$.did. CN-106684487-$.did. WO-2018218358-$.did. EP-2741357-$.did. CN-105021024-$.did. CA-3043947-$.did. JP-6124001-$.did. JP-H11167936-$.did. DE-19924707-$.did. WO-2002023651-$.did. CA-2391865-$.did. EP-1269554-$.did. KR-20190078653-$.did. CN-108306050-$.did. DE-102017212266-$.did. WO-2005015668-$.did. EP-1652253-$.did. CA-2730336-$.did. JP-H06338352-$.did. EP-1056146-$.did. US-20040028585-$.did. EP-1269554-$.did. CN-105594056-$.did. US-6524737-$.did. KR-20150135304-$.did. US-7192564-$.did. DE-102015010955-$.did. CN-105594056-$.did. WO-2014155784-$.did. DE-102018100158-$.did. US-9937481-$.did. US-7198865-$.did. EP-1041659-$.did. CN-109777957-	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 13:05

		$.did. CN-107910610-$.did. EP-3063811-$.did. WO-1994007277-$.did. JP-3079849-$.did. TW-I543431-$.did. US-20150080630-$.did. CA-2311792-$.did. CN-109449525-$.did. DE-19518643-$.did. US-5352270-$.did. TW-201724628-$.did. DE-4118001-$.did. DE-202014007301-$.did. DE-202013006631-$.did. US-9450277-$.did. DE-202013006632-$.did. WO-2015063007-$.did. US-20170012328-$.did. AT-407753-$.did. JP-2016207463-$.did. RU-2201018-$.did.				
S18	156	US-20170170529-$.did. KR-100300829-$.did. JP-2016207464-$.did. US-9620828-$.did. WO-2003061056-$.did. US-6511639-$.did. DE-19532626-$.did. EP-1472756-$.did. DE-102013111853-$.did. US-20070292746-$.did. JP-2017084707-$.did. KR-19980064595-$.did. CN-1442928-$.did. EP-1333523-$.did. DE-102013105676-$.did. AU-2003205087-$.did. US-7858216-$.did. US-20180198115-$.did. EP-1472756-$.did. CA-2309773-$.did. EP-1333522-$.did. US-9843077-$.did. US-20170207504-$.did. WO-2017000157-$.did. KR-20070090851-$.did. US-20160049699-$.did. US-20030186110-$.did. EP-3063811-$.did. DE-10049097-$.did. DE-102012109032-$.did. JP-2001332237-$.did. EP-1333523-$.did. US-9912022-$.did. WO-2017073686-$.did. KR-20030039376-$.did. JP-6245412-$.did. DE-19547151-$.did. JP-4187804-$.did. JP-2017084706-$.did. US-20030180604-$.did. DE-10049097-$.did. WO-2019150403-$.did. JP-5050845-$.did. JP-2000223123-$.did. JP-H0785889-$.did. CN-104412426-$.did. US-20130122398-$.did. WO-2015046211-$.did. KR-100997349-$.did. US-7833646-$.did. CN-109478473-$.did. US-20040096746-$.did. US-20180198179-$.did. DE-102007012693-$.did. JP-2014222582-$.did. US-9728807-$.did. US-20150188137-$.did. EP-854530-$.did. CN-107910610-$.did. DE-102014226241-$.did. JP-2019008980-$.did. CN-1442928-$.did. JP-2012204131-$.did. US-6789757-$.did. US-20030038197-$.did. JP-2016152226-$.did. JP-5894688-$.did. WO-1998044580-$.did. EP-1330299-$.did. CN-107403930-$.did. US-5972531-$.did. EP-854530-$.did. DE-102013012250-$.did. US-8999009-$.did. DE-102010020738-$.did. KR-20160064104-$.did. US-6986967-$.did. WO-2002028500-$.did. US-6461766-$.did. JP-2014160575-$.did. JP-2015179666-$.did. EP-2894702-$.did. US-20130232772-$.did. JP-2013146680-$.did. EP-972313-$.did. US-7910242-$.did. CA-2285275-$.did. CA-2418527-$.did. CN-109193064-$.did. KR-20040071776-$.did. CA-2409218-$.did. EP-1408575-$.did. CN-103959553-$.did. TW-201418387-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 13:06
S19	172	JP-2016207465-$.did. TW-201318972-$.did. JP-5708121-$.did. US-6335122-$.did.	US-PGPUB; USPAT;	OR	ON	2019/09/04 13:06

		WO-2000051806-$.did. JP-3540085-$.did. JP-2018022670-$.did. JP-2016511916-$.did. DE-19842658-$.did. US-20080199768-$.did. CN-105144464-$.did. WO-2017073687-$.did. CN-104412425-$.did. KR-100838943-$.did. WO-2019060996-$.did. US-20180309126-$.did. US-20140290438-$.did. JP-4753220-$.did. CA-2225699-$.did. CN-103959553-$.did. JP-2016170878-$.did. CN-107591583-$.did. JP-5966457-$.did. US-20150180020-$.did. WO-2014120718-$.did. TW-201545976-$.did. EP-3370286-$.did. US-20060138056-$.did. DE-102015223136-$.did. KR-20150110797-$.did. WO-1998035396-$.did. WO-2001091210-$.did. EP-958626-$.did. CN-104412425-$.did. KR-20160143829-$.did. EP-1293003-$.did. EP-2951872-$.did. KR-20090020888-$.did. US-6444356-$.did. TW-I644859-$.did. CN-108281729-$.did. EP-3132485-$.did. DE-102016208407-$.did. WO-2018034155-$.did. CN-108808156-$.did. US-6503432-$.did. KR-101025415-$.did. EP-3191195-$.did. WO-2016038207-$.did. AU-2003205087-$.did. US-20170047586-$.did. EP-794587-$.did. CA-2280622-$.did. CA-2471015-$.did. WO-2015158557-$.did. JP-2016152225-$.did. US-9780419-$.did. EP-1408575-$.did. IN-201917003070-$.did. EP-622862-$.did. WO-2017057146-$.did. JP-5691636-$.did. KR-20070090850-$.did. WO-2014034859-$.did. EP-794587-$.did. EP-84381-$.did. JP-2016152224-$.did. DE-102013219604-$.did. CA-2199096-$.did. IN-198823-$.did. US-20010008723-$.did. JP-2012190768-$.did. JP-2004355977-$.did. JP-6315198-$.did. AU-701597-$.did. WO-2015129200-$.did. KR-20000019850-$.did. RO-106045-$.did. US-20030054245-$.did. JP-3371713-$.did. JP-6345116-$.did. JP-2015092465-$.did. US-20160268597-$.did. US-7666310-$.did. WO-2019158177-$.did. WO-2014034857-$.did. JP-2010086885-$.did. DE-102012000914-$.did. US-20050247914-$.did. JP-6079651-$.did. JP-H08279358-$.did. KR-20020025480-$.did. US-6329096-$.did. JP-5647377-$.did. US-20180323433-$.did. CN-101944585-$.did. WO-2015136722-$.did. JP-6311877-$.did. US-20020039686-$.did. WO-2006088021-$.did. EP-1168479-$.did. KR-100880525-$.did.	USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB			
S20	198	AU-2017347388-$.did. CA-3040793-$.did. US-20180301769-$.did. EP-3312922-$.did. CA-2983768-$.did. EP-3312922-$.did. DE-102016120046-$.did. WO-2018073101-$.did. EP-3529841-$.did. WO-2016174156-$.did. EP-3289627-$.did. DE-102015207843-$.did. DE-102015207843-$.did. CN-109845008-$.did. KR-20190084985-$.did. CN-107636875-$.did. WO-2019149698-$.did. EP-2446497-$.did. WO-2010149611-$.did. EP-2446497-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 13:26

		DE-102013011470-$.did. DE-102018102026-$.did. DE-102009027179-$.did. EP-3517641-$.did. DE-102013011470-$.did. EP-2742559-$.did. WO-2013023640-$.did. DE-102012024876-$.did. DE-4118001-$.did. EP-2742559-$.did. DE-102011110083-$.did. DE-4424825-$.did. DE-102011110083-$.did. DE-102015206153-$.did. AT-407753-$.did. IN-285315-$.did. US-20100162619-$.did. CA-2710719-$.did. AU-2007340905-$.did. US-9512376-$.did. DE-19547151-$.did. US-6789757-$.did. US-20030038197-$.did. CA-2391865-$.did. AU-2007340905-$.did. WO-2002023651-$.did. US-20170283725-$.did. EP-1269554-$.did. CA-2409218-$.did. WO-2019015892-$.did. EP-1269554-$.did. CA-2730336-$.did. US-20040028585-$.did. EP-2741357-$.did. CA-2710719-$.did. CA-3031475-$.did. TW-201728531-$.did. CH-678131-$.did. AU-2013201163-$.did. US-6461766-$.did. US-7192564-$.did. DE-102008058444-$.did. JP-H06338353-$.did. DE-19518643-$.did. JP-3240814-$.did. WO-2001091210-$.did. EP-1293003-$.did. EP-1215023-$.did. EP-1652253-$.did. WO-2005015668-$.did. WO-2008074352-$.did. EP-2376597-$.did. JP-3495707-$.did. WO-2008081408-$.did. EP-673075-$.did. WO-2018218358-$.did. CN-106684487-$.did. DE-102011082187-$.did. DE-202013010308-$.did. WO-2008081407-$.did. EP-2106435-$.did. CA-3043947-$.did. JP-H06338352-$.did. CN-106684487-$.did. WO-1994007277-$.did. WO-2008081409-$.did. WO-2008081410-$.did. US-5352270-$.did. EP-673075-$.did. EP-1333523-$.did. JP-3935594-$.did. WO-2008119525-$.did. US-5679477-$.did. US-5612150-$.did. JP-H11167936-$.did. US-20150080630-$.did. DE-10062947-$.did. DE-10045241-$.did. EP-3063811-$.did. US-9450277-$.did. JP-3079849-$.did. JP-2013229326-$.did. EP-1333522-$.did. US-20170012328-$.did. RO-106045-$.did. DE-10021383-$.did. EP-1333523-$.did. WO-2015063007-$.did. US-9620828-$.did. US-8067107-$.did. CN-105144464-$.did. US-6223449-$.did. US-20030180604-$.did. DE-102008055508-$.did. JP-6124001-$.did. DE-19809400-$.did. US-20050244704-$.did. DE-10228471-$.did. US-6524737-$.did. DE-19547151-$.did. US-7833646-$.did. US-20170207504-$.did.				
S21	5959	y02W30/84.cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:19
S22	1437	(y02p10/212.cpc. b02c21/$.cpc. (b02c23/08,10).cpc. b09b3/008.cpc. (b01d53/02,002).cpc. b01d11/00.cpc.	US-PGPUB; USPAT; USOCR;	OR	ON	2019/09/04 14:31

		b09b3/0058.cpc. b01d2253/102.cpc.) AND (battery batteries)	FPRS; EPO; JPO; DERWENT; IBM_TDB			
S23	3	wo-2010102377-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S24	3	de-102011110083-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S25	2	de-102012024876-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S26	2	wo-2010149611-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S27	1	kr-20060101683-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S28	0	wo-2106174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S29	2	wo-2016174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S30	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S31	2	cn-101394015-$.did.	US-PGPUB;	OR	ON	2019/09/04

			USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB			14:37
S32	3	de-102015207843-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S33	3	de-602005001937-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S34	3	cn-102496752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S35	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S38	352	S22 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S39	2006	S21 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S40	3	de-19954998-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 19:41
S41	2	de-29803442-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT;	OR	ON	2019/09/04 19:41

			IBM_TDB			
S42	1	(S38 S39) AND (liquid WITH gas WITH jet WITH (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:19
S44	484	(liquid WITH gas WITH jet WITH (ejector pump)) WITH vacuum	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:21
S45	8	(liquid WITH gas WITH jet WITH (ejector pump) WITH vacuum) SAME pH	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:21
S46	21	(jet ADJ (ejector pump)) WITH ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:26
S47	169	(S38 S39) AND ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:27
S48	3	WO-2013140039-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:31
S49	3	(S38 S39) AND (jet ADJ (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:39
S50	8	(jet ADJ (ejector pump)) WITH fluoride	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:41
S51	1	(jet ADJ (ejector pump)) WITH (fluoride fluorine) WITH (carbonate)	US-PGPUB; USPAT; USOCR; FPRS;	OR	ON	2019/09/04 20:42

			EPO; JPO; DERWENT; IBM_TDB			
S53	174	(S38 S39) AND (electrolyte WITH (remov$4 weight))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 21:02
S54	28	(S38 S39) AND (electrolyte WITH (remov$4 vaporiz$3) WITH (dri$2 dry$3))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 21:06
S55	5959	y02W30/84.cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34
S56	658	S55 and lithium and (dry$3 dri$2)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34
S61	11	(venturi adj pump) SAME (fluorine fluoride carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 10:54
S62	12	"9780419" "20050241943" "7713396"	US-PGPUB; USPAT; USOCR	OR	ON	2019/09/05 11:07
S63	3	wo-2010102377-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S64	3	de-102011110083-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S65	2	de-102012024876-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT;	OR	ON	2019/09/05 11:07

			IBM_TDB			
S66	2	wo-2010149611-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S67	1	kr-20060101683-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S68	0	wo-2106174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S69	2	wo-2016174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S70	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S71	2	cn-101394015-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S72	3	de-102015207843-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S73	3	de-602005001937-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S74	3	cn-102496752-$.did.	US-PGPUB; USPAT; USOCR; FPRS;	OR	ON	2019/09/05 11:07

			EPO; JPO; DERWENT; IBM_TDB			
S75	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
S76	27	S63 S64 S65 S66 S67 S68 S69 S70 S71 S72 S73 S74 S75	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:07
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S82	204	(vacuum WITH scrub$4) SAME (fluorine fluoride carbonate)	US-PGPUB; USPAT; USOCR;	OR	ON	2019/09/05 11:14

			FPRS; EPO; JPO; DERWENT; IBM_TDB			
S83	204	(vacuum WITH scrub$4) SAME (fluorine fluoride carbonate)S82 and (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:14
S84	9	S82 and (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:15
S85	111	(vacuum WITH scrub$4) SAME (fluorine fluoride)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:28
S86	4	(vacuum WITH scrub$4) SAME ((fluorine fluoride) AND carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 11:28
S87	2	wo-2013023640-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 16:16
S88	106	((sodium adj hydroxide) NaOH) WITH ((jet adj pump) ejector)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 13:48
S89	23	(US-20190260101-$ or US-20180301769-$ or US-20180205122-$ or US-20030186110-$ or US-20070134546-$ or US-20160251740-$ or US-20160043450-$ or US-20140290438-$ or US-20100230518-$ or US-20040113588-$ or US-20030180604-$ or US-20140003568-$).did. or (US-9780419-$ or US-6524737-$ or US-5632863-$).did. or (DE-4424825-$ or CA-2311792-$).did. or (WO-2010102377-$ or DE-102011110083-$ or WO-2013140039-$ or CN-102496752-$ or CN-101183740-$ or EP-1333522-$).did.	US-PGPUB; USPAT; FPRS; DERWENT	OR	ON	2019/09/07 13:50
S91	8	S89 and (fluor$3)	US-PGPUB; USPAT;	OR	ON	2019/09/07 13:50

			USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB			
S92	1	(carbonate WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:24
S93	0	(base WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:24
S94	0	((basic alkalin$3) WITH ((jet adj pump) ejector) SAME (fluorine fluoride)) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S95	18	((bas$2 alkalin$3) WITH ((jet adj pump) ejector) SAME (fluorine fluoride))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S96	3	(carbonate WITH ((jet adj pump) ejector) SAME (fluorine fluoride))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/07 20:25
S97	2	"20070134546"	US-PGPUB; USPAT; USOCR	OR	ON	2019/09/08 12:20
S99	2	wo-2013023640-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/08 14:28

3/4/2020 9:54:26 PM
C:\Users\gswiatocha\Documents\EAST\Workspaces\15569484.wsp

PTO/SB/06 (09-11)
Approved for use through 1/31/2014. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 15/569,484	Filing Date 10/26/2017	☐ To be Mailed

ENTITY: ☑ LARGE ☐ SMALL ☐ MICRO

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA		RATE ($)	FEE ($)
☐ BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A		N/A	
☐ SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A		N/A	
☐ EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A		N/A	
TOTAL CLAIMS (37 CFR 1.16(i))	minus 20 =	*		x $80 =	
INDEPENDENT CLAIMS (37 CFR 1.16(h))	minus 3 =	*		x $420 =	
☐ APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).				
☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))					
* If the difference in column 1 is less than zero, enter "0" in column 2.				TOTAL	

APPLICATION AS AMENDED - PART II

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT	01/10/2020	CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	* 21	Minus	** 20	= 1		x $100 =	100
	Independent (37 CFR 1.16(h))	* 5	Minus	*** 3	= 2		x $460 =	920
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	1020

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	*	Minus	**	=		x $0 =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=		x $0 =	
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.	LIE
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".	/ANGELONA D JONES/
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".	
The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.	

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of

HANISCH et al.

Application No. 15/569,484

Filed October 26, 2017

Confirmation No. 9373

Group Art Unit 3725

Examiner Swiatocha

For METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

PETITION FOR EXTENSION OF TIME AND

AMENDMENT UNDER 37 C.F.R. § 1.111

Sir:

Applicant hereby petitions for a one month extension of time. Commissioner is hereby authorized to charge Deposit Account No. 50-2041 for any deficiency in the petition fee required to gain entry and consideration of this amendment.

In response to the Office Action mailed September 10, 2019, Applicant respectfully requests entry of the following Amendment and reconsideration.

Amendments to the Specification: begin on page 2.

Amendments to the Abstract: begin on page 4.

Amendments to the Drawings: Replacement sheets for Figures 1-3 are attached hereto.

Amendments to the Claims: begin on page 5.

Remarks: begin on page 10.

Ascend Elements EX1002 Page 244

Duesenfeld Ex. 2014-244

Electronic Patent Application Fee Transmittal

Application Number:	15569484
Filing Date:	26-Oct-2017
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Timothy Bryce Miller
Attorney Docket Number:	03101160US

Filed as Large Entity

Filing Fees for U.S. National Stage under 35 USC 371

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
CLAIMS IN EXCESS OF 20	1615	1	100	100
INDEPENDENT CLAIMS IN EXCESS OF 3	1614	2	460	920
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Extension - 1 month with $0 paid	1251	1	200	200
Miscellaneous:				
		Total in USD ($)		**1220**

Electronic Acknowledgement Receipt	
EFS ID:	38261423
Application Number:	15569484
International Application Number:	
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
Filer:	Timothy Bryce Miller
Filer Authorized By:	
Attorney Docket Number:	03101160US
Receipt Date:	10-JAN-2020
Filing Date:	26-OCT-2017
Time Stamp:	12:23:55
Application Type:	U.S. National Stage under 35 USC 371

Payment information:

Submitted with Payment	yes
Payment Type	CARD
Payment was successfully received in RAM	$1220
RAM confirmation Number	E202010C24193352
Deposit Account	
Authorized User	
The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		2019-01-10_03101160US_res1111.pdf	168313 de6fdcb00a7931f651b89560c3b2518cf78eec87	yes	20

Multipart Description/PDF files in .zip description

Document Description	Start	End
Drawings-only black and white line drawings	18	20
Applicant Arguments/Remarks Made in an Amendment	10	17
Claims	5	9
Abstract	4	4
Specification	2	3
Amendment/Req. Reconsideration-After Non-Final Reject	1	1

Warnings:

Information:

2	Fee Worksheet (SB06)	fee-info.pdf	34031 d3365caa75150cd2f3a021caba758b3b5bcd03d6	no	2

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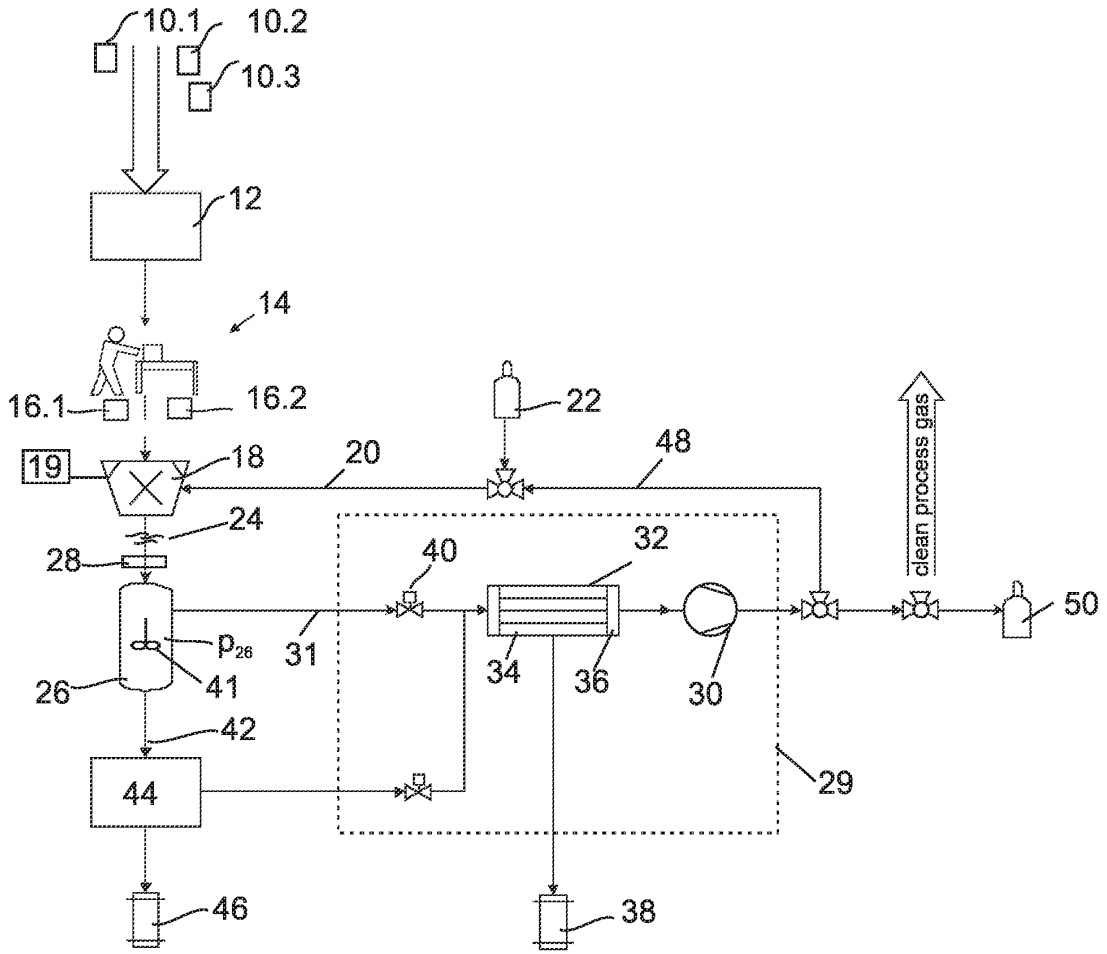


Fig. 1

REPLACEMENT SHEET

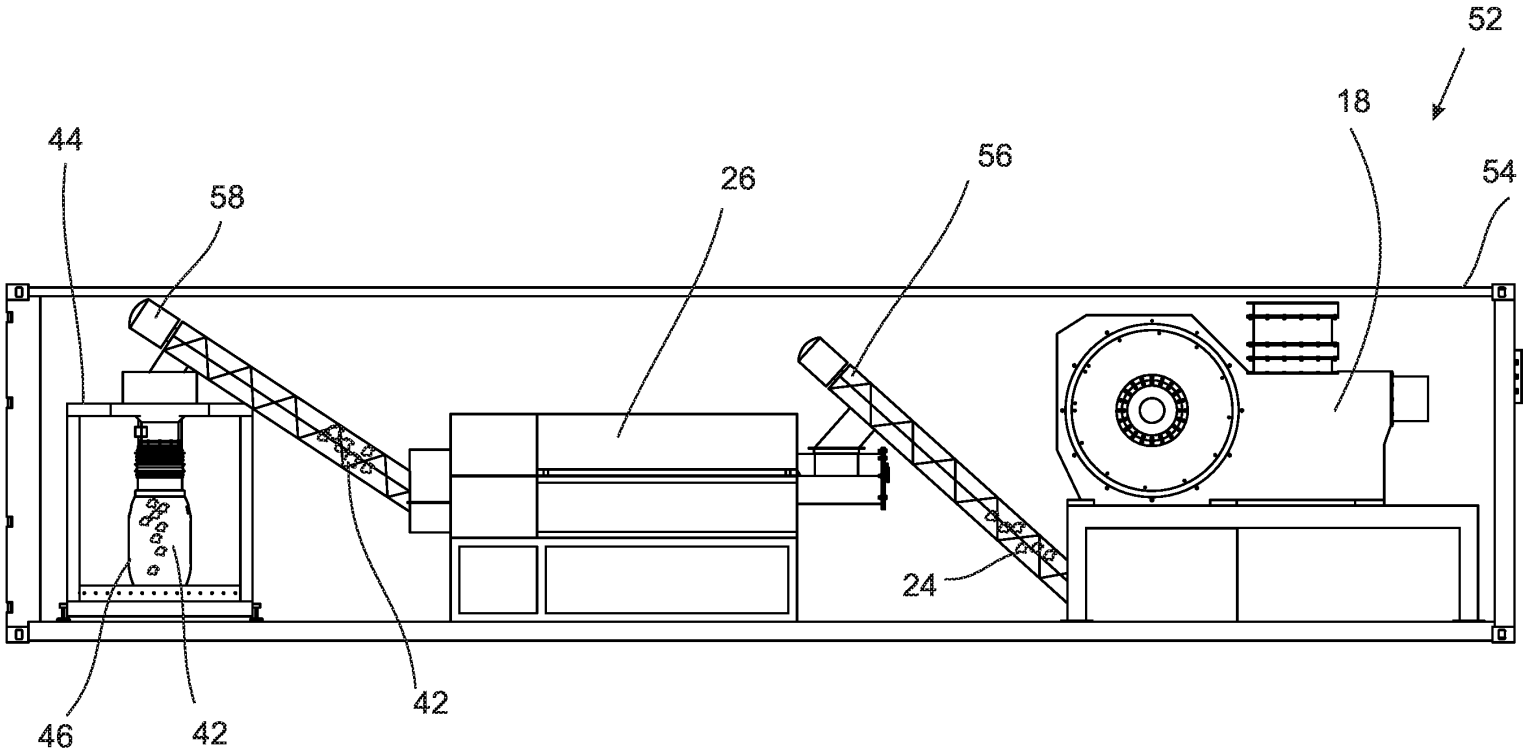
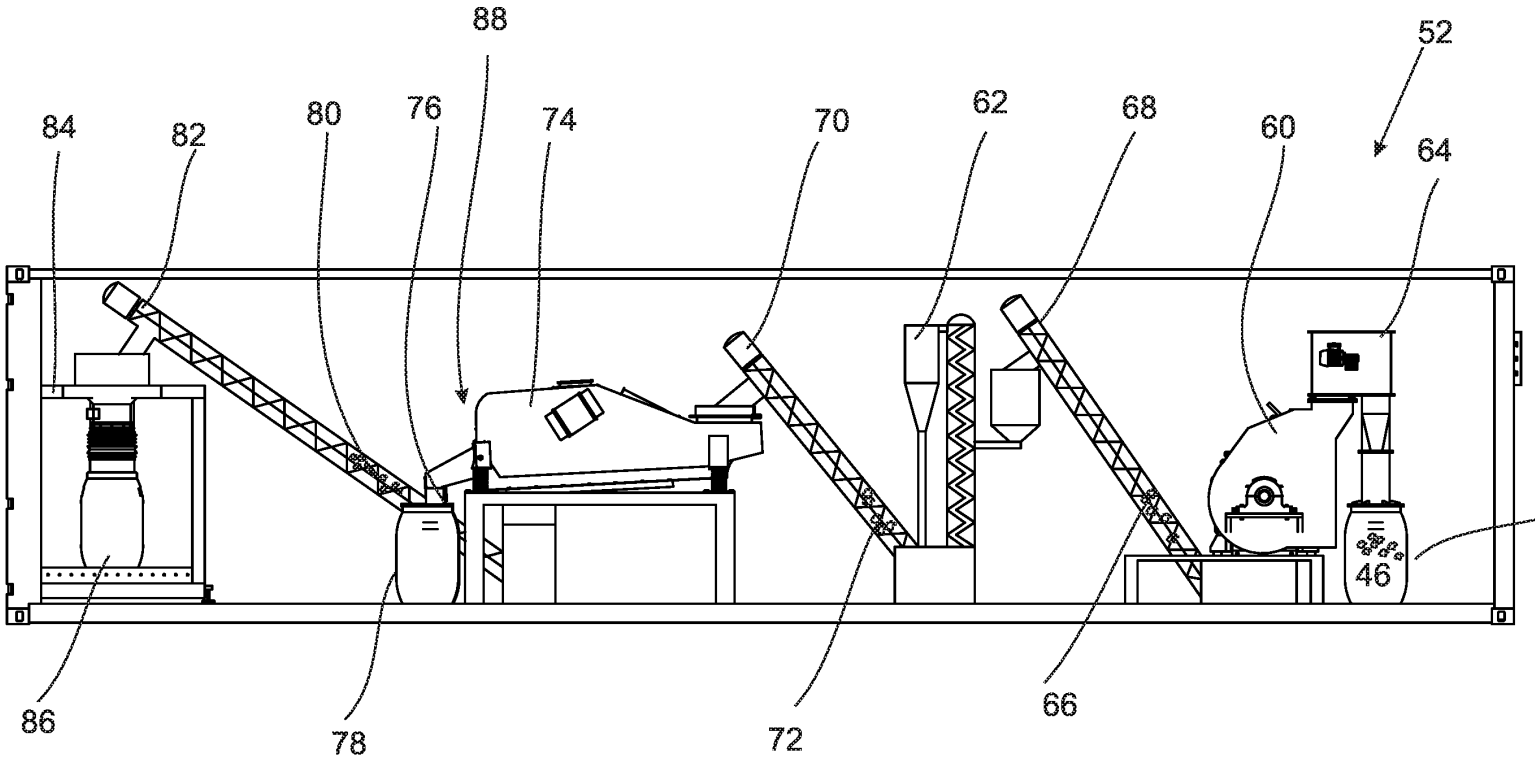


Fig. 2

REPLACEMENT SHEET



REPLACEMENT SHEET

Fig. 3

REMARKS

Claims 1, 4, 5, 6, 7, 11, 12, 13, 15, and 20 are amended. Claim 21 is newly added (see, e.g., page 4 of the specification). Applicant respectfully requests reconsideration of the claims above in view of the remarks presented below.

Specification and Drawings

The drawings were objected to. Replacement sheets for Figures 1-3 are attached hereto. No new matter is introduced.

The abstract was objected to for containing language such as "invention". The abstract is amended herein to comply with formalities.

The disclosure was objected to for its use of reference character "26" with two different nouns. The paragraph on page 13 lines 15-19 of the specification is amended herein to correct the typographical error.

Claim Objections

Claims 1, 5, 7, and 15 were objected to for various informalities. The examiner's suggested changes on page 4 of the action have been adopted in the claim set above, obviating these objections.

The examiner also indicated a lack of clarity in grammatical structures in claims 1, 5, 6, and 11. These claims are amended herein to improve clarity.

35 U.S.C. 112(f)

Several claim terms which employ the noun "device" have been interpreted under 35 U.S.C. 112(f). Applicant respectfully traverses.

As the examiner indicates in the Action, "a claim limitation that **does not use the term "means" or "step"** will trigger the rebuttable **presumption** that 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. **112, sixth paragraph does not apply**" (see supporting case law cited in MPEP 2181, emphasis added). Thus, because the instant claims do not use "means plus function" formulations, presumptively 35 U.S.C. 112 should not apply. In addition, "The standard is whether the words of the claim are understood by persons of ordinary skill in the art to have a

Ascend Elements EX1002 Page 253

Duesenfeld Ex. 2014-253

sufficiently definite meaning as the name for structure." *Williamson,* 792 F.3d at 1349, 115 USPQ2d at 1111; see also *Greenberg v. Ethicon Endo-Surgery, Inc.,* 91 F.3d 1580, 1583, 39 USPQ2d 1783, 1786 (Fed. Cir. 1996). Respectfully, applicant submits that the terms interpreted under 35 U.S.C. 112(f) by the examiner have sufficiently defined meaning to persons of ordinary skill in the art to be the names of structures.

What's more, the three-prong test for 112(f) (which the examiner cites on page 5 of the Action) *disfavors* interpretation of the claims under 112(f). Each of the three prongs (A), (B), and (C) must be satisfied before applying 112(f) to a claim term. Respectfully, prong (C) highlights that a supposedly generic placeholder "**modified by** sufficient structure, material, **or acts for performing the claimed function**" should NOT be interpreted under 112(f). In other words, sufficient specificity of what a "device" is configured or otherwise required to do places such "device" outside of 112(f) interpretation.

"an inactivation device" – Assuming even for the sake of argument that "inactivation device" ought to be interpreted under 112(f), the claims do in fact recite more structure for this device, namely a "drying device."

"a drying device" – This terms supplies sufficient structure and should not be interpreted under 112(f). "Dryer" and "drying device" are substantively identical terms, and "dryer" is widely used as the name of a non-generic structure and not merely a functional placeholder. Moreover, the application clearly supplies more than just "vacuum circulation system" as exemplary drying devices / dryers. Page 4 of the specification, for instance, gives ample description, including "vacuum drying, contact drying, convection drying and/or infra-red drying". All of these terms imply adequate structure according to their plain meanings as understood to persons of ordinary skill in the art and bolster the presumption in favor of NOT applying 112(f). Even if 112(f) is nevertheless applied, "drying device" is not so limited as to encompass only "vacuum circulation system" and its equivalents, since the applicant's specification gives many further instances of drying device techniques besides "vacuum circulation".

"a filling device" and **"second filling device"** – Here prong (C) of the 112(f) test supports the conclusion that a 112(f) interpretation should not be applied. Claim 7 does not merely recite "a filling device" but rather modifies "filling device" with the requirements that it be physically capable of *filling a transport container* and be able to handle *inactivated*

Ascend Elements EX1002 Page 254

Duesenfeld Ex. 2014-254

comminuted battery materials. These limitations supply the requisite "acts for performing the claimed function" under prong (C) of the 112(f) test to make 112(f) interpretation inapplicable.

"separation device" – The examiner has interpreted this to be an air jet sieving device or comminuting device. As clear from page 16 first paragraph of the specification, separation may be achieved by any sieve, not strictly air jet sieves. Moreover, claim 12 (where "separation device" appears) goes on to specifically modify the term "separation device" by requiring it be "configured to separately fill the active material fraction and the carrier fraction in respective containers." Under prong (C) of the 112(f) test, these limitations supply the requisite modifying "acts for performing the claimed function" to render 112(f) inapplicable.

35 U.S.C. 112(b)

Claim 6 is rejected under 35 U.S.C. 112(b) for indefiniteness over "the drying" and "the drying process". The redundancy has been eliminated by this amendment, obviating this rejection. Issue was also raised with respect to the phrase "the subsequent processing" in claim 6. This phrase has been removed.

35 U.S.C. 112(a)

Claim 13 is rejected under 35 U.S.C. 112(a) as failing the written description requirement. Claim 13 is amended for clarity, and applicant submits that amended claim 13 should obviate the rejection.

35 U.S.C. 103

Claims 1, 3, 5, 7, 12, 13, 16, and 17 are rejected under 35 U.S.C. 103 as being unpatentable over Stevens (US 8,210,456) in view of Kellermann (DE 4424825). This rejection is respectfully traversed.

Stevens describes a method of recycling sealed batteries involving heating as well as drawing air from a pre-shredder, shredder, oven conveyer, and a sieve using a vacuum source (see, e.g., column 2 lines 54-60).

Kellermann discloses inactivating batteries by deep freezing and then shredding them in a frozen state (cf. figure 1). Freezing requires a lot of energy. The shredded material undergoes a floating procedure (Flotation 19) that removes the organic compounds from the flotation liquid.

Ascend Elements EX1002 Page 255

Duesenfeld Ex. 2014-255

As the examiner acknowledges on page 10 of the action, Stevens fails to disclose "filling a transport container with the inactivated comminuted material" as recited in claim 1. Stevens also fails to disclose "inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa" as recited in claim 1 as amended.

Stevens describes a rotary kiln in which the comminuted material is heated. Gases from the kiln are removed by suction. However, nothing in Stevens discloses anything close to or suggestive of drying at a maximum pressure of 300 hPa. The person skilled in the art learns from Stevens that lithium cells should be heated at an average temperature of approximately 148°C, with the resulting gases being sucked off. But, as the present inventors have found, this can lead to the formation of highly toxic organic fluorine compounds. Sucking off gases does not lead to a pressure below 300 hPa. As best understood from Stevens disclosure, Stevens' air uptakes 13, 15, 21, and 51, described at column 2 lines 54-60, are not so different from range hoods, such as used for stove tops in many kitchens. They "draw air" as expressly stated at column 2 line 59, but drawing air does not itself result in a pressure drop from ambient anywhere near to the 300 hPa threshold now recited in claim 1. Moreover, since the oven of Stevens has no airlock, no vacuum can form in any case. Simply put, a pressure of 300 hPa cannot be achieved with suction devices disclosed or suggested by Stevens.

Kellermann does not cure Stevens' deficiency in respect to the drying and pressure requirements of amended claim 1. As a general matter, Kellermann voices low concern for gases and vapors from the recycled batteries, indicating that very little harmful gases and vapors are generated as a result of a deep freeze approach. Some gases are moved from one location to another by pumps (8 and 16 for example) but in no instance does Kellerman disclose or suggest pumping gases from a container of such design as to actually create an appreciable pressure drop from ambient pressures. Moreover, Kellermann does not employ inactivation by drying, using instead inactivation by freezing, making Kellermann that much further unlike amended claim 1 which recites "inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa".

Further distinguishing Kellermann, claim 1 provides for safe and effective battery recycling and removing the electrolyte without the need for expensive liquid nitrogen. By contrast, in Kellermann, the electrolyte comes into contact with the floating liquid and is disposed of as exhaust gases in device 23.

Ascend Elements EX1002 Page 256

Duesenfeld Ex. 2014-256

In sum, Stevens does not describe the element that the drying takes place at a pressure of at most 300 hPa and that this is carried out in such a way that the comminuted material is filled into a transport container. These distinguishing features have the technical effect that the comminuted material can be safely inactivated and transported to a central processing unit and the electrolyte recycled. Kellermann teaches a different approach, i.e. inactivation by liquid nitrogen, that cannot be combined with Stevens. Kellermann, like Stevens, does not disclose a pressure of less than 300 hPa. A combination of Stevens and Kellermann would inherit their mutual deficiencies. Any of the references or any purported combination thereof would fail to arrive at a method for the treatment of used batteries as recited in claim 1, especially "inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa" as recited in claim 1 as amended.

Independent claim 7 is also distinguished from Stevens, Kellermann, and their purported combination. Amended claim 7 positively recites a battery processing installation having "a vacuum installation connected to the drying device and configured to generate a vacuum in the drying device." As mentioned above, the apparatuses of Stevens and Kellermann are not designed to be capable of generating anything that might quality as a "vacuum" according to the ordinary meaning of that word. A pump that draws air may cause displacement of air but does not alone produce anything that may be qualify as a "vacuum." To use the same analogy as above, the references disclose apparatuses for drawing off air which may be compared to a stove range hood in most kitchens. It should be readily apparent to any range hood user that a "vacuum" is not generated nor generatable over the stove burners with such a configuration. Claim 7 is new and non-obvious over the references of record and their purported combination on at least this basis.

Claims 2, 6, and 8 are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Sloop (US 2003/0186110). This rejection is respectfully traversed.

Sloop uses yet another method to remove the electrolyte from batteries, i.e. using supercritical CO2. Supercritical CO2 requires a pressure of more than 73,75 bar (1060 psi). Thus Sloop's teaching contradicts claim 1's recitation of "inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa". Sloop thus cannot cure the deficiencies of Stevens

Ascend Elements EX1002 Page 257

Duesenfeld Ex. 2014-257

and Kellermann discussed above. As claims 2, 6, and 8 all depend from claim 1, they are new and nonobvious over any combination of the cited references for at least the same reasons as claim 1.

Claim 9 is rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Meador (US 5,632,863). This rejection is respectfully traversed.

Meador discloses a battery reclamation system and method which centers around pyrolysis, in particular decomposition temperatures between 350° and 650° F (see, e.g., Abstract). "Drying" and "drying device" as described in the disclosure at, e.g., page 4 of the specification, is distinguished from Meador's furnace 17. Accordingly, Meador fails to even have a "drying device" much less a "comminution unit and…drying device [that] are arranged in a container" as recited in claim 9.

Claims 14 and 18 have been rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Hanisch et al., herein "Hanisch," (WO 2013/023640), to which the examiner references using corresponding U.S. Patent No. 9,780,419. This rejection is respectfully traversed.

Like Meador, Hanisch involves heating to extremely high decomposition temperatures. In Hanisch's case, the desired temperatures are 350 to 800°C. A combination of Stevens, Kellermann, and Hanisch fails to arrive at the subject matter of the claims as amended herein. Claims 14 and 18 are new and nonobvious for at least the same reasons discussed above in respect of the independent claims from which they depend.

Claims 15 and 19 have been rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Sloop (US 2016/0043450), herein "Sloop2". This rejection is respectfully traversed. Sloop2 describes a method of battery recycling which does not include or suggest ""inactivating the comminuted material…by drying…at a maximum pressure of 300 hPa" as recited in claim 1 or "a vacuum installation connected to the drying device and configured to generate a vacuum in the drying device" as recited in claim 7. Claims 15 and 19, which depend from these independent claims, are new and nonobvious on at least this basis.

Ascend Elements EX1002 Page 258

Duesenfeld Ex. 2014-258

New claim 21 is added and recites “The method according to claim 1 wherein drying occurs at a temperature of less than 80°C.”

Stevens discloses in col. 5, lines 28-30 that the temperature should be below 454 ° F (234°C), but does not disclose that high temperatures can lead to the formation of highly toxic fluorine compounds and does not say that low temperatures could be beneficial. On the contrary, the person skilled in the art knows that high temperatures accelerate drying. The person skilled in the art will therefore choose a temperature which is as high as possible but below 234 ° C. In col. 4, lines 10-12 300° F (148° C) are referred to as "low temperature" and in col. 5, line 44 Stevens teaches that a "mid temperature" should be used for lithium batteries, i.e. a temperature higher than 148° C. Accordingly, Stevens does not disclose (and in fact teaches away from) claim 21 herein.

Kellermann uses ultra-low temperatures in order to inactivate the batteries, but does not dry them, cf. 5th para. “because the residual energy capacity of the batteries by their freezing before crushing is not or substantially no longer a chemical and physical reaction in air capable under normal pressure and temperature conditions when the protective metal casing of the batteries has been opened or removed”. Thus, Kellerman disclose an altogether different approach from what is claimed herein. Kellermann (inactivation by freezing and electrolyte removal by flotation) is incompatible with Stevens (inactivation by heating). The references, alone or in combination, would not lead one of ordinary skill in the art to claim 1 or claim 21.

Allowable Subject Matter

Applicant acknowledges with appreciation indication of claims 4, 11, and 20 as being drawn to allowable subject matter. Each of these claims has been rewritten in independent form by this amendment, placing them in condition for allowance.

Conclusion

In view of the foregoing, it is respectfully requested that the application be reconsidered, that claims 1-9 and 11-21 be allowed, and that the application be passed to issue.

Although the present communication may include alterations to the application or claims, or characterizations of claim scope or referenced art, Applicant is not conceding in this application that previously pending claims are not patentable over the cited references. Rather,

Ascend Elements EX1002 Page 259

Duesenfeld Ex. 2014-259

any alterations or characterizations are being made to facilitate expeditious prosecution of the application. Applicant reserves the right to pursue at a later time any previously pending or other broader or narrower claims that capture any subject matter supported by the present disclosure, including subject matter found to be specifically disclaimed herein or by any prior prosecution.

Accordingly, reviewers of this or any parent, child or related prosecution history shall not reasonably infer that Applicant has made any disclaimers or disavowals of any subject matter supported by the present application.

Should the Examiner find the application to be other than in condition for allowance, the Examiner is requested to contact the undersigned at the local telephone number listed below to discuss any other changes deemed necessary in a telephonic, WebEx, or personal interview.

A provisional petition is hereby made for any extension of time necessary for the continued pendency during the life of this application. Please charge any fees for such provisional petition and any deficiencies in fees and credit any overpayment of fees to Attorney's Deposit Account No. 50-2041.

Respectfully submitted,

/T. Bryce Miller/

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Ascend Elements EX1002 Page 260

Duesenfeld Ex. 2014-260

Listing of the Claims:

The following is a complete listing of all the claims in the application, with an indication of the status of each:

1. (currently amended) A method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained;~~,~~

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained;~~,~~ and

(c) filling a transport container with the inactivated comminuted material;~~,~~

wherein the inactivating step is performed by drying the comminuted material, and

wherein the drying occurs at a maximum pressure of 300 hPa.

2. (previously presented) The method according to claim 1, wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

3. (previously presented) The method according to claim 1, wherein the drying occurs under vacuum.

4. (currently amended) ~~The~~ A method for the treatment of used batteries, comprising the steps: ~~according to claim 3,~~

(a) comminuting the batteries such that comminuted material is obtained;

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained; and

(c) filling a transport container with the inactivated comminuted material;

wherein the inactivating step is performed by drying the comminuted material,

wherein the drying occurs under vacuum,

wherein the vacuum is created by means of a jet pump, and

wherein a pump liquid of the jet pump has at least one of

a pH value of at least 8, and

~~contains~~ a substance that precipitates fluoride.

Ascend Elements EX1002 Page 261

Duesenfeld Ex. 2014-261

5. (currently amended) The method according to claim 1, further comprising the steps of:

removing ~~removal of~~ the comminuted material from the transport container; and~~,~~

one or more of

detaching ~~detachment of~~ hard parts, ~~and~~

separating ~~separation of~~ active material from a carrier, such that an active material fraction and a carrier fraction ~~(80)~~ are obtained, and

separately ~~separate~~ packing ~~of~~ the active material fraction and carrier fraction in respective further ~~transport~~ containers.

6. (currently amended) The method according to claim 1, wherein the drying of the comminuted material is only completed if one or more of the following conditions is met:~~,~~

~~after the completion of the drying process,~~ no flammable or explosive gas mixture is formable ~~can form~~ above the comminuted material in the transport container, and ~~and/or if~~

the comminuted material is so dry that no flammable or explosive gas mixture is able to emerge ~~can emerge~~ in the transport container ~~or during the subsequent processing~~.

7. (currently amended) A battery processing installation for treatment of used batteries, comprising:

(a) a comminution unit configured to comminute ~~for comminuting~~ the batteries such that comminuted material is obtained;~~,~~

(b) an inactivation device comprising a drying device configured to inactivate ~~for inactivating~~ the comminuted material; ~~and~~

(c) a filling device ~~for filling~~ configured to fill a transport container with the inactivated comminuted material; and~~,~~

(d) a vacuum installation connected to the drying device and configured to generate a vacuum in the drying device.

~~wherein~~

~~the inactivation device comprises a drying device, and~~

~~the battery processing installation has a vacuum installation that is connected to the drying device for generating a vacuum in the drying device.~~

Ascend Elements EX1002 Page 262

Duesenfeld Ex. 2014-262

8. (previously presented) The battery processing installation according to claim 7, wherein the drying device is configured to dry the comminuted material until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

9. (previously presented) The battery processing installation according to claim 7, wherein the comminution unit and the drying device are arranged in a container.

10. (canceled)

11. (currently amended) ~~The~~ A battery processing installation for treatment of used batteries, comprising: ~~according to claim 7, wherein~~

(a) a comminution unit configured to comminute the batteries such that comminuted material is obtained;

(b) an inactivation device configured to inactivate the comminuted material, wherein the inactivation device comprises a drying device;

(c) a filling device configured to fill a transport container with the inactivated comminuted material;

(d) a vacuum installation that is connected to the drying device and configured to generate a vacuum in the drying device;

wherein

[[-]] the vacuum installation has a jet pump with a pump liquid, ~~and wherein~~

[[-]] the pump liquid is conducted within a circuit, and

[[-]] the pump liquid contains a substance that reacts with hydrogen fluoride.

12. (currently amended) The battery processing installation according to claim 7 further comprising:

[[-]] a hard metal detachment device and/or a light fraction detachment device;~~,~~

[[-]] a separation device configured to separate ~~for separating~~ active material from a carrier such that an active material fraction and a carrier fraction occur;~~,~~ and

[[-]] a second filling device configured to separately fill ~~for separate filling of~~ the active material fraction and the carrier fraction in respective containers.

Ascend Elements EX1002 Page 263

Duesenfeld Ex. 2014-263

13. (currently amended) The battery processing installation of claim 12 wherein ~~the active material fraction and~~ the carrier fraction are ~~respectively~~ aluminum and copper foils.

14. (previously presented) The battery processing installation of claim 12, wherein the separation device is an air jet sieving device.

15. (currently amended) The battery processing installation of claim 12 wherein the separation device is a second comminution device. ~~stage.~~

16. (previously presented) The battery processing installation of claim 7 wherein the batteries are lithium batteries.

17. (previously presented) The method according to claim 1 wherein the batteries are lithium batteries.

18. (previously presented) The method according to claim 5 wherein separation of the active material from the carrier is performed by air jet sieving.

19. (previously presented) The method according to claim 5 wherein separation of the active material from the carrier is performed using a second comminution stage.

20. (currently amended) ~~The method according to claim 4~~ A method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained,

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained, and

(c) filling a transport container with the inactivated comminuted material,

wherein the inactivating step is performed by drying the comminuted material,

wherein the drying occurs under vacuum,

wherein the vacuum is created by means of a jet pump, and

Ascend Elements EX1002 Page 264

Duesenfeld Ex. 2014-264

wherein a pump liquid of the jet pump has a ~~the~~ pH value ~~is~~ of at least 9.

21. (new) The method according to claim 1 wherein drying occurs at a temperature of less than 80°C.

Ascend Elements EX1002 Page 265

Duesenfeld Ex. 2014-265

Amendments to the Abstract:

Please amend the abstract as follows:

~~The invention relates to a~~ A method is described for the treatment of used batteries ~~(10)~~, in particular lithium batteries, containing the steps: comminuting the batteries ~~(10)~~ such that comminuted material ~~(24)~~ is obtained, inactivating the comminuted material ~~(24)~~ such that inactivated comminuted material ~~(42)~~ is obtained, and filling a transport container ~~(46)~~ with the inactivated comminuted material. ~~(42). It is provided according to the invention that the~~ The inactivation is performed by drying the comminuted material, ~~(24)~~ and the comminuted material ~~(24)~~ is dried until an electrolyte content is so low that an electrochemical reaction is not possible.

Ascend Elements EX1002 Page 266

Duesenfeld Ex. 2014-266

Amendments to the Specification:

Please amend the specification as follows:

Insert the following header at page 1 line 3:

FIELD OF THE INVENTION

Insert the following header at page 1 line 17:

BACKGROUND

Amend the paragraph at page 1 lines 24-31 as follows:

DE 10 2012 024 876 A1 describes a system for transferring transport-critical electrolyte cells, in which they are initially comminuted under inert gas and then dusted with a deactivation powder so as to prevent the electrochemically active material from spontaneously combusting. The disadvantage of this is that the resulting material still poses a comparatively high hazard potential and that the dusting ~~power~~ powder itself poses a risk of explosion and that the formation of a flammable and explosive atmosphere in the transport container cannot be ruled out.

Insert the following header at page 2 line 6:

SUMMARY

Insert the following header at page 12 line 17:

BRIEF DESCRIPTION OF THE DRAWINGS

Insert the following header at page 12 line 28:

DETAILED DESCRIPTION

Amend the paragraph on page 13 lines 9-13 as follows:

The comminution unit 18 comminutes the batteries 10 under shielding gas 20, which is extracted, for example, from a shielding gas cylinder 22. Alternatively or additionally, liquid nitrogen from a liquid nitrogen source 19 may be ~~may be~~ injected. The shielding gas may refer, for example, to nitrogen, a noble gas, carbon dioxide, nitrous oxide or another gas which is preferably not toxic.

Ascend Elements EX1002 Page 267

Duesenfeld Ex. 2014-267

Amend the paragraph on page 13 lines 15-19 as follows:

Comminuted material 24 is produced during the comminuting; the material is fed into an inactivation device in the form of a drying device 26. An airlock 28 is arranged between the comminution unit 18 and the drying device 26, the airlock being so gas-tight that the drying ~~pressure~~ device 26 is - to a good approximation - separated from the comminution unit 18 so as to be gas-tight.

Ascend Elements EX1002 Page 268

Duesenfeld Ex. 2014-268

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(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

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	1	20140290438	A1	2014-10-02	C. HANISCH; W. HASELRIEDER; A. KWADE	

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	1	2010/102337	WO	A1	2010-09-16	W. STEVENS		
	2	103959553	CN	A	2014-07-30	UNIV. BRAUNSCHWEIG TECH	Equivalent to WO2010/102337	
	3	102496752	CN	A	2012-06-13	CHINA NAT OFFSHORE OIL CORP ET AL.		X

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
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Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2019-09-12
Name/Print	Michael E. Whitham	Registration Number	32635

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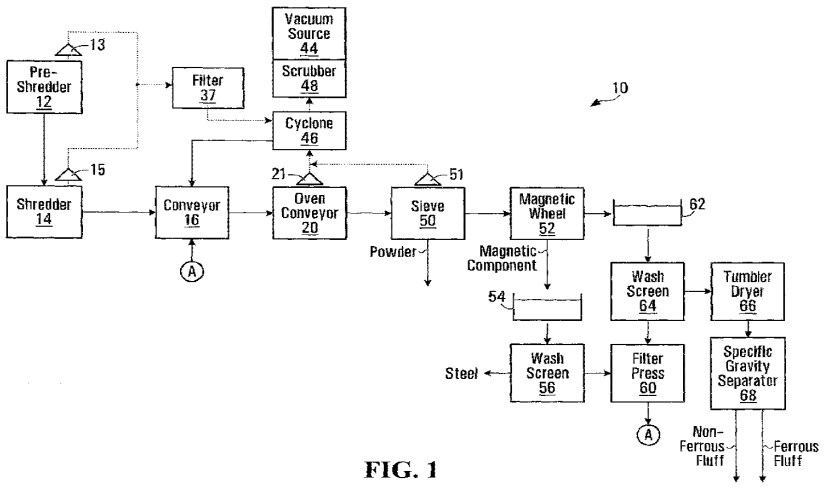

FIG. 1

(57) Abstract: In a method of recycling sealed batteries, the batteries are shredded to form a shredded feedstock. The shredded feedstock is heated above ambient temperature and rolled to form a dried material. The dried material is screen separated into a coarse fraction and a powder fraction and the powder fraction is output. A system for recycling sealed cell batteries comprises an oven with a first conveyor extending into the oven. A rotatable tunnel extends within the oven from an output of the first conveyor. The tunnel has a spiral vane depending from its inner surface which extends along a length of the tunnel. A second conveyor is positioned below an output of the rotatable tunnel.

WO 2010/102377 A1

BATTERY RECYCLING

BACKGROUND

[0001] The present invention relates to a system and process for recycling sealed cell batteries.

[0002] Ninety-five percent of portable batteries are household batteries. The vast majority of these are sealed cell alkaline batteries. Once spent, most of these batteries are simply discarded and find their way to landfill sites.

[0003] A known method for recycling alkaline batteries involves mechanically removing the casing from the battery cell and then using chemical processes to separate the solid materials of the cell. Major solid components of the cells are carbon, zinc, potassium, and manganese. There are several problems with chemical separation processes. Firstly, the component materials involved are of low value while chemical processing is expensive. Also, additional waste streams are created with the chemical processes. In view of these drawbacks, this recycling method has not found widespread use.

[0004] This invention seeks to overcome drawbacks of known sealed cell battery recycling processes.

SUMMARY

[0005] In a method of recycling sealed batteries, the batteries are shredded to form a shredded feedstock. The shredded feedstock is heated above ambient temperature and rolled to form a dried material. The dried material is screen separating into a coarse fraction and a powder fraction and the powder fraction is output.

Ascend Elements EX1002 Page 274

Duesenfeld Ex. 2014-274

[0006] A system for recycling sealed cell batteries comprises an oven with a first conveyor extending into the oven. A rotatable tunnel extends within the oven from an output of the first conveyor. The tunnel has a spiral vane depending from its inner surface which extends along a length of the tunnel. A second conveyor is positioned below an output of the rotatable tunnel.

[0007] Other features and advantages will be apparent from the following description in conjunction with the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] In the figures which illustrate an example embodiment of the invention,

[0009] **FIG. 1** is a schematic diagram of a system for battery recycling in accordance with this invention,

[0010] **FIG. 2** is a partially cut-away perspective view of a portion of the system of **FIG. 1**,

[0011] **FIG. 3** is a schematic diagram of another system for battery recycling in accordance with this invention, and

[0012] **FIG. 4** is a schematic diagram of a portion of another system for battery recycling in accordance with this invention.

DETAILED DESCRIPTION

[0013] Technically, a battery is a series of battery cells. Thus, a 9 V battery is a true battery, whereas AAA through D size batteries are cells. In this specification, the term battery is used to mean either true batteries or cells.

[0014] The central core of an alkaline cylindrical button cell is the anode which is a dispersion of zinc oxide powder in a gel containing a potassium hydroxide electrolyte. This core is surrounded by a separator which is a non-woven layer of

cellulose (paper) or a synthetic polymer (plastic). Surrounding the separator is an annular cathode which is a compressed paste of manganese dioxide with carbon (graphite) powder to increase conductivity. The anode, separator, and cathode are sealed in a drawn steel casing. Although some alkaline batteries are mercury-free, many spent alkaline batteries contain mercury, with an average overall concentration of 100 ppm.

[0015] Turning to **FIG. 1**, a system **10** for recycling sealed cell alkaline batteries has a pre-shredder **12** for pre-shredding the feedstock in order to rupture their sealed steel casings. Next the batteries may be further shredded in secondary shredder **14**, which may be a granulator, and are then conveyed downstream by conveyor **16**. The conveyor, which may be a chain conveyor or a belt conveyor with flights, assists in breaking up the feedstock into a more uniform material feed stream.

[0016] From the conveyor **16**, the feedstock passes to an oven conveyor **20**. The oven conveyor **20** is detailed in **FIG. 2**. Turning to **FIG. 2**, oven conveyor **20** has an input belt conveyor **22** with an overlying blade **24** which levels the feedstock. The input belt conveyor dispenses to an upstream screw tunnel **26**. The upstream screw tunnel **26** has a spiral vane depending from its inner surface that extends along the length of the tunnel. The upstream screw tunnel **26** rotates to roll the feedstock. The upstream screw tunnel **26** in turn dispenses to a middle belt conveyor **28** with an overlying blade **30** which levels the feedstock. The middle belt conveyor dispenses to a downstream screw tunnel **32** having a similar configuration as the upstream screw tunnel. The downstream screw tunnel dispenses to an output belt conveyor **34**. The output belt conveyor has an overlying blade **36** which levels the feedstock. An oven **40** surrounds all but the upstream end of the input belt conveyor and downstream end of the output belt conveyor.

[0017] Feedstock traveling on the input belt conveyor **22** is levelled by blade **24**. The material then drops into upstream screw tunnel **26**. This screw tunnel rotates

Ascend Elements EX1002 Page 276

Duesenfeld Ex. 2014-276

to roll the feedstock and feed it downstream. At the downstream end of the upstream screw tunnel, the feedstock drops to the middle belt conveyor **28** where it is levelled by blade **30** and conveyed into downstream screw tunnel **32**. The downstream screw tunnel rotates to roll the feedstock and convey it to output belt conveyor **34**, where the feedstock is levelled by blade **36** and then dispensed from the oven conveyor.

[0018] By repeatedly conveying, rolling and levelling the feedstock at elevated temperatures in the oven conveyor, the feedstock is dried and evaporation of mercury in the feedstock is promoted.

[0019] Returning to **FIG. 1**, there are air uptakes **13**, **15**, **21**, and **51** associated with each of pre-shredder **12**, shredder **14**, oven conveyer **20** and a sieve **50**, respectively. Each of the air uptakes feed ultimately to a cyclone **46**, with air uptakes **13** and **15** feeding to the cyclone through a particulate filter **37**. A vacuum source **44** draws air through the cyclone **46** to a scrubber **48** before the air leaves the system **10**. The cyclone swirls the moving air to drop out any powder or other solids entrained in the moving air. The scrubber sequesters vapours in the moving air which, with the alkaline battery feedstock, will be mercury vapours. The scrubber **48** may be a venturi scrubber with a pH control system that ensures the solubility of vapours. It may also be any other type known in the art to be effective in these conditions. With a venturi scrubber, the scrubber itself acts as the vacuum source. Filter **37** traps any paper and plastics drawn off by air intakes **13** and **15** so that the scrubber is not obstructed by this material and so that any fire that develops in the scrubber is not additionally fed by these paper and plastics.

[0020] While mercury has a boiling point of about 357°C (about 675 °F) , it is nevertheless volatile even at room temperature and therefore off gases at the various air uptakes as well as off gassing strongly in the oven conveyor.

[0021] Feedstock leaving the oven conveyor passes to a sieve **50** which may be a shaker table with a mesh size #30. This separates a coarser fraction of the

feedstock from a finer fraction. The mesh size is such that the finer fraction which drops through the sieve is a zinc oxide, manganese dioxide, and potassium hydroxide powder. This powder is recovered as a finished product of the process.

[0022] The coarser fraction, which comprise the shredded steel casing along with brass, cellulose (paper), graphite (carbon), and plastic, collectively known as fluff, pass to a magnetic separator **52**, which may be a magnetic wheel.

[0023] The separated steel (magnetic component) is immersed in a first water bath **54**. Water from the bath **54** drains through a wash screen **56**. Any remaining powder will pass through the screen and therefore remain in the water. Larger solids, namely the separated steel, are blocked by the screen and are washed off the screen to recover the steel as a second finished product of the process.

[0024] The water which passes through the screen **56** passes on to filter press **60**.

[0025] The non-magnetic component of the fluff which is separated by the magnetic separator is immersed in a second water bath **62**. Water from the bath **62** drains through a wash screen **64**. Any remaining powder in the water will pass through the screen with the water. Larger solids, namely the fluff, are blocked by the screen and are washed off the screen. This fluff passes to a tumbler dryer **66** where it is dried and then to a specific gravity separator **68**. The specific gravity separator may be of the forced air type known in the art. The specific gravity separator **68** separates the ferrous component of the fluff (graphite and brass) from the nonferrous component (paper and plastic). These two output streams are finished products of the process.

[0026] The water which passes through screen **64** passes on to filter press **60**.

[0027] The water passing to the filter press from screens **56** and **64** passes through the membranes of the filter press such that any powder which had been in

Ascend Elements EX1002 Page 278

Duesenfeld Ex. 2014-278

the water is recovered as a wet powder. This wet powder is fed back to the conveyor **16** in the system **10**. The water, which at this stage is normally caustic, then passes to a holding tank (not shown) wherein its pH is adjusted so that it may be reused in the water baths.

[0028] The recovered powder of zinc oxide, manganese dioxide, and potassium hydroxide may be used in fertilizer provided its mercury content has been sufficiently reduced. In this regard, typically, a powder with a mercury content of less than 75 ppm is suitable for use in fertilizer. With the subject system, mercury concentrations down to undetectable levels may be achieved.

[0029] The recovered steel may be used in the steel industry. The recovered paper and plastic from the fluff may be burned for energy (e.g., in oven **40**).

[0030] The same process described for alkaline batteries may also be used for any of zinc carbon, zinc chloride, and manganese batteries. The composition of the recovered powder will vary somewhat depending upon the type of battery which forms the feedstock. However, for all of these types of batteries, the recovered powder may be used in fertilizer.

[0031] The time during which the feedstock remains in the oven conveyor and the temperature of this conveyor are determined based on characteristics of the feedstock and desired properties of the powder output from the system. More specifically, a customer of the powder may specify a required dryness and mercury content for the powder. Characteristics of the feedstock which impact the required heating time and temperature are the size of the battery cells, the age of the cells (newer batteries need to be run slower at higher temperatures), mercury content, and the type of battery. Regardless of customer requirements, the feedstock must at least be sufficiently dry so that it separates at the screen separator sieve **50**.

[0032] While the temperature of the oven can be varied as required, typically, a temperature of between a low temperature of 300°F and a high temperature of

800°F is sufficient where the feedstock remains in the oven conveyor for 1 to 10 minutes. The speed of the conveyors determines the time in the oven conveyor. By way of example only one minute in the oven conveyor may correspond to a high speed whereas ten minutes in the oven conveyor may correspond to a low speed.

[0033] For example, for a mixed feedstock of alkaline, zinc carbon, zinc chloride, and manganese batteries and a 26 ft long oven conveyor, the speed through the oven conveyor may be 2.6 ft/min and the temperature may be set at 800° F.

[0034] The mesh size of the screen separator can be varied as required provided it is sufficiently small to separate the powder fraction of the feedstock. However, a #30 mesh size is suitable where the powder is to be used in the fertilizer industry and also allows the powder to pelletize well.

[0035] The described system may be modified for use with nickel metal hydride sealed cell batteries. More specifically, turning to **FIG. 3**, system **100** for use with a feedstock of nickel metal hydride batteries is with some exceptions the same as system **10** of **FIG. 1**, and like parts have been given like reference numerals. The differences are as follows. System **100** includes a tumbler dryer **76** at the output of the first wash screen **56** which feeds to a specific gravity separator **78**.

[0036] With nickel metal hydride batteries, the anode is nickel oxyhydroxide and the cathode is most commonly a lanthanoid mixture with nickel, cobalt, manganese, and/or aluminum. Some cathodes may include other metals, such as iron or chromium. The electrolyte is most commonly potassium hydroxide.

[0037] The processing of nickel metal hydride batteries in system **100** proceeds identically to the processing of alkaline batteries in system **10** of **FIG. 1** except as follows. Vapours generated from drying the feedstock are water and volatile organic compounds (VOCs). The speed of the oven conveyor is adjusted so that the feedstock dries sufficiently through the oven conveyor at the chosen oven

Ascend Elements EX1002 Page 280

Duesenfeld Ex. 2014-280

temperature. The scrubber (which may be a venturi scrubber as used in the system **10** of **FIG. 1**) sequesters the vapours from the drying process.

[0038] When the feedstock leaves the oven conveyor and reaches the sieve, the powder component which passes through the sieve comprises nickel oxyhydroxide powder, cobalt oxide, and lanthanoids. As when operating with alkaline batteries, sieve **50** may have a #30 mesh size. The recovered powder may be used in the coatings industry for pigmentation or the powder may be used in the stainless steel industry.

[0039] The solid component, which does not pass through the sieve, contains steel, fluff and nickel alloys. These nickel alloys include iron. The steel and nickel alloys form the magnetic component which is separated from the fluff by the magnetic wheel **52**. However, the nickel alloys of this magnetic component typically encapsulate some of the fluff as a consequence of the upstream shredding process. This encapsulated fluff remains with the magnetic component leaving the magnetic wheel. The magnetic component is directed through bath **54** to the first wash screen **56**. The solid component leaving the wash screen passes to tumbler dryer **76** where it is dried. The dried solid component then inputs specific gravity separator **78** to separate nonferrous fluff residues from the nickel alloys and to separate the nickel alloys from the steel. The steel and nickel alloys are finished products that may be used in the steel industry.

[0040] System **100** may be modified to operate with a feedstock of lithium button cells, namely lithium ion batteries, which are rechargeable, or lithium polymer batteries. With a lithium battery, the anode is a lithium metal or lithium compound. The cathode is, most commonly, manganese dioxide and the electrolyte is, most commonly, lithium perchlorate in propylene carbonate and dimethoxyethane.

[0041] **FIG. 4** illustrates a portion of a system **1000** for use with lithium button cells. System **1000** is identical to system **100** of **FIG. 3** except as follows. The scrubber **48** outputs to a water atomizer **84** and the atomizer **84** outputs to a tank

86. These modifications are employed because of the highly combustible nature of lithium and the volatile organic compounds (VOCs) in the batteries.

[0042] In this modified system, when the lithium batteries are shredded and conveyed by the conveyor, lithium, which gas off at any of the air pick up points and pass to the scrubber **48** where they are sequestered. Any lithium or VOCs which escape the scrubber are condensed by the atomized water from the atomizer **84** and the condensed lithium passes to the water tank **86**. The pH of the water in the atomizer and the tank is controlled with, for example, the addition of sodium hydroxide to a concentration of 50%, to ensure the gas remains condensed in the water. Any paper drawn off or drawn off is trapped by filter **70** before it reaches the scrubber so that it will not feed any fire that might occur at the scrubber or obstruct the scrubber. Because of the risk of combustion with lithium ion battery feedstock, the temperature in the oven conveyor is maintained below the combustion temperature of paper (454°F).

[0043] With lithium button cells, at the sieve **50**, the powder which drops out is mostly lithium cobalt oxide. This powder can be used in the coatings industry. As before, a mesh size #30 is suitable to limit contamination of this powder.

[0044] The magnetic component separated by the magnetic wheel includes steel and magnetic alloys as well as any aluminum and copper that is trapped by the steel and magnetic alloys. The specific gravity separator **78** (**FIG. 3**) separates out non-ferrous fluff encapsulated with this magnetic component and also separates the steel, magnetic alloys, aluminum, and copper from each other.

[0045] In general, with system **10** run with alkaline batteries, the oven conveyor is run at a low speed and high temperature (to promote evaporation of mercury), with system **1000** run with lithium batteries, the oven conveyor is run at a mid speed and temperature (to avoid combustion), and when the system **100** is run with nickel metal hydride batteries, the oven conveyor is run at high speeds and temperatures.

[0046] While three different systems have been described, they may be combined in a single system and components of that system switched in or out, or on or off, as appropriate, to adapt the system to the different kinds of feedstock.

[0047] Rather than providing a vacuum source to draw vapours into the scrubber, blowers could be used for this purpose.

[0048] There is a minimal amount of paper and light plastics released with the shredding of alkaline batteries. Accordingly, optionally, filter **37** may be omitted when running system **10**.

[0049] Other modifications will be apparent to those skilled in the art and, therefore, the invention is defined in the claims.

Ascend Elements EX1002 Page 283

Duesenfeld Ex. 2014-283

WHAT IS CLAIMED IS:

1. A method of recycling sealed batteries, comprising:

shredding said batteries to form a shredded feedstock;

heating said shredded feedstock above ambient temperature while rolling said shredded feedstock to form a dried material;

screen separating said dried material into a coarse fraction and a powder fraction; and

outputting said powder fraction.

2. The method of claim 1 further comprising levelling said shredded feedstock while heating said feedstock above ambient temperature.

3. The method of claim 1 wherein said batteries are alkaline batteries containing mercury, said heating comprises heating said shredded feedstock above ambient temperature and below the boiling point of mercury, said heating while rolling evaporates a portion of said mercury from said feedstock, and wherein said powder fraction comprises zinc oxide, manganese dioxide, and potassium hydroxide.

4. The method of claim 3 further comprising drawing air through said shredded material during said heating and rolling to draw off mercury vapour from said shredded feedstock.

5. The method of claim 4 further comprising sequestering said mercury in a scrubber.

6. The method of claim 5 further comprising swirling said air drawn from said shredded material to drop out any particulates from said air and returning said particulates to said shredded material.

7. The method of claim 1 further comprising magnetically separating said coarse fraction into a magnetic component and a non-magnetic component.

Ascend Elements EX1002 Page 284

Duesenfeld Ex. 2014-284

8. The method of claim 7 further comprising immersing said magnetic component in a first water bath and passing water from said first water bath through a screen to separate magnetic solids, and outputting said magnetic solids.

9. The method of claim 8 further comprising separating said magnetic solids based on their specific gravity prior to said outputting said magnetic solids and wherein said outputting said magnetic solids comprises outputting separated constituents of said magnetic solids.

10. The method of claim 8 further comprising immersing said non-magnetic component in a second water bath and passing water from said second water bath through a screen to separate fluff, and outputting said fluff.

11. The method of claim 10 further comprising separating said fluff based on specific gravity of constituents of said fluff prior to said outputting said fluff and wherein said outputting said fluff comprises outputting separated constituents of said fluff.

12. The method of claim 10 further comprising combining water from said first water bath and said second water bath as a water component and filtering said water component to recover a powder component.

13. The method of claim 12 further comprising adding said powder component to said shredded material.

14. A method of recycling sealed batteries containing mercury, comprising:

shredding said batteries to form a shredded feedstock;

heating said shredded feedstock above ambient temperature and below the boiling point of mercury while rolling said shredded feedstock to evaporate a portion of said mercury from said feedstock to form a mercury reduced dried material;

screen separating said dried material into a coarse fraction and a powder fraction; and

outputting said powder fraction.

15. A system for recycling sealed cell batteries, comprising:

an oven;

a first conveyor extending into said oven;

a rotatable tunnel with a spiral vane depending from an inner surface of said tunnel extending along a length of said tunnel, said rotatable tunnel disposed within said oven and extending from an output of said first conveyor;

a second conveyor below an output of said rotatable tunnel.

16. The system of claim 15 further comprising a sieve downstream of an output of said second conveyor.

17. The system of claim 16 further comprising a shredder upstream of an input of said first conveyor.

18. The system of claim 17 further comprising a leveller associated with said first conveyor.

19. The system of claim 15 further comprising a cyclone and a scrubber above said oven, and means for moving air upwardly through said oven, said cyclone and said scrubber.

20. The system of claim 19 further comprising at least one particulate filter between said oven and said scrubber.

Ascend Elements EX1002 Page 286

Duesenfeld Ex. 2014-286

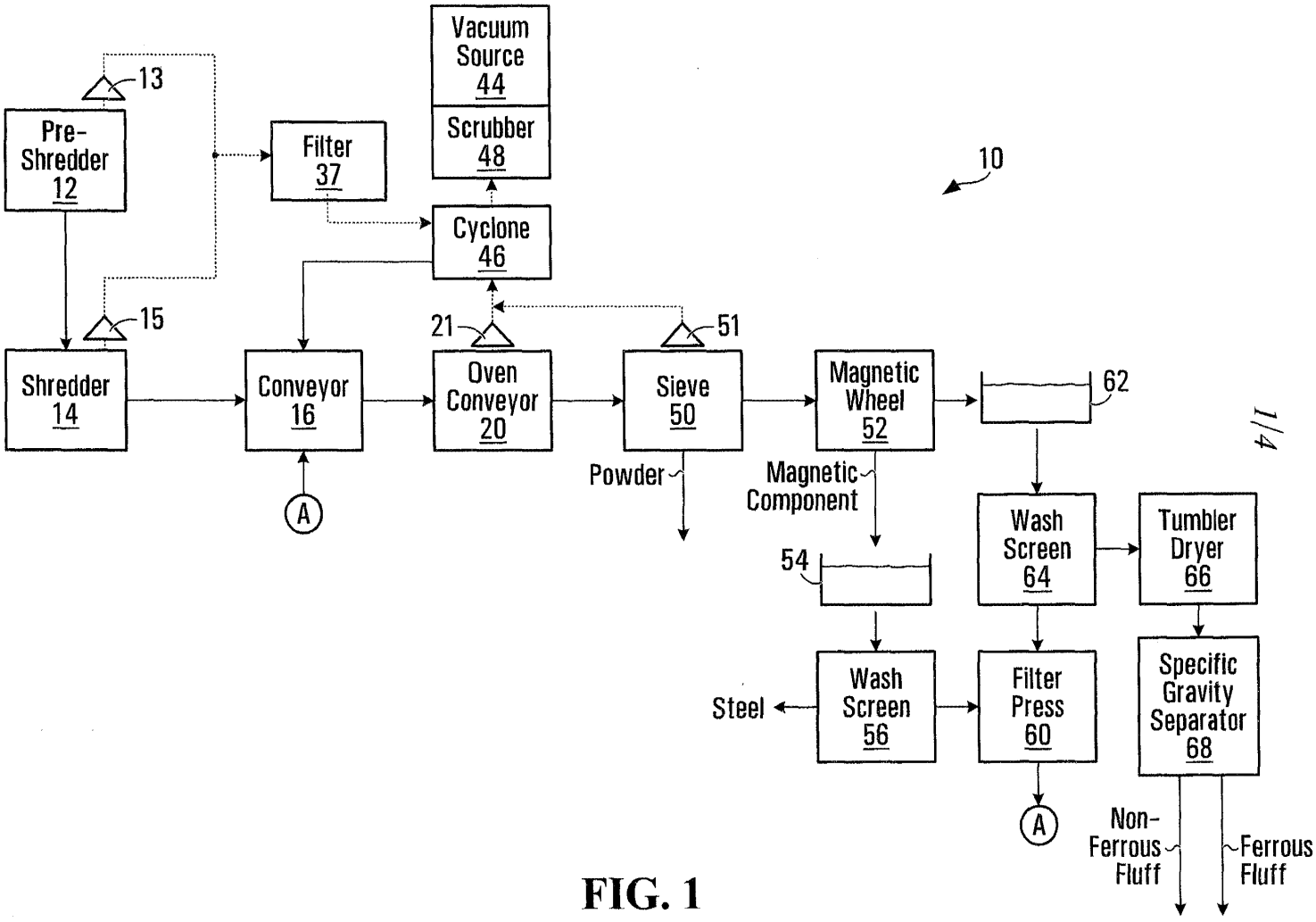
Vacuum Source 44
Scrubber 48
Cyclone 46
Filter 37
Pre-Shredder 12
13
15
Shredder 14
Conveyor 16
A
21
Oven Conveyor 20
51
Sieve 50
Powder
Magnetic Wheel 52
62
Magnetic Component
54
Steel
Wash Screen 56
Filter Press 60
A
Wash Screen 64
Tumbler Dryer 66
Specific Gravity Separator 68
Non-Ferrous Fluff
Ferrous Fluff
10

FIG. 1

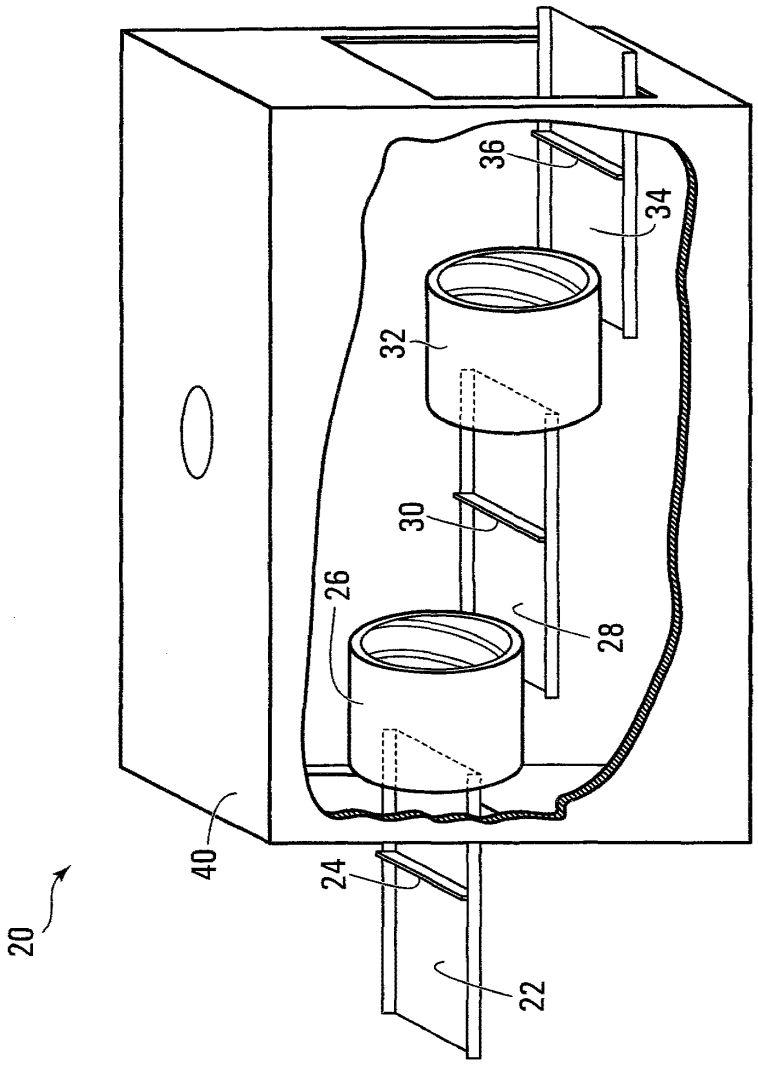

FIG. 2

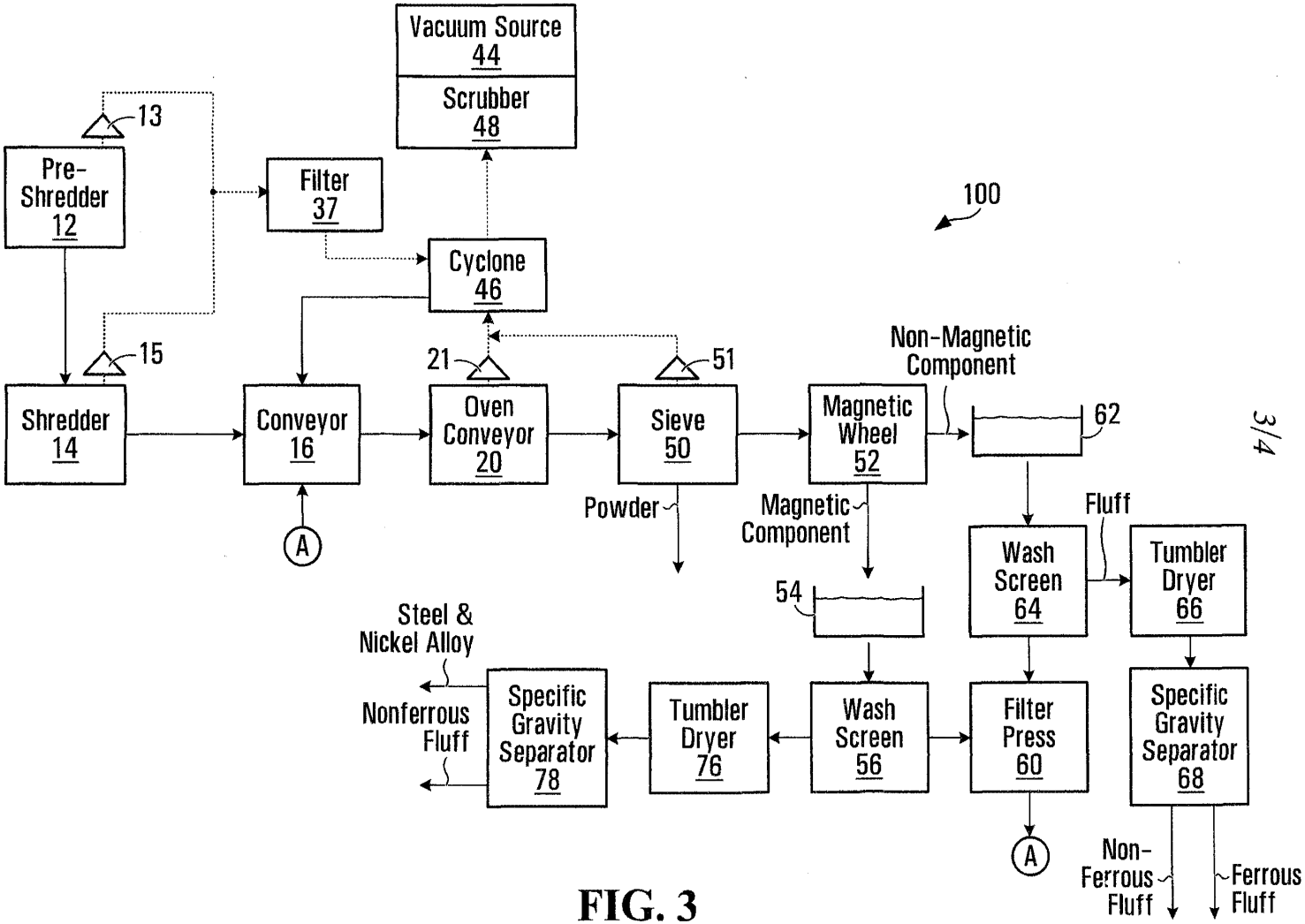
Vacuum Source
44
Scrubber
48
13
Pre-
Shredder
12
Filter
37
100
Cyclone
46
15
21
51
Non-Magnetic
Component
Shredder
14
Conveyor
16
Oven
Conveyor
20
Sieve
50
Magnetic
Wheel
52
62
A
Powder
Magnetic
Component
Fluff
Wash
Screen
64
Tumbler
Dryer
66
54
Steel &
Nickel Alloy
Nonferrous
Fluff
Specific
Gravity
Separator
78
Tumbler
Dryer
76
Wash
Screen
56
Filter
Press
60
Specific
Gravity
Separator
68
A
Non-
Ferrous
Fluff
Ferrous
Fluff

FIG. 3

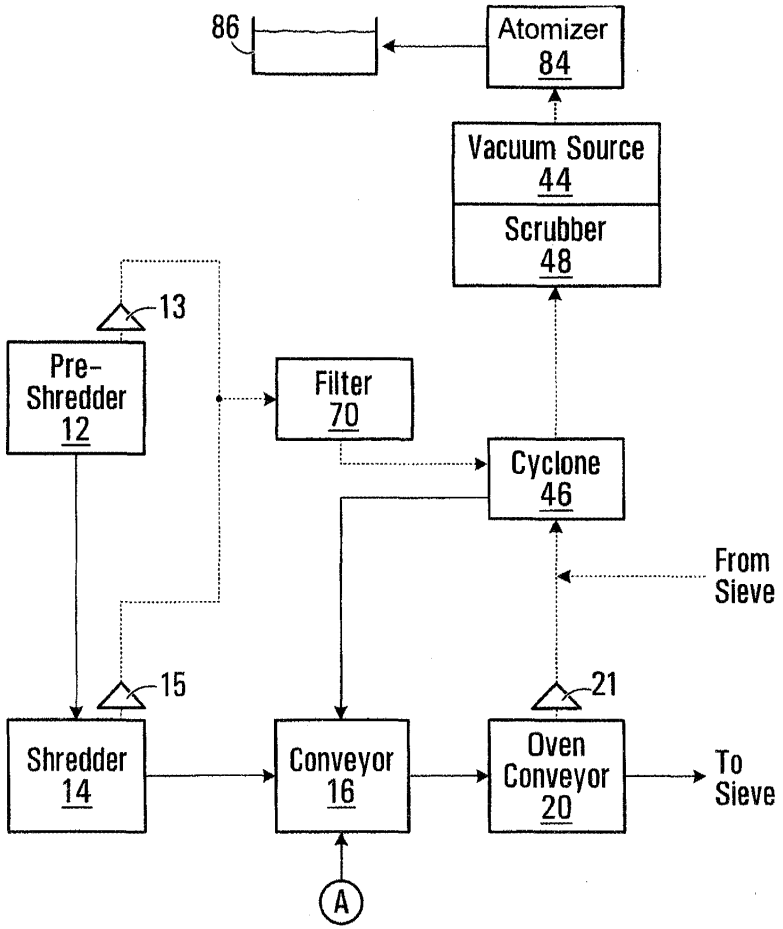
86
Atomizer
84
Vacuum Source
44
Scrubber
48
1000
13
Pre-
Shredder
12
Filter
70
Cyclone
46
From
Sieve
15
21
Shredder
14
Conveyor
16
Oven
Conveyor
20
To
Sieve
A

FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2009/001583

A. CLASSIFICATION OF SUBJECT MATTER
IPC: ***H01M 6/52*** (2006.01) , ***A62D 3/30*** (2007.01) , ***B02C 23/08*** (2006.01) , ***B02C 23/14*** (2006.01) , ***H01M 10/54*** (2006.01)
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
No classification restrictions

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
Canadian Patent Database, Intellect, Epoque, Delphion, Patent Abstracts of Japan

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,026,477 (Tremolada) 31 May 1977 (31.05.1977) col.1, lines 10-12, 32-45, Example, claim 1	1
Y		7
P, Y	US 2009/0095127 A1 (Petterson) 16 April 2009 (16.04.2009)	7
P, A	whole description and Figures 1-2	1-13
Y	WO 2009/028795 A2 (IM, Hyong Hag) 05 March 2009 (05.03.2009)	7
A	abstract, [12], [17] - [51] and claims	1-13
A	US 6,686,086 B1 (Martin et al) 03 February 2004 (03.02.2004) abstract, col.1, lines 18-39, col.2, lines 60-63, col.4, lines 35-37, col.6, line 5 - col.7, line 4 and claim 1	1-5, 14 and 15
A	CA 2,418,527 (BATREC INDUSTRIE AG, CH) 01 August 2003 (01.08.2003) abstract, page 1 to page 10, line 14	1

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents :
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
27 January 2010 (27-01-2010)	01 February 2010 (01-02-2010)

Name and mailing address of the ISA/CA	Authorized officer
Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 001-819-953-2476	Irina Kargina (819) 997-3059

Ascend Elements EX1002 Page 291

Duesenfeld Ex. 2014-291

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2009/001583

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CA 2,391,865 (AVESTOR) 21 March 2002 (21.03.2002) abstract, whole description	1

Ascend Elements EX1002 Page 292

Duesenfeld Ex. 2014-292

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CA2009/001583

Patent Document Cited in Search Report	Publication Date	Patent Family Member(s)	Publication Date
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		BE675058A	03-05-1966
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		EP1269554A1	02-01-2003
		JP2004508694T	18-03-2004
		US7192564B2	20-03-2007
		US2004028585A1	12-02-2004
		WO0223651A1	21-03-2002

Ascend Elements EX1002 Page 293

Duesenfeld Ex. 2014-293

(19)中华人民共和国国家知识产权局

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(43)申请公布日 2014.07.30

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(22)申请日 2012.08.10
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2014.03.12
(86)PCT国际申请的申请数据
PCT/DE2012/000811 2012.08.10
(87)PCT国际申请的公布数据
WO2013/023640 DE 2013.02.21
(71)申请人 莱昂工程公司
地址 德国不伦瑞克
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A·夸德
(74)专利代理机构 永新专利商标代理有限公司 72002
代理人 侯鸣慧
(51)Int.Cl.
H01M 10/54(2006.01)
B07B 4/08(2006.01)

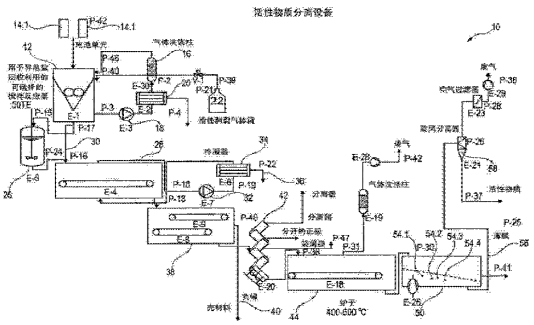
权利要求书2页 说明书6页 附图2页

(54)发明名称

用于从伽伐尼电池回收活性材料的方法和活性材料分离设备、尤其活性金属分离设备

(57)摘要

本发明涉及一种用于回收伽伐尼电池的活性材料的方法，所述电池包括活性材料、用于所述活性材料的载体和用于连接所述活性材料和所述载体的粘合剂，所述方法具有以下步骤：(a)尤其在惰性气体下或在真空中粉碎所述电池，从而至少也产生固体的电池碎片，(b)加热所述固体的电池碎片到分解温度(T_z)，所述分解温度选择得如此高，使得所述粘合剂分解和/或蒸发，优选在惰性气体下或在真空中，从而产生已热处理的电池碎片，和(c)对已热处理的电池碎片分类，其中，(d)所述分类包括空气喷气筛子并且(e)所述空气喷气筛子如此实施，使得活性材料从所述载体分离。



CN 103959553 A

1. 用于回收伽伐尼电池的活性材料的方法，所述电池包括活性材料、用于所述活性材料的载体和用于连接所述活性材料和所述载体的粘合剂，所述方法具有以下步骤：

（a）尤其在惰性气体下或在真空中粉碎所述电池，从而至少也产生固体的电池碎片，

（b）加热所述固体的电池碎片到分解温度（T_z），所述分解温度选择得如此高，使得所述粘合剂分解和/或蒸发，优选在惰性气体下或在真空中，从而产生已热处理的电池碎片，和

（c）对已热处理的电池碎片分类，

其特征在于，

（d）所述分类包括空气喷气筛选，

（e）其中，所述空气喷气筛选如此实施，使得活性材料从所述载体分离。

2. 根据权利要求 1 所述的方法，其特征在于，

- 使用伽伐尼电池（14），所述电池的活性材料由颗粒构成，

- 所述颗粒具有颗粒直径分布，其中，所述颗粒直径分布具有 90% 直径，所述 90% 直径相当于大于全部颗粒的 90% 的直径并且小于全部颗粒的 10% 的直径的那个直径，和

- 空气喷气筛选通过筛子进行，所述筛子的筛孔径最大相当于所述 90% 直径的 200 倍。

3. 根据上述权利要求之一所述的方法，其特征在于，所述筛子的筛孔径为最大 200 微米。

4. 根据上述权利要求之一所述的方法，其特征在于，所述伽伐尼电池（14）每个包括至少一个正极和至少一个负极并且所述正极和负极共同被加热，并且，产生的热处理过的电池碎片在一共同的空气喷气筛子（50）中被分类。

5. 根据上述权利要求之一所述的方法，其特征在于以下步骤，

（a2）在加热所述电池碎片到所述分解温度（T_z）之前

- 在干燥温度（T_t）下干燥所述电池碎片，从而产生干燥的电池碎片和干燥蒸汽并且

- 所述干燥蒸汽的至少一部分出于回收所述电解质溶液介质的目的被冷凝。

6. 根据上述权利要求之一所述的方法，其特征在于，加热所述电池碎片到所述分解温度（T_z）在惰性气体下或在真空中实施。

7. 根据上述权利要求之一所述的方法，其特征在于以下步骤，

（a3）借助于磁性分离器处理所述干燥的电池碎片，从而产生不含铁的电池碎片，和

（a4）风力风选所述不含铁的电池碎片。

8. 根据上述权利要求之一所述的方法，其特征在于，所述伽伐尼的电池（14）具有电解质和/或当在惰性气体下粉碎所述电池时至少也排出所述电解质。

9. 根据上述权利要求之一所述的方法，其特征在于，所述电池的粉碎如此实施，使得产生的薄膜碎片的最长直径的平均值不低于 200 微米。

10. 活性材料分离设备、尤其活性金属分离设备（10），其被构造用于回收伽伐尼电池（14）的活性材料、尤其锂，所述电池包括活性材料、用于所述活性材料的载体、用于连接所述活性材料和所述载体的粘合剂和电解质，所述活性材料分离设备具有

（a）用于粉碎所述电池的电池粉碎装置（12），其中，所述电池粉碎装置是不透气的并且包括惰性气体供给设备，从而所述电池能够在惰性气体下粉碎成电池碎片，和

（b）炉子（44），所述炉子至少间接地与所述电池粉碎装置（12）连接并且被设置用于加热所述电池碎片到分解温度（T_z），所述分解温度选择得如此高，使得所述粘合剂分解，

Ascend Elements EX1002 Page 295

Duesenfeld Ex. 2014-295

其特征在于，

（c）空气喷气筛装置，所述空气喷气筛装置至少间接地与所述炉子（44）连接并且被设置用于对活性材料和载体分类，其方式是，所述空气喷气筛选如此实施，使得所述活性材料与所述载体分开。

11. 根据权利要求 10 所述的活性材料分离设备，其特征在于，所述空气喷气筛装置具有筛子、挡板和喷嘴板条，其中，所述筛子相对于穿过所述空气喷气筛装置的气流布置在所述喷嘴板条和挡板之间。

12. 根据权利要求 11 所述的活性材料分离设备，其特征在于，所述挡板具有与所述筛子至少 30 微米、尤其至少 40 微米的间距。

13. 根据权利要求 11 或 12 所述的活性材料分离设备，其特征在于，所述挡板具有与所述筛子至多 250 微米、尤其至少 200 微米的间距。

Ascend Elements EX1002 Page 296

Duesenfeld Ex. 2014-296

用于从伽伐尼电池回收活性材料的方法和活性材料分离设备、尤其活性金属分离设备

[0001] 本发明涉及一种用于从伽伐尼电池回收活性材料、尤其锂或锂化合物的方法，所述电池包括活性材料、用于该活性材料的载体、用于连接所述活性材料和所述载体的粘合剂和必要时电解质，所述方法具有以下步骤：(a)尤其在惰性气体下粉碎所述电池，从而至少也产生固体的电池碎片，(b)加热所述固体的电池碎片到分解温度(T_z)，该分解温度选择得如此高，使得所述粘合剂分解和/或蒸发，从而产生已热处理的电池碎片，和(c)对已热处理的电池碎片分类。

[0002] 根据第二方面，本发明涉及一种活性金属分离设备，该活性金属分离设备被构造用于回收伽伐尼电池的活性材料、尤其锂，所述电池包括活性材料、用于所述活性材料的载体、用于连接所述活性材料和所述载体的粘合剂和电解质，所述活性材料分离设备具有(a)用于粉碎所述电池的电池粉碎装置，其中，所述电池粉碎装置是不透气的并且包括惰性气体供给设备，从而所述电池能够在惰性气体下粉碎成电池碎片，和(b)炉子，该炉子至少间接地与所述电池粉碎装置连接并且被设置用于加热所述电池碎片到分解温度，该分解温度如此高地选择，使得所述粘合剂分解。

[0003] 在从锂蓄电池或电池中回收活性材料、尤其锂时，存在化学过程相对费事的问题。对此，原因在于，仅仅不充分地达到所述活性材料与其载体的分开。

[0004] 从US2005/0241943A1中已知一种方法，其中，电池首先被粉碎并且然后进行热处理。然后，电池被粉碎并且加热到分解温度。接着，已热处理的电池碎片通过震动筛选分开。然而表明，该方法是费事的。本发明的任务在于，改进从旧的电池或蓄电池中回收活性材料、尤其锂或锂化合物。

[0005] 从DE19954998A1中已知一种切割碾磨机，其中，由切割转子切割的物料连续地如此输送穿过筛子、尤其空气喷气筛子，使得仅仅未超过预给定大小的颗粒能够离开所述装置。

[0006] 从DE29803442U1中已知一种用于分析的空气喷气筛子，通过该空气喷气筛子能够在较短的时间内实施高数量的分析并且出于该目的，所述空气喷气筛子具有多个彼此相对分开的筛面。该空气筛子用于确定在微颗粒范围内的颗粒大小分布。

[0007] 从AT386769B中已知一种空气喷气多层筛机，借助于该空气喷气多层筛机能够在一工序中把干燥的、散粒状的小颗粒松散物料分类成至多25组分。

发明内容

[0008] 本发明通过所述类型的方法解决所述问题，其中，所述分类包括空气喷气筛选。根据第二方面，本发明通过活性金属分离设备解决所述问题，该活性金属分离设备具有空气喷气筛装置，该空气喷气筛装置至少间接地与所述炉子连接并且被设置用于对活性材料和载体、尤其载体薄膜分类。

[0009] 本发明的优点是，能够以高纯度获得活性材料。其原因在于，在空气喷气筛选时能够使用非常小的筛孔径，从而也阻止小的杂质颗粒。

Ascend Elements EX1002 Page 297

Duesenfeld Ex. 2014-297

[0010] 更有利地，也能够以高纯度回收载体材料。例如经常使用铝薄膜作为载体，只有当所述铝薄膜在纯度方面满足某种最低要求时，才能够有效地回收所述铝薄膜。例如具有多于 0.03% 杂质的载体薄膜仅仅少量地适用于回收。在其他湿法冶金的加工中，当由一种酸性的锂溶液组成的过渡金属盐溶液在传统的分离中包含通常大约 1% 质量百分比的铝污染物时，该过渡金属盐溶液易于聚结。该方面的原因在于，形成牢固连接所述聚结物的氢氧化铝化合物。该固体聚结物在传统的过程中不能完全被磨碎，这导致材料损耗。相应地制成的被铝污染的过渡金属盐溶液则必须在磨碎之前高成本地提纯。在铝含量降低到 0.1% 以下时，不再能观察该效果。以仅仅 0.05 至 0.06% 质量百分比的铝污染物所达到的结果仍能够在生态上和经济上有利地回收所述材料。通过在空气喷气筛选时碰撞到筛子上时拍掉活性材料的方式，能够达到对于载体、尤其载体薄膜来说非常高的纯度。

[0011] 本发明基于以下知识，将电池碎片加热到分解温度虽然在理论上肯定足以如此程度地破坏粘合剂，使得活性材料被释放，但是，活性材料仍然很难地从载体脱离。是由于什么作用，活性材料在没有粘合剂的情况下也附着在载体上，至今还不清楚。

[0012] 在空气喷气筛选时发生，具有还附着的活性材料的载体猛烈地碰撞到所述筛子上。该碰撞分离还附着的活性材料，从而它能够被回收。

[0013] 由于所述载体碰撞到所述筛子上和由此导致的分离作用，所述空气喷气筛选也可以被称为碰撞分离。类似地，所述空气喷气筛装置也可以被称为碰撞分离装置并且所述空气喷气筛也可以被称为碰撞分离器。

[0014] 因此，根据本发明，也提出一种活性材料分离设备，该活性材料分离设备是碰撞分离装置，该碰撞分离装置至少间接地与所述炉子连接并且被设置用于对活性材料和载体分类，其方式是，空气喷气筛选如此实施，使得所述活性材料至少也通过碰撞到所述碰撞分离装置的筛子上和 / 或碰撞到挡板上而与所述载体分开。

[0015] 已知所述空气喷气筛选作为用于分开小颗粒与其他小颗粒的实验室方法。然而，已热处理的电池碎片具有在数量级上比所述活性材料的颗粒更大的直径，从而用于分开分别具有小直径的两种类型的颗粒的空气喷气筛选的能力对于根据本发明的方法意义不大。在根据本发明的方法中，所述空气喷气筛选也作为附加的分离步骤使用。

[0016] 在本说明书的范围内，所述电池的粉碎尤其理解为切碎、打碎、剁碎或压碎。这能够在 -195.79℃至 100℃之间的温度进行，从而在温度管理方面仅仅存在低要求。

[0017] 固体电池碎片的加热尤其理解为在不透气的容器中的加热，从而防止例如氧气进入。所述加热尤其在惰性气体下或在真空中进行。

[0018] 所述活性材料尤其理解为能够可逆地吸收和放出锂的化合物。在负极侧上例如是锂镍锰钴氧化物、锂镍铝氧化物和锂铁磷酸盐，在正极侧上例如是石墨。

[0019] 用于活性材料的载体尤其理解为这样的载体薄膜，在该载体薄膜上施加呈颗粒形状的活性材料。所述载体薄膜例如为铝薄膜。所述粘合剂例如为聚偏氟乙烯（PVDF）。

[0020] 电池的粉碎和 / 或固体电池碎片的加热优选在惰性气体下、例如在氮气下进行。有益的是，电池的粉碎在环境温度的情况下、尤其在最高 30℃的温度下进行，其中，有利的可以是，冷却粉碎电池所用的粉碎装置，从而对电池的热影响保持低下。

[0021] 根据本发明的方法优选实施为连续的过程，也就是非按批次的。

[0022] 优选地，生产量为大于每年 100 吨。所述空气喷气筛选至今在实验室范围内使用，

Ascend Elements EX1002 Page 298

Duesenfeld Ex. 2014-298

在那里所述空气喷气筛选用于批量地处理样品。此外，所述样品非常小。所述空气喷气筛选在大量方法中的使用至今未知。

[0023] 根据优选实施方式使用伽伐尼电池，所述电池的活性材料由颗粒构成，其中，所述颗粒具有颗粒直径分布并且其中，该颗粒直径分布具有90%直径，该90%直径相当于大于全部颗粒的90%的直径并且小于全部颗粒的10%的直径的那个直径，其中，空气喷气筛选通过筛子进行，所述筛子的筛孔径最大相当于所述90%直径的200倍。提出的颗粒直径是通过筛选得到的那个颗粒直径。

[0024] 优选地，所述筛子的筛孔径为最高250微米、尤其最高100微米。现代的锂离子电池具有这样的活性材料，该活性材料由具有小于200微米的直径的颗粒构成。在传统的筛选方法、例如在震动筛选中，必须选择明显较大的筛孔径，以使筛选时间不变得如此长，使得所述方法变得不经济。然而，较大的筛孔径导致，较多的杂质颗粒穿过所述筛子，从而获得的活性材料的纯度下降。

[0025] 优选地，所述伽伐尼电池每个包括至少一个正极和至少一个负极，其中，所述正极和负极共同被加热并且产生的热处理过的电池碎片在一共同的空气喷气筛子中被分类。所述正极和负极共同被加热的特征理解为由两者组成的混合物在同一个装置中被加热。这是可能的，因为所述空气喷气筛选导致足够种类纯粹地分开集流薄膜和涂层颗粒。

[0026] 优选地，所述方法包括以下步骤，在加热电池碎片到所述分解温度之前，在干燥温度下干燥所述电池碎片，从而产生干燥的电池碎片和干燥蒸汽，其中，所述干燥蒸汽的至少一部分被冷凝。这具有能够回收所述电解质溶液介质的优点。在此，有利的是，干燥温度处于100℃以下、尤其80℃以下。

[0027] 优选地，加热所述电池碎片到所述分解温度在惰性气体下或在真空中实施。这具有铜不被氧化的优点，从而不仅正极而且负极能够共同被处理。这又导致特别简单的分离方法，该分离方法仍提供高种类纯粹度的回收产物。惰性气体理解为在现有的环境条件下既不与铜也不与铝(集流薄膜)反应的气体或气体混合物。铜则能够作为贵重成分以高纯度被回收。

[0028] 优选地，所述方法包括借助于磁性分离器处理所述干燥的电池碎片的步骤，从而产生不含铁的电池碎片。有利的是，所述不含铁的电池碎片用风力风选，例如通过锯齿形风力风选机。然后，所述不含铁的电池碎片被加热到所述粘合剂的分解温度。

[0029] 所述分解温度优选为至少350℃和最高800℃。已经证实，在该温度下粘合剂可靠地被分解。

[0030] 优选地，在惰性气体下粉碎所述电池时至少也排出所述电解质介质。这提高了回收率。

[0031] 根据优选的实施方式，所述电池的粉碎如此实施，使得产生的薄膜碎片的最长直径的平均值不低于200微米。根据优选的实施方式，所述电池的粉碎如此实施，使得所述薄膜碎片至少80%具有超过200微米的直径，其中，该直径通过筛选确定。

[0032] 根据优选的实施方式，所述空气喷气筛选如此进行，使得达到每小时每平方米筛面积至少250立方米空气、尤其每小时每平方米筛面积至少500立方米空气的空气通过量。特别有利的是，每小时每平方米筛面积至少750立方米空气。然后，得到如此高的颗粒速度，使得所述碰撞导致高的分离度。

Ascend Elements EX1002 Page 299

Duesenfeld Ex. 2014-299

[0033] 优选地，在所述空气喷气筛子的后面布置有旋风分离器。此外，有利的是，载体空气在空气喷气筛子的后面被吸走。

[0034] 为了尽可能小的能量消耗，有利的是，至少在很大程度上整面地以含颗粒的气流加载所述筛子。

[0035] 所述活性材料分离设备优选具有预干燥器，该预干燥器沿材料流布置在所述电池粉碎装置的后面并且在所述炉子的前面。所述预干燥器被设置用于加热所述电池碎片到干燥温度。

[0036] 此外，优选所述活性材料分离设备包括沿材料流在所述预干燥器的后面并且在所述炉子的前面的磁性分离器。

[0037] 有利的是，所述活性材料分离设备具有用于导电盐回收的搅拌反应器，该搅拌反应器与所述电池粉碎装置连接并且从所述电池粉碎装置中排出所述液态组分的至少一部分。

[0038] 用于回收所述伽伐尼电池的活性材料的方法尤其也理解为这样的方法，其中，加工由电极生产形成的电极，而所述电极没有装入伽伐尼电池中，但能装入伽伐尼电池中。换句话说，本方法也可以是用于过程中回收的方法。

[0039] 根据优选的实施方式，相对于气流和电池碎片流在所述筛子的后面布置有挡板。这导致仅仅与所述筛子有微弱的碰撞的活性材料颗粒在碰撞到所述挡板上时从载体上分离，这提高了分离作用。

[0040] 根据优选的实施方式，所述电池粉碎装置包括至少一个碰撞碾磨机。通过所述碰撞碾磨机中的大量的相撞过程使活性材料和载体之间的结合松动。所述电池粉碎装置优选还包括至少一个破碎机和/或一个切碎装置，所述切碎装置特别有利地沿材料流方向布置在碰撞碾磨机之前。

[0041] 此外，根据本发明，在用于例如从旧的电池和/或蓄电池中以及从生产电池和/或蓄电池时的废品中回收活性材料、尤其锂和/或锂化合物的方法、尤其连续的方法中，使用空气喷气筛子。

[0042] 以下借助附图详细解释本发明。在此示出：

[0043] 图 1 用于实施根据本发明的方法的根据本发明的活性材料分离设备的流程图，和

[0044] 图 2 根据图 1 的活性材料分离设备的碰撞分离器的实施方式的分解图。

[0045] 图 1 示出根据本发明的活性材料分离设备 10 的流程图，该活性材料分离设备 10 具有电池粉碎装置 12，给该电池粉碎装置供应伽伐尼电池 14.1、14.2、……。所述电池粉碎装置 12 是不透气的并且包括气体洗涤柱 16、循环泵 18 和冷凝器 20，借助于它们循环和净化惰性气体、例如氮气。此外，电池粉碎装置包括惰性气体箱 22，用于调节电池粉碎装置 12 中的惰性气体压力。冷凝器 20 具有电解质排出装置 24，通过该电解质排出装置能够排出已冷凝的电解质。

[0046] 在材料流动方向上在电池粉碎装置 12 的后面能够布置有搅拌反应器 26，该搅拌反应器被布置用于从电池粉碎装置 12 中排出液态组分并且用于搅拌它们，从而能够从伽伐尼电池 14（没有数字后缀的附图标记表示对象本身）中回收导电盐。

[0047] 在材料流动方向上在电池粉碎装置 12 的后面布置有预干燥器 28，该预干燥器通过管道 30 与所述电池粉碎装置 12 连接。在粉碎所述伽伐尼电池 14 时所产生的电池碎片

Ascend Elements EX1002 Page 300

Duesenfeld Ex. 2014-300

通过管道 30 到达预干燥器 28 中。在那里，存在优选 T_t=80℃的干燥温度 T_t。预干燥器 28 包括循环泵 32 和用于冷凝电解质的冷凝器 34，该电解质能够通过管道 36 排出。预干燥器 28 不透气地构造并且借助惰性气体通过循环泵 32 冲洗。

[0048] 在材料流动方向上在预干燥器 28 的后面布置有分离器 38，该分离器包括磁性分离器和必要时附加包括涡流分离器。磁性的材料通过排出管道 40 排出。非磁性的材料到达风力风选机 42、例如锯齿形风力风选机中。风力风选机 42、如分离器 38 是可选的并且特别当如在现有情况下仅仅应该继续使用伽伐尼电池的负极时是有利的。正极同样如必要时存在的袋薄膜那样被取出。

[0049] 在材料流动方向上在风力风选机 42 的后面布置有炉子 44，在该炉子中存在 400 至 600℃的分解温度 T_z。由此，分解粘合剂、例如聚偏氟乙烯（PVDF）。分解产物通过排出管道 46 排出并且导入气体洗涤柱 48 中。

[0050] 在材料流动方向上在炉子 44 的后面布置有空气喷气筛子 50。该空气喷气筛子 50 包括筛子元件 52。来自炉子 44 的已热处理的电池碎片被输送到空气喷气筛子上。该空气喷气筛子的特征在于，在细筛孔的筛子元件（20-200 微米）的上方或下方供给气流。该气流在筛子的下方排出。由于空气流动，电极碎片被卷起并且然后被机械式地负荷。该负荷支持载体薄膜和涂层的分离。活性材料颗粒则在气流中通过筛孔排出并且随后通过旋风分离器从气流中分离。薄膜组分（铝和铜）通过筛子元件挡住并且能够作为金属组分被获得并且被重新使用。

[0051] 图 2 示出碰撞分离器或空气喷气筛子的分解图。待分开的已热处理的电池碎片供应到筛子的一侧上，从另一侧将运动的空气喷气例如通过运动的喷嘴板条向筛子的方向吹。由此，电池碎片被抛向呈挡盖形式的挡板并且在那里分成载体和涂层。

[0052] 空气喷气从筛子的一侧被吹到筛子上，在该侧存在吸气接管，借助于该吸气接管吹入的空气与涂层一起排出。挡板通过隔离环与筛子间隔地保持。

[0053] 附图标记列表

[0054] 10 活性材料分离设备

[0055] 12 电池粉碎装置

[0056] 14 伽伐尼电池

[0057] 16 气体洗涤柱

[0058] 18 循环泵

[0059] 20 冷凝器

[0060] 22 惰性气体箱

[0061] 24 电解质排出装置

[0062] 26 搅拌反应器

[0063] 28 预干燥器

[0064] 30 管道

[0065] 32 循环泵

[0066] 34 冷凝器

[0067] 36 管道

[0068] 38 分离器

Ascend Elements EX1002 Page 301

Duesenfeld Ex. 2014-301

[0069] 40 排出管道

[0070] 42 风力风选机

[0071] 44 炉子

[0072] 46 排出管道

[0073] 48 气体洗涤柱

[0074] 50 空气喷气筛子

[0075] 52 筛子元件

[0076] 54 颗粒

[0077] 56 排出管道

[0078] 58 旋风分离器

[0079] T_t 干燥温度

[0080] T_z 分解温度

Ascend Elements EX1002 Page 302

Duesenfeld Ex. 2014-302

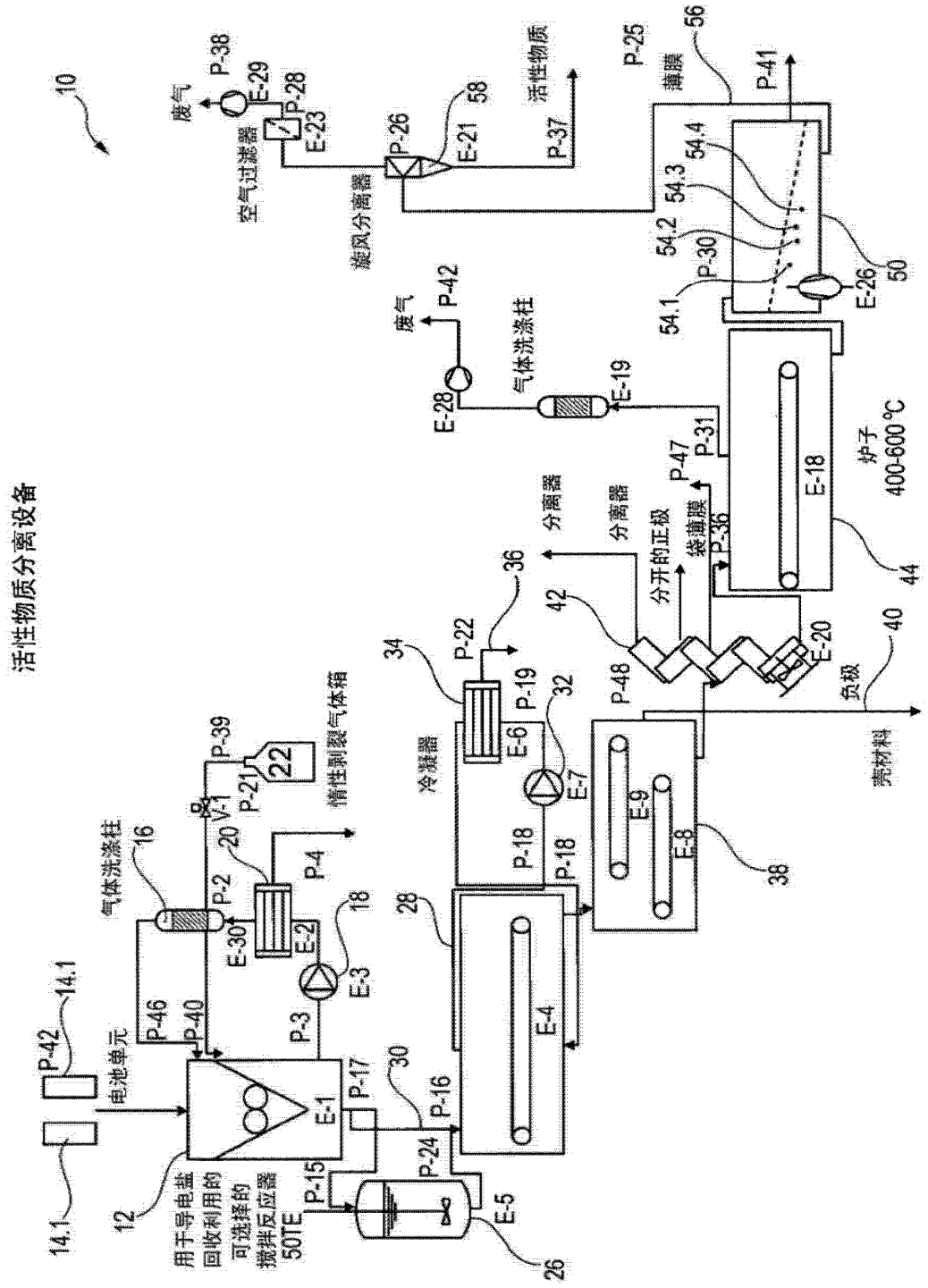


图 1

Ascend Elements EX1002 Page 303

Duesenfeld Ex. 2014-303

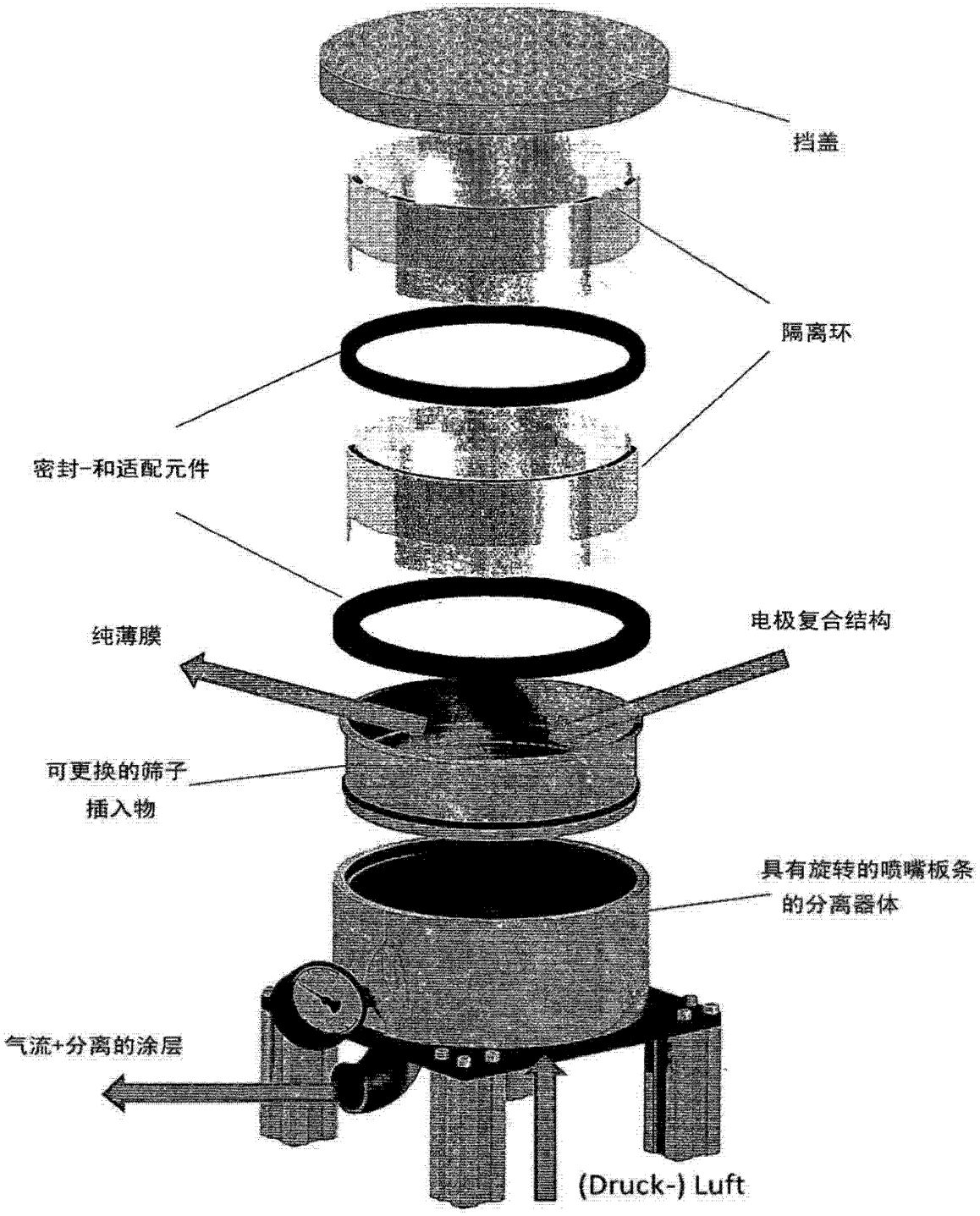


图 2

Ascend Elements EX1002 Page 304

Duesenfeld Ex. 2014-304

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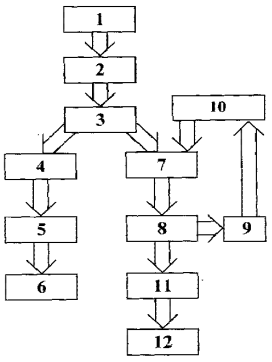
(54)发明名称

一种回收废旧锂离子电池电解液的方法

(57)摘要

本发明为一种回收废旧锂离子电池电解液的方法。所述的方法主要包括以下步骤：将收集的锂离子电池清洁干净，放电后放入干燥间或惰性气体保护的手套箱中。把电池打开，将电解液小心取出放入料罐中，高真空减压精馏分离得到电解液所含有机溶剂，精馏纯化后回收。将六氟磷酸锂粗品放入溶解釜中，加入氟化氢溶液溶解回收的六氟磷酸锂。然后将该溶液过滤后放入结晶釜中进行结晶提纯，筛分，干燥，包装，回收得到产品六氟磷酸锂。本方法工艺简单、实用高效、易于控制且清洁环保，实现了经济效益和环境社会效益的紧密结合。



CN 102496752 A

1. 一种回收废旧锂离子电池电解液的方法；其特征在于：

包括以下步骤：

1) 将收集的各种锂离子电池，包括钴酸锂电池、磷酸亚铁锂电池、锰酸锂电池、三元材料电池；以及在锂离子电池生产制造过程中产生的不符合使用标准的残次品锂离子电池；都清洁干净；使用前将锂离子电池充分放电；

2) 把电池和准备好的料罐一起放入干燥间或惰性气体保护的手套箱中；将电池打开，将其中的电解液小心取出放入料罐中；干燥间水分在 1% -2%间；手套箱中所使用的惰性气体包括：高纯氮气、高纯氦气、高纯氖气、高纯氩气，其水分含量在 1% -100ppm 之间；用于保护电解液，防止发生水解反应和氧化反应；

3) 然后高真空减压精馏分离得到电解液所含有机溶剂；使用有机溶剂洗涤电池，采用高真空精馏技术处理回收的六氟磷酸锂溶液，有效防止六氟磷酸锂的分解副反应；精馏操作的条件：真空度 0 至 20 千帕，温度在 20 至 120℃之间，采用连续精馏或者间歇精馏方法操作；回收的有机溶剂为碳酸乙烯酯、碳酸丙烯酯、碳酸二甲酯、碳酸二乙酯、碳酸甲乙酯、γ- 丁内酯、四氢呋喃、碳酸亚乙烯酯中的一种或几种混合物；

4) 将得到固体六氟磷酸锂物料加入六氟磷酸锂溶解釜中，加入氟化氢溶液溶解回收的六氟磷酸锂；溶解六氟磷酸锂粗品过程中所使用的氟化氢采用市售、一般级别、蒸馏提纯后的氟化氢或者生产六氟磷酸锂结晶过程中取到的母液；在氟化氢溶液中溶解六氟磷酸锂粗品时；温度没有特殊要求，在 20℃至 -30℃之间；

5) 将该溶液过滤后放入结晶釜中进行结晶提纯；温度 -80℃到 10℃之间；结晶时间在 4 小时到 48 小时之间；结晶分离方法采用自然过滤、或加压过滤、或离心过滤方法；

6) 筛分，干燥，干燥操作过程中的温度在 40℃到 140℃之间；干燥的时间在 4 小时到 48 小时之间；然后包装，得到六氟磷酸锂产品。

Ascend Elements EX1002 Page 306

Duesenfeld Ex. 2014-306

一种回收废旧锂离子电池电解液的方法

技术领域

[0001] 本发明属于锂离子电池回收技术领域，具体涉及一种从废旧锂离子电池电解液中回收六氟磷酸锂和溶剂的方法。

背景技术

[0002] 锂离子电池自 1990 年商业化以来，以其电压高、容量大、质量轻等优点逐渐取代传统二次电池成为市场的主流，在手机、数码相机、数码摄像机、笔记本电脑等电子产品中得到广泛地应用。2000 年世界锂离子电池的消费量为 5 亿只，2008 年达到了 28 亿只，随着科技的发展和时代的进步，以及电动汽车行业的迅猛发展，人们对锂离子电池的需求量急剧增加。锂离子电池属于易耗品，预计未来每年将产生 10 万吨以上的废旧锂离子电池。锂离子电池主要由正极、负极、隔膜、电解液组成，含有钴、锰、铝、铜、锂、磷、氟等有用成分，如果不对锂离子电池所含有用成分进行回收，不但造成有用资源的极大浪费，而且将对环境造成严重污染。因此，回收处理锂离子电池成为全社会十分迫切的问题。

[0003] 锂离子电池经拆解得到的外壳、隔膜材料组成单一，易于回收。有关电池的正极、负极材料回收领域的文献专利较多，正极回收方法主要有 :1. 湿法冶金 ;2. 火法冶金 ;3. 电化学法等。负极回收方法主要有 :1. 高温法 ;2. 比重发 ;3. 粉碎法 ;4. 浸泡法等。CN200910017377.7、CN200910040963.3、CN201010262198.2、CN201010295586.0 等专利介绍了锂离子电池中所含有的有用成分回收方法，但并未提及电解液六氟磷酸锂的回收。

[0004] 锂离子电池电解液主要是各种有机溶剂溶解六氟磷酸锂形成的溶液。有关锂离子电池电解液领域回收利用的研究报道很少，我们知道，锂离子电池电解液中含有六氟磷酸锂和小分子有机化合物，如果不加处理将其随意放置，容易污染环境，甚至引起火灾。

[0005] 目前，针对锂离子电池的回收方法已经开展了相关的研究工作。CN201010523257.7 报道了一种从废旧锂离子电池及极片中回收锂的方法，通过将电池和极片破碎，高温去除粘结剂，加入酸、碱反应除杂，最后以氟盐沉淀得到锂化合物。CN201010605151.1 报道了一种废旧电池中锂的回收方法，通过采用有机溶剂处理废旧锂离子电池湿法处理过程中的含锂萃余液，将锂回收。CN201010141128.1 介绍采用酸溶锂离子电池电芯碎片，加碱分别析出金属氢氧化物，最后将含锂滤液中加入碳酸钠生成碳酸锂沉淀，从而回收电极中的锂化合物。这些方法存在着如下缺点 :1) 浪费原材料，能耗高、效率低、工艺复杂 ;2) 有些动力电池体积较大，处理不好容易起火燃烧，比较危险 ;3) 在回收电极材料过程中同时处理电解液，电解液中含有的六氟磷酸锂被分解，生成氟化物、磷化合物和锂化合物，和其他的金属离子混合在一起，后续处理困难，容易造成环境污染。因此，很有必要开发一种锂离子电池电解液所含有的六氟磷酸锂和溶剂的回收方法来解决这一问题。

[0006] 本专利针对锂离子电池资源化现状，采用环境友好的方法实现了电解液有用成分六氟磷酸锂和溶剂的资源化分离回收。六氟磷酸锂为热敏物料，在温度较高时易于分解，而且它性质非常活泼，很容易和空气、水分等发生副反应，因此本专利采用高真空精馏技术处理回收的六氟磷酸锂溶液。

Ascend Elements EX1002 Page 307

Duesenfeld Ex. 2014-307

发明内容

[0007] 本发明的目的在于提供一种工序简单、成本低廉、行之有效的锂离子电池电解液材料的回收方法，以实现对电解液中所含有的六氟磷酸锂和溶剂的回收利用。

[0008] 因此，针对上述技术问题，本发明提供一种从废旧锂离子电池中有效回收六氟磷酸锂和溶剂的方法，以简化传统回收工艺，减轻对外界自然环境造成的污染。

[0009] 本发明为一种回收废旧锂离子电池电解液的方法；其特征在于：

[0010] 包括以下步骤：

[0011] 1）将收集的各种锂离子电池，包括钴酸锂电池、磷酸亚铁锂电池、锰酸锂电池、三元材料电池；以及在锂离子电池生产制造过程中产生的不符合使用标准的残次品锂离子电池；都清洁干净；使用前将锂离子电池充分放电；

[0012] 2）把电池和准备好的料罐一起放入干燥间或惰性气体保护的手套箱中；将电池打开，将其中的电解液小心取出放入料罐中；干燥间水分在1%-2%间；手套箱中所使用的惰性气体包括：高纯氮气、高纯氦气、高纯氖气、高纯氩气，其水分含量在1%-100ppm之间；用于保护电解液，防止发生水解反应和氧化反应；

[0013] 3）然后高真空减压精馏分离得到电解液所含有机溶剂；使用有机溶剂洗涤电池，采用高真空精馏技术处理回收的六氟磷酸锂溶液，有效防止六氟磷酸锂的分解副反应；精馏操作的条件：真空度0至20千帕，温度在20至120度之间，采用连续精馏或者间歇精馏方法操作；回收的有机溶剂为碳酸乙烯酯、碳酸丙烯酯、碳酸二甲酯、碳酸二乙酯、碳酸甲乙酯、γ-丁内酯、四氢呋喃、碳酸亚乙烯酯中的一种或几种混合物；

[0014] 4）将得到固体六氟磷酸锂物料加入六氟磷酸锂溶解釜中，加入氟化氢溶液溶解回收的六氟磷酸锂；溶解六氟磷酸锂粗品过程中所使用的氟化氢采用市售、一般级别、蒸馏提纯后的氟化氢或者生产六氟磷酸锂结晶过程中取到的母液；在氟化氢溶液中溶解六氟磷酸锂粗品时；温度没有特殊要求，在20℃至-30℃之间；

[0015] 5）将该溶液过滤后放入结晶釜中进行结晶提纯；温度-80℃到10℃之间；结晶时间在4小时到48小时之间；结晶分离方法采用自然过滤、加压过滤、离心过滤方法；

[0016] 6）筛分，干燥，干燥操作过程中的温度在40℃到140℃之间；干燥的时间在4小时到48小时之间；然后包装，得到六氟磷酸锂产品。

[0017] 同现有技术相比较，本发明的优点在于：

[0018] 1. 本发明实现了对锂离子电池电解液的简单处理，不需要复杂的化学过程。整个工艺简单、高效、易于控制且清洁环保。

[0019] 2 本工艺回收的六氟磷酸锂经过纯化后可以直接使用。

[0020] 3 本工艺回收的有机溶剂经过纯化后可以直接使用。

[0021] 本发明所提及的废旧锂离子电池回收电解液有效成分的方法，分别得到电解质盐六氟磷酸锂和各种有机溶剂，经上述方法获得的化合物，可以直接作为锂离子电池材料加以利用，有效地实现了电解质盐、有机溶剂资源的回收利用。这种方法工艺简单，设备投入少，生产周期短、回收成本低，回收效率高，回收过程不会造成二次污染。对于降低电池生产成本、节约资源、保护环境都可起到积极的作用。

Ascend Elements EX1002 Page 308

Duesenfeld Ex. 2014-308

具体实施方式

[0022] 下面通过具体的实施方式，对本发明所述回收废旧锂离子电池电解液有效成分六氟磷酸锂和溶剂的方法进行进一步的详细说明。

[0023] 所述的废旧锂离子电池包括钴酸锂、磷酸亚铁锂、锰酸锂、三元材料等种类的锂离子电池。

[0024] 所述的有机溶剂回收过程包括间歇或者连续的减压精馏、高真空精馏等方式。

[0025] 所述的方法回收的溶剂包括如碳酸二甲酯、碳酸二乙酯、碳酸甲乙酯、γ－丁内酯、四氢呋喃、碳酸乙烯酯等。

附图说明

[0026] 图 1 是本发明的流程示意图：

[0027] 1、锂离子电池清洗放电；2、打开电池，取出电解液；3、减压精馏；4、有机溶剂；5 精馏；6、纯化回收利用；7、溶解六氟磷酸锂；8、结晶、过滤；9、母液回用；10、结晶母液；11、粉碎、筛分、干燥；12、成品。

具体实施方式

[0028] 实施例一、

[0029] 将锂离子电池清洁干净，进行完全放电处理。把电池和准备好的料罐一起放入高纯氮气保护的手套箱中，将电池打开，小心取出电解液放入料罐中；然后减压精馏分离得到电解液所含有机溶剂，回收利用。得到 2.9 公斤固体物料移出手套箱，放入六氟磷酸锂溶解釜中，加入 16 公斤生产六氟磷酸锂结晶过程中取到的母液溶解回收的六氟磷酸锂；将该溶液过滤后放入结晶釜中在零下 50 度 24 小时内进行结晶提纯；粉碎筛分，高纯氮气保护下 120 度在干燥机中干燥 8 小时，包装，得到产品六氟磷酸锂 2.4 公斤，回收率 82.7％。

[0030] 实施例二、

[0031] 将锂离子电池清洁干净，进行完全放电处理。把电池在干燥间打开，小心取出电解液放入料罐中；然后减压分离得到电解液所含有机溶剂，回收利用。得到 3.1 公斤固体物料放入六氟磷酸锂溶解釜中，加入 17 公斤例 1 操作中得到的氟化氢母液，溶解回收的六氟磷酸锂；将该溶液过滤后放入结晶釜中在零下 65 度 36 小时内进行结晶提纯；粉碎筛分，高纯氮气保护下 130 度在干燥机中干燥 12 小时，包装，得到产品六氟磷酸锂 2.5 公斤，回收率 80.6％。

Ascend Elements EX1002 Page 309

Duesenfeld Ex. 2014-309

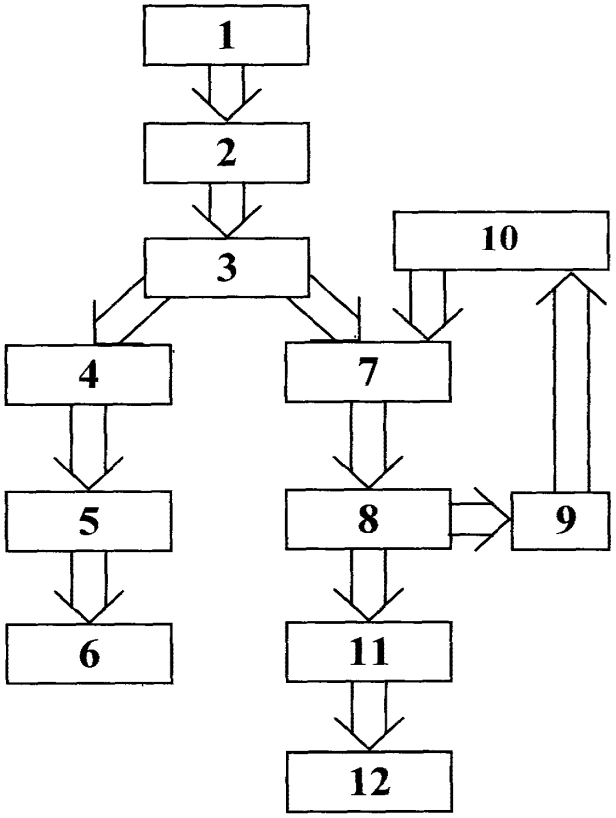


图 1

Ascend Elements EX1002 Page 310

Duesenfeld Ex. 2014-310

Abstract of CN 102496752 A

The invention discloses a method for recycling the electrolyte of a waste lithium ion battery. The method mainly comprises the following steps of: collecting and cleaning the lithium ion battery; after discharge, putting the lithium ion battery in a drying room or a glove box under protection of inert gas; opening the battery, carefully taking out the electrolyte and placing in a material tank; performing high-vacuum decompressed rectification and separation to obtain the organic solvent contained in the electrolyte; performing rectification and purification for recycling; putting a rough product of lithium hexafluorophate into a dissolution kettle, and adding a hydrogen fluoride solution for dissolving the recycled lithium hexafluorophate; filtering the solution and then putting into a crystallization kettle for crystallization and purification; and screening, drying, packaging and recycling to obtain the product lithium hexafluorophate. The method has simple technology, is practical, efficient, clean and environment-friendly and easy to control, and realizes close integration of economic benefits and environmental social benefits.

Electronic Acknowledgement Receipt

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First Named Inventor/Applicant Name:	Christian HANISCH
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Ascend Elements EX1002 Page 312

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The time period for reply, if any, is set in the attached communication.

Office Action Summary	Application No. 15/569,484	Applicant(s) HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	Art Unit 3725	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☑ Responsive to communication(s) filed on 16 May 2019.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL.** 2b) ☑ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5) ☑ Claim(s) 1-9 and 11-20 is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6) ☐ Claim(s) _____ is/are allowed.

7) ☑ Claim(s) 1-3,5-9 and 12-19 is/are rejected.

8) ☑ Claim(s) 4,11 and 20 is/are objected to.

9) ☐ Claim(s) _____ are subject to restriction and/or election requirement

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov.**

Application Papers

10)☑ The specification is objected to by the Examiner.

11)☑ The drawing(s) filed on 26 October 2017 is/are: a)☐ accepted or b)☑ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☑ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☑ All b)☐ Some** c)☐ None of the:

1.☑ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☑ Notice of References Cited (PTO-892)

2) ☑ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date Attached.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date _____.

4) ☑ Other: See Continuation Sheet.

Continuation of Attachment(s) 4) Other: DE 4424825 and Machine Translation; WO 2013/023640 and Machine Translation

DETAILED ACTION

Notice of Pre-AIA or AIA Status

1) The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

2) In the event the determination of the status of the application as subject to AIA 35 U.S.C. 102 and 103 (or as subject to pre-AIA 35 U.S.C. 102 and 103) is incorrect, any correction of the statutory basis for the rejection will not be considered a new ground of rejection if the prior art relied upon, and the rationale supporting the rejection, would be the same under either status.

3) This application currently names joint inventors. In considering patentability of the claims the examiner presumes that the subject matter of the various claims was commonly owned as of the effective filing date of the claimed invention(s) absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and effective filing dates of each claim that was not commonly owned as of the effective filing date of the later invention in order for the examiner to consider the applicability of 35 U.S.C. 102(b)(2)(C) for any potential 35 U.S.C. 102(a)(2) prior art against the later invention.

Priority

4) Acknowledgment is made of applicant's claim for foreign priority under 35 U.S.C. 119 (a)-(d).

5) Receipt is acknowledged of certified copies of papers required by 37 CFR 1.55.

Information Disclosure Statement

6) The information disclosure statements (IDS) are in compliance with the provisions of 37 CFR 1.97. Accordingly, the information disclosure statements are being considered by the examiner.

Drawings

7) The drawings are objected to because of the following formalities:

- The text and lines of the drawings is unclear

8) Corrected drawing sheets in compliance with 37 CFR 1.121(d) are required in reply to the Office action to avoid abandonment of the application. Any amended replacement drawing sheet should include all of the figures appearing on the immediate prior version of the sheet, even if only one figure is being amended. The figure or figure number of an amended drawing should not be labeled as "amended." If a drawing figure is to be canceled, the appropriate figure must be removed from the replacement sheet, and where necessary, the remaining figures must be renumbered and appropriate changes made to the brief description of the several views of the drawings for consistency. Additional replacement sheets may be necessary to show the renumbering of the remaining figures. Each drawing sheet submitted after the filing date of an application must be labeled in the top margin as either "Replacement Sheet" or "New Sheet" pursuant to 37 CFR 1.121(d). If the changes are not accepted by the examiner, the applicant will be notified and informed of any required corrective action in the next Office action. The objection to the drawings will not be held in abeyance.

Specification

9) Applicant is reminded of the proper language and format for an abstract of the disclosure.

The abstract should be in narrative form and generally limited to a single paragraph on a separate sheet within the range of 50 to 150 words in length. The abstract should describe the disclosure sufficiently to assist readers in deciding whether there is a need for consulting the full patent text for details.

The language should be clear and concise and should not repeat information given in the title. It should avoid using phrases which can be implied, such as, "The disclosure concerns," "The disclosure defined by this invention," "The disclosure describes," etc. In addition, the form and legal phraseology often used in patent claims, such as "means" and "said," should be avoided.

10) The abstract of the disclosure is objected to because of the following informalities:

- The abstract uses phrases which can be implied - "The invention relates to" (line 1)

11) Correction is required. See MPEP § 608.01(b).

12) The disclosure is objected to because of the following informalities:

- Reference character "26" has been used to designate both a "drying device" (Page 13, line 16) and a "pressure device" (Page 13, line 18).

Appropriate correction is required.

Claim Objections

13) Claims 1, 5, 7, and 15 are objected to because of the following informalities:

Claim 1 -

- "material." (line 9). Examiner suggests, "material;"

Claim 5 -

- "in transport containers" (line 13). Examiner suggests, "in respective further containers"

Claim 7 -

- "with inactivated comminuted material" (line 9). Examiner suggests, "with the inactivated comminuted material"

Claim 15 -

- "a second comminution stage" (line 2). Examiner suggests, "a second comminution device"

Examiner notes that claims have not been submitted in a clear format. Therefore, there grammatical structures such as periods and commas are unclear as found in at least Claims 1, 5, 6, and 11.

Appropriate correction is required.

Claim Interpretation

14) The following is a quotation of 35 U.S.C. 112(f):

(f) Element in Claim for a Combination. – An element in a claim for a combination may be expressed as a means or step for performing a specified function without the recital of structure, material, or acts

in support thereof, and such claim shall be construed to cover the corresponding structure, material, or acts described in the specification and equivalents thereof.

The following is a quotation of pre-AIA 35 U.S.C. 112, sixth paragraph:

An element in a claim for a combination may be expressed as a means or step for performing a specified function without the recital of structure, material, or acts in support thereof, and such claim shall be construed to cover the corresponding structure, material, or acts described in the specification and equivalents thereof.

15) The claims in this application are given their broadest reasonable interpretation using the plain meaning of the claim language in light of the specification as it would be understood by one of ordinary skill in the art. The broadest reasonable interpretation of a claim element (also commonly referred to as a claim limitation) is limited by the description in the specification when 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is invoked.

As explained in MPEP § 2181, subsection I, claim limitations that meet the following three-prong test will be interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph:

(A) the claim limitation uses the term "means" or "step" or a term used as a substitute for "means" that is a generic placeholder (also called a nonce term or a non-structural term having no specific structural meaning) for performing the claimed function;

(B) the term "means" or "step" or the generic placeholder is modified by functional language, typically, but not always linked by the transition word "for" (e.g., "means for") or another linking word or phrase, such as "configured to" or "so that"; and

(C) the term "means" or "step" or the generic placeholder is not modified by sufficient structure, material, or acts for performing the claimed function.

Use of the word "means" (or "step") in a claim with functional language creates a rebuttable presumption that the claim limitation is to be treated in accordance with 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph. The presumption that the claim limitation is interpreted under 35 U.S.C.

112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is rebutted when the claim limitation recites sufficient structure, material, or acts to entirely perform the recited function.

Absence of the word "means" (or "step") in a claim creates a rebuttable presumption that the claim limitation is not to be treated in accordance with 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph. The presumption that the claim limitation is not interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, is rebutted when the claim limitation recites function without reciting sufficient structure, material or acts to entirely perform the recited function.

Claim limitations in this application that use the word "means" (or "step") are being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, except as otherwise indicated in an Office action. Conversely, claim limitations in this application that do not use the word "means" (or "step") are not being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, except as otherwise indicated in an Office action.

16) This application includes one or more claim limitations that do not use the word "means," but are nonetheless being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, because the claim limitation(s) uses a generic placeholder that is coupled with functional language without reciting sufficient structure to perform the recited function and the generic placeholder is not preceded by a structural modifier. Such claim limitation(s) is/are: **"an inactivation device" (Claim 7, line 6); "a filling device" (Claim 7, line 8); "a drying device" (Claim 7, line 11); "a hard metal detachment device and/or light fraction detachment device" (Claim 12, line 3); "a separation device" (Claim 12, line 5); and "a second filling device" (Claim 12, line 10).**

Because this/these claim limitation(s) is/are being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, it/they is/are being interpreted to cover the corresponding structure described in the specification as performing the claimed function, and equivalents thereof. **The following appears to be the corresponding structure as disclosed:**

Inactivation device - vacuum circulation system

Filling device - a platform or frame (Fig. 2)

Drying device - vacuum circulation system

Hard metal detachment device - magnet or other separator

Light fraction detachment device - zigzag separator or air separator

Separation device - air jet sieving device or comminuting device

Second filling device - a platform or frame (Fig. 2)

If applicant does not intend to have this/these limitation(s) interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph, applicant may: (1) amend the claim limitation(s) to avoid it/them being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph (e.g., by reciting sufficient structure to perform the claimed function); or (2) present a sufficient showing that the claim limitation(s) recite(s) sufficient structure to perform the claimed function so as to avoid it/them being interpreted under 35 U.S.C. 112(f) or pre-AIA 35 U.S.C. 112, sixth paragraph.

Claim Rejections - 35 USC § 112

17) The following is a quotation of 35 U.S.C. 112(b):
(b) CONCLUSION.—The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the inventor or a joint inventor regards as the invention.

The following is a quotation of 35 U.S.C. 112 (pre-AIA), second paragraph:
The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

18) Claim 6 is rejected under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention.

19) Regarding Claim 6, the recitation of "the drying" (line 3) and "the drying process" (line 4) renders the claim indefinite. Claim 6 appears to be claiming that drying is completed after drying is completed. Therefore the phrase, "after the completion of the drying process" appears redundant.

20) Claim 6 recites the limitation "the subsequent processing" in line 8 of the claim. There is insufficient antecedent basis for this limitation in the claim. It is unclear what subsequent processing is being recited.

Claim Rejections - 35 USC § 112

21) The following is a quotation of the first paragraph of 35 U.S.C. 112(a):

> (a) IN GENERAL.—The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor or joint inventor of carrying out the invention.

The following is a quotation of the first paragraph of pre-AIA 35 U.S.C. 112:

> The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor of carrying out his invention.

22) Claim 13 is rejected under 35 U.S.C. 112(a) or 35 U.S.C. 112 (pre-AIA), first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor or a joint inventor, or for pre-AIA the inventor(s), at the time the application was filed, had possession of the claimed invention. **Regarding Claim 13, the Specification does not disclose that the active material fraction and the carrier fraction are aluminum and copper foils, respectively. Rather, Page 16 of the Specification discloses "a heavy material fraction with carrier foils (aluminum and copper)." Further Page 9 of the Specification discloses that, "The active material should be understood to mean the material that reacts electrochemically during operation of the batteries. The**

carrier for the active material should be understood particularly to mean a carrier foil to which the active material is applied in the form of particles. For example, the carrier foil refers to a foil made of aluminium or an aluminium alloy."

Claim Rejections - 35 USC § 103

23) The following is a quotation of 35 U.S.C. 103 which forms the basis for all obviousness rejections set forth in this Office action:

> A patent for a claimed invention may not be obtained, notwithstanding that the claimed invention is not identically disclosed as set forth in section 102, if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a person having ordinary skill in the art to which the claimed invention pertains. Patentability shall not be negated by the manner in which the invention was made.

24) The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103 are summarized as follows:

1. Determining the scope and contents of the prior art.

2. Ascertaining the differences between the prior art and the claims at issue.

3. Resolving the level of ordinary skill in the pertinent art.

4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

25) **Claims 1, 3, 5, 7, 12, 13, 16, and 17 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens (U.S. 8,210,456) in view of Kellermann (DE 4424825).**

26) Regarding Claim 1, Stevens discloses a method for the treatment of used batteries, comprising the steps:

(a) comminuting the batteries such that comminuted material is obtained (Lithium batteries are shredded: Col 5, L16-17),

(b) inactivating the comminuted material such that an inactivated comminuted material is obtained (Oven conveyor Fig. 3, #20 & Dried material: Col 1, L37 & VOCs: Col 5, L15);

wherein the inactivating step is performed by drying the comminuted material (Dried material: Col 1, L37). Stevens is not explicit to filling a transport container with the inactivated comminuted material. **Kellermann** teaches a method for the disposal of lithium batteries including filling a transport container with the inactivated comminuted material (The fractional fractions of the crushed batteries are collected in collection vessels: Page 2) in order to further process the batteries. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to collect the inactivated comminuted material of Stevens in a transport container, as taught by Kellermann, in order to further process the batteries.

27) Regarding Claim 3, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses wherein the drying occurs under vacuum (Air uptake: Fig. 4, #21 & Vacuum source: Fig. 4, #44).

28) Regarding Claim 5, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses one or more of detachment of hard parts (magnetic component separated by the magnetic wheel: Col 5, L35), and separation of active material from a carrier, such that an active material fraction and a carrier fraction are obtained (The specific gravity separator separates out nonferrous fluff encapsulated with this magnetic component and also separates the steel, magnetic alloys, aluminum, and copper from each other: Col 5, L38-41). Kellermann further teaches removal of the comminuted material from the transport container (The fractional fractions of the crushed batteries are collected in collection vessels and sent to a pool of a flotation station where a first separation treatment of fractions takes place: Page 2. Examiner notes that the comminuted material would be removed from the collection vessels in order to be put into the separation pool) and separate packing of the active material fraction and carrier fraction in transport

containers (Dried parts are fed in a container: Page 3 & The iron component are deposited: Page 3 & The non-ferrous components are deposited: Page 3).

29) Regarding Claim 7, Stevens discloses (a) a comminution unit for comminuting the batteries such that comminuted material is obtained (Shredder: Fig. 3, #14),

(b) an inactivation device for inactivating the comminuted material (Oven conveyor Fig. 3, #20 & Dried material: Col 1, L37 & VOCs: Col 5, L15) wherein

the inactivation device comprises a drying device (Oven conveyor Fig. 3, #20 & Dried material: Col 1, L37), and

the battery processing installation has a vacuum installation that is connected to the drying device for generating a vacuum in the drying device (Air uptake: Fig. 4, #21 & Vacuum source: Fig. 4, #44). Stevens is not explicit to a filling device for filling a transport container with inactivated comminuted material. **Kellermann** teaches a device for the disposal of lithium batteries including a filling device for filling a transport container with inactivated comminuted material (Collection vessels: Fig. 1, #18. Examiner notes that the filling vessels are located along the process path or within the shredder device and therefor would include a filling device or frame where the collection vessels would be filled) in order to further process the batteries. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to collect the inactivated comminuted material of Stevens in a transport container with a filling device, as taught by Kellermann, in order to further process the batteries.

30) Regarding Claim 12, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses a hard metal detachment device (Magnetic wheel: Fig. 3, #52) and/or a light fraction detachment device,

a separation device, for separating active material from a carrier such that an active material fraction and a carrier fraction occur (Specific gravity separator: Fig. 3, #68). Kellermann further teaches a second

filling device for separate filling of the active material fraction and the carrier fraction (Dried parts are fed in a container: Page 3 & The iron component are deposited: Page 3 & The non-ferrous components are deposited: Page 3. Examiner notes that depositing of the material would occur with a filling device.).

31) Regarding Claim 13, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Steven further discloses wherein the active material fraction and the carrier fraction are respectively aluminum and copper foils (Aluminum and copper: Col 5, L36-37).

32) Regarding Claim 16, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses wherein the batteries are lithium batteries (Lithium battery: Col 5, L5).

33) Regarding Claim 17, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses wherein the batteries are lithium batteries (Lithium battery: Col 5, L5).

34) **Claims 2, 6, and 8 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Sloop (U.S. 2003/0186110).**

35) Regarding Claim 2, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried material (Col 5, L19), but is not explicit to wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

Sloop teaches the recycling of lithium batteries including wherein the comminuted material is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible (Dry, non-flammable, minimally reactive form: Par. 0042) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens would be such that an electrolyte content in the comminuted material is so low that an electrochemical

reaction is impossible, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

36) Regarding Claim 6, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried material (Col 5, L19), but is not explicit to wherein the drying of the comminuted material is only completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material in the transport container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing.

Sloop teaches the recycling of lithium batteries including wherein the drying of the comminuted material is only completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material in the transport container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing (Dry, non-flammable, minimally reactive form: Par. 0042. Examiner notes that if the dried material in non-flammable and minimally reactive, no flammable or explosive gas would emerge) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens would be such that no flammable or explosive gas mixture can form above the comminuted material in the transport container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

37) Regarding Claim 8, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. Stevens further discloses that VOCs are removed from the dried

material (Col 5, L19), but is not explicit to wherein the drying device is configured to dry the comminuted material until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

Sloop teaches the recycling of lithium batteries including wherein the drying device is configured to dry the comminuted material until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible (After the electrolyte is removed . . . Dry, non-flammable, minimally reactive form: Par. 0042) as a known result of drying the batteries to remove the electrolyte. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention that the dried batteries of Stevens would be such that an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible, as taught by Sloop, as a known result of drying the batteries to remove the electrolyte.

38) **Claim 9 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Meador (U.S. 5,632,863).**

39) Regarding Claim 9, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein the comminution device and the drying device are arranged in a container.

Meador teaches a battery recycling process wherein the comminution device (27) and the drying device (17) are arranged in a container (See frame of device & Col 4, L36-53) in order to prevent oxygen from entering the device which could cause an explosion hazard. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to arrange the comminution device and drying device of Stevens in a container, as taught by Meador, in order to prevent oxygen from entering the device (Meador: Col 4, L52).

40) **Claims 14 and 18 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Hanisch et al., hereinafter Hanisch, (WO 2013/023640). Note that citations for Hanisch are taken from corresponding U.S. Patent No. 9,780,419.**

41) Regarding Claim 14, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein the separation device is an air jet sieving device.

Hanisch teaches a separation device for an active material and a carrier wherein the separation device is an air jet sieving device (Air jet sieve: Fig. 1, #50) in order to separate materials with a high degree of purity. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the air jet sieve of Hanisch in order to separate materials with a high degree of purity (Hanisch: Col 2, L6).

42) Regarding Claim 18, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein separation of the active material from the carrier is performed by air jet sieving.

Hanisch teaches a separation device for an active material and a carrier wherein separation of the active material from the carrier is performed by air jet sieving (Air jet sieve: Fig. 1, #50) in order to separate materials with a high degree of purity. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the air jet sieve of Hanisch in order to separate materials with a high degree of purity (Hanisch: Col 2, L6).

43) **Claims 15 and 19 is/are rejected under 35 U.S.C. 103 as being unpatentable over Stevens in view of Kellermann and Sloop (U.S. 2016/0043450).**

44) Regarding Claim 15, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein the separation device is a second comminution stage.

Sloop teaches a device for the recycling of lithium batteries including a comminuting stage (14), drying stage (20), and separating stage (22, 24) wherein the separation device is a second comminution stage (Grinding step: Par. 0019) in order to improve yield in subsequent sieving. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the grinder of Sloop in order to improve yield in subsequent sieving (Sloop: Par. 0019).

45) Regarding Claim 19, the combination of Stevens and Kellermann teach all elements of the claimed invention as stated above. The combination of Steven and Kellerman do not teach wherein separation of the active material from the carrier is performed using a second comminution stage.

Sloop teaches a device for the recycling of lithium batteries including a comminuting stage (14), drying stage (20), and separating stage (22, 24) wherein separation of the active material from the carrier (24) is performed using a second comminution stage (Grinding step: Par. 0019) in order to improve yield in subsequent sieving. Therefore, it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to replace the specific gravity separator of Stevens with the grinder of Sloop in order to improve yield in subsequent sieving (Sloop: Par. 0019).

Allowable Subject Matter

46) Claims 4, 11, and 20 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims.

47) The following is a statement of reasons for the indication of allowable subject matter:

48) Regarding Claims 4 and 11, it is the opinion of the Examiner that the art of record neither anticipates nor renders obvious wherein "*a pump liquid of the jet pump has at least one of a pH value of at least 8, and contains a substance that precipitates fluoride*" or "*wherein the pump liquid is conducted within a circuit and the pump liquid contains a substance that reacts with hydrogen fluoride*" in combination with the rest of the claimed limitations set forth in the dependent claim. Searching by the Examiner yielded prior art as cited below:

49) Stevens discloses the removal of an electrolyte including lithium from the recycled battery using a vacuum system, scrubber, and an atomizer using a 50% concentration of sodium hydroxide in water, and this discloses a liquid with a pH of at least 9. Sodium hydroxide is also known to react with hydrogen fluoride to precipitate fluoride. However, the electrolyte disclosed by Stevens is lithium perchlorate in propylene carbonate and dimethoxyethane. While, **Tanii et al. (U.S. 6,524,737)** discloses the recycling of lithium batteries containing lithium phosphohexafluoride as the electrolyte, and thus the electrolyte of Stevens could be replaced with lithium phosphohexafluoride, the fluoride compounds are treated in a wastewater tank instead of with a jet pump. Further, while the use of jet pumps containing alkaline liquids such as sodium hydroxide is known from **Eckardt et al. (U.S. 2014/0003568)** such a use is not disclosed for the recycling of a lithium electrolyte battery.

50) Therefore, Stevens, Tanii, and Eckardt alone, or in combination with any other prior art, do not anticipate or render obvious the claimed invention.

Conclusion

51) The prior art made of record and not relied upon is considered pertinent to applicant's disclosure: Hashimoto (U.S. 2007/0134546).

52) Any inquiry concerning this communication or earlier communications from the examiner should be directed to GREGORY D. SWIATOCHA whose telephone number is (571)272-7022. The examiner can normally be reached on Monday-Friday (9-5).

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Shelley Self can be reached on (571) 272-4524. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3725

Notice of References Cited	Application/Control No. 15/569,484	Applicant(s)/Patent Under Reexamination HANISCH et al.	
	Examiner GREGORY D SWIATOCHA	Art Unit 3725	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	CPC Classification	US Classification
*	A	US-8210456-B2	07-2012	Stevens; Wayne C.	B03B9/061	241/19
*	B	US-9780419-B2	10-2017	Hanisch; Christian	H01M10/54	1/1
*	C	US-20030186110-A1	10-2003	Sloop, Steven E.	H01G9/038	429/49
*	D	US-20070134546-A1	06-2007	Hashimoto; Zenzo	C22B7/005	429/49
*	E	US-20160043450-A1	02-2016	Sloop; Steven E.	C01G53/50	252/182.1
*	F	US-5632863-A	05-1997	Meador; W. R.	C10B47/44	201/25
*	G	US-6524737-B1	02-2003	Tanii; Tadaaki	C22B7/005	429/49
*	H	US-20140003568-A1	01-2014	Eckardt; Bernd	B01D47/10	376/283
	I					
	J					
	K					
	L					
	M					

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*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	CPC Classification
	N	DE-4424825-A1	01-1996	DE	KELLERMANN THAMMO	B03B9/061
	O	WO-2013023640-A1	02-2013	WO	KWADE ARNO	H01M10/54
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	Machine Translation of DE 4424825, Translated 9/5/2019, 3 Pages. (Year: 1996)
	V	Machine Translation of WO 2013/023640, Translated 9/5/2019, 5 Pages (Year: 2013)
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

Index of Claims	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	**Art Unit**
	GREGORY D SWIATOCHA	3725

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

CLAIMS

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIM		DATE								
Final	Original	09/05/2019								
	1	✓								
	2	✓								
	3	✓								
	4	O								
	5	✓								
	6	✓								
	7	✓								
	8	✓								
	9	✓								
	10	-								
	11	O								
	12	✓								
	13	✓								
	14	✓								
	15	✓								
	16	✓								
	17	✓								
	18	✓								
	19	✓								
	20	O								

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3725

CPC - Searched*		
Symbol	**Date**	**Examiner**
Y02W30/84	09/04/2019	G.S.
Y02P10/212	09/04/2019	G.S.
B02C23/08, 10	09/04/2019	G.S.
B09B3/008, 0058	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102	09/04/2019	G.S.

CPC Combination Sets - Searched*		
Symbol	**Date**	**Examiner**

US Classification - Searched*			
Class	**Subclass**	**Date**	**Examiner**

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

/GREGORY D SWIATOCHA/ Primary Examiner, Art Unit 3725	

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/569,484	HANISCH et al.
	Examiner	Art Unit
	GREGORY D SWIATOCHA	3725

Search Notes		
Search Notes	**Date**	**Examiner**
EAST Inventor search, Assignee search, CPC search, Text search, Combination class../text search	09/04/2019	G.S.
Espacenet search	09/04/2019	G.S.
IP.com search	09/04/2019	G.S.
NPL Search	09/04/2019	G.S.
Y02W30/84 (See search history printout)	09/04/2019	G.S.
Y02P10/212 (See search history printout)	09/04/2019	G.S.
B02C23/08, 10 (See search history printout)	09/04/2019	G.S.
B09B3/008, 0058 (See search history printout)	09/04/2019	G.S.
B01D53/05, 002; 11/00; 2253/102 (See search history printout)	09/04/2019	G.S.

Interference Search			
US Class/CPC Symbol	**US Subclass/CPC Group**	**Date**	**Examiner**

/GREGORY D SWIATOCHA/
Primary Examiner, Art Unit 3725

Duesenfeld Ex. 2014-337

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (02-18)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99)**	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

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	1	2010/149611	WO	A1	2010-12-29	SB LIMOTIVE COMPANY LTD.		☒
	2	20060101683	KR		2005-03-21			☒

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1	HIGGELKE: "Infinite Power Solutions stellt papierdünnen Akku vor", "(Infinite Power Solution presents paper-thin Battery)", 2012-06-06	☒
	2	VDMA-Norm 24351 Trocknungstechnik (VDMA-German Engineering Federation, VDMA Standared 24351 - Drying Technology - Basic terms and definitions), 1999-01	☐
	3	HERAUSGEBER: "Florke/Wolff-Kursthemen Chemie - Allgemeine und Physikalische Chemie", "(Course Issues Chemisty- General and Physical Chemistry)", 1985	☒

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EXAMINER SIGNATURE

Examiner Signature	/GREGORY D SWIATOCHA/	Date Considered	09/04/2019

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	15569484
	Filing Date	2017-10-26
	First Named Inventor	Christian HANISCH
	Art Unit	
	Examiner Name	
	Attorney Docket Number	03101160US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

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Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2019-04-18
Name/Print	Michael E. Whitham	Registration Number	32635

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Ascend Elements EX1002 - Page 341

Duesenfeld Ex. 2014-341

(19) BUNDESREPUBLIK DEUTSCHLAND

DEUTSCHES PATENTAMT

(12) **Offenlegungsschrift**

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B 03 B 9/06

(21) Aktenzeichen: P 44 24 825.3
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DE 44 24 825 A 1

(71) Anmelder:
Ingenieurkontor Lübeck Prof. Gabler Nachf. GmbH, 23568 Lübeck, DE

(74) Vertreter:
H. Wilcken und Kollegen, 23552 Lübeck

(72) Erfinder:
Kellermann, Thammo, 23568 Lübeck, DE

(54) Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien

(57) Das Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, umfaßt ein Zerkleinern der Batterien durch beispielsweise eine Shreddereinrichtung. Um das Shreddern gefahrlos durchführen zu können, werden die Batterien vor dem Zerkleinern auf eine solche tiefe Temperatur abgekühlt, daß eine chemische Reaktion der vom Metallgehäuse wenigstens teilweise befreiten Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet. Danach kann dann eine Trennbehandlung der entstandenen Fraktionsanteile der Batterien mit herkömmlicher Technik erfolgen.

DE 44 24 825 A 1

Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen

BUNDESDRUCKEREI 11. 95 508 063/308

Ascend Elements EX1002 Page 342

Duesenfeld Ex. 2014-342

Beschreibung

Die Erfindung betrifft ein Verfahren zum Entsorgen von entladenen und geladenen elektrischen Feststoff-Batterien gemäß dem Oberbegriff des Patentanspruchs 1.

Das Problem bei der Entsorgung von Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, auch wenn sie entladen bzw. verbraucht sind, besteht darin, daß die Bestandteile der Batterien beim Zerkleinerungsvorgang, zum Beispiel durch Shreddern, bei Normalbedingungen in Luft noch chemisch und physikalisch heftig miteinander reagieren und dabei Knall- und Verbrennungsgeräusche verursachen, weshalb die Zerkleinerungsanlage mit umfangreichen Schutzmaßnahmen versehen sein muß, was mit einem erheblichen Material- und Kostenaufwand verbunden ist. Desweiteren werden dabei auch schädliche Gase, Dämpfe und feste Stoffe freigesetzt, die Mensch und Umwelt belasten und ebenfalls mit erheblichem Material- und Kostenaufwand einzukapseln und zu entsorgen sind.

Die Aufgabe der Erfindung besteht darin, ein verbessertes Verfahren der eingangs angeführten Art vorzuschlagen, das ein gefahrloses Öffnen bzw. Zerkleinern von gasdicht gekapselten Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, ermöglicht und eine einfache Weiterverarbeitung der zerkleinerten Batterien gestattet.

Die Lösung dieser Aufgabe geht von dem einleitend angeführten Verfahren aus und ist durch die Merkmale des Patentanspruchs 1 gekennzeichnet.

Durch die erfindungsgemäße Lösung ist ein gefahrloses Öffnen und Zerkleinern von zu entsorgenden Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, ermöglicht, denn das Restenergievermögen der Batterien ist durch deren Tiefkühlung vor dem Zerkleinern nicht oder im wesentlichen nicht mehr zu einer chemischen und physikalischen Reaktion in Luft unter normalen Druck- und Temperaturbedingungen fähig, wenn das schützende Metallgehäuse der Batterien geöffnet bzw. entfernt worden ist. Die Batterien können also ohne Gefahr in Luft bei Normalbedingungen mechanisch zerkleinert werden, zum Beispiel durch Shreddern, wobei keinerlei Knall- und Verbrennungsgeräusche unter Rauch- und/oder Flammenentwicklung mehr entstehen. Weiterhin entstehen während der Tiefkühlbehandlung nur geringfügig schädliche Gase und Dämpfe. Die sich an die vollständige Zerkleinerung der Batterien anschließende Trennbehandlung aller entstandenen Fraktionsanteile sorgt dafür, daß sich die langsam wieder auf etwa Raumtemperatur erwärmten, nun losen Batteriebestandteile ebenfalls nicht mehr oder nur noch in einem unbedeutenden Umfang unkontrolliert chemisch reagieren können. Die Trennbehandlung kann mit herkömmlichen Verfahren und Vorrichtungen durchgeführt werden, derart, daß wenigstens einige der Bestandteile der zerstörten Batterien als Recycle-Rohstoff einer Wiederverwendung zugeführt werden können und nur noch wenige, nach Möglichkeit keine Reststoffe auf eine Deponie gegeben werden müssen.

Eine bevorzugte Tiefkühltemperatur beträgt wenigstens etwa −190° Celcius und als Tiefkühlmittel wird vorzugsweise flüssiger Stickstoff oder auch Argon verwendet.

Falls bei einigen zu entsorgenden Batterien nach dem Tiefkühlen beim Shreddern noch eine geringe chemische Reaktion zu befürchten ist, kann aus dem beim Tiefkühlen verwendeten Tiefkühlmittel ein gasförmiger Anteil abgezweigt und für eine chemisch inerte Atmosphäre beim Zerkleinern der tiefgekühlten Batterien verwendet werden. Hierdurch erfolgt das Shreddern in einer eine mögliche chemische Reaktion unterbindenden Atmosphäre.

Die Trennbehandlung der Fraktionsanteile der zerstörten Batterien wird vorzugsweise durch einen Flotationsvorgang eingeleitet, wobei das Flotationsgemisch einer Leichtfraktionsbehandlung und einer Schwerfraktionsbehandlung unterworfen wird, um eine Grobtrennung vorzunehmen.

Die Erfindung ist nachstehend anhand des anliegenden Ablaufdiagramms beispielsweise näher erläutert. In **Fig.** 1 ist ein erster Teil des Ablaufdiagramms gezeigt, während in **Fig.** 2 der zweite Teil des Ablaufdiagramms gezeigt ist.

Das vorgeschlagene Verfahren eignet sich zur Entsorgung von Feststoff-Batterien **1** und **2**, die zum Beispiel in Zylinderform und Quaderform im Handel erhältlich sind, und zwar insbesondere für solche Batterien, die Lithium enthalten. Lithiumbatterien wiederum enthalten je nach Zellentyp außer Lithium noch Mangandioxid, Schwefeldioxid, Thionylchlorid und Acetonnitril als elektrolytische Reaktoren. Derartige Batterien enthalten, auch wenn sie entladen oder fast entladen sind, noch eine relativ hohe Energiedichte, so daß eine chemische und physikalische Reaktion der genannten Substanzen stattfindet, wenn das schützende Metallgehäuse der Batterien bei Normalbedingungen in Luft geöffnet wird.

Gemäß dem vorgeschlagenen Verfahren nach den **Fig.** 1 und 2 werden die zu entsorgenden Batterien in einer Packstation **3** zu einer vorbestimmten Menge zusammengesetzt, welche dann in wenigstens einen Schleppkasten **4** eines Vorkühlraumes **5** gegeben wird. Die in die Schlepp- oder Transportkästen **4** eingefüllten Batterien durchlaufen dann mit Hilfe eines Transportsystems eine Vorkühlstrecke **6**, innerhalb der sie von Raumtemperatur auf eine Temperatur von etwa −30° bis −50° Celsius abgekühlt werden. Die Durchlaufdauer durch die Vorkühlstrecke **6** wird von der Temperatur und der Art des zugeführten Kühlgases und der Batteriemasse bestimmt und kann beispielsweise 15−30 Minuten betragen.

Der Vorkühlraum **5** wird mit einem Vorkühlgas beschickt, welches beispielsweise eine Temperatur von −80° bis −190° Celsius aufweisen kann. Vorzugsweise wird das Kühlgas aus einem Hauptkühlraum **7** abgezweigt, in welchem die vorgekühlten Batterien mittels der Kästen **4** eingeführt werden. Das Vorkühlgas wird mittels einer Pumpe **8** aus dem Hauptkühlraum **7** abgesaugt und über eine Leitung **9** in den Vorkühlraum **5** eingeleitet.

Die vorgekühlten Batterien in den Kästen **4** durchlaufen dann mittels eines Transportsystems für eine vorbestimmte Zeit den Hauptkühlraum und werden darin auf eine solche tiefe Temperatur abgekühlt, daß eine chemische und physikalische Reaktion der später vom Metallgehäuse wenigstens teilweise befreiten Batterie-Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet. Eine solche Tiefkühltemperatur beträgt wenigstens etwa −80° Celsius, vorzugsweise etwa −190° Celsius.

Ein geeignetes Kühlmittel ist flüssiger Stickstoff (LIN), mit dem die vorgekühlten Batterien in Kontakt gebracht werden. Eine solche Kontaktdauer kann bei-

Duesenfeld Ex. 2014-343

spielsweise 15 bis 30 Minuten oder mehr betragen, was davon abhängig ist, in welcher Form die vorgekühlten Batterien mit dem flüssigen Stickstoff in Kontakt kommen. Hierfür kann beispielsweise ein Eintauchvorgang in Frage kommen, so daß die Batterien in den flüssigen Stickstoff eingetaucht werden. Es kann aber auch eine Besprühung oder Begießen der vorgekühlten Batterien mit flüssigem Stickstoff in Frage kommen. Auch eine Mischung aller oder zweier Kontaktarten ist möglich. Der flüssige Stickstoff weist eine Temperatur von wenigstens −196° Celsius auf und wird mittels einer Pumpe **10** aus einem Vorratsbehälter **11** über eine Leitung **12** in den Hauptkühlraum **7** eingeleitet.

Während des Tiefkühlvorganges der Batterien in dem Hauptkühlraum **7** entsteht in diesem Raum aus dem flüssigen Stickstoff ein gasförmiger Anteil, der für verschiedene Weiterbehandlungsvorgänge der Batterien bzw. Batterieteile verwendet wird. Wie bereits angedeutet, wird ein gewisser Anteil für die Vorkühlung der Batterien in dem Vorkühlraum **5** verwendet.

Das nicht mehr benötigte Vorkühlgas in dem Vorkühlraum **5** wird aus diesem, gegebenenfalls zusammen mit Abluft, abgesaugt und für die Wiederverwendung im Hauptkühlraum **7** vorbereitet. Hierzu ist eine Rückgewinnungseinrichtung **13** vorgesehen, aus welcher der wieder flüssige Stickstoff über eine Leitung **14** dem Vorratsbehälter **11** zugeführt wird. Gegebenenfalls vorhandene Abluft wird aus der Einrichtung **13** ins Freie abgeleitet.

Die auf die vorbestimmte Tiefkühltemperatur abgekühlten Batterien werden dann in eine Zerkleinerungsvorrichtung **15** gegeben, wo sie zerkleinert werden. Diese Vorrichtung **15** kann beispielsweise aus einer mehrstufigen Shreddervorrichtung bestehen. Es können aber auch Sägeeinrichtungen oder andere Einrichtungen alternativ oder zusätzlich eingesetzt werden, um die tiefkalten Batterien zu ausreichend kleinstückigen Fraktionsanteilen zu zerstören. In der Vorrichtung **15** herrscht vorzugsweise ein Druck von 1 bar (10^5 Pa) und eine Temperatur von maximal etwa 20° Celsius. Im übrigen kann der Innenraum der Zerkleinerungsvorrichtung **15** mit Luft gefüllt sein.

Wenn es aus bestimmten Sicherheitsgründen erforderlich ist, anstelle von Luft in der Vorrichtung **15** ein inertes Gas zu verwenden, so kann dazu ebenfalls gasförmiger Stickstoff aus dem Hauptkühlraum **7** abgezweigt werden, der mittels einer Pumpe **16** über eine Leitung **17** in die Vorrichtung **15** eingeleitet wird.

Die Fraktionsanteile der zerkleinerten Batterien werden in Auffangbehältern **18** gesammelt und einem Bekken einer Flotationsstation **19** zugeführt, wo eine erste Trennungsbehandlung der Fraktionsanteile stattfindet. Zur Durchführung des Flotationsvorganges befindet sich in dem Becken der Flotationsstation **19** neben einem aufbereiteten Wasser auch ein alkalisches Lösungsmittel, zum Beispiel Natronlauge, welche zusammen mit dem Wasser eine Suspension ergibt. Weiterhin wird neben Luft auch ein inertes Gas eingeleitet, welches wiederum gasförmiger Stickstoff sein kann und vorzugsweise aus dem Hauptkühlraum **7** über eine Leitung **20** und eine Pumpe **21** entnommen wird. Dieses Gas kann ebenfalls eine tiefe Temperatur aufweisen, beispielsweise −180° Celsius.

Durch die Flotation erfolgt eine Grobtrennung der Fraktionsanteile voneinander, und zwar eine Trennung der schwereren Fraktionsanteile, wie zum Beispiel Eisenmetalle, Nichteisenmetalle, Glas- und Keramikanteile, von den leichteren Fraktionsanteilen, die insbesondere aus Kunststoff bestehen. Die für die Flotation insgesamt in die Station **19** eingeleiteten Gase, die während der Flotation ihre Aufgabe erfüllt haben, und die während der Flotation freigewordenen Gase, insbesondere organische Gase, werden über eine Leitung **22** abgeführt und einer Einrichtung **23** zum Unschädlichmachen dieser Abgase zugeführt. Dieser Einrichtung **23** können auch die nicht mehr benötigten Gase aus der Shreddervorrichtung **15** zugeführt werden, wie es mit der Leitung **24** angegeben ist.

Nachdem sich die schwereren von den leichten Fraktionsanteilen der zerstörten Batterien in der Flotationsstation abgesondert haben, ist es ohne weiteres möglich, die so grob getrennten Fraktionsanteile aus der Flotationsstation **19** zu entnehmen und weiteren Trennungsbehandlungen zu unterwerfen. Die verbrauchte Lösungssuspension wird ebenfalls aus der Station **19** abgeführt und einer Behandlung zum Unschädlichmachen und Wiederaufbereiten unterworfen, die jedoch nicht Gegenstand dieser Erfindung ist.

Die aus der Flotationsstation **19** entnommenen Leichtfraktionsanteile werden zunächst einer Trocknereinrichtung **25** zugeführt, wonach die getrockneten Anteile in einem Behältnis **26** einer Sichteinrichtung **27** zugeführt werden, wo nochmals, falls erforderlich, eine Trennung der Kunststoffteile vorgenommen wird. Die so gewonnenen Kunststoffteile können als Recyclerohstoff in der chemischen Industrie wiederverwertet werden.

Auch die Schwerfraktionsanteile aus der Flotationsstation **19** werden zunächst einer Trocknereinrichtung **29** zugeführt, um sie dann besser weiterbehandeln zu können. Nach dem Trocknen können die Schwerfraktionsanteile zur weiteren und feineren Zerkleinerung einer weiteren Shreddereinrichtung **30** zugeführt werden. Von dort gelangen sie dann in eine erste Abscheideeinrichtung **31**, wo mittels bekannter Technik die Eisenanteile abgeschieden werden. In einer dann folgenden zweiten Abscheideeinrichtung **32** werden die Nichteisenanteile, insbesondere Kupfer, abgeschieden. Die Eisenanteile und die Nichteisenanteile können dann als Recyclematerial als wiederverwertbare Rohstoffe zur Verfügung stehen und mittels der Verhüttungstechnik verarbeitet werden. Die nun noch verbleibenden Restanteile, wie insbesondere Glas, können deponiert oder beispielsweise in der Glasindustrie oder im Straßenbau wiederverwendet werden. Erforderlichenfalls können durch entsprechende Trenneinrichtungen auch die Restanteile nochmals getrennt werden, um zum Beispiel die Keramikanteile abzuscheiden und einer Wiederverwertung zuzuführen.

Bevorzugt wird als Tiefkühlmittel im Hauptkühlraum **7** flüssiger Stickstoff verwendet. Davon abgezweigter Stickstoff in Gasform wird bevorzugt zur Vorkühlung im Vorkühlraum **5** und für die anderen, vorstehend verwendeten Zwecke eingesetzt. Stattdessen kann jedoch auch ein anderes Gas verwendet werden, zum Beispiel Argon. Obwohl das vorstehende Verfahren hauptsächlich für Lithium enthaltende Feststoff-Batterien vorgesehen ist, können auch andere Feststoff-Batterien, beispielsweise Nickel-Hydrid-Batterien, wie vorstehend beschrieben, entsorgt werden.

Patentansprüche

1. Verfahren zum Entsorgen von entladenen und geladenen elektrische Feststoff-Batterien, insbesondere von Lithium enthaltenden Batterien, die

Duesenfeld Ex. 2014-344

aus einem Metallgehäuse und darin enthaltenen festen Inhaltsstoffen bestehen, gemäß dem die zu entsorgenden Batterien mechanisch zerkleinert werden, **dadurch gekennzeichnet,** daß die Batterien vor dem Zerkleinern auf eine solche tiefe Temperatur abgekühlt werden, daß eine chemische und physikalische Reaktion der vom Metallgehäuse wenigstens teilweise befreiten Inhaltsstoffe bei normalen Temperatur- und Druckbedingungen und Sauerstoffeinwirkung im wesentlichen nicht mehr stattfindet und daß das nach dem Zerkleinern erhaltene Gemisch der Fraktionsanteile der zerstörten Batterien zur Gewinnung von wiederverwendbarem Ausgangsmaterial einer Trennbehandlung unterworfen wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Tiefkühlen der Batterien durch in berührungbringen derselben mit einem chemisch inerten Tiefkühlmittel erfolgt, dessen Temperatur wenigstens etwa −196° Celsius beträgt.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß als Tiefkühlmittel flüssiger Stickstoff verwendet wird.

4. Verfahren nach Anspruch 2 oder 3, dadurch gekennzeichnet, daß aus dem beim Tiefkühlen verwendeten Tiefkühlmittel eine gasförmiger Anteil abgezweigt und zum Durchführen einer Vorkühlung der Batterien verwendet wird.

5. Verfahren nach Anspruch 1, 2, 3 oder 4, dadurch gekennzeichnet, daß das Zerkleinern der tiefgekühlten Batterien in einer chemisch inerten Atmosphäre durchgeführt wird.

6. Verfahren nach Anspruch 5, dadurch gekennzeichnet, daß aus dem beim Tiefkühlen verwendeten Tiefkühlmittel ein gasförmiger Anteil abgezweigt und für eine chemisch inerte Atmosphäre beim Zerkleinern der tiefgekühlten Batterien verwendet wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß das Gemisch der Fraktionsanteile der zerstörten Batterien einem Flotationsvorgang unterworfen wird, um eine Grobtrennung des Flotationsgemisches in Form eines Leichtfraktionsvorganges und eines Schwerfraktionsvorganges durchzuführen.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß der Flotationsvorgang wenigstens im wesentlichen in einer chemisch inerten Atmosphäre durchgeführt wird.

9. Verfahren nach Anspruch 7 oder 8, dadurch gekennzeichnet, daß sich an den Leichtfraktions- und den Schwerfraktionsvorgang jeweils weitere Trennvorgänge zur Gewinnung einzelner Materialsorten aus den Fraktionsanteilen der zerstörten Batterien anschließen und daß wenigstens einige der gewonnenen Materialsorten der Wiederverwendung zugeführt werden.

Hierzu 2 Seite(n) Zeichnungen

ZEICHNUNGEN SEITE 1

Nummer: DE 44 24 825 A1
Int. Cl.⁶: A 62 D 3/00
Offenlegungstag: 18. Januar 1996

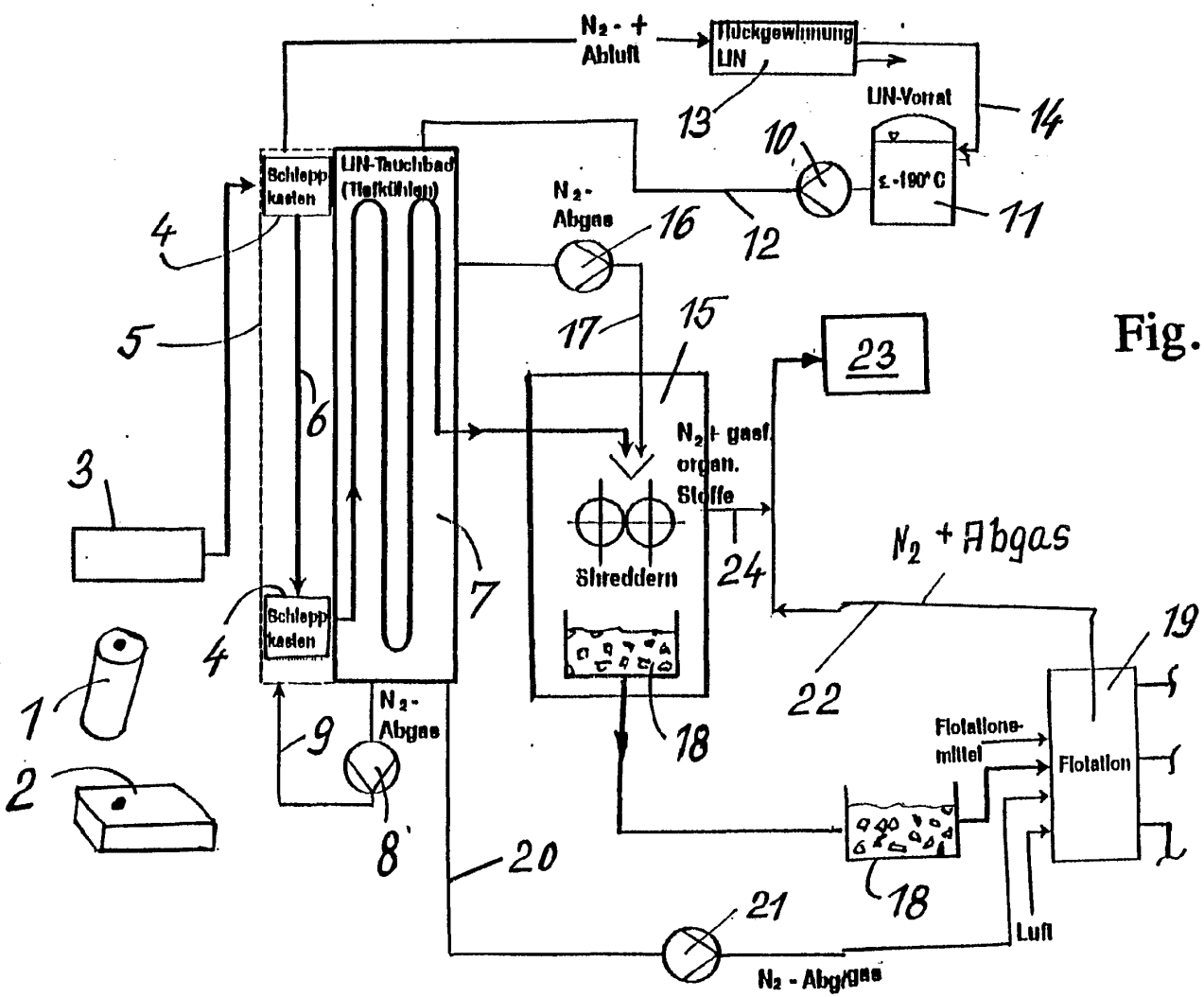

Fig. 1

508 063/308

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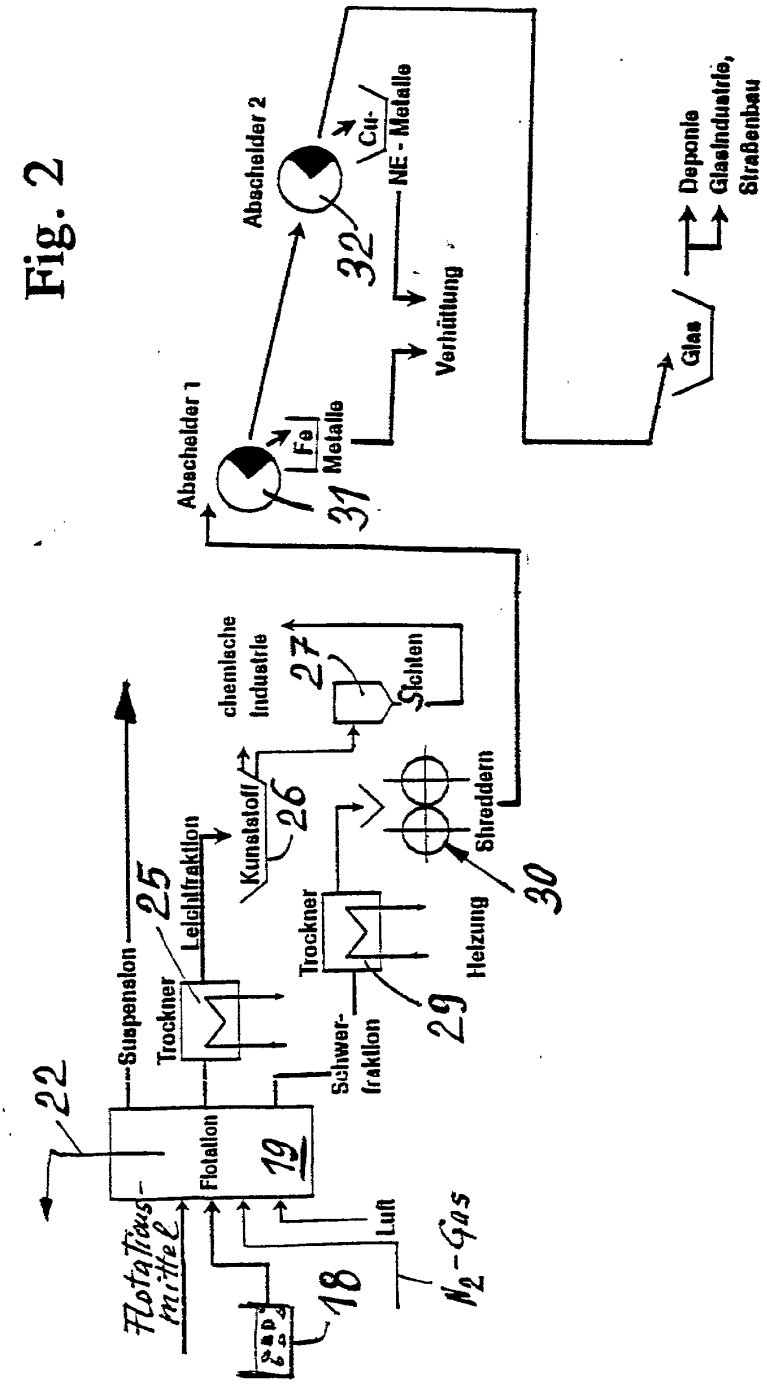
Offenlegungstag: 18. Januar 1996

Fig. 2



508 063/308

Ascend Elements EX1002 Page 347
Duesenfeld Ex. 2014-347

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TITLE: Safe disposal of dry batteries and recovery of materials
PUBN-DATE: January 18, 1996
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INVENTOR-INFORMATION:

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APPLICANT:

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APPL-NO: DE 4424825 A
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CPC-CURRENT:

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CPCI	B03B9/061 20130101
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CPCI	C22B1/005 20130101
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IPCS	H01M10/54 20060101
IPCS	C22B7/00 20060101
IPCS	C22B1/00 20060101

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ABSTRACT:

Process for the disposal of spent, or partly spent, dry, metal-cased, batteries, especially those containing lithium, involves cooling the batteries before disintegration to such a low temperature that the partially freed components cannot react chemically or physically under normal conditions of temperature, pressure and the presence of oxygen. Further provision is made for the separation and recovery of materials which can be recycled. Batteries are cooled by direct contact with, pref., liquid nitrogen at -196 deg C. The gasified nitrogen is used in a pre-cooling chamber and also to provide an inert atmosphere in the disintegration section, including the materials separation chambers. A coarse separation is carried out in a flotation process followed by a further separation of individual materials, some of which will be used for recycling.

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Verified and Acknowledged: /GREGORY D SWIATOCHA/ Examiner's Signature | Initials

Title: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

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CONTINUING DATA

This application is a 371 of PCT/EP2016/059526 04/28/2016

FOREIGN APPLICATIONS

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EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
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S22	1437	(y02p10/212.cpc. b02c21/$.cpc. (b02c23/08,10).cpc. b09b3/008.cpc. (b01d53/02,002).cpc. b01d11/00.cpc. b09b3/0058.cpc. b01d2253/102.cpc.) AND (battery batteries)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:31
S23	3	wo-2010102377-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S24	3	de-102011110083-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S25	2	de-102012024876-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT;	OR	ON	2019/09/04 14:37

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S27	1	kr-20060101683-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S28	0	wo-2106174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S29	2	wo-2016174156-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S30	2	wo-2016164752-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S31	2	cn-101394015-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S32	3	de-102015207843-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S33	3	de-602005001937-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S34	3	cn-102496752-$.did.	US-PGPUB; USPAT; USOCR; FPRS;	OR	ON	2019/09/04 14:37

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S35	3	cn-103959553-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S36	27	S23 S24 S25 S26 S27 S28 S29 S30 S31 S32 S33 S34 S35	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 14:37
S37	12	"9780419" "20050241943" "7713396"	US-PGPUB; USPAT; USOCR	OR	ON	2019/09/04 14:37
S38	352	S22 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S39	2006	S21 and lithium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 16:40
S40	3	de-19954998-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 19:41
S41	2	de-29803442-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 19:41
S42	1	(S38 S39) AND (liquid WITH gas WITH jet WITH (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:19
S44	484	(liquid WITH gas WITH jet WITH (ejector pump)) WITH vacuum	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT;	OR	ON	2019/09/04 20:21

			IBM_TDB			
S45	8	(liquid WITH gas WITH jet WITH (ejector pump) WITH vacuum) SAME pH	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:21
S46	21	(jet ADJ (ejector pump)) WITH ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:26
S47	169	(S38 S39) AND ((sodium calcium) ADJ carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:27
S48	3	WO-2013140039-$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:31
S49	3	(S38 S39) AND (jet ADJ (ejector pump))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:39
S50	8	(jet ADJ (ejector pump)) WITH fluoride	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:41
S51	1	(jet ADJ (ejector pump)) WITH (fluoride fluorine) WITH (carbonate)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 20:42
S53	174	(S38 S39) AND (electrolyte WITH (remov$4 weight))	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/04 21:02
S54	28	(S38 S39) AND (electrolyte WITH (remov$4 vaporiz$3) WITH (dri$2 dry$3))	US-PGPUB; USPAT; USOCR; FPRS;	OR	ON	2019/09/04 21:06

			EPO; JPO; DERWENT; IBM_TDB			
S55	5959	y02W30/84.cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34
S56	658	S55 and lithium and (dry$3 dri$2)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/09/05 07:34

9/ 5/ 2019 11:06:40 AM
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Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
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	Art Unit	
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Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	9780419	B2	2017-10-03	LION ENGINEERING GMBH	

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	1	20050241943	A1	2005-11-03	KAWASAKI JUKOGYO KABUSHIKI KAISHA	

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	1	2010/102377	WO	A1	2010-09-16	STEVENS, WAYNE, C.		
	2	10 2011 110 083	DE	A1	2013-02-14	TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA		
	3	10 2012 024 876	DE	A1	2014-06-26	ELECTROCYCLING GMBH; VOLKSWAGEN AKTIENGESELLSCHAFT		

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Ascend Elements EX1002 Page 361

Duesenfeld Ex. 2014-361

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[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

Ascend Elements EX1002 Page 362

Duesenfeld Ex. 2014-362

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	
	First Named Inventor	
	Art Unit	
	Examiner Name	
	Attorney Docket Number	

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Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2017-10-25
Name/Print	Michael E. Whitham	Registration Number	32635

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Ascend Elements EX1002 Page 363

Duesenfeld Ex. 2014-363

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Ascend Elements EX1002 - Page 364

Duesenfeld Ex. 2014-364

(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

(19) Weltorganisation für geistiges Eigentum
Internationales Büro

(43) Internationales Veröffentlichungsdatum
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[Fortsetzung auf der nächsten Seite]

(54) **Title**: METHOD FOR RECLAIMING ACTIVE MATERIAL FROM A GALVANIC CELL, AND AN ACTIVE MATERIAL SEPARATION INSTALLATION, PARTICULARLY AN ACTIVE METAL SEPARATION INSTALLATION

(54) **Bezeichnung** : VERFAHREN ZUM WIEDERGEWINNEN VON AKTIVMATERIAL AUS EINER GALVANISCHEN ZELLE UND AKTIVMATERIAL-SEPARATIONSANLAGE, INSBESONDERE AKTIVMETALL-SEPARATIONSANLAGE

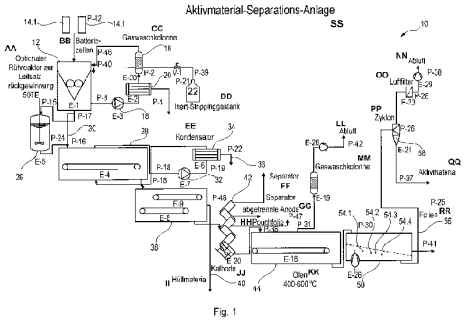

Fig. 1

SS Active material separation installation
AA Optional stirred reactor for recovering conducting salt
BB Battery cells
CC Gas-scrubbing column
DD Inert stripping-gas tank
EE Condenser
FF Separator
GG Separated anodes
HH Pouch film
II Cladding material
JJ Cathode
KK Furnace
LL Exhaust air
MM Gas-scrubbing column
NN Exhaust air
OO Air filter
PP Cyclone
QQ Active material
RR Films

(57) **Abstract**: The invention relates to a method for reclaiming active material of a galvanic cell that comprises an active material, a carrier for said active material, and a binder for binding the active material and the carrier, said method comprising the following steps:(a) comminuting the cells, particularly under inert gas or in a vacuum, such that solid cell fragments are at least also produced, (b) heating the solid cell fragments to a decomposition temperature (T_z) selected to be high enough that the binder decomposes and/or evaporates, preferably under inert gas or in a vacuum, so as to produce thermally treated cell fragments, and (c) classifying these thermally treated cell fragments, wherein (d) the classification comprises an air-jet sieving process and (e) the air-jet sieving is implemented such that active material is separated from the carrier.

(57) **Zusammenfassung**: Die Erfindung betrifft ein Verfahren zum Wiedergewinnen von Aktivmaterial einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial und einen Binder zum Verbinden des Aktivmaterials und des Trägers umfasst, mit den Schritten: (a) Zerkleinern der Zellen, insbesondere unter Inertgas oder im Vakuum, so dass zumindest auch feste Zellenfragmente entstehen, (b) Erwärmen der festen Zellenfragmente

[Fortsetzung auf der nächsten Seite]

WO 2013/023640 A1

Erklärungen gemäß Regel 4.17:

— *hinsichtlich der Berechtigung des Anmelders, ein Patent zu beantragen und zu erhalten (Regel 4.17 Ziffer ii)*

— *Erfindererklärung (Regel 4.17 Ziffer iv)*

Veröffentlicht:

— *mit internationalem Recherchenbericht (Artikel 21 Absatz 3)*

— *vor Ablauf der für Änderungen der Ansprüche geltenden Frist; Veröffentlichung wird wiederholt, falls Änderungen eingehen (Regel 48 Absatz 2 Buchstabe h)*

auf eine Zersetzungs-Temperatur (T_z), die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, vorzugsweise unter Intergas oder im Vakuum so dass wärmebehandelte Zellenfragmente entstehen, und (c) Klassieren der wärmebehandelten Zellenfragmente, wobei (d) das Klassieren ein Luftstrahlsieben umfasst und (e) das Luftstrahlsieben so durchgeführt wird, dass Aktivmaterial von dem Träger getrennt wird.

Verfahren zum Wiedergewinnen von Aktivmaterial aus einer galvanischen Zelle und Aktivmaterial-Separationsanlage, insbesondere Aktivmetall-Separationsanlage

Die Erfindung betrifft ein Verfahren zum Wiedergewinnen von Aktivmaterial, insbesondere von Lithium oder Lithiumverbindungen, aus einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und gegebenenfalls einen Elektrolyten umfasst, mit den Schritten: (a) Zerkleinern der Zellen, insbesondere unter Inertgas, so dass zumindest auch feste Zellenfragmente entstehen, (b) Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur, die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, so dass wärmebehandelte Zellenfragmente entstehen, und (c) Klassieren der wärmebehandelten Zellenfragmente.

Gemäß einem zweiten Aspekt betrifft die Erfindung eine Aktivmetall-Separationsanlage, die ausgebildet ist zum Wiedergewinnen von Aktivmaterial, insbesondere Lithium, einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und einen Elektrolyten umfasst, mit (a) einer Zellenzerkleinerungsvorrichtung zum Zerkleinern der Zellen, wobei die Zellenzerkleinerungsvorrichtung gasdicht ist und eine Inertgas-Zuführvorrichtung umfasst, so dass die Zellen unter Inertgas zu Zellenfragmenten zerkleinerbar sind, und (b) einem Ofen, der zumindest mittelbar mit der Zellenzerkleinerungsvorrichtung verbunden und eingerichtet ist zum Erwärmen der Zellenfragmente auf eine Zersetzungstemperatur, die so hoch gewählt ist, dass der Binder sich zersetzt.

Bestätigungskopie

Ascend Elements EX1002 Page 367

Duesenfeld Ex. 2014-367

Beim Recycling von Aktivmaterial, insbesondere von Lithium aus Lithium-Akkumulatoren oder Batterien, besteht das Problem, dass die chemischen Prozesse relativ aufwendig sind. Der Grund dafür ist, dass die Trennung des Aktivmaterials von seinem Träger nur unzureichend gelingt.

Aus der US 2005/0241943 A1 ist ein Verfahren bekannt, bei dem die Zellen zunächst zerkleinert und dann einer Wärmebehandlung unterzogen werden. Danach werden die Zellen geschreddert und auf die Zersetzungstemperatur erwärmt. Anschließend werden die wärmebehandelten Zellenfragmente durch Vibrationssieben getrennt. Es hat sich jedoch gezeigt, dass dieses Verfahren aufwendig ist. Der Erfindung liegt die Aufgabe zugrunde, das Wiedergewinnen von Aktivmaterial, insbesondere von Lithium oder Lithiumverbindungen, aus alten Batterien oder Akkumulatoren zu verbessern.

Aus der DE 199 54 998 A1 ist eine Schneidmühle bekannt, bei der das von einem Schneidrotor geschnittene Gut kontinuierlich so durch ein Sieb, insbesondere ein Luftstrahlsieb, gefördert wird, das nur diejenigen Partikel die Vorrichtung verlassen können, die eine vorgegebene Größe unterschreiten.

Aus der DE 29 803 442 U1 ist ein Luftstrahlsieb für die Analyse bekannt, mit dem eine hohe Zahl an Analysen in kurzer Zeit durchführbar ist und das zu diesem Zweck mehrere gegeneinander abgetrennte Siebflächen aufweist. Dieses Luftsieb wird zur Bestimmung von Korngrößenverteilungen im Feinkornbereich eingesetzt.

Aus der AT 386 769 B ist eine Luftstrahl-Mehrdeck-Siebmaschine bekannt, mittels der trockene, rieselförmige Schuttgüter kleiner Korngröße in bis zu 25 Fraktionen in einem Arbeitsgang klassifiziert werden können.

Die Erfindung löst das Problem durch ein gattungsgemäßes Verfahren, bei dem das Klassieren ein Luftstrahlsieben umfasst. Gemäß einem zweiten Aspekt löst die Erfindung das Problem durch eine Aktivmetall-Separationsanlage, die eine Luftstrahlsiebvorrichtung besitzt, die zumindest unmittelbar mit dem Ofen verbunden und eingerichtet ist zum Klassieren von Aktivmaterial und Träger, insbesondere Trägerfolie.

Vorteilhaft an der Erfindung ist, dass das Aktivmaterial in hoher Reinheit gewonnen werden kann. Der Grund dafür ist, dass beim Luftstrahlsieben sehr kleine Maschenweiten einsetzbar sind, so dass auch kleine Verunreinigungspartikel ferngehalten werden.

Vorteilhafterweise kann auch das Trägermaterial in hoher Reinheit zurückgewonnen werden kann. Beispielsweise wird als Träger häufig Aluminiumfolie verwendet, die nur dann effizient recycelt werden kann, wenn sie gewisse Mindestanforderungen hinsichtlich der Reinheit erfüllt. Beispielsweise eignet sich eine Trägerfolie mit mehr als 0,03 % Verunreinigungen nur wenig zum Recyceln. In der weiteren hydrometallurgischen Aufarbeitung neigen Übergangsmetallsalzlösungen aus einer sauren Lithiumlösung zur Agglomeration, wenn diese die bei der konventionellen Trennung üblichen ca. 1 Massen-% Aluminiumkontamination enthalten. Dieser Aspekt liegt darin begründet, dass sich Aluminiumhydroxid-Komplexe bilden, welche die Agglomerate fest verbinden. Diese festen Agglomerate lassen sich in einem herkömmlichen Prozess nicht vollständig vermahlen, was zu einem Materialverlust führt. Entsprechend hergestellte Aluminium-kontaminierte Übergangsmetallsalzlösungen müssten so vor der Vermahlung kostenintensiv aufgereinigt werden. Bei einer Reduktion des Aluminiumsgehalts auf unter 0,1 % ist dieser Effekt nicht mehr zu beobachten. Mit dem erreichten Resultat einer Al-Kontamination von nur 0,05-0,06 Massen-% lassen sich die Materialien demnach ökologisch und ökonomisch vorteilhaft recyceln. Dadurch, dass beim Aufprall auf das Sieb beim Luftstrahlsieben Aktivmaterial abgeklopft wird, lassen sich sehr hohe Reinheiten für den

Träger, insbesondere die Trägerfolie, erreichen.

Der Erfindung liegt die Erkenntnis zugrunde, dass das Erwärmen der Zellenfragmente auf die Zersetzungstemperatur zwar theoretisch ausreichen müsste, um den Binder so weit zu zerstören, dass das Aktivmaterial freigesetzt wird, dass aber dennoch das Aktivmaterial nur schwer vom Träger zu lösen ist. Aufgrund welchen Effekts das Aktivmaterial auch ohne Binder am Träger anhaftet, ist bislang noch ungeklärt.

Beim Luftstrahlsieben kommt es zu einem heftigen Aufprall des Trägers mit noch anhaftendem Aktivmaterial auf das Sieb. Dieser Aufprall löst noch anhaftendes Aktivmaterial ab, so dass es wiedergewonnen werden kann.

Wegen des Aufpralls des Trägers auf dem Sieb und den dadurch herbeigeführten Trenneffekt kann das Luftstrahlsieben auch als Prallsepararieren bezeichnet werden. Analog kann die Luftstrahlsiebvorrichtung auch als Prallseparierverrichtung und das Luftstrahlsieb auch als Prallseparator bezeichnet werden.

Erfindungsgemäß ist damit auch eine Aktivmaterial-Separationsanlage, die eine Prallseparationsvorrichtung, die zumindest mittelbar mit dem Ofen verbunden und eingerichtet ist zum Klassieren von Aktivmaterial und Träger, indem das Luftstrahlsieben so durchgeführt wird, dass das Aktivmaterial zumindest auch durch einen Aufprall auf ein Sieb der Prallseparationsvorrichtung und/oder auf eine Prallplatte von dem Träger getrennt wird.

Das Luftstrahlsieben ist bekannt als Laborverfahren zum Trennen von kleinen Partikeln von anderen kleinen Partikeln. Die wärmebehandelten Zellenfragmente haben jedoch einen um Größenordnungen größeren Durchmesser als die Partikel des Aktivmaterials, so dass die Fähigkeit des

Luftstrahlsiebens zur Trennung von zwei Arten von Partikeln mit jeweils kleinen Durchmessern für das erfindungsgemäße Verfahren irrelevant ist. Im erfindungsgemäßen Verfahren wird das Luftstrahlsieben auch als zusätzlicher Trennschritt eingesetzt.

Im Rahmen der vorliegenden Beschreibung wird unter dem Zerkleinern der Zellen insbesondere ein Zerschneiden, Zerschlagen, Zerhäckseln oder Zerdrücken verstanden. Das kann bei Temperaturen zwischen -195,79 °C -100 °C erfolgen, so dass nur geringe Anforderungen hinsichtlich des Temperaturregimes bestehen.

Unter dem Erwärmen der festen Zellenfragmente wird insbesondere ein Erwärmen in einem gasdichten Behälter verstanden, so dass beispielsweise Sauerstoff am Zutritt gehindert wird. Insbesondere erfolgt das Erwärmen unter Inertgas oder im Vakuum.

Unter dem Aktivmaterial wird insbesondere eine Verbindung verstanden, welche Lithiumionen reversibel aufnehmen und abgeben kann. Auf der Kathodenseite beispielsweise Lithiumnickelmangankobaltoxid, Lithiumnickelaluminiumoxid und Lithiumeisenphosphat, auf der Anodenseite beispielsweise Graphit.

Unter dem Träger für das Aktivmaterial wird insbesondere eine Trägerfolie verstanden, auf der das Aktivmaterial in Form von Partikeln aufgebracht ist. Beispielsweise handelt es sich bei der Trägerfolie um Aluminiumfolie. Bei dem Binder handelt es sich beispielsweise um Polyvenylidenfluorid (PVDF).

Das Zerkleinern der Zellen und/oder das Erwärmen der festen Zellenfragmente erfolgt bevorzugt unter Inertgas, beispielsweise unter Stickstoff. Günstig ist es, wenn das Zerkleinern der Zellen unter Umgebungstemperatur, insbesondere unter bei höchstens 30°, erfolgt, wobei es vorteilhaft

sein kann, wenn die Zerkleinerungsvorrichtung, mit der die Zellern zerkleinert werden, gekühlt wird, so dass der Wärmeeinfluss auf die Zellen gering bleibt.

Das erfindungsgemäße Verfahren wird vorzugsweise als kontinuierlicher Prozess durchgeführt, also nicht chargenweise.

Vorzugsweise beträgt der Durchsatz mehr als 100 Tonnen pro Jahr. Das Luftstrahlsieben wird bislang im Labormaßstab eingesetzt, wo es zur chargenweisen Bearbeitung von Proben genutzt wird. Diese Proben sind zudem sehr klein. Der Einsatz des Luftstrahlsiebens in einem Massenverfahren ist bislang unbekannt.

Gemäß einer bevorzugten Ausführungsform werden galvanische Zellen verwendet, deren Aktivmaterial aus Partikeln gebildet ist, wobei die Partikel eine Partikeldurchmesserverteilung haben und wobei diese Partikeldurchmesserverteilung einen 90%-Durchmesser besitzt, der demjenigen Durchmesser entspricht, der größer ist als der Durchmesser von 90 % aller Partikel und der kleiner ist als der Durchmesser von 10 % aller Partikel, wobei das Luftstrahlsieben mit einem Sieb erfolgt, dessen Maschenweite maximal dem 200-fachen des 90 %-Durchmessers entspricht. Der angegebene Partikeldurchmesser ist derjenige, der durch Siebung erhalten wird.

Vorzugsweise beträgt die Maschenweite des Siebs höchstens 250 µm, insbesondere höchstens 100 µm. Moderne Lithium-Ionen-Batterien besitzen ein Aktivmaterial, das aus Partikeln mit einem Durchmesser von weniger als 200 µm aufgebaut ist. Bei klassischen Siebverfahren, beispielsweise dem Vibrationssieben, muss eine deutlich größere Maschenweite gewählt werden, damit nicht die Siebzeit so groß wird, dass das Verfahren unwirtschaftlich wird. Eine größere Maschenweite jedoch hat zur Folge, dass mehr Schmutzpartikel durch das Sieb gelangen, so dass die Reinheit

des gewonnen Aktivmaterials sinkt.

Vorzugsweise umfassen die galvanischen Zellen je zumindest eine Anode und zumindest eine Kathode, wobei die Anoden und Kathoden gemeinsam erwärmt und die entstehenden wärmebehandelten Zellenfragmente in einem gemeinsamen Luftstrahlsieb klassiert werden. Unter dem Merkmal, dass die Anoden und die Kathoden gemeinsam erwärmt werden, wird verstanden, dass ein Gemisch aus beiden in ein und derselben Vorrichtung erwärmt wird. Das ist möglich, da das Luftstrahlsieben zu einer hinreichend sortenreinen Trennung von Stromsammlerfolie und Beschichtungspartikeln führt.

Vorzugsweise umfasst das Verfahren den Schritt, dass vor dem Erwärmen der Zellenfragmente auf die Zersetzungstemperatur die Zellenfragmente bei einer Trocknungs-Temperatur getrocknet werden, so dass trockene Zellenfragmente und Trocknungsdämpfe entstehen, wobei zumindest ein Teil der Trocknungsdämpfe kondensiert wird. Das hat den Vorteil, dass das Elektrolytlöschungsmittel zurückgewonnen werden kann. Es ist dabei vorteilhaft, wenn die Trocknungs-Temperatur unter 100 °C liegt, insbesondere unter 80 °C.

Vorzugsweise wird das Erwärmen der Zellenfragmente auf die Zersetzungs-Temperatur unter Inertgas oder im Vakuum durchgeführt. Das hat den Vorteil, dass Kupfer dann nicht oxidiert, so dass sowohl die Anode als auch die Kathode gemeinsam verarbeitet werden können. Das wiederum führt zu einem besonders einfachen Trennverfahren, das dennoch hoch sortenreine Recyclingprodukte zur Verfügung stellt. Unter Inertgas wird ein Gas oder Gasgemisch verstanden, das unter den vorliegenden Umgebungsbedingungen weder mit Kupfer noch mit Aluminium (Stromsammelfolien) reagiert. Das Kupfer kann so als wertvolle Komponente in hoher Reinheit zurückgewonnen werden.

Vorzugsweise umfasst das Verfahren den Schritt eines Behandelns der trockenen Zellfragmente mittels Magnetabscheider, so dass eisenfreie Zellenfragmente entstehen. Günstig ist es, wenn diese eisenfreien Zellenfragmente gesichtet werden, beispielsweise durch einen Zickzacksichter. Die eisenfreien Zellenfragmente werden dann auf die Zersetzungs-Temperatur des Binders erhitzt.

Die Zersetzungs-Temperatur beträgt vorzugsweise zumindest 350 °C und höchstens 800 °C. Es hat sich herausgestellt, dass bei diesen Temperaturen der Binder sicher zersetzt wird.

Vorzugweise wird beim Zerkleinern der Zellen unter Inertgas zumindest auch das Elektrolytmittel abgezogen. Das steigert die Recyclingquote.

Gemäß einer bevorzugten Ausführungsform wird das Zerkleinern der Zellen so durchgeführt, dass der Median der längsten Durchmesser der resultierenden Folienfragmente 200 µm nicht unterschreitet. Gemäß einer bevorzugten Ausführungsform wird das Zerkleinern der Zellen so durchgeführt, dass die Folienfragmente zu zumindest 80% einen Durchmesser von über 200 µm haben, wobei der Durchmesser durch Siebung bestimmt wird.

Gemäß einer bevorzugten Ausführungsform erfolgt das Luftstrahlsieben so, dass ein Luftdurchsatz von mindestens 250 Kubikmeter Luft pro Stunden und Quadratmeter Siebfläche erreicht wird, insbesondere mindestens 500 Kubikmeter Luft pro Stunden und Quadratmeter Siebfläche. Besonders günstig sind mindestens 750 Kubikmeter Luft pro Stunden und Quadratmeter Siebfläche. Es ergibt sich dann eine so hohe Partikelgeschwindigkeit, dass der Aufprall zu einem hohen Separationsgrad führt.

Vorzugsweise ist hinter dem Luftstrahlsieb ein Zyklon angeordnet. Günstig ist es zudem, wenn die Trägerluft hinter dem Luftstrahlsieb abge-

saugt wird.

Für einen möglichst geringen Energieverbrauch ist es günstig, wenn das Sieb zumindest weitgehend vollflächig mit dem partikelhaltigen Luftstrom beaufschlagt wird.

Die Aktivmaterial-Separationsanlage besitzt vorzugsweise einen Vortrockner, der im Materialfluss hinter der Zellenzerkleinerungsvorrichtung und vor dem Ofen angeordnet ist. Der Vortrockner ist eingerichtet, um die Zellenfragmente auf die Trocknungs-Temperatur zu erwärmen.

Vorzugsweise umfasst die Aktivmaterial-Separationsanlage zudem einen Magnetabscheider im Materialfluss hinter dem Vortrockner und vor dem Ofen.

Günstig ist es, wenn die Aktivmaterial-Separationsanlage einen Rührreaktor zur Leitsalzrückgewinnung aufweist, der mit der Zellenzerkleinerungsvorrichtung verbunden ist und aus der Zellenzerkleinerungsvorrichtung zumindest einen Teil der Flüssigfraktion abzieht.

Unter dem Verfahren zum Wiedergewinnen von Aktivmaterial der galvanischen Zelle wird insbesondere auch ein Verfahren verstanden, bei dem Elektroden aus der Elektrodenproduktion bearbeitet werden, ohne dass die Elektroden in eine galvanische Zelle eingebaut worden sind, jedoch in eine galvanische Zelle eingebaut werden könnten. In anderen Worten kann es sich bei dem Verfahren auch um ein Verfahren zum In-Prozess-Recycling handeln.

Gemäß einer bevorzugten Ausführungsform ist bezüglich eines Luft- und Zellfragmentstroms hinter dem Sieb eine Prallplatte angeordnet. Diese führt dazu, dass sich die Partikel aus Aktivmaterial, die nur eine schwache Kollision mit dem Sieb hatten, beim Aufprall auf die Prallplatte vom

Träger lösen, was die Trennwirkung erhöht.

Gemäß einer bevorzugten Ausführungsform umfasst die Zellenzerkleinerungsvorrichtung zumindest eine Prallmühle. Durch die Vielzahl an Zusammenprallvorgängen in der Prallmühle wird die Bindung zwischen Aktivmaterial und Träger gelockert. Vorzugsweise umfasst die Zellenzerkleinerungsvorrichtung zudem zumindest einen Schredder und/oder eine Zerschneidvorrichtung, die besonders günstig in Materialflussrichtung vor der Prallmühle angeordnet ist.

Erfindungsgemäß ist zudem die Verwendung eines Luftstrahlsiebs in einem Verfahren, insbesondere einem kontinuierlichen Verfahren, zum Wiedergewinnen von Aktivmaterial, insbesondere von Lithium und/oder Lithiumverbindungen, beispielsweise aus gebrauchten Batterien und/oder Akkumulatoren sowie aus Ausschuss bei der Produktion von Batterien und/oder Akkumulatoren.

Im Folgenden wird die Erfindung anhand der beigefügten Zeichnungen näher erläutert. Dabei zeigt

Figur 1 ein Flussdiagramm einer erfindungsgemäßen Aktivmaterial-Separationsanlage zum Durchführen eines erfindungsgemäßen Verfahrens und

Figur 2 eine Explosionszeichnung einer Ausführungsform eines Prallseparators der Aktivmaterial-Separationsanlage nach Figur 1.

Figur 1 zeigt ein Flussdiagramm einer erfindungsgemäßen Aktivmaterial-Separationsanlage 10, die eine Zellenzerkleinerungsvorrichtung 12 besitzt, der galvanische Zellen 14.1, 14.2, ... zugeführt werden. Die Zellenzerkleinerungsvorrichtung 12 ist gasdicht und umfasst eine Gaswaschkolonne 16, eine Umwälzpumpe 18 und einen Kondensator 20, mittels denen ein Inertgas, beispielsweise Stickstoff, umgewälzt und gereinigt wird. Die Zellenzerkleinerungsvorrichtung umfasst zudem einen Inertgastank 22 zum Einstellen eines Inertgasdrucks in der Zellenzerkleinerungsvorrichtung 12. Der Kondensator 20 besitzt einen Elektrolytabzug 24, über den kondensierter Elektrolyt abgezogen werden kann.

In Materialstromrichtung hinter der Zellenzerkleinerungsvorrichtung 12 kann ein Rührreaktor 26 angeordnet sein, der zum Abziehen einer Flüssigfraktion aus der Zellenzerkleinerungsvorrichtung 12 und zum Rühren derselben angeordnet ist, so dass Leitsalz aus den galvanischen Zellen 14 (Bezugszeichen ohne Zählsuffix bezeichnen das Objekt als solches) zurückgewonnen werden kann.

In Materialflussrichtung hinter der Zellenzerkleinerungsvorrichtung 12 ist ein Vortrockner 28 angeordnet, der über eine Leitung 30 mit der Zellenzerkleinerungsvorrichtung 12 verbunden ist. Beim Zerkleinern der galvanischen Zellen 14 entstandene Zellenfragmente gelangen durch die Leitung

30 in den Vortrockner 28. Dort herrscht eine Trocken-Temperatur T_t von vorzugsweise T_t = 80 °C. Der Vortrockner 28 umfasst eine Umwälzpumpe 32 und einen Kondensator 34 zum Abkondensieren von Elektrolyt, der über eine Leitung 36 abgezogen werden kann. Der Vortrockner 28 ist gasdicht ausgebildet und wird mit Inertgas durch die Umwälzpumpe 32 gespült.

In Materialstromrichtung hinter dem Vortrockner 28 ist ein Abscheider 38 angeordnet, der einen Magnetabscheider und gegebenenfalls zusätzlich einen Wirbelstromabscheider umfasst. Das magnetische Material wird über eine Ableitung 40 abgezogen. Das nicht magnetische Material gelangt in einen Sichter 42, beispielsweise einen Zickzacksichter. Der Sichter 42 ist wie der Abscheider 38 optional und besonders dann vorteilhaft, wenn wie im vorliegenden Fall nur die Kathode der galvanischen Zellen weiterverwendet werden soll. Die Anoden werden ebenso wie gegebenenfalls vorliegende Pouchfolien abgezogen.

In Materialflussrichtung hinter dem Sichter 42 ist ein Ofen 44 angeordnet, in dem eine Zersetzungstemperatur T_z von 400 bis 600 °C herrscht. Dadurch zersetzt sich der Binder, beispielsweise Polyvenylidenfluorid (PVDF). Das Zersetzungsprodukt wird über eine Ableitung 46 abgeleitet und in eine Gaswaschkolonne 48 geführt.

In Materialflussrichtung hinter dem Ofen 44 ist ein Luftstrahlsieb 50 angeordnet. Das Luftstrahlsieb 50 umfasst ein Siebelement 52. Die aus dem Ofen 44 kommenden wärmebehandelten Zellenfragmente werden auf ein Luftstrahlsieb gefördert. Dieses ist dadurch charakterisiert, dass ober- oder unterhalb des feinmaschigen Siebelements (20-200 µm) ein Luftstrom zugeführt wird. Dieser wird unterhalb des Siebes abgezogen. Durch die Luftströmung werden die Elektrodenfragmente aufgewirbelt und dann mechanisch beansprucht. Diese Beanspruchung unterstützt die Separation von Trägerfolie und Beschichtung. Die Aktivmaterial-Partikel werden so im Luftstrom durch die Maschen abzogen und im Folgenden durch einen Zyk-

lon aus dem Luftstrom abgetrennt. Die Folienfraktion (Aluminium und Kupfer) wird durch das Siebelement zurückgehalten und kann als Metallfraktion gewonnen und wieder verwendet werden.

Figur 2 zeigt eine Explosionsansicht eines Prallseparators oder Luftstrahlsiebs. Die zu trennenden wärmebehandelten Zellenfragmente werden auf eine Seite des Siebs zugeführt, von der anderen Seite wird ein sich bewegender Luftstrahl, beispielsweise durch eine sich bewegende Düsenleiste in Richtung auf das Sieb geblasen. Dadurch werden die Zellenfragmente gegen die Prallplatte in Form des Pralldeckels geschleudert und trennen sich dort in den Träger und die Beschichtung.

Auf der Seite des Siebs, von der der Luftstrahl auf das Sieb geblasen wird, befindet sich ein Ansaugstutzen, mittels dem die eingeblasene Luft zusammen mit der Beschichtung abgezogen wird. Die Prallplatte wird durch Distanzringe vom Sieb beabstandet gehalten.

Bezugszeichenliste

10	Aktivmaterial-Separationsanlage	40	Ableitung
12	Zellenzerkleinerungsvorrichtung	42	Sichter
14	galvanische Zelle	44	Ofen
16	Gaswaschkolonne	46	Ableitung
18	Umwälzpumpe	48	Gaswaschkolonne
20	Kondensator	50	Luftstrahlsieb
22	Inertgastank	52	Siebelement
24	Elektrolytabzug	54	Partikel
26	Rührreaktor	56	Ableitung
28	Vortrockner	58	Zyklon
30	Leitung	T_t	Trocken-Temperatur
32	Umwälzpumpe	T_z	Zersetzungs-Temperatur
34	Kondensator		
36	Leitung		
38	Abscheider		

Patentansprüche:

1. Verfahren zum Wiedergewinnen von Aktivmaterial einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial und einen Binder zum Verbinden des Aktivmaterials und des Trägers umfasst,
mit den Schritten:
   (a) Zerkleinern der Zellen, insbesondere unter Inertgas oder im Vakuum, so dass zumindest auch feste Zellenfragmente entstehen,
   (b) Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur (T_z), die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, vorzugsweise unter Intergas oder im Vakuum so dass wärmebehandelte Zellenfragmente entstehen, und
   (c) Klassieren der wärmebehandelten Zellenfragmente,
dadurch gekennzeichnet, dass
   (d) das Klassieren ein Luftstrahlsieben umfasst,
   (e) wobei das Luftstrahlsieben so durchgeführt wird, dass Aktivmaterial von dem Träger getrennt wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass
   - galvanische Zellen (14) verwendet werden, deren Aktivmaterial aus Partikeln gebildet ist,
   - die Partikel eine Partikeldurchmesserverteilung haben, wobei die Partikeldurchmesserverteilung einen 90%-Durchmesser besitzt, der demjenigen Durchmesser entspricht, der größer ist als der Durchmesser von 90% aller Partikel und der kleiner ist als der Durchmesser von 10% aller Partikel, und
   - das Luftstrahlsieben mit einem Sieb erfolgt, dessen Maschenweite maximal dem 200-fachen des 90%-Durchmessers entspricht.

3. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die Maschenweite des Siebs höchstens 200 µm beträgt.

4. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die galvanischen Zellen (14) je zumindest eine Anode und zumindest eine Kathode umfassen und die Anoden und Kathoden gemeinsam erwärmt werden und die entstehenden wärmebehandelten Zellenfragmente in einem gemeinsamen Luftstrahlsieb (50) klassiert werden.

5. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch den Schritt:

(a2) vor dem Erwärmen der Zellenfragmente auf die eine Zersetzungs-Temperatur (T_z)

- Trocknen der Zellenfragmente bei einer Trocknungs-Temperatur (T_t), so dass trockene Zellenfragmente und Trocknungsdämpfe entstehen und
- Kondensieren zumindest eines Teils der Trocknungsdämpfe zum Zweck der Rückgewinnung der Elektrolytlösemittel.

6. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Erwärmen der Zellenfragmente auf die Zersetzungs-Temperatur (T_z) unter Inertgas oder in einem Vakuum durchgeführt wird.

7. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch die Schritte:

(a3) Behandeln der trockenen Zellenfragmente mittels Magnetabscheidung, so dass eisenfreie Zellenfragmente entstehen, und

(a4) Sichten der eisenfreien Zellenfragmente.

8. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die galvanischen Zellen (14) einen Elektrolyten aufweisen und/oder beim Zerkleinern der Zellen unter Inertgas zumindest auch der Elektrolyt abgezogen wird.

9. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Zerkleinern der Zellen so durchgeführt wird, dass der Median der längsten Durchmesser der resultierenden Folienfragmente 200 µm nicht unterschreitet.

10. Aktivmaterial-Separationsanlage, insbesondere Aktivmetall-Separationsanlage (10), die ausgebildet ist zum Wiedergewinnen von Aktivmaterial, insbesondere Lithium, einer galvanischen Zelle (14), die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und einen Elektrolyten umfasst, mit

(a) einer Zellenzerkleinerungsvorrichtung (12) zum Zerkleinern der Zellen, wobei die Zellenzerkleinerungsvorrichtung gasdicht ist und eine Inertgaszuführvorrichtung umfasst, so dass die Zellen unter Inertgas zu Zellenfragmenten zerkleinerbar sind, und

(b) einem Ofen (44), der zumindest mittelbar mit der Zellenzerkleinerungsvorrichtung (12) verbunden und eingerichtet ist zum Erwärmen der Zellenfragmente auf eine Zersetzungs-Temperatur (T_z), die so hoch gewählt ist, dass der Binder sich zersetzt,

gekennzeichnet durch

(c) eine Luftstrahlsiebvorrichtung, die zumindest mittelbar mit dem Ofen (44) verbunden und eingerichtet ist zum Klassieren von Aktivmaterial und Träger, indem das Luftstrahlsieben so durchgeführt wird, dass das Aktivmaterial von dem Träger getrennt wird.

11. Aktivmaterial-Separationsanlage nach Anspruch 10, dadurch gekennzeichnet, dass die Luftstrahlsiebvorrichtung ein Sieb, eine Prallplatte und eine Düsenleiste aufweist, wobei das Sieb bezüglich eines Luftstroms durch die Luftstrahlsiebvorrichtung zwischen Düsenleiste und Prallplatte angeordnet ist.

12. Aktivmaterial-Separationsanlage nach Anspruch 11, dadurch gekennzeichnet, dass die Prallplatte einen Abstand von mindestens 30 Millimetern, insbesondere mindestens 40 Millimetern, vom Sieb hat.

13. Aktivmaterial-Separationsanlage nach Anspruch 11 oder 12, dadurch gekennzeichnet, dass die Prallplatte einen Abstand von höchstens 250 Millimetern, insbesondere mindestens 200 Millimetern, vom Sieb hat.

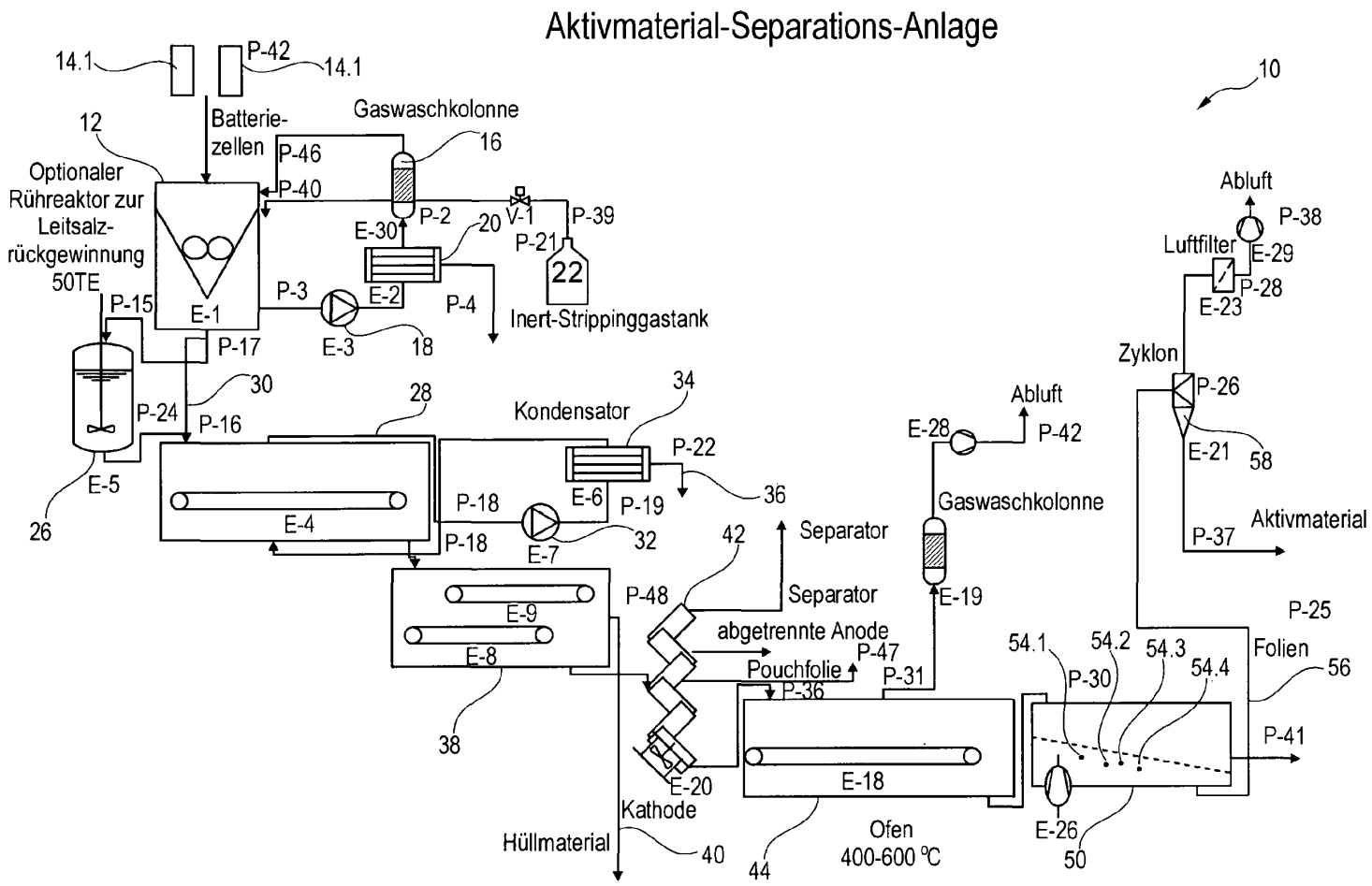
Aktivmaterial-Separations-Anlage
14.1
P-42
14.1
Batterie-
zellen
Gaswaschkolonne
16
12
P-46
P-40
Optionaler
Rührreaktor zur
Leitsalz-
rückgewinnung
50TE
E-30
P-2
20
V-1
P-39
P-21
22
Inert-Strippinggastank
P-15
E-1
P-3
E-2
P-4
E-3
18
P-17
30
P-24
P-16
28
Kondensator
34
P-22
E-5
26
E-4
P-18
E-6
P-19
36
P-18
E-7
32
42
Separator
P-48
Separator
E-9
E-8
abgetrennte Anode
P-47
Pouchfolie
P-36
38
E-20
Kathode
Hüllmaterial
40
E-18
Ofen
400-600 °C
44
10
Abluft
P-38
Luftfilter
E-29
P-28
E-23
Zyklon
P-26
E-21
58
Abluft
E-28
P-42
Gaswaschkolonne
E-19
P-37
Aktivmaterial
P-25
54.1
54.2
54.3
54.4
Folien
56
P-30
P-31
P-41
E-26
50

Fig. 1

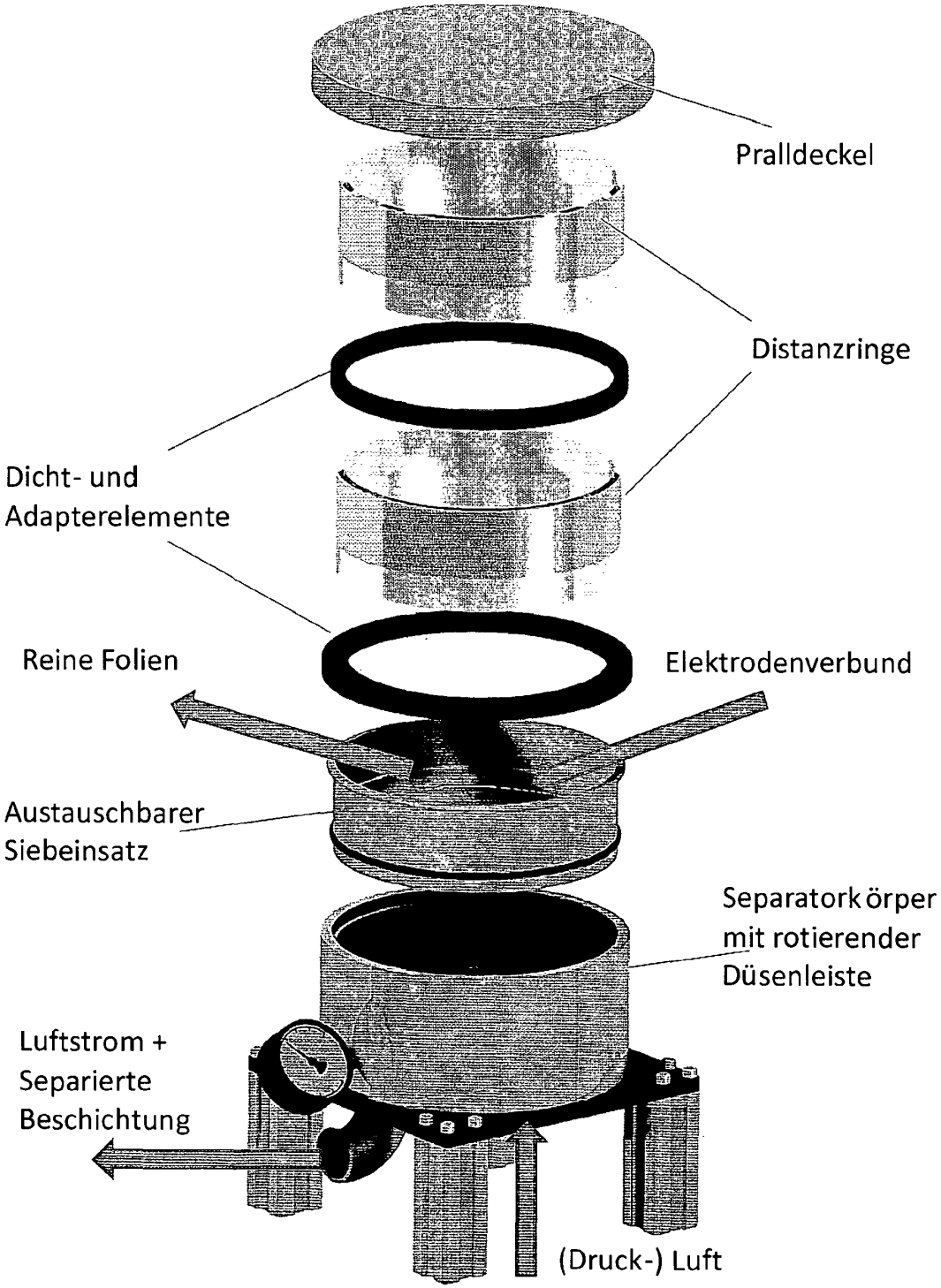
Pralldeckel
Distanzringe
Dicht- und
Adapterelemente
Reine Folien
Elektrodenverbund
Austauschbarer
Siebeinsatz
Separatorkörper
mit rotierender
Düsenleiste
Luftstrom +
Separierte
Beschichtung
(Druck-) Luft

Fig. 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/DE2012/000811

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M10/54 B07B4/08
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M B07B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 198 42 658 A1 (TOSHIBA KAWASAKI KK [JP]) 1 April 1999 (1999-04-01) page 4, line 10 - line 15 page 4, line 36 - line 37 page 7, line 19 - line 24 page 9, line 50 - line 60 figure 1	1-13
A	EP 1 215 742 A1 (TOSHIBA KK [JP]) 19 June 2002 (2002-06-19) paragraphs [0106], [0107]	1-13
A	DE 298 03 442 U1 (RHEINISCHE WERKZEUG & MASCHF [DE]) 12 November 1998 (1998-11-12) cited in the application the whole document	1-13

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
3 December 2012	11/12/2012

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Schwake, Andree

Form PCT/ISA/210 (second sheet) (April 2005)

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CHRISTIAN HANISCH ET AL: "Recovery of Active Materials from Spent Lithium-Ion Electrodes", GLOCALIZED SOLUTIONS FOR SUSTAINABILITY IN MANUFACTURING-PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING; PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING, SPRINGER, BERLIN, DE; TECHNISCH, 1 January 2011 (2011-01-01), pages 85-89, XP008158000, DOI: 10.1007/978-3-642-19692-8_15 ISBN: 978-3-642-19691-1 the whole document -----	1-13
A	KRUEGER S ET AL: "Recycling Of Lithium-Ion Batteries", 18TH INTERNATIONAL CONFERENCE ON SOLID STATE IONICS (SSI-18), WARSAW UNIVERSITY OF TECHNOLOGY, WARSZAWA, POLAND, 1 January 2011 (2011-01-01), XP008158002, the whole document -----	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/DE2012/000811

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		JP 11097076 A	09-04-1999
EP 1215742 A1	19-06-2002	CA 2374245 A1	28-12-2000
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		EP 1215742 A1	19-06-2002
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		US 6811923 B1	02-11-2004
		WO 0079621 A1	28-12-2000
DE 29803442 U1	12-11-1998	NONE	

INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
PCT/DE2012/000811

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
INV. H01M10/54 B07B4/08
ADD.

Nach der Internationalen Patentklassifikation (IPC) oder nach der nationalen Klassifikation und der IPC

B. RECHERCHIERTE GEBIETE

Recherchierter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)
H01M B07B

Recherchierte, aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen

Während der internationalen Recherche konsultierte elektronische Datenbank (Name der Datenbank und evtl. verwendete Suchbegriffe)
EPO-Internal, WPI Data

C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
A	DE 198 42 658 A1 (TOSHIBA KAWASAKI KK [JP]) 1. April 1999 (1999-04-01) Seite 4, Zeile 10 - Zeile 15 Seite 4, Zeile 36 - Zeile 37 Seite 7, Zeile 19 - Zeile 24 Seite 9, Zeile 50 - Zeile 60 Abbildung 1 -----	1-13
A	EP 1 215 742 A1 (TOSHIBA KK [JP]) 19. Juni 2002 (2002-06-19) Absätze [0106], [0107] -----	1-13
A	DE 298 03 442 U1 (RHEINISCHE WERKZEUG & MASCHF [DE]) 12. November 1998 (1998-11-12) in der Anmeldung erwähnt das ganze Dokument -----	1-13

-/--

[X] Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen [X] Siehe Anhang Patentfamilie

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Datum des Abschlusses der internationalen Recherche	Absendedatum des internationalen Recherchenberichts
3. Dezember 2012	11/12/2012

Name und Postanschrift der Internationalen Recherchenbehörde	Bevollmächtigter Bediensteter
Europäisches Patentamt, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Schwake, Andree

Formblatt PCT/ISA/210 (Blatt 2) (April 2005)

Duesenfeld Ex. 2014-390

C. (Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
A	CHRISTIAN HANISCH ET AL: "Recovery of Active Materials from Spent Lithium-Ion Electrodes", GLOCALIZED SOLUTIONS FOR SUSTAINABILITY IN MANUFACTURING-PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING; PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING, SPRINGER, BERLIN, DE; TECHNISCH, 1. Januar 2011 (2011-01-01), Seiten 85-89, XP008158000, DOI: 10.1007/978-3-642-19692-8_15 ISBN: 978-3-642-19691-1 das ganze Dokument -----	1-13
A	KRUEGER S ET AL: "Recycling Of Lithium-Ion Batteries", 18TH INTERNATIONAL CONFERENCE ON SOLID STATE IONICS (SSI-18), WARSAW UNIVERSITY OF TECHNOLOGY, WARSZAWA, POLAND, 1. Januar 2011 (2011-01-01), XP008158002, das ganze Dokument -----	1-13

INTERNATIONALER RECHERCHENBERICHT

Angaben zu Veröffentlichungen, die zur selben Patentfamilie gehören

Internationales Aktenzeichen
PCT/DE2012/000811

Im Recherchenbericht angeführtes Patentdokument	Datum der Veröffentlichung	Mitglied(er) der Patentfamilie	Datum der Veröffentlichung
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PUB-NO: WO2013023640A1
DOCUMENT-IDENTIFIER: WO 2013023640 A1
TITLE: METHOD FOR RECLAIMING ACTIVE MATERIAL FROM A GALVANIC CELL, AND AN ACTIVE MATERIAL SEPARATION INSTALLATION, PARTICULARLY AN ACTIVE METAL SEPARATION INSTALLATION
PUBN-DATE: February 21, 2013
PATENT-FAMILY-ID: 46875602

INVENTOR-INFORMATION:

NAME	COUNTRY
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HASELRIEDER WOLFGANG	DE
HANISCH CHRISTIAN	DE

APPLICANT:

NAME	COUNTRY
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HASELRIEDER WOLFGANG	DE
HANISCH CHRISTIAN	DE
TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA	DE

APPL-NO: DE 2012000811 W
APPL-DATE: August 10, 2012

PRIORITY-DATA:

PRIORITY-NO	PRIORITY-APPL-DATE
DE 102011110083 A	20110812

CPC-CURRENT:

TYPE	CPC/DATE
CPCI	H01M10/54 20130101
CPCI	B07B4/08 20130101
CPCI	C22B26/12 20130101
CPCI	B02C23/06 20130101
CPCI	B02C23/08 20130101

CPCA	H01M10/052 20130101
CPCA	Y02W30/84 20150501
CPCA	B02C23/00 20130101

INT-CL-CURRENT:

TYPE	**IPC/DATE**
IPCP	H01M10/54 20060101
IPCS	B07B4/08 20060101

REF-CITED:

PHASE-COUNTRY/NUMBER/KIND/DATE-NPL-INFO

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KRUEGER S ET AL: "Recycling Of Lithium-Ion Batteries", 18TH INTERNATIONAL CONFERENCE ON SOLID STATE IONICS (SSI-18), WARSAW UNIVERSITY OF TECHNOLOGY, WARSZAWA, POLAND, 1 January 2011 (2011-01-01), XP008158002

CHRISTIAN HANISCH ET AL: "Recovery of Active Materials from Spent Lithium-Ion Electrodes", GLOCALIZED SOLUTIONS FOR SUSTAINABILITY IN MANUFACTURING-PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING; PROCEEDINGS OF THE 18TH CIRP INTERNATIONAL CONFERENCE ON LIFE CYCLE ENGINEERING, SPRINGER, BERLIN, DE; TECHNISCH, 1 January 2011 (2011-01-01), pages 85 - 89, XP008158000, ISBN: 978-3-642-19691-1, DOI: 10.1007/978-3-642-19692-8_15

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ABSTRACT:

The invention relates to a method for reclaiming active material of a galvanic cell that comprises an active material, a carrier for said active material, and a binder for binding the active material and the carrier, said method comprising the following steps:(a) comminuting the cells, particularly under inert gas or in a vacuum, such that solid cell fragments are at least also produced, (b) heating the solid cell fragments to a decomposition temperature (Tz) selected to be high enough that the binder decomposes and/or evaporates, preferably under inert gas or in a vacuum, so as to produce thermally treated cell fragments, and (c) classifying these thermally treated cell fragments, wherein (d) the classification comprises an air-jet sieving process and (e) the air-jet sieving is implemented such that active material is separated from the carrier.

PTO/SB/08a (02-18)
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(Not for submission under 37 CFR 1.99)

Application Number	15569484
Filing Date	2017-10-26
First Named Inventor	Christian HANISCH
Art Unit	
Examiner Name	
Attorney Docket Number	03101160US

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	2	20060101683	KR		2005-03-21			☒

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	1	HIGGELKE: "Infinite Power Solutions stellt papierdunnen Akku vor", "(Infinite Power Solution presents paper-thin Battery)", 2012-06-06	☒
	2	VDMA-Norm 24351 Trocknungstechnik (VDMA=German Engineering Federation; VDMA Standared 24351 - Drying Technology - Basic terms and definitions), 1999-01	☐
	3	HERAUSGEBER: "Florke/Wolff Kursthemen Chemis - Allgemeine und Phsyikalische Chemie", "(Course Issues Chemisty- General and Physical Chemistry)", 1985	☒

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The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

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A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2019-04-18
Name/Print	Michael E. Whitham	Registration Number	32635

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DESCRIPTION KR20060101683

(Dry-method of separating Co from battery scarp)

The present invention relates to a method of cobalt dry separation of waste batteries. Lithium-ion batteries (including primary and secondary batteries) are widely used as power sources for small electric and electronic equipment due to their high energy density and light weight and light weight characteristics. A spent lithium ion battery has been obtained by a specific method in terms of environmental pollution and resource recycling. Generally, techniques for obtaining cobalt or cobalt oxide from a spent lithium ion battery are disclosed in Korean Patent Registration No. 10-0448273 (2004.09.01), Korean Patent Registration No. 10-040742 (October 27, 2003) and Korean Patent Publication No. 2003- 0011737 (2003.02.11) and the like, and a technique for obtaining a lithium-cobalt oxide is disclosed in Korean Patent Publication No. 2003-0093583 (2003.12.11). These techniques are capable of obtaining high purity cobalt (or cobalt oxide) and lithium-cobalt oxide by the wet process. but since the process of obtaining is very complicated and requires precise control. it is not possible to process large amounts of waste lithium- Of course, there is a problem that strong acid or strong alkali is used, which can cause secondary pollution. An object of the present invention, which is created to solve the above-mentioned problems, is to provide a dry battery capable of obtaining cobalt or cobalt oxide from a spent lithium ion battery by a dry method without secondary contamination which may be caused in the step of obtaining cobalt or cobalt oxide. And a cobalt dry separation method using the same. The object of the present invention is achieved by a method of manufacturing a lithium ion secondary battery comprising the steps of: (a) disrupting at least one spent lithium ion battery; (b) drying the crushed material to remove the electrolyte; (c) finely pulverizing the crushed material from which the electrolyte is removed. and separating the cobalt compound from the finely pulverized material using a particle size difference; and (d) reducing the cobalt compound to obtain pure cobalt. Preferably, the step (a) of the present invention is such that the waste lithium ion battery has an area of 100 to 1,600 mm 2.

Preferably, in the step (b) of the present invention, the pulverized product is dried by hot air at 300 to 550 ° C to completely remove the electrolyte. Preferably, the step (c) of the present invention comprises finely pulverizing the crushed material using a vibrating mill or a ball mill, wherein the cobalt compound is pulverized to a powder having a particle size of 50 to 100 mesh. A step (c) of dispersing the cobalt compound powder by supplying air at a high pressure to the fine pulverized product; and (2) a step (c) of dispersing the powder of the cobalt compound by passing through a filter for removing impurities to remove impurities, (C) -3 step of obtaining a compound powder. Preferably, the step (c) of the present invention is a step (c) of finely pulverizing the crushed material using a vibrating mill or a ball mill, wherein the cobalt compound is pulverized to a powder having a particle size of 50 to 100 meshes And (c) 5-5 steps of providing the fine pulverized product on the upper side of the screen device in which the pulverization is performed, thereby to obtain a cobalt compound powder by free fall.

Hereinafter, the construction of the present invention will be described in detail. Step (a) - At least one waste lithium ion battery crushed waste lithium ion battery is crushed to have an area of 100 to 1,600 mm 2 by using a crusher. This method is obtained by mixing a case part made of iron and a content part in a crushed product. Another method is to separate the contents of the case of the waste lithium ion battery from the contents of the case to separate the contents from the case by cutting the center portion or both ends of the waste lithium ion battery and then crushing the contents using a crusher . (B) Step - Drying the lump to remove the electrolyte The lump is dried at a temperature of 300 to 550 (deg.) C The electrolyte is completely removed by hot air drying. When the drying process is performed at a temperature of only 300 ° C, the drying time is prolonged and the drying performance is not good. When drying is performed at a temperature higher than 550 " C, the paste and organic matter There is a problem that pollutants are generated. The drying time is determined by the amount of the crushed material. (C) Step - Crushing of the crushed material and separation of the cobalt compound The crushed material is dried and pulverized by means of a vibrating mill or a ball mill. Here, it is very important to pulverize the cobalt compound to a powder having a particle size of 50 mesh to 100 mesh. The cobalt compound is present as a powder having a particle size of 50 to 100 meshes at the time of fine grinding, and the aluminum as the positive electrode component and the copper as the negative electrode component are each present as a thin plate. Nickel and iron are present at a ratio of 5 to 15 times it has grain size. The powder (powder) is sprayed only with the above-described cobalt compound powder by the supplied high-pressure air. The aluminum and the

copper thin plate except for the cobalt compound powder are pressed on the surface of the mill, and nickel And iron components remain in the wheat.

Here, the supplied high-pressure air has a pressure and an air amount corresponding thereto such that only the cobalt compound powder is scattered (air classification method). The scattered cobalt compound powder is collected after being flowed through a closed path such as a duct or the like. Here, since impurities (particularly aluminum or copper foil) may be mixed in the scattered cobalt compound powder, it is preferable to obtain the pure cobalt compound powder by removing the above-mentioned impurities via the filter for removing impurities in the pre-collecting step . The method of obtaining a simple cobalt compound powder in addition to the air classification method described above is to supply finely pulverized powder to an upper part of a swinging screen device provided with a mesh, It is preferable that the particle size is large. (D) Step - Reduction of Cobalt Compound Powder to Obtain Pure Cobalt Powder [0051] The cobalt compound powder obtained in the step (c) described above is obtained by reducing the powder of cobalt compound to the lower portion of the screen device. Since a small amount of carbon powder may be contained in the compound powder, pure cobalt powder can be obtained by removing carbon and then reducing it by heating at a high temperature of 300 ° C or higher. While the present invention has been described in connection with what is presently considered to be practical exemplary embodiments, it is to be understood that the invention is not limited to the disclosed embodiments, but, on the contrary, It is obvious that it belongs to the technical idea of.

The present invention having the above-mentioned constitution has an effect of obtaining a large amount of pure cobalt at a high price from a spent lithium ion battery. In addition, the present invention having such a configuration is characterized in that the process is not dangerous because strong acid or strong alkali is not used in the recycling process of the waste lithium ion battery, and secondary pollution due to the work process is prevented.

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CLAIMS KR20060101683

1.

(a) breaking at least one spent lithium ion battery; (b) drying the crushed material to remove the electrolyte; (c) finely pulverizing the crushed material from which the electrolyte is removed, and separating the cobalt compound from the finely pulverized material using a particle size difference; (d) reducing the cobalt compound to obtain pure cobalt.

2.

The method of claim 1, wherein the step (a) is performed such that the waste lithium ion battery has an area of 100 to 1,600 mm 2.

3.

The method of claim 1, wherein the step (b) comprises completely removing the electrolyte by hot-air drying the crushed product at 300 to 550 ° C.

4.

The method according to claim 1, wherein step (c) comprises: (c) pulverizing the pulverized product using a vibrating mill or a ball mill so that the cobalt compound is a powder having a particle size of 50 to 100 mesh; (C) a step (c) of dispersing the cobalt compound powder by supplying air at a high pressure to the fine pulverized product, and (2) a step of passing the pure cobalt compound powder through the impurity removing filter by passing the scattered cobalt compound powder through a filter for removing impurities (C) - 3 steps of capturing and collecting the cobalt dry separation of waste batteries.

5.

The method according to claim 1, wherein the step (c) comprises: (c) pulverizing the crushed material using a vibrating mill or a ball mill so that the cobalt compound is a powder having a particle size of 50 to 100 mesh; And (c) 5-5 steps of providing the fine pulverized product on the upper part of the screen device to be shaken to obtain a cobalt compound powder by free fall. [5] The cobalt dry separation method of claim 5,

(19)대한민국특허청(KR)
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심사청구 : 있음

(54) 폐건전지의 코발트 건식 분리방법

요약

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것으로, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 구성됨으로써, 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있음은 물론, 폐리튬 이온 전지의 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2차 오염을 원천 방지할 수 있는 효과가 있다.

색인어

폐건전지, 건전지 재생, 코발트, 건식 분리

명세서

발명의 상세한 설명

발명의 목적

발명이 속하는 기술 및 그 분야의 종래기술

본 발명은 폐건전지의 코발트 건식 분리 방법에 관한 것이다.

리튬 이온 전지(1차 및 2차 전지 포함)는 고 에너지 밀도 및 경박 경량의 특성으로 인하여 소형 전기, 전자장비의 전원 등으로 폭넓게 활용되고 있다. 사용이 완료된 폐리튬 이온 전지는 환경 오염 및 자원 재활용의 측면에서 특정 방법에 의해 유가 물질이 수득되고 있다.

통상적으로, 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 수득하는 기술은 대한민국 특허등록번호 10-0448273(2004.09.01), 대한민국 특허등록번호 10-040742(2003.10.27) 및 대한민국 특허공개번호 특2003-0011737(2003.02.11) 등에 다양하게 개시되어 있으며, 리튬-코발트 산화물을 수득하는 기술은 대한민국 특허공개번호 특2003-0093583호(2003.12.11)에 개시되어 있다.

이러한 기술들은 습식 방법에 의해 고순도의 코발트(또는 코발트 산화물) 및 리튬-코발트 산화물을 수득할 수 있으나, 수득 공정이 매우 복잡하고 정밀 제어가 요구되기 때문에 다량의 폐리튬 이온 전지를 처리할 수 없을은 물론, 강산 또는 강알카리가 사용됨으로써 2차 오염을 유발할 수 있는 문제점이 있다.

발명이 이루고자 하는 기술적 과제

상기와 같은 문제점을 해소하기 위하여 창출된 본 발명의 목적은, 코발트 또는 코발트 산화물 수득 공정시 야기될 수 있는 2차 오염없이 폐리튬 이온 전지로부터 코발트 또는 코발트 산화물을 건식 방법으로 수득할 수 있는 폐건전지의 코발트 건식 분리 방법을 제공함에 있다.

이러한 본 발명의 목적은, (a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와; (b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와; (c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와; (d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법에 의해 달성될 수 있다.

바람직하게는, 본 발명의 상기 (a) 단계는 상기 폐리튬 이온 전지가 100 내지 1,600㎠의 면적을 갖도록 파쇄된다.

바람직하게는, 본 발명의 상기 (b) 단계는 상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거된다.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시(mesh)의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와, 상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와, 상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함한다.

바람직하게는, 본 발명의 상기 (c) 단계는 상기 파쇄물을 진동 밀 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와, 상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함한다.

발명의 구성 및 작용

이하, 본 발명의 구성을 상세히 설명하면 다음과 같다.

(a) 단계 - 적어도 하나 이상의 폐리튬 이온 전지 파쇄

폐리튬 이온 전지는 파쇄기를 이용하여 100 내지 1,600㎠ 의 면적이 되도록 파쇄되는 것이 바람직하다. 이러한 방법은 철 재질인 케이스 부분과 내용물 부분이 파쇄물에 혼합된 상태로 수득된다.

다른 방법으로는 폐리튬 이온 전지의 철 재질인 케이스와 케이스에 내장된 내용물을 분리시키는 것으로 폐리튬 이온 전지의 중심 부분이나 양단부를 절단하여 케이스로부터 내용물을 분리한 다음, 내용물을 파쇄기를 이용하여 파쇄하는 것이다.

이러한 방법은 내용물만을 파쇄하기 때문에 전술된 방법(케이스가 포함되는 경우) 보다는 보다 적은 면적을 갖도록 파쇄하는 것이 바람직하다.

(b) 단계 - 파쇄물을 건조하여 전해질을 제거

파쇄물은 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거한다.

Ascend Elements EX1002 Page 403

Duesenfeld Ex. 2014-403

여기서, 건조 과정이 300℃ 이만의 온도에서 건조가 수행되는 경우에는 작업 건조 시간이 길어져서 작업성이 양호치 못하고, 550℃ 초과의 온도에서 건조가 수행되는 경우에는 파쇄물 중의 폐이스트 및 유기물 등이 착화되어 오염 물질이 발생되는 문제점이 있다.

그리고, 건조 시간은 파쇄물의 양에 의해 결정된다.

(c) 단계 - 파쇄물 미세 분쇄 및 코발트 화합물 분리

건조가 완료된 파쇄물은 진동 밀 또는 볼 밀 등에 의해 미세 분쇄된다. 여기서, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 것이 매우 중요하다.

미세 분쇄시 코발트 화합물은 50 내지 100 메시의 입도를 갖는 분말로 존재하고, 양극 성분인 알루미늄과 음극 성분인 구리는 각기 박판으로 존재하게 되며, 니켈 및 철은 코발트 화합물 분말에 비하여 5 내지 15배의 입도를 갖게 된다.

그리고, 분쇄물(분말)은 공급되는 고압의 공기에 의해 전술된 코발트 화합물 분말만이 비산되고, 코발트 화합물 분말을 제외한 알루미늄 및 구리 박판은 밀 표면에 압착되어 있으며 코발트 화합물 분말보다 상대적으로 중량인 니켈과 철 성분은 밀에 잔류된다. 여기서, 공급되는 고압의 공기는 코발트 화합물 분말 만이 비산(공기 분급 방법)되도록 이에 상응하는 압력과 공기량을 갖는다.

비산된 코발트 화합물 분말은 덕트 등과 같은 폐쇄 경로를 통해 유동된 후 포집된다. 여기서, 비산된 코발트 화합물 분말 중에 불순물(특히, 알루미늄 또는 구리 박판)이 혼입될 수 있기 때문에 포집 전 단계에서 불순물 제거용 필터를 경유시켜 전술된 불순물을 제거한 후 순수한 코발트 화합물 분말을 수득하는 것이 바람직하다.

또한, 전술된 공기 분급 방법 이외에 간단한 코발트 화합물 분말을 수득하는 방법은 망체(網體)가 구비된 요동되는 스크린 장치의 상부에 미세 분쇄된 분말을 공급하는 것으로, 망체의 메시는 코발트 화합물 분말보다는 적어도 입도가 큰 것이 바람직하다. 따라서, 코발트 화합물 분말만이 스크린 장치의 하부로 자유 낙하되기 때문에 순수한 코발트 화합물을 수득할 수 있다.

(d) 단계 - 코발트 화합물 분말을 환원시켜 순순한 코발트 분말 수득

전술된 (c) 단계에서 수득된 코발트 화합물 분말 속에는 미량의 탄소 분말이 함유될 수 있기 때문에 우선적으로, 300℃ 이상의 고열로 가열함으로써 탄소를 제거한 후 환원처리하면 순수한 코발트 분말을 수득할 수 있다.

이상에서 설명된 본 발명은 전술된 실시예에 한정되어 설명되었지만, 이에 한정되지 않고 본 발명이 속하는 분야의 통상적인 기술분야에서 통상의 지식을 가진 자가 용이하게 실시할 수 있는 정도의 변형은 본 발명의 기술적 사상에 속하는 것임은 자명하다.

발명의 효과

이상의 구성을 갖는 본 발명은 폐리튬 이온 전지로부터 고가(高價)의 순수한 코발트를 다량 수득할 수 있는 효과가 있다. 또한, 이러한 구성을 갖는 본 발명은 폐리튬 이온 전지의 재활용 과정에서 강산 또는 강알칼리를 사용하지 않기 때문에 작업 과정이 위험하지 않으며 이로 인한 2차 오염을 원천 방지할 수 있는 특징이 있다.

(57) 청구의 범위

청구항 1.

(a) 적어도 하나 이상의 폐리튬 이온 전지를 파쇄하는 단계와;

(b) 상기 파쇄물을 건조하여 전해질을 제거하는 단계와;

Ascend Elements EX1002 Page 404

Duesenfeld Ex. 2014-404

(c) 상기 전해질이 제거된 파쇄물을 미세 분쇄하고, 상기 미세 분쇄물로부터 코발트 화합물을 입도차를 이용하여 분리하는 단계와;

(d) 상기 코발트 화합물을 환원시켜 순수한 코발트를 수득하는 단계를 포함하여 폐건전지의 코발트 건식 분리 방법.

청구항 2.

제 1 항에 있어서,

상기 (a) 단계는,

상기 폐리튬 이온 전지가 100 내지 1,600㎠의 면적을 갖도록 파쇄되는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 3.

제 1 항에 있어서,

상기 (b) 단계는,

상기 파쇄물을 300 내지 550℃로 열풍 건조하여 전해질을 완전 제거하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 4.

제 1 항에 있어서,

상기 (c) 단계는,

상기 파쇄물을 진동 및 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 1 단계와,

상기 미세 분쇄물에 고압의 공기를 공급하여 코발트 화합물 분말을 비산시키는 (c) - 2 단계와,

상기 비산된 코발트 화합물 분말을 불순물 제거용 필터를 통과시켜 불순물을 제거하여 순수한 코발트 화합물 분말을 포집 수득하는 (c) - 3 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

청구항 5.

제 1 항에 있어서,

상기 (c) 단계는,

상기 파쇄물을 진동 및 또는 볼 밀을 이용하여 미세 분쇄하되, 코발트 화합물이 50 내지 100 메시의 입도를 갖는 분말이 되도록 분쇄하는 (c) - 4 단계와,

상기 미세 분쇄물을 요동되는 스크린 장치 상부에 제공하여 자유 낙하에 의해 코발트 화합물 분말을 선별 수득하는 (c) - 5 단계를 포함하는 것을 특징으로 하는 폐건전지의 코발트 건식 분리 방법.

Ascend Elements EX1002 Page 405

Duesenfeld Ex. 2014-405

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Warnings:

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3	Foreign Reference	03101160US_frn1-eng.pdf	74452 229b5472a4e3cbcf97086e1d969b61ce39fe2148	no	1
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5	Non Patent Literature	03101160US_npl1.pdf	793541 0a6031ec766838fb1392db27f7ec0298bb289853	no	10
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6	Non Patent Literature	03101160US_npl1-eng.pdf	81601 179722b550d3b58716c56766d92662ff8968a51b	no	2
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7	Non Patent Literature	03101160US_npl2.pdf	2828930 c88d2c56090bb5b0646c458b04d83213b7ddbe8a	no	58
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Ascend Elements EX1002 Page 407

8	Non Patent Literature	03101160US_npl3.pdf	1072411 3be3293d54c26fdbaeec5b7472146076550f1584	no	6
Warnings:					
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9	Non Patent Literature	03101160US_npl3-eng.pdf	136973 4eae7033059fe56c224e59e36c3d363bc2bbea7d	no	2
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If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.
<u>National Stage of an International Application under 35 U.S.C. 371</u>
If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.
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(54) Title: METHOD FOR SAFELY COMMINUTING LITHIUM-ION BATTERIES

(54) Bezeichnung : VERFAHREN ZUR SICHEREN ZERKLEINERUNG VON LITHIUM-IONEN-BATTERIEN

(57) Abstract: The invention relates to a method for safely comminuting batteries, comprising the steps of: a) providing one or more batteries to be comminuted; and b) mechanically comminuting the batteries, wherein the comminuting process takes place in the presence of: i) at least one metal flame retardant suitable for preventing or suppressing combustion of the batteries; and ii) at least one binding agent suitable for binding acids and/or bases.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zur sicheren Zerkleinerung von Batterien, umfassend die Schritte: a) Bereitstellen einer oder mehrerer zu zerkleinernder Batterien; und b) mechanische Zerkleinerung der bereitgestellten Batterien, wobei der Zerkleinerungsprozess stattfindet in Anwesenheit von: i) mindestens einem Metallbrandschutzmittel, welches geeignet ist einen Brand der Batterien zu unterdrücken oder zu vermindern; und von ii) mindestens einem Bindemittel, welches geeignet ist Säuren und/oder Basen zu binden.

Beschreibung

Titel

Verfahren zur sicheren Zerkleinerung von Lithium-Ionen-Batterien

Stand der Technik

Lithium-Ionen-Batterien und Lithium-Ionen-Polymer-Batterien werden derzeit nach Beendigung ihrer Betriebszeit (auch als EOL für ‚end of life' bezeichnet) entsprechend bekannter Recycling-Prozesse entsorgt, wobei wertvolle Elemente wie beispielsweise Kobalt und Nickel wieder gewonnen werden können. Ein bekanntes Beispiel für ein Entsorgungs- und Recyclingkonzept von Lithium-Ionen-Batterien ist das Schmelzverfahren (VAL'ES^{TM}) der Firma Umicore. Bei diesem Verfahren werden im großtechnischen Maßstab Lithium-Ionen- oder Lithium-Ionen-Polymer-Zellen beispielsweise von Handys oder Laptops der Entsorgung zugeführt und als Einzelzellen oder assemblierte Einheiten bei sehr hohen Temperaturen eingeschmolzen bzw. verbrannt.
Allerdings sind die bekannten Verfahren derzeit für das Recycling von Lithium-Ionen-Systemen auf Lithium-Ionen-Zellen oder -Module ausgelegt, die aus dem Consumer-Markt kommen. Hierbei handelt es sich um kommerziell etablierte Produkte wie Lithium-Ionen-Wickel-Zellen im Hardcase oder im Softpack sowie Lithium-Ionen-Stapelzellen im Softpack. Als Softpacks oder Pouch-Zellen werden Zelleinheiten bezeichnet, die mit einer Aluminium-Verbundfolie verpackt sind; auch die Bezeichnung Lithium-Ionen-Polymer-Zellen wird dafür verwendet. Die nominalen Kapazitäten der zugrunde liegende Lithium-Ionen- bzw. Lithium-Ionen-Polymer-Einzel-Zeilen, die heutzutage mit solchen Prozessen großtechnisch recycelt werden, liegen typischerweise zwischen 0,1Ah und 3 Ah.

Für Lithium-Ionen-Batterien, deren Applikation im Automobilbereich liegt, können diese beschriebenen Recycling-Verfahren derzeit nicht angewandt werden, da ihr Volumen, Gewicht und nominale Kapazitäten zu groß für die bestehenden Anlagenkonzepte sind. Nominale Kapazitäten von Lithium-Ionen-Einzel-Zellen für

PKWs oder LKWs liegen im Bereich von 3 - 300Ah und typischerweise zwischen 4 und 80 Ah. Lithium-Ionen-Zellen, -Module oder -Batterien mit solch hohen nominalen Kapazitäten können mit den derzeitigen grosstechnischen Recycling-Konzepten nicht vernichtet und recycelt werden.

Ein weiterer Mangel ist die Tatsache, dass Lithium-Ionen-Batterien, insbesondere Lithium-Ionen-Batterien mit einer nominalen Kapazität von mindestens 3Ah, in einem undefinierten Zustand überhaupt nicht transportiert werden dürfen und somit einem dezentralen Recycling-System gar nicht ohne weiteres zugeführt werden können. Neben den fehlenden Rohstoffen, die dann nicht wiedergewonnen werden können, verbleibt zudem noch ein großes Sicherheitsrisiko auf dem Werksgelände des Zellherstellers, des Assemblierers oder auch in den Räumlichkeiten des Testers (insbesondere nach sogenannten Abuse-Tests im vollgeladenen Zustand, in der Regel grösser oder gleich 4,2V). Beispiele für relevante Batterietests sind so genannte Droptests, bei denen Zellen bei 4,25V Betriebsspannung eine Distanz von ca. 1,5m senkrecht herunter fallen oder gemäß dem so genannte Ofentest nach UL (Lagerung bei 130°C; 4.2V Betriebsspannung für 10 Minuten (UL 1642)). Selbst wenn die Zellen den Test gemäß Test-Vorschrift bestehen, zeigt sich in der Praxis jedoch, dass diese getesteten Zellen während einer anschließenden Lagerung, beispielsweise auf einem Werks- oder Testgelände, sich noch nach Tagen oder Wochen spontan entzünden können. Dies stellt ein sehr großes Gefährdungspotential dar.

Aufgabe der vorliegenden Erfindung ist es einen oder mehrere Nachteile des Standes der Technik zu vermindern oder zu vermeiden. Insbesondere ist es Aufgabe der Erfindung ein Verfahren bereitzustellen mit dem Batterien sicher für die Entsorgung vorbereitet werden können.

Offenbarung der Erfindung

Die Aufgabe wird gelöst durch Bereitstellung eines Verfahrens zur sicheren Zerkleinerung von Batterien, umfassend die Schritte:
a) Bereitstellen einer oder mehrerer zu zerkleinernder Batterien; und

b) mechanische Zerkleinerung der bereitgestellten Batterien, wobei der Shredder-Prozess stattfindet in Anwesenheit von:

i) mindestens einem Metallbrandschutzmittel, welches geeignet ist einen Brand der Batterien zu unterdrücken oder zu vermindern; und von

ii) mindestens einem Bindemittel, welches geeignet ist Säuren und/oder Basen zu binden.

Das erfindungsgemäße Verfahren erlaubt es Batterien, insbeondere höher kapazitive Lithium-Ionen-Batterien mit einer nominalen Kapazität von typischerweise größer 3Ah pro Zelle, sicher zu vernichten und die freigesetzten Energiemengen sowie gefährlichen Batterie- und auftretende Prozessstoffe kontrolliert und ohne Gefahr für Mensch und Umwelt zu binden und zu eliminieren.

Überraschenderweise zeigt sich, dass durch das erfindungsgemäße Verfahren Batterien, insbesondere Lithium-Ionen-Batterien mit nominalen Kapazitäten von mindestens 3Ah, gefahrlos und kontrolliert zerkleinert werden können. Anschließend können diese Bestandteile einem eigentlichen Recycling-Prozess zugeführt werden, z.B. dem VAL'ES^{TM}–Verfahren der Firma Umicore, da die Materialien nach dem erfindungsgemäßen Verfahren kompatibel zum eigentlichen großindustriellen Recycling-Prozess sind. Bei einem erindungsgemäßen mechanischen Zerkleinerungsprozess an hochkapazitiven Zellen, Modulen und Batterien wird erfindungsgemäß in Anwesenheit von bekannten Brandschutzmitteln das Batteriematerial mechanisch zerkleinert. Völlig unerwartet läuft die Vernichtung der Batterie auch im vollgeladenen Zustand moderat ab. Die Anwesenheit von Metallbrandschutzmitteln ermöglicht es, dass Brandstellen lokal abgedeckt werden und nicht noch in weiterem Maße andere Zellen der Gesamtbatterie oder umliegender Batterien in einen „Thermal Runaway" treiben. Das Bindemittel bindet entstandene Säure- und/oder Basenbestandteile, im Fall von Lithium-Ionen-Batterien insbesondere von entstandener Flusssäure (HF) unter Entstehung von vorzugsweise Kalziumfluorit (CaF_2). Bei Lithium-Ionen-Batterien entsteht Flusssäure, die hoch toxische und korrosive Eigenschaften hat, durch Hydrolyse bzw. thermische Zersetzung des Leitsalzes Lithium-Hexa-Fluoro-Phosphat ($LiPF_6$). Die so zerkleinerten und behandelten Materialen können in einem Wasserbad, das durch die Anwesenheit von Bindemitteln, wie Kaliumhydroxid ($Ca(OH)_2$), leicht basisch ist, aufgefangen werden, um weitere Flusssäure (HF), die eventuell noch anwesend ist, unter Bildung von CaF_2 zu binden. Durch das wässrige Me-

dium werden auch Reste an lithiiertem Graphit hydrolysiert; es entstehen Lithium-Salze wie Lithium-Hydroxid (LiOH). Die hydrolysierten Materialien können abgefiltert, luftgetrocknet und einem anschließenden Recyclingprozess zugeführt werden. Somit wird ein Verfahren bereitgestellt, mit welchem sich ggf. vor Ort hochkapazitive Lithium-Ionen-Batterien sicher zerkleinern und transportfähig machen lassen, um dann einer geeigneten, entfernt gelegenen, zentralen Recyclinganlage zugeführt zu werden.

In einem ersten Schritt werden eine oder mehrere Batterien bereit gestellt, die sicher zerkleinert werden sollen. Dabei können sowohl Batterien ausschließlich eines Typs, als auch Mischungen von Batterien unterschiedlicher Typen, Bauweisen und nominaler Kapazitäten gemeinsam zerkleinert werden. Einzelne, mehrere oder alle Batterien können sich dabei sowohl in voll geladenem, teilgeladenem und/oder ungeladenem Zustand befinden. Dabei können eine, mehrere oder alle Batterien intakt, in voll assemblierten Zustand, teilassembliert und/oder in undefiniertem Zustand befinden. Dabei umfasst ein undefinierter Zustand insbesondere solche Batteriezustände nach Umwelttests, Abuse- und/oder Missuse-Tests, havarierte Batterien und auch solche Batteriezustände, die bei Unfällen entstehen können.

Im erfindungsgemäßen Verfahren können grundsätzlich Batterien jeden Typs und jeder Bauart zerkleinert werden. Unter dem Begriff „Batterie“ werden hier elektrochemische Energiespeicher verstanden, insbesondere Batterien oder Akkumulatoren aller gebräuchlichen Akkumulatortechnologien sein. Es können Batterien oder Akkumulatoren vom Typ Pb – Bleiakku, NiCd - Nickel-Cadmium-Akku, NiH2 - Nickel-Wasserstoff-Akkumulator, NiMH - Nickel-Metallhydrid-Akkumulator, Li-Ion - Lithium-Ionen-Akku, LiPo - Lithium-Polymer-Akku, LiFe - Lithium-Metall-Akku, Li-Mn - Lithium-Mangan-Akku, $LiFePO_4$ - Lithium-Eisen-Phosphat-Akkumulator, LiTi - Lithium-Titanat-Akku, RAM - Rechargeable Alkaline Manganese, Ni-Fe - Nickel-Eisen-Akku, Na/NiCl - Natrium-Nickelchlorid-Hochtemperaturbatterie, SCiB - Super Charge Ion Battery, Silber-Zink-Akku, Silikon-Akku, Vanadium-Redox-Akkumulator und/oder Zink-Brom-Akku verwendet werden. Insbesondere können Batterien vom Typ der Blei/Säure-, Nickel-Cadmium-, Nickel-Metallhydrid- und/oder Natrium/Natriumnickelchlorid-Batterie eingesetzt werden. Besonders bevorzugt werden Batterien vom Typ der Lithium-Ionen-Batterie verwendet. Der Begriff „Batterie“ wird dabei sowohl für einzelne Zellen, als

auch für Module aus mehreren Zellen, als auch für komplexere Architekturen umfassend mehrere Zellen und/oder Module verwendet.

Bevorzugt werden hochkapazitive Batterien eingesetzt mit einer nominalen Kapazität von mindestens 3Ah. Besonders bevorzugt von 3Ah bis 300Ah, insbesondere von 4Ah bis 80Ah. Nominale Kapazitäten von Lithium-Ionen-Einzel-Zellen für PKWs oder LKWs liegen im Bereich von 3 - 300Ah und typischerweise zwischen 4 und 80 Ah. Dabei können sich die angegebenen nominalen Kapazitätswerte entweder auf die ganze intakte Batterie beziehen oder auf eine einzelne Zelle einer Batterie.

Bevorzugt sind die bereitgestellten Batterien teilweise oder ausschliesslich Lithium-Ionen- und/oder Lithium-Ionen-Polymer-Zellen, -Module und/oder -Batterien mit einer nominalen Kapazität von mindestens 3Ah pro Zelle.

Im erfindungsgemäßen Verfahren werden die bereitgestellten Batterien einer mechanischen Zerkleinerung unterzogen. Dies kann beispielsweise ein Shredderprozess sein. Dazu kann ein Shredder eingesetzt werden. Ein Shredder ist ein mechanisches Gerät zum Zerkleinern von unterschiedlichsten Materialien, für die Zwecke der Erfindung ist der Shredder so ausgelegt, dass er in der Lage ist ohne wesentliche Beeinträchtigung eine Mehrzahl von Batterien zu zerkleinern. Dem Prinzip des Schredderns liegt der Betrieb eines Mahlwerks zugrunde, welches beispielsweise durch entsprechend dimensionierte Elektro- oder Verbrennungsmotoren angetrieben werden kann. Das Mahlwerk kann beispielsweise aus einem durch versetzt angebrachte Walzen aufgebauten Getriebe bestehen. Wird eine Batterie in den Schredder gegeben, wird diese durch Zerreiben, Zermahlen, Zerreißen oder Zerschneiden in kleine Bestandteile aufgelöst. Dem Fachmann sind Shredder in verschiedensten Ausführungsformen, -größen und –dimensionen bekannt, sodass der Fachmann keine Schwierigkeiten hat für ausgewählte Batterien einen geeigneten Shredder zu wählen oder anzupassen.

Im erfindungsgemäßen Verfahren werden die bereitgestellten Batterien einer mechanischen Zerkleinerung unterzogen. Dies kann beispielsweise in einem Laserschneideprozess geschehen. Dazu kann ein Laserschneider bekannter Bauart eingesetzt werden, wie er bereits in vielen industriellen Anwendungen zum

Schneiden von metallischen und/oder nichtmetallischen Werkstoffen Verwendung findet. Ein Laserschneider ist ein mechanisches Gerät zum Zerkleinern von unterschiedlichsten Materialien, für die Zwecke der Erfindung ist der Laserschneider so ausgelegt, dass er in der Lage ist ohne wesentliche Beeinträchtigung eine Mehrzahl von Batterien zu zerkleinern. Geeignete Laserschneideprozesse und Anordnungen sind dem Fachmannn bekannt und ohne Schwierigkeiten für das erfindungsgemäße Verfahren einsetzbar und/oder adaptierbar.

Im erfindungsgemäßen Verfahren werden die bereitgestellten Batterien einer mechanischen Zerkleinerung unterzogen. Dies kann beispielsweise ein Wasserschneideprozess sein. Dazu kann eine Wasserschneidemaschiene eingesetzt werden. Eine Wasserstrahlschneidemaschine ist eine Werkzeugmaschine die sich zum Trennen von Materialien eines Hochdruckwasserstrahles bedient. Dabei trennt der Hochdruckwasserstrahl das zu bearbeitende Material. Dieser Hochdruckwasserstrahl kann einen Druck von 6000 bar oder mehr aufweisen und es können Austrittsgeschwindigkeiten von ca. 1000 m/s oder mehr erreicht werden. Beim Reinwasserschneiden wird im Wesentlichen die Strahlenergie des Wassers ausgenutzt. Dies ermöglicht eine Schneidleistung bei der der Schnittspalt sehr eng sein und lediglich 0,1 mm betragen kann. Zur Strahlbündelung können Polymere zugesetzt werden. Zur Erhöhung der Schneidleistung kann dem Strahl ein Schneidmittel, ein sogenanntes Abrasiv, zugesetzt werden. Durch die Beimengung eines solchen Abrasivs (wie z. B. Granat, Aluminiumoxid oder Korund) ist es möglich, härtere Materialien noch effektiver zu trennen, die mit einem reinem Wasserstrahl nicht trennbar sind, oder deren Bearbeitung mit Purwasser nicht wirtschaftlich ist, bzw. wo eine höhere Schnittqualität verlangt wird. Um mit einem Abrasiv schneiden zu können, verfügt die Wasserstrahlanlage über einen Schneidkopf, der im wesentlichen aus drei Komponenten besteht: einer Reinwasserdüse, einer Abrasiv-Mischkammer und einer Abrasivfokussierdüse bzw. einem Fokussierrohr. Zuerst wird durch die Reinwasserdüse das hochkomprimierte Wasser zu einem Strahl bevorzugt von etwa 0,25–0,4 mm (düsenabhängig) geformt. Dieser Strahl schießt durch die Mischkammer mit einer hohen Geschwindigket, z.B. mit bis zu 1000 m/s, und erzeugt somit ein Vakuum im Schneidkopf. Durch eine kleine Öffnung im Schneidkopf kann jetzt Abrasivmittel in die Mischkammer gesaugt und mit dem Wasserstrahl vermischt werden. Das Wasser-Abrasiv-Gemisch wird dann fokussiert und tritt mit einem vorher festgelegten Durchmesser aus. Das Abrasivmittel kann auch CNC-gesteuert dosiert

und dem Schneidkopf zugeführt werden, entweder mit Luft als Träger, oder in einer Emulsion.

Ein großer Vorteil bei der mechanischen Zerkleinerung mittels eines Wasserschneideprozesses besteht darin, dass sich das Schneidgut nicht nennenswert erwärmt. Auch ein Schneiden unter Wasser, etwa durch Erhöhung des Wasserspiegels im Strahlfänger und/oder durch Umgebung der Düse mit einer Wasserglocke, ist möglich.

Die erfindungsgemäße mechanischen Zerkleinerung mittels eines Wasserschneideprozesses erlaubt das Zerschneiden von allen Zell-Komoneneten in einem komplexen Lithium-Ionen-System. Beispielsweise 5 mm starkes Edelstahl, welches häufig als Aussen-Gehäuse der assemblierten Li-Ionen-Batterie eingesetzt wird kann damit zertrennt werden. Weitere Beispiele sind das Zertrennen anderer Bestandteile, insbesondere des Zell-Gehäuses, welches beispielsweise aus 2 mm starken Aluminiumblechen bestehen kann. Weiterhin können problemlos Elektroden und metallische Kollektorfolien getrennt werden; die Dicke von den gängigen Aluminium- und Kupfer-Kollektorfolien liegen in der Regel bei 8 - 20pm, bevorzugt zwischen 10 und 15pm.

Der Einsatz eines Wasserschneideprozesses hat weitere Vorteile. Die Temperatur der Batterie, insbesondere der Lithium-Ionen-Batterie wird durch den Schneidevorgang mittels Wasserstrahl nicht relevant erhöht. Das ist insbesondere bei einer Zerstörung von hochgeladenen Lithium-Ionen-Batterien vorteilhaft, weil keine weiteren Sekundärenergien auftreten, die die Batterie oder umliegende Batterien unkontrolliert weiter in ein thermisches Durchgehen treiben (im englischen als „thermal runaway" bezeichnet: darunter versteht man bei Lithium-Ionen-Akkumulatoren Vorgänge wie zum Beispiel lokal entstandene innere Kurzschlüsse, zum Beispiel Einsticke mit metallischen Gegenständen (z. Bsp. Skalpelle), die betroffene Stellen soweit aufheitzen, dass diese in Mitleidenschaft gezogen werden). In der Praxis beobachtet man dann eine Zerstörung des Separators und es kommt zur Erhöhung des Kurzschlussstromes und der Erwärmung und die Vorgänge schaukeln sich auf und die gespeicherte Energie wird schlagartig frei. Dabei kann es auch zu einer heftigen Explosion kommen. Ein weiterer Vorteil ist die Tatsache, dass Gefügeveränderungen an den Schnittkanten weitgehend ausge-

schlossen werden. Dies ist sehr wichtig, wenn man in der Lithium-tonen-Zelle den Verbund aus positiver Elektrode, Separator und negativer Elektrode durchtrennt; etwaige Schnittgrade können hohe innere Kurzschlüsse und damit hohe Kurzschlussströme herbeiführen, die wiederum für die Batterie kritisch sein können und gerade in der Phase der Zerkleinerung Explosionen herbeiführen können. Auch das Wasser als trennendes Medium hat große Vorteile gegenüber den herkömmlichen auf Metall-Basis trennenden Werkstücken, weil es im Lithium-Ionen-Akkumulator keine inneren Kurzschlüsse verursacht. Gerade Metalle, die während dem Zerkleinern auf Lithium-Ionen-Batterien einwirken, bewirken unkontrollierbare innere Kurzschlüsse, die zur starken Erwärmung oder sogar zu Explosionen führen können. Zudem sind diese Metalle während dem Zerstörungsvorgang ggf. stark ätzenden und korrosiven Säuren, wie beispielsweise Flusssäure (HF), ausgesetzt. Sollte dennoch während dem Wasserstrahlschneiden ein „Thermal Runaway" auftreten, findet eine in-sito Kühlung durch das umgebende Wasser statt.

Erfindungsgemäß findet der Zerkleinerungsprozess in Anwesenheit mindestens eines Metallbrandschutzmittels statt. Das Metallbrandschutzmittel ist dabei derart ausgewählt, dass es einen Brand der zu zerkleinernden Batterien vermeiden, unterdrücken, vermindern oder löschen kann. Dies kann beispielsweise dadurch geschehen, dass das Metallbrandschutzmittel dazu führt, dass die Menge an zur Verfügung stehendem Oxidationsmittel beeinflusst wird oder dass die entstandene Energie, insbesondere thermische Energie, vom Metallbrandschutzmittel absorbiert wird, bevor dadurch weitere Batterien oder Batteriebestandteile in den Brand miteinbezogen werden. Die Wahl der Metallbrandschutzmittels kann dabei von der Art und Zusammensetzung der zu zerkleinernden Batterien abhängen. Bevorzugt kann das mindestens eine Metallbrandschutzmittel Wasser, Natriumchlorid (NaCl) und/oder Quarzsand (SiO_2) umfassen oder daraus bestehen. Wird zur mechanischen Zerkleinerung der Batterien ein Wasserschneideprozess eingesetzt, so kann bereits das Wasser des Wasserschneiders gleichzeitig auch das Metallbrandschutzmittel im Sinne der Erfindung darstellen. Auch im Zerkleinerungsraum vorhandenes Wasser kann erfindungsgemäß als das mindestens eine Metallbrandschutzmittel dienen.

Im erfindungsgemäßen Verfahren findet der mechanische Zerkleinerungsprozess zusätzlich unter Anwesenheit mindestens eines Bindemittels statt, welches geeignet ist Säuren und/oder Basen zu binden. Je nach aufzuarbeitendem Batteriematerial werden bei der Zerkleinerung unterschiedliche Säuren und/oder Basen freigesetzt oder können als Reaktionsprodukt entstehen, wenn vorher getrennte Bestandteile der Batterien nun in Kontakt treten. Daher kann die Auswahl des oder der Bindemittel davon abhängen, welche Batterietypen zerkleinert werden sollen. Insbesondere bei der Aufbereitung von Lithium-Ionen-Batterien kann das Bindemittel basisch und geeignet sein Säuren, insbesondere Flusssäure, zu binden. Es können Bindemittel verwendet werden, die geeignet sind Fluor zu binden, sogenannte Fluor-Fänger. Geeignete Bindemittel oder Säureregulatoren sind dem Fachmann bekannt. Bevorzugt kann das mindestens eine Bindemittel Kalziumoxid (CaO), Kalziumchlorid ($CaCl_2$) und/oder Kaliumhydroxid ($Ca(OH)_2$) umfassen oder daraus bestehen.

Es können auch mehr als ein Bindemittel jeweils einzeln oder als Gemsich eingesetzt werden. Das Bindemittel, die Bindemittel oder das Bindemittelgemisch können jeweils unabhängig voneinander in fester, flüssiger und/oder gasförmiger Form eingesetzt werden.

Die Menge an Metallbrandschutzmittel und/oder Bindemittel, die bei einem erfindungsgemäßen mechanischen Zerkleinerungsprozess eingesetzt werden, hängen nicht nur von der Wahl des Metallbrandschutzmittels bzw. des Bindemittels ab, sondern könnnen von Volumen und Beschaffenheit des Zerkleinerungsraums und von der Menge, Gewicht, Kapazität, Art und Zusammensetzung der zu zerkleinernden Batterien mitbestimmt werden. So kann es notwendig sein, für einen bestimmungsgemäßen Betrieb einer bestimmten Vorrichtung zur Ausführung des erfindungsgemäßen Verfahrens in Abhängigkeit von Gewicht und Kapazität der zu zerkleinernden Batterien die erforderlichen Mengen und/oder Arten an Metallbrandschutzmittel und/oder Bindemittel zunächst in einem Testlauf zu bestimmen. Der Fachmann stößt bei dieser Bestimmung auf keine Schwierigkeiten, da ihm die Parameter und Möglichkeiten der Einflussnahme bekannt und für ihn ohne weiteres kontrollierbar sind. Die Bestimmung der erforderlichen Mengen an Metallbrandschutzmittel und Bindemittel liegen somit im Bereich von Routineexperimenten, die der Fachmann vor dem Hintergrund seines Fachwissens ohne weitere Anleitung selbständig ausführt.

Solide Metallbrandschutzmittel und/oder Bindemittel werden bevorzugt als feinkörniges, mindestens vorwiegend festes Material eingesetzt. Besonders bevorzugt als Material mit einem d_{50}-Wert von nicht mehr als 5µm, ganz besonders bevorzugt mit einem d_{50}-Wert von nicht mehr als 3µm. Enthält das Metallbrandschutzmittel NaCl und/oder Quarzsand, so wird das Material bevorzugt in einer Körnung mit einem d_{50}-Wert von 2µm +/- 0,5 eingesetzt. Enthält das Bindemittel Kalziumoxid und/oder Kaliumhydroxid, so wird das Mittel in einer Körnung mit einem d_{50}-Wert von 1µm +/-0,25 eingesetzt.

Es können auch flüssige Brandschutzmittel verwendet werden.

Um unerwünschte Reaktionen, insbesondere Oxidationen, soweit als möglich zu vermeiden, kann der Zerkleinerungsprozess ganz oder teilweise unter einer Atmosphäre mit einem Inertgas oder einem Inertgasgemisch durchgeführt werden, die, insbesondere mit Batteriebestandteilen, wenig reaktiv ist. Bevorzugt wird der mechanische Zerkleinerungsprozess ganz oder teilweise unter einer Atmosphäre mit einem Anteil an einem Inertgases oder einem Inertgasgemisch von mehr als 30% durchgeführt, bevorzugt mehr als 60%, besonders bevorzugt mehr als 90%. Dem Fachmann sind geeignete Inertgase oder Mischungen von Inertgasen bekannt. Bevorzugt kann die Inertgasatmosphäre Stickstoff (N_2) und/oder Argon (Ar) enthalten oder daraus bestehen.

Teile der Reaktionen, die beim Betrieb des erfindungsgemäßen Verfahrens ablaufen sind exotherm, so dass die Temperatur während des mechanischen Zerkleinerungsprozesses zunimmt. Daher kann es sinnvoll sein, den mechanischen Zerkleinerungsprozess ganz oder teilweise gekühlt ablaufen zu lassen. Dies kann beispielsweise dadurch erreicht werden, dass der Zerkleinerungsraum doppelwandig ausgestaltet ist und zwischen den beiden Wänden ein Kühlmedium vorhanden ist. Alternativ oder zusätzlich kann der Raum zur Ausführung des Zerkleinerungsprozesses ein Kühlmittel enthalten. Dieses kann beispielsweise vor und/oder während des Zerkleinerungsprozesses zugegeben werden. Beispielsweise können die Gase der Atmosphäre unter der der Shredderprozess abläuft gekühlt vorliegen oder fortlaufend gekühlt werden. Es ist aber auch möglich die zu zerkleinernden Batterien vorzukülen und dann erst nach Erreichen einer vorher festgelegten niedrigen Temperatur dem erfindungsgemäßen Verfahren zuzu-

führen. Alternativ können die zu zerkleinernden Batterien vor, nach und/oder während des Zerkleinerungsverfahrens mit Kühlmedium versetzt oder vermischt werden. Voraussetzung dafür ist, dass das verwendete Kühlmedium im Wesentlichen inert ist bzgl. der Batteriebestandteile und der Vorrichtung zur Ausführung des Verfahrens. Das Kühlmittel kann aus einer einzigen Substanz oder Verbindung bestehen oder eine Mischung von verschiedenen Substanzen oder Verbindungen aufweisen. Beispielsweise kann zur Kühlung Wasser, flüssiger Stickstoff und/oder Trockeneis eingesetzt werden.

Um eine besonders vorteilhafte Umgebund für den mechanischen Zerkleinerungsprozess zu schaffen, kann der Zerkleinerungsprozess ganz oder teilweise unter Wasser durchgeführt werden. Dies hat den Vorteil, dass das Wasser sowohl als Medium mit nicht unerheblicher eigener Wärmekapazität (und damit als das mindestens eine Metallbrandschutzmittel im Sinne der Erfindung), sowie als Wärmeleiter zu einem weiteren Kühlmedium fungieren kann, als auch als Lösungsmittel für das mindestens eine Bindemittel und gegebenenfalls für weitere Metallbrandschutzmittel. Dadurch wird die Bereitstellung von kontrollierten Reaktionsbedingungen an möglichst vielen Oberflächen der zu zerkleinernden bzw. der bereits ganz oder teilweise zerkleinerten Batterien erleichtert, da das Metallbrandschutzmittel und das Bindemittel in genau definierten Konzentrationen gelöst und gleichmäßig verteilt im Zerkleinerungsraum vorliegen. In dem wässrigen Millieu wird, im Fall von Lithium-Ionen-Batterien, lithiiertes Graphit hydrolysiert. Es entstehen dabei Lithiumsalze wie beispielsweise Lithium-Hydroxid (LiOH).

Das erfindungsgemäße Verfahren kann weitere Schritte aufweisen, die sich an den mechanischen Zerkleinerungsprozess anschließen.

In einem ersten weiteren Schritt werden die zerkleinerten Materialien in ein Wasserbad überführt bzw. darin aufgefangen. Dieses Wasserbad enthält ein Bindemittel, welches dieselben Eigenschaften aufweisen kann, wie das Bindemittel welches während des mechanischen Zerkleinerungsprozesses zum Einsatz kommt. Dabei muss in einer Ausführungsform des Verfahrens während des Zerkleinerungsprozesses und im anschließenden Wasserbad nicht das gleiche Bindemittel verwendet werden. Es können in beiden Schritten unterschiedliche Bindemittel eingesetzt werden. Bevorzugte Bindemittel sind bereits oben für den mechanischen Zerkleinerungsprozess beschrieben.

An das Wasserbad kann sich ein weiterer zusätzlicher Verfahrensschritt anschließen. In diesem zweiten zusätzlichen Schritt wird der gegebenenfalls hydrolysierte Austrag des mechanischen Zerkleinerungsprozesses abfiltirert. Sinn und Zweck dieses Schrittes ist es, die unlöslichen Bestandteile des Austrags des Zerkleinerungsprozesses von den flüssigen Bestandteilen zu trennen. Gegebenfalls können diese Fraktionen dann einzeln weiteren Verwertungsschritten und/oder einem Recyclingprozess zugeführt werden. Geeignete Filterlösungen sind dem Fachmann bekannt. Gegebenfalls kann das Filtrat anschließend getrocknet werden. Dieser Trocknungsschritt reduziert z.B. das Gewicht des Filtrats, was insbesondere dann von Vorteil ist, wenn das Filtrat gelagert und/oder über längere Strecken transportiert werden muss, bevor es einer weiteren Verwertung zugeführt wird.

Die vorliegende Erfindung bezieht sich auch auf ein Verfahren zum Recycling von Batterien, bevorzugt von Lithium-Ionen-Batterien mit einer nominalen Kapazität von mindestens 3Ah. Bei diesem Verfahen werden die Batterien zunächst nach einem oben beschriebenen erfindungsgemäßen Verfahren zerkleinert und anschließend einem Recycling-Prozess zugeführt. Unter einem Recycling-Prozess oder Recyclingverfahren wird dabei ein Prozess oder Verfahren verstanden, bei dem mindestens ein Rohstoff, der in den zerkleinerten Batterien enthalten ist isoliert und einer weiteren Verwendung zugeführt wird. Ein Beispiel für einen solchen Recycling-Prozess ist das VAL'ES^{TM}-Verfahren der Firma Umicore, welches in der Patentschrift EP1 589 121 B1 beschreiben ist. Andere alternative oder zusätzliche Recycling-Prozesse sind dem Fachmann bekannt.

Die vorliegende Erfidung bezieht sich auch auf eine Vorrichtung zur Ausführung eines erfindungsgemäßen Verfahrens zur sicheren Zerkleinerung von Batterien. Dazu weist die Vorrichtung Mittel auf zur Durchführung des erfindungsgemäßen Zerkleinerungsverfahrens. Das offenbarte erfindungsgemäße Verfahren lässt sich durch dem Fachmann bekannte Mittel umsetzen. Erfolgt die mechanische Zerkleinerung mindestens teilweise durch einen Shredderprozess, so kann als Basis ein Shredder nach bekannter Bauart dienen. Erfolgt die mechanische Zerkleinerung mindestens teilweise durch einen Laserschneideprozess, so kann als Basis ein Laserschneider bekannter Bauart eingesetzt werden. Erfolgt die mechansiche Zerkleinerung mindestens teilweise durch einen Wasserschneidepro-

zess, so kann ein Wasserschneider nach bekannter Bauart als Basis dienen. Mittel zur Zu- und Ableitung des Metallbrandschutzmittels bzw. des Bindemittels sowie geeignete Ausgestaltungen des Zerkleinerungsraums sind entweder bekannt oder liegen im Bereich von Routinetätigkeiten des verfahrenstechnisch bzw. maschinenbaulich vorgebildeten Fachmanns.

Zur Steuerung des erfindungsgemäßen Verfahrens kann die Vorrichtung eine sogenannten CNC-Steuerung (CNC steht für Computerized Numerical Control) aufweisen. Es ist möglich die CNC-Steuerung der erfindungsgemäßen Vorichtung mit einem konstruktiven CAD-Programm (CAD steht für Computer Aided Design) des jeweiligen Lithium-Ionen-Systems zu korrelieren, um besonders unkritisch eine Zerkleinerung zu erreichen und dementsprechend an unkritischen Positionen im Pack anzusetzen. CNC-Steuerungen sind sehr präzise und insbesondere in einer Serienproduktion ist es nicht nötig viel Personal an der Anlage selbst einzusetzen. Dies minimiert die Gefährdung des Menschen und erhöht die Akzeptanz solcher Anlagen für die sehr gefährlichen Lithium-Ionen-Batterie-Zerkleinerungsvorgänge.

Ansprüche

1. Verfahren zur sicheren Zerkleinerung von Batterien, umfassend die Schritte:
a) Bereitstellen einer oder mehrerer zu zerkleinernder Batterien; und
b) mechanische Zerkleinerung der bereitgestellten Batterien, wobei der Zerkleinerungsprozess stattfindet in Anwesenheit von:
i) mindestens einem Metallbrandschutzmittel, welches geeignet ist einen Brand der Batterien zu unterdrücken oder zu vermindern; und von
ii) mindestens einem Bindemittel, welches geeignet ist Säuren und/oder Basen zu binden.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die bereitgestellten Batterien teilweise oder ausschliesslich Lithium-Ionen- und/oder Lithium-Ionen-Polymer-Zellen, -Module und/oder -Batterien mit einer nominalen Kapazität von mindestens 3Ah pro Zelle umfassen.

3. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass das mindestens eine Metallbrandschutzmittel Wasser, Natriumchlorid und/oder Quarzsand umfasst oder daraus besteht.

4. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass das mindestens eine Bindemittel Kalziumoxid, Kalziumchlorid und/oder Kaliumhydroxid umfasst oder daraus besteht.

5. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass der Zerkleinerungsprozess teilweise oder ganz unter einer Atmosphäre mit einem Anteil an einem Inertgas oder einem Inertgasgemisch von mehr als 30% durchgeführt wird, bevorzugt mehr als 60%, besonders bevorzugt mehr als 90%.

6. Verfahren nach Anspruch 5, dadurch gekennzeichnet, dass die Inertgasatmosphäre Stickstoff und/oder Argon umfasst oder daraus besteht.

7. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass der Zerkleinerungsprozess teilweise oder ganz gekühlt durchgeführt wird.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, dass zur Kühlung Wasser, flüssiger Stickstoff und/oder Trockeneis eingesetzt wird.

9. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass der Zerkleinerungsprozess ganz oder teilweise unter Wasser durchgeführt wird.

10. Verfahren nach einem der vorhergehenden Ansprüche, umfassend den zusätzlichen Schritt
c) Auffangen der zerkleinerten Materialien in einem Wasserbad enthaltend mindestens ein Bindemittel, welches geeignet ist Säuren und/oder Basen zu binden.

11. Verfahren nach Anspruch 10, umfassend die zusätzlichen Schritte
d) Abfilterung des Austrags des Zerkleinerungsprozesses; und
e) gegebenenfalls Trocknung des Filtrats.

12. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass bei der mechanischen Zerkleinerung ein Wasserschneideprozess, ein Laserschneideprozess und/oder ein Shredderprozess zum Einsatz kommt.

13. Verfahren zum Recycling von Batterien, bevorzugt von Lithium-Ionen- und/oder Lithium-Ionen-Polymer-Zellen, -Module und/oder –Batterien, umfassend die Schritte:
- Zerkleinerung der Batterien nach einem der Verfahren gemäß einem der Ansprüche 1 bis 12; und
- Zuführung der zerkleinerten Batterien zu einem Recycling-Prozess.

14. Vorrichtung umfassend Mittel zur Durchführung eines Verfahrens gemäß einem der Ansprüche 1 bis 12.

15. Vorrichtung nach Anspruch 14, dadurch gekennzeichnet, dass die Vorrichtung Mittel zur Ausführung eines Wasserschneideprozesses, eines Laserschneideprozesses und/oder eines Shredderprozesses aufweist.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2010/058716

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M6/52 H01M10/54 A62D1/00 B02C19/00 B02C23/00
B09B3/00 C22B7/00
ADD.
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M A62D B02C B09B C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 303 872 A (MILEWITS MARVIN [US] ET AL) 19 April 1994 (1994-04-19)	1-3, 7-10,12, 14,15
A	column 2, line 15 - line 32 column 3, line 64 - column 4, line 13 column 5, lines 30-44 column 6, lines 40-44; claim 4	4-6,11, 13

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
17 August 2010	24/08/2010

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Maxisch, Thomas

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 1 041 659 A1 (MITSUBISHI HEAVY IND LTD [JP]) 4 October 2000 (2000-10-04) paragraph [0094] paragraph [0074] paragraph [0077] paragraph [0080] paragraph [0085] paragraph [0090] paragraph [0143] - paragraph [0149] paragraph [0119] - paragraph [0122] paragraph [0128]; figures 1-27	1-15
X	EP 0 613 198 A1 (CANON KK [JP]) 31 August 1994 (1994-08-31) page 4, left-hand column, line 18 - page 5, left-hand column, line 17 page 7, left-hand column, lines 10-42; figures 1,2	1-15
X	WO 02/23651 A1 (AVESTOR [CA]; CARDARELLI FRANCOIS [CA]; DUBE JONATHAN [CA]) 21 March 2002 (2002-03-21) page 8, line 21 - page 10, line 11	1-15

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2010/058716

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EP 1041659 A1	04-10-2000	CA 2311792 A1	06-04-2000
		WO 0019557 A1	06-04-2000
		JP 3495707 B2	09-02-2004
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		EP 1269554 A1	02-01-2003
		JP 2004508694 T	18-03-2004
		US 2004028585 A1	12-02-2004

INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
PCT/EP2010/058716

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
INV. H01M6/52 H01M10/54 A62D1/00 B02C19/00 B02C23/00
B09B3/00 C22B7/00
ADD.
Nach der Internationalen Patentklassifikation (IPC) oder nach der nationalen Klassifikation und der IPC

B. RECHERCHIERTE GEBIETE

Recherchierter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)
H01M A62D B02C B09B C22B

Recherchierte, aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen

Während der internationalen Recherche konsultierte elektronische Datenbank (Name der Datenbank und evtl. verwendete Suchbegriffe)
EPO-Internal

C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
X	US 5 303 872 A (MILEWITS MARVIN [US] ET AL) 19. April 1994 (1994-04-19)	1-3, 7-10,12, 14,15
A	Spalte 2, Zeile 15 - Zeile 32 Spalte 3, Zeile 64 - Spalte 4, Zeile 13 Spalte 5, Zeilen 30-44 Spalte 6, Zeilen 40-44; Anspruch 4	4-6,11, 13
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[X] Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen [X] Siehe Anhang Patentfamilie

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"&" Veröffentlichung, die Mitglied derselben Patentfamilie ist

Datum des Abschlusses der internationalen Recherche	Absendedatum des internationalen Recherchenberichts
17. August 2010	24/08/2010

Name und Postanschrift der Internationalen Recherchenbehörde	Bevollmächtigter Bediensteter
Europäisches Patentamt, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Maxisch, Thomas

Formblatt PCT/ISA/210 (Blatt 2) (April 2005)

C. (Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
X	EP 1 041 659 A1 (MITSUBISHI HEAVY IND LTD [JP]) 4. Oktober 2000 (2000-10-04) Absatz [0094] Absatz [0074] Absatz [0077] Absatz [0080] Absatz [0085] Absatz [0090] Absatz [0143] - Absatz [0149] Absatz [0119] - Absatz [0122] Absatz [0128]; Abbildungen 1-27	1-15
X	EP 0 613 198 A1 (CANON KK [JP]) 31. August 1994 (1994-08-31) Seite 4, linke Spalte, Zeile 18 - Seite 5, linke Spalte, Zeile 17 Seite 7, linke Spalte, Zeilen 10-42; Abbildungen 1,2	1-15
X	WO 02/23651 A1 (AVESTOR [CA]; CARDARELLI FRANCOIS [CA]; DUBE JONATHAN [CA]) 21. März 2002 (2002-03-21) Seite 8, Zeile 21 - Seite 10, Zeile 11	1-15

INTERNATIONALER RECHERCHENBERICHT

Angaben zu Veröffentlichungen, die zur selben Patentfamilie gehören

Internationales Aktenzeichen
PCT/EP2010/058716

Im Recherchenbericht angeführtes Patentdokument		Datum der Veröffentlichung	Mitglied(er) der Patentfamilie		Datum der Veröffentlichung
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			JP	3495707 B2	09-02-2004
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			EP	1269554 A1	02-01-2003
			JP	2004508694 T	18-03-2004
			US	2004028585 A1	12-02-2004

Espacenet

Bibliographic data: WO2010149611 (A1) — 2010-12-29

METHOD FOR SAFELY COMMINUTING LITHIUM-ION BATTERIES

Inventor(s): WOEHRLE THOMAS [DE]; KERN RAINER [DE] ± (WOEHRLE, THOMAS, ; KERN, RAINER)

Applicant(s): SB LIMOTIVE CO LTD [KR]; SB LIMOTIVE GERMANY GMBH [DE]; WOEHRLE THOMAS [DE]; KERN RAINER [DE] ± (SB LIMOTIVE COMPANY LTD, ; SB LIMOTIVE GERMANY GMBH, ; WOEHRLE, THOMAS, ; KERN, RAINER)

Classification: - international: ***A62D1/00; B02C19/00; B02C23/00; B09B3/00; C22B7/00; H01M6/52***
- cooperative: **B02C19/0056**; **B02C23/04**; **B02C23/18**; **B02C23/36**; **C22B1/248**; **C22B7/00**; **H01M10/54**; **H01M6/52**; C22B26/12; Y02P10/212; Y02W30/84

Application number: WO2010EP58716 20100621 Global Dossier

Priority number(s): DE20091027179 20090625

Also published as: DE102009027179 (A1) EP2446497 (A1) EP2446497 (B1)

Abstract of WO2010149611 (A1)

The invention relates to a method for safely comminuting batteries, comprising the steps of: a) providing one or more batteries to be comminuted; and b) mechanically comminuting the batteries, wherein the comminuting process takes place in the presence of: i) at least one metal flame retardant suitable for preventing or suppressing combustion of the batteries; and ii) at least one binding agent suitable for binding acids and/or bases.

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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
15/569,484	10/26/2017	Christian HANISCH	03101160US

CONFIRMATION NO. 9373

30743
W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

PUBLICATION NOTICE

Title:METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

Publication No.US-2018-0301769-A1
Publication Date:10/18/2018

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

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In addition, information on the status of the application, including the mailing date of Office actions and the dates of receipt of correspondence filed in the Office, may also be accessed via the Internet through the Patent Electronic Business Center at www.uspto.gov using the public side of the Patent Application Information and Retrieval (PAIR) system. The direct link to access this status information is currently https://portal.uspto.gov/pair/PublicPair. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

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Office of Data Managment, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

MULTIPLE DEPENDENT CLAIM FEE CALCULATION SHEET

Substitute for Form PTO-1360
(For use with Form PTO/SB/06)

Application Number	Filing Date
15569484	

Applicant(s) Christian HANISCH

* May be used for additional claims or amendments

CLAIMS	AS FILED		AFTER FIRST AMENDMENT		AFTER SECOND AMENDMENT	
	Indep	Depend	Indep	Depend	Indep	Depend
1	1		1			
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U.S. APPLICATION NUMBER NO.	FIRST NAMED INVENTOR	ATTY. DOCKET NO.
15/569,484	Christian HANISCH	03101160US

30743
W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

INTERNATIONAL APPLICATION NO.	
PCT/EP2016/059526	
I.A. FILING DATE	PRIORITY DATE
04/28/2016	04/28/2015

CONFIRMATION NO. 9373
371 ACCEPTANCE LETTER

OC000000100863347

Date Mailed: 07/11/2018

NOTICE OF ACCEPTANCE OF APPLICATION UNDER 35 U.S.C 371 AND 37 CFR 1.495

The applicant is hereby advised that the United States Patent and Trademark Office, in its capacity as a Designated / Elected Office (37 CFR 1.495), has ACCEPTED the above identified international application for national patentability examination in the United States Patent and Trademark Office.

The United States Application Number assigned to the application is shown above. A Filing Receipt will be issued for the present application in due course. **THE DATE APPEARING ON THE FILING RECEIPT AS THE "FILING DATE or 371(c) DATE" IS THE DATE ON WHICH THE LAST OF THE 35 U.S.C. 371 (c)(1) and (c)(2) REQUIREMENTS HAS BEEN RECEIVED IN THE OFFICE. THIS DATE IS SHOWN BELOW.** The filing date of the above identified application is the international filing date of the international application (Article 11(3) and 35 U.S.C. 363)

10/26/2017
DATE OF RECEIPT OF 35 U.S.C.
371(c)(1) and (c)(2) REQUIREMENTS

The following items have been received:

- Copy of the International Application filed on 10/26/2017
- English Translation of the IA filed on 10/26/2017
- Copy of the International Search Report filed on 10/26/2017
- Preliminary Amendments filed on 10/26/2017
- Information Disclosure Statements filed on 10/26/2017
- Inventor's Oath or Declaration filed on 11/20/2017
- Request for Immediate Examination filed on 10/26/2017
- U.S. Basic National Fees filed on 10/26/2017
- Assignment filed on 11/20/2017
- Authorize Access to Search Results filed on 10/26/2017
- Priority Documents filed on 10/26/2017
- Authorization to Permit Access filed on 10/26/2017
- Application Data Sheet (37 CFR 1.76) filed on 10/26/2017

Applicant is reminded that any communications to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no. shown above (37 CFR 1.5)

ROSA M WEST

Telephone: (571) 272-1019

PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 15/569,484

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY FEE($)
BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A			N/A	300
SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A	N/A			N/A	520
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A			N/A	760
TOTAL CLAIMS (37 CFR 1.16(i))	19 minus 20 =	*			OR	x 100 =	0.00
INDEPENDENT CLAIMS (37 CFR 1.16(h))	2 minus 3 =	*				x 460 =	0.00
APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						0.00
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))							0.00
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL			TOTAL	1580

APPLICATION AS AMENDED - PART II

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY ADDITIONAL FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY ADDITIONAL FEE($)
AMENDMENT A	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	RATE($)	ADDITIONAL FEE($)		RATE($)	ADDITIONAL FEE($)
AMENDMENT B	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".
The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.

Duesenfeld Ex. 2014-437

UNITED STATES PATENT AND TRADEMARK OFFICE

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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
15/569,484	10/26/2017		1620	03101160US	19	2

CONFIRMATION NO. 9373

30743
W&C IP
11491 SUNSET HILLS ROAD
SUITE 340
RESTON, VA 20190

FILING RECEIPT

OC000000100863346

Date Mailed: 07/11/2018

Receipt is acknowledged of this non-provisional patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Inventor(s)

Christian HANISCH, Braunschweig, GERMANY;
Bastian WESTPHAL, Braunschweig, GERMANY;
Wolfgang HASELRIEDER, Braunschweig, GERMANY;
Martin SCHOENITZ, Braunschweig, GERMANY;

Applicant(s)

DUESENFELD GMBH, Braunschweig, GERMANY;

Power of Attorney: None

Domestic Priority data as claimed by applicant

This application is a 371 of PCT/EP2016/059526 04/28/2016

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.)
GERMANY 10 2015 207 843.4 04/28/2015

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 07/10/2018

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/569,484**

Projected Publication Date: 10/18/2018

Non-Publication Request: No

Early Publication Request: No
Title

METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

Preliminary Class

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at http://www.uspto.gov/web/offices/pac/doc/general/index.html.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, http://www.stopfakes.gov. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

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NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

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PATENT ASSIGNMENT COVER SHEET

Electronic Version v1.1
Stylesheet Version v1.2

EPAS ID: PAT4697332

SUBMISSION TYPE:	NEW ASSIGNMENT
NATURE OF CONVEYANCE:	ASSIGNMENT

CONVEYING PARTY DATA

Name	Execution Date
CHRISTIAN HANISCH	11/08/2017
BASTIAN WESTPHAL	11/08/2017
WOLFGANG HASELRIEDER	11/09/2017
MARTIN SCHOENITZ	11/09/2017

RECEIVING PARTY DATA

Name:	DUESENFELD GMBH
Street Address:	FRIEDLANDWEG 5
City:	BRAUNSCHWEIG
State/Country:	GERMANY
Postal Code:	38114

PROPERTY NUMBERS Total: 1

Property Type	Number
Application Number:	15569484

CORRESPONDENCE DATA

Fax Number:

Correspondence will be sent to the e-mail address first; if that is unsuccessful, it will be sent using a fax number, if provided; if that is unsuccessful, it will be sent via US Mail.

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Address Line 4: RESTON, VIRGINIA 20190

ATTORNEY DOCKET NUMBER:	03101160US
NAME OF SUBMITTER:	MICHAEL E. WHITHAM
SIGNATURE:	/Michael E. Whitham/
DATE SIGNED:	11/20/2017
	This document serves as an Oath/Declaration (37 CFR 1.63).

Total Attachments: 2
source=03101160US_dec#page1.tif

source=03101160US_dec#page2.tif

DECLARATION (37 C.F.R. 1.63) FOR UTILITY PATENT APPLICATION USING AN APPLICATION DATA SHEET (37 C.F.R. 1.76) AND ASSIGNMENT

Title of Invention: **METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES AND BATTERY PROCESSING INSTALLATION**

As a below named inventor, I hereby declare that:

This declaration and assignment document is directed to:

[ ] the attached application;

[X] United States application number 15/569,484 filed on October 26, 2017;

[X] PCT international application number PCT/EP2016/059526 filed on April 28, 2016.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I have reviewed and understand the contents of the application, including the claims.

I am aware of the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in 37 CFR Section 1.56.

Whereas, the undersigned inventor(s) has/have made certain inventions, improvements, and discoveries (herein referred to as the "Invention") disclosed in the above-identified patent application;

Whereas, **DUESENFELD GMBH**, having a place of business at Friedlandweg 5, 38114 Braunschweig, DE (herein referred to as "ASSIGNEE"), desires to acquire, and each undersigned inventor desires to grant to ASSIGNEE, the entire worldwide right, title, and interest in and to the Invention and in and to any and all patent applications and patents directed thereto;

Now, therefore, for good and valuable consideration, the receipt and sufficiency thereof being hereby acknowledged, each undersigned inventor ("ASSIGNOR") hereby sells or has sold, assigns or has assigned, and otherwise transfers or has transferred to the ASSIGNEE, its successors, legal representatives, and assigns, the entire worldwide right, title, and interest in and to the Invention, the above-identified United States patent application, and any and all other patent applications and patents for the Invention which may be applied for or granted therefor in the United States and in all foreign countries and jurisdictions, including all divisions, continuations, reissues, reexaminations, renewals, extensions, counterparts, substitutes, and extensions thereof, and all rights of priority resulting from the filing of such applications and granting of such patents. In addition, each undersigned inventor hereby authorizes and requests the Director of the United States Patent and Trademark Office to issue any United States Patent, and foreign patent authorities to issue any foreign patent, granted for the Invention, to ASSIGNEE, its successors, legal representatives, and assigns, the entire worldwide right, title, and interest in and to the same to be held and enjoyed by ASSIGNEE, its successors, legal representatives, and assigns to the full end of the terms for which any and all such patents may be granted, as fully and entirely as would have been held and enjoyed by the undersigned had this Assignment not been made; and each undersigned inventor agrees to execute any and all documents and instruments and perform all

lawful acts reasonably related to recording this Assignment or perfecting title to the Invention and all related patents and applications, in ASSIGNEE, its successors, legal representatives, and assigns, whenever requested by ASSIGNEE, its successors, legal representatives, or assigns.

Each undersigned inventor acknowledges their prior and ongoing obligations to sell, assign, and transfer the rights under this Assignment to ASSIGNEE and is unaware of any reason why they may not have the full and unencumbered right to sell, assign, and transfer the rights hereby sold, assigned, and transferred, and has not executed, and will not execute, any document or instrument in conflict herewith. Each undersigned inventor also hereby grants ASSIGNEE, its successors, legal representatives, and assigns, the right to insert in this Assignment any further identification (including, but not limited to, patent Application Number) which may be necessary or desirable for recordation of this Assignment.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

I hereby authorize and request any attorney of WHITHAM, CURTIS & COOK, P.C., Customer No. 30743, to insert or correct any information concerning the identity of ASSIGNOR, ASSIGNEE, or the above-mentioned application (including the serial no. and the filing date).

(1) Legal Name of Inventor: **Christian HANISCH**

Signature: _____ Date: 08.11.2017

(2) Legal Name of Inventor: **Bastian WESTPHAL**

Signature: _____ Date: 09.11.2017

(3) Legal Name of Inventor: **Wolfgang HASELRIEDER**

Signature: _____ Date: 08.11.2017

(4) Legal Name of Inventor: **Martin SCHOENITZ**

Signature: _____ Date: 09.11.2017

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of

Hanisch	Confirmation No. (not assigned)
Serial No. (not assigned)	Group Art Unit (not assigned)
Filed Concurrently Herewith	Examiner (not assigned)

For METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

This application is a Rule 371 National Stage filing from PCT/EP2016/059526 filed on April 28, 2016, which claims priority to German Application 10 2015 207 843.4 filed April 28, 2015

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

PRELIMINARY AMENDMENT

Sir:

Prior to calculation of the filing fees and examination on the merits, please amend the above-identified patent application as follows:

Amendments to the Claims: begin on page 2

Remarks: begin on page 6

Listing of the Claims:

The following is a complete listing of all the claims in the application, with an indication of the status of each:

1.(currently amended) A method ~~Method~~ for the treatment of used batteries, comprising ~~(10), in particular lithium batteries, containing~~ the steps:

(a) comminuting the batteries ~~(10)~~ such that comminuted material ~~(24)~~ is obtained,

(b) inactivating the comminuted material ~~(24)~~ such that an inactivated comminuted material ~~(42)~~ is obtained, and

(c) filling a transport container ~~(46)~~ with the inactivated comminuted material ~~(42)~~,

~~characterised by the fact that~~

~~(d)~~ wherein the inactivating step ~~inactivation~~ is performed by drying the comminuted material ~~(24)~~.

2.(currently amended) The method ~~Method~~ according to claim 1, wherein ~~characterised by the fact that~~ the comminuted material ~~(24)~~ is dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

3.(currently amended) The method according to claim 1, wherein ~~Method according to one of the above claims, characterised by the fact that~~ the drying occurs under vacuum.

4.(currently amended) The method according to claim 3, wherein ~~Method according to one of the above claims, characterised by the fact that~~ the vacuum is created by means of a jet pump, and

a pump liquid of the jet pump has at least one of

a pH value of at least 8, and ~~especially 9, and/or~~

contains a substance that precipitates fluoride.

5.(currently amended) The method according to claim 1, further comprising the steps of ~~Method according to one of the above claims, characterised by the steps~~:

~~(e)~~ removal of the comminuted material ~~(24)~~ from the transport container ~~(46)~~,

~~(f)~~ one or more of

~~detachment of hard parts~~, and ~~and/or~~

separation of active material from a ~~the~~ carrier,

~~particularly through air jet sieving or a second comminution stage~~

such that an active material fraction ~~(76)~~ and a carrier fraction (80) are obtained, and

~~(g)~~ separate packing of the active material fraction ~~(76)~~ and carrier fraction ~~(80)~~ in transport containers ~~(78)~~.

6.(currently amended) The method according to claim 1, wherein ~~Method according to one of the above claims, characterised by the fact that~~ the drying of the comminuted material is only completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material ~~that has been filled~~ in the transport container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing.

7.(currently amended) A battery ~~Battery~~ processing installation for ~~the~~ treatment of used batteries, comprising ~~in particular lithium batteries, with~~:

(a) a comminution unit ~~(18)~~ for comminuting the batteries ~~(10)~~ such that comminuted material ~~(24)~~ is obtained,

(b) an inactivation device ~~(26)~~ for inactivating the comminuted material ~~(24)~~ and

(c) a filling device ~~(44)~~ for filling a transport container ~~(46)~~ with ~~the~~ inactivated comminuted material ~~(24)~~,

~~characterised by the fact that~~ wherein

~~(d)~~ the inactivation device comprises a drying device, ~~(26)~~ and

~~(e)~~ the battery processing installation has a vacuum installation that is connected to the drying device ~~(26)~~ for ~~the purpose of~~ generating a vacuum in the drying device ~~(26)~~.

8.(currently amended) The battery ~~Battery~~ processing installation according to claim 7, wherein ~~characterised by the fact that~~ the drying device ~~(26)~~ is configured to dry the comminuted material ~~(21)~~ until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible.

9.(currently amended) The battery ~~Battery~~ processing installation according to claim 7, wherein ~~or 8, characterised by the fact that~~ the comminution unit and the drying device ~~(26)~~ are arranged in a ~~standard~~ container ~~(54)~~.

10. (canceled)

11. (currently amended) The battery ~~Battery~~ processing installation according to claim 7, wherein ~~10, characterised by the fact that~~

- the vacuum installation ~~(29)~~ has a jet pump with a pump liquid, and wherein
- the pump liquid is conducted within a circuit and
- the pump liquid contains a substance that reacts with hydrogen fluoride.

12. (currently amended) The battery ~~Battery~~ processing installation according to claim 7 further comprising: ~~one of the claims 7 to 11, characterised by~~

- a hard metal detachment device and/or a light fraction detachment device,
- a separation device~~, especially a classification device (74),~~ for separating active material from a ~~the~~ carrier~~, in particular by means of air jet sieving and/or a second comminution stage~~ such that an active material fraction

~~(76)~~ and a carrier fraction occur~~, in particular aluminium and copper foils~~, and

- a second filling device ~~(44)~~ for ~~the~~ separate filling of the active material fraction ~~(76)~~ and the carrier fraction ~~(80), in particular aluminium and copper foils~~.

13. (New) The battery processing installation of claim 12 wherein the active material fraction and the carrier fraction are respectively aluminum and copper foils.

14. (New) The battery processing installation of claim 12, wherein the separation device is an air jet sieving device.

15. (New) The battery processing installation of claim 12 wherein the separation device is a second comminution stage.

16. (New) The battery processing installation of claim 7 wherein the batteries are lithium batteries.

17. (New) The method according to claim 1 wherein the batteries are lithium batteries.

18. (New) The method according to claim 5 wherein separation of the active material from the carrier is performed by air jet sieving.

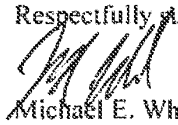
19. (New) The method according to claim 5 wherein separation of the active material from the carrier is performed using a second comminution stage.

20. (New) The method according to claim 4 wherein the pH value is at least 9.

REMARKS

Claim 10 is cancelled. Claims 1-9 and 11-12 are amended. Claims 13-20 are added. The amendments eliminate multiple dependent claim format, provide for antecedent basis, and amend the language to a form more suited to US practice. Please calculate the filing fee based on claims 1-9 and 11-20, and proceed to examination on the merits.

Respectfully submitted.



Michael E. Whitham
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Tel. (703) 787-9400
Fax. (703) 787-7557

Customer No.: 30743

Electronic Patent Application Fee Transmittal

Application Number:	
Filing Date:	
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIS, AND BATTERY PROCESING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Filer:	Michael E. Whitham/Kyle Comiskey
Attorney Docket Number:	03101160US

Filed as Large Entity

Filing Fees for U.S. National Stage under 35 USC 371

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
NATIONAL STAGE FEE	1631	1	280	280
NATL STAGE SEARCH FEE - REPORT PROVIDED	1642	1	480	480
NATIONAL STAGE EXAM - ALL OTHER CASES	1633	1	720	720
Pages:				
Claims:				
Miscellaneous-Filing:				
OATH/DECL > 30 MOS FROM 371 COMMENCEMENT	1617	1	140	140
Petition:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Miscellaneous:				
		Total in USD ($)		**1620**

Electronic Acknowledgement Receipt	
EFS ID:	30767446
Application Number:	15569484
International Application Number:	PCT/EP2016/059526
Confirmation Number:	9373
Title of Invention:	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIS, AND BATTERY PROCESING INSTALLATION
First Named Inventor/Applicant Name:	Christian HANISCH
Customer Number:	30743
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Filer Authorized By:	Michael E. Whitham
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Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		03101160US_app.pdf	1220144 5a9ec60acde03fa84252a612aaca1b57931609c0	yes	60

Multipart Description/PDF files in .zip description

Document Description	Start	End
Transmittal Letter	1	3
Specification - Not in English	4	32
Specification	33	50
Claims	51	54
Abstract	55	55
Drawings-only black and white line drawings	56	58
Documents submitted with 371 Applications	59	60

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Information:

2	Application Data Sheet	03101160US_ADS.pdf	1823198 932894798705dfee771682d7da6ae9859ca173cf	no	9

Warnings:

Information:

3	Information Disclosure Statement (IDS) Form (SB08)	03101160US_IDS.pdf	1035130 1e4280bded390b13e9cdfbc06c3c918a6dfbac01	no	4

Warnings:

Information:

4	Foreign Reference	03101160US_frn1.pdf	908180 600a11f658014655747042b000043fbd2895e98c	no	21
Warnings:					
Information:					
5	Foreign Reference	03101160US_frn2.pdf	1652744 dcd40243811ae098e9a54964942c60c866e9e1f6	no	10
Warnings:					
Information:					
6	Foreign Reference	03101160US_frn3.pdf	1282496 a8faf51ba840bb17bd0f90ba6f4bf8267d2e0ae5	no	14
Warnings:					
Information:					
7		03101160US_prelim.pdf	90006 5bb88166fb023d5d151472c6418ef3cf5da21db7	yes	6

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Preliminary Amendment	1	1
	Claims	2	5
	Applicant Arguments/Remarks Made in an Amendment	6	6

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8	Fee Worksheet (SB06)	fee-info.pdf	37101 ca8ddf8a7d10f3b4a0b473b210746a3e971c860b	no	2
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National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

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PTO-1390 (05-13)
Approved for use through 6/30/2013. OMB 0651-0021
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A SUBMISSION UNDER 35 U.S.C. 371

Attorney Docket No.	U.S. Application No. (if known, see 37 CFR 1.5)
03101160US	

International Application No.	International Filing Date	Priority Date Claimed
PCT/EP2016/059526	2016-04-28	2015-04-28

Title of Invention
METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

First Named Inventor
Christian HANISCH

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. ☑ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). NOTE: The express request under 35 U.S.C. 371(f) will not be effective unless the requirements under 35 U.S.C. 371(c)(1), (2), and (4) for payment of the basic national fee, copy of the International Application and English translation thereof (if required), and the oath or declaration of the inventor(s) have been received.
2. ☑ A copy of the International Application (35 U.S.C. 371(c)(2)) is attached hereto (not required if the International Application was previously communicated by the International Bureau or was filed in the United States Receiving Office (RO/US)).
3. An English language translation of the International Application (35 U.S.C. 371(c)(2))
   a. ☐ is attached hereto.
   b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
4. An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4))
   a. ☐ is attached.
   b. ☐ was previously filed in the international phase under PCT Rule 4.17(iv).

Items 5 to 8 below concern amendments made in the international phase.

PCT Article 19 and 34 amendments

5. ☐ Amendments to the claims under PCT Article 19 are attached (not required if communicated by the International Bureau) (35 U.S.C. 371(c)(3)).
6. ☐ English translation of the PCT Article 19 amendment is attached (35 U.S.C. 371(c)(3)).
7. ☐ English translation of annexes (Article 19 and/or 34 amendments only) of the International Preliminary Examination Report is attached (35 U.S.C. 371(c)(5)).

Cancellation of amendments made in the international phase

8a. ☐ Do not enter the amendment made in the international phase under PCT Article 19.

8b. ☐ Do not enter the amendment made in the international phase under PCT Article 34.

NOTE: A proper amendment made in English under Article 19 or 34 will be entered in the U.S. national phase application absent a clear instruction from applicant not to enter the amendment(s).

The following items 9 to 17 concern a document(s) or information included.

9. ☑ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
10. ☑ A preliminary amendment.
11. ☑ An Application Data Sheet under 37 CFR 1.76.
12. ☐ A substitute specification. NOTE: A substitute specification cannot include claims. See 37 CFR 1.125(b).
13. ☐ A power of attorney and/or change of address letter.
14. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13*ter*.3 and 37 CFR 1.821-1.825.
15. ☐ Assignment papers *(cover sheet and document(s))*. Name of Assignee: _____
16. ☐ 37 CFR 3.73(c) Statement *(when there is an Assignee)*.

This collection of information is required by 37 CFR 1.414 and 1.491-1.492. The information is required to obtain or retain a benefit by the public, which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 15 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop PCT, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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U.S. APPLN. No. (if known – see 37 CFR 1.5)	INTERNATIONAL APPLICATION No.	ATTORNEY DOCKET No.
	PCT/EP2016/059526	03101160US

17. [✓] Other items or information:

INTERNATIONAL SEARCH REPORT

The following fees have been submitted.	CALCULATIONS	PTO USE ONLY
18. [✓] Basic national fee (37 CFR 1.492(a)) $280	$ 280	
19. [✓] Examination fee (37 CFR 1.492(c)) If the written opinion prepared by ISA/US or the international preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4) ..... $0 All other situations $720	$ 720	
20. [✓] Search fee (37 CFR 1.492(b)) If the written opinion prepared by ISA/US or the international preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4) ..... $0 Search fee (37 CFR 1.445(a)(2)) has been paid on the international application to the USPTO as an International Searching Authority ..... $120 International Search Report prepared by an ISA other than the US and provided to the Office or previously communicated to the US by the IB ..... $480 All other situations $600	$ 480	
TOTAL OF 18, 19, and 20 =	$ 1480	
[ ] Additional fee for specification and drawings filed in paper over 100 sheets (excluding sequence listing in compliance with 37 CFR 1.821(c) or (e) in an electronic medium or computer program listing in an electronic medium) (37 CFR 1.492(j)). Fee for each additional 50 sheets of paper or fraction thereof $400		

Total Sheets	Extra Sheets	Number of each addition 50 or fraction thereof (round up to a whole number)	RATE	
- 100 =	/ 50 =		x $400	$

Surcharge of $140.00 for furnishing any of the search fee, examination fee, or the oath or declaration after the date of commencement of the national stage (37 CFR 1.492(h)).	$ 140	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	- 20 =		x $80	$	
Independent claims	- 3 =		x $420	$	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ $780	$	
Processing fee of $140.00 for furnishing the English translation later than 30 months from the earliest claimed priority date (37 CFR 1.492(i)).			+	$	
TOTAL OF ABOVE CALCULATIONS =				$ 1620	
[ ] Applicant asserts small entity status. See 37 CFR 1.27. Fees above are reduced by ½.					
[ ] Applicant certifies micro entity status. See 37 CFR 1.29. Fees above are reduced by ¾. Applicant must attach form PTO/SB/15A or B or equivalent.					
TOTAL NATIONAL FEE =				$ 1620	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property.			+	$	
TOTAL FEES ENCLOSED =				$ 1620	
				Amount to be refunded:	$
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ii. ☑ any required fee except for excess claims fees required under 37 CFR 1.492(d) and (e) and multiple dependent claim fee required under 37 CFR 1.492(f).

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Correspondence Address

☑ The address associated with Customer Number: 38743 OR ☐ Correspondence address below

Name					
Address					
City		State		Zip Code	
Country			Telephone		
Email					

Signature	/Michael E. Whitham/	Date	10/26/17
Name (Print/Type)	Michael E. Whitham	Registration No. (Attorney/Agent)	32635

(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

(19) Weltorganisation für geistiges Eigentum
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WO 2016/174156 A1

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Erklärungen gemäß Regel 4.17:

— *hinsichtlich der Berechtigung des Anmelders, ein Patent zu beantragen und zu erhalten (Regel 4.17 Ziffer ii)*

[Fortsetzung auf der nächsten Seite]

(54) Title: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

(54) Bezeichnung : VERFAHREN ZUM BEHANDELN GEBRAUCHTER BATTERIEN, INSBESONDERE WIEDER AUFLADBARER BATTERIEN UND BATTERIE-VERARBEITUNGSANLAGE

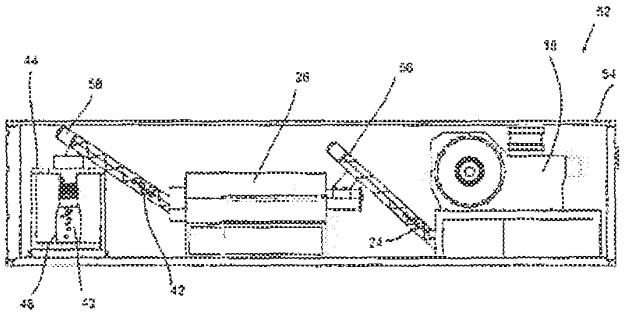

Fig. 2

(57) Abstract: The invention relates to a method for the treatment of used batteries (10), in particular lithium batteries, having the steps: comminuting the batteries (10) such that comminuted material (24) is obtained, inactivating the comminuted material (24) such that inactivated comminuted material (42) is obtained, and filling a transport container (46) with the inactivated comminuted material (42). It is provided according to the invention that the inactivation is performed by drying the comminuted material (24), and the comminuted material (24) is dried until an electrolyte content is so low that an electrochemical reaction is not possible.

(57) Zusammenfassung:

[Fortsetzung auf der nächsten Seite]

WO 2016/174156 A1

WO 2016/174156 A1

Veröffentlicht:

mit internationalem Recherchenbericht (Artikel 21 Absatz 3)

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten: Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird, Inaktivieren des Zerkleine- rungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehäl- ter (46). Erfindungsgemäß ist vorgesehen, dass das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt und das Zerkleinerungsgut (24) solange ge- trocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

Verfahren zum Behandeln gebrauchter Batterien, insbesondere wieder aufladbarer Batterien und Batterie-Verarbeitungsanlage

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien, insbesondere gebrauchter Lithium-Batterien, beispielsweise Lithium-Ionen-Batterien, mit den Schritten (a) Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) Inaktivieren des Zerkleinerungsguts, sodass ein inaktiviertes Zerkleinerungsgut erhalten wird, und (c) Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Gemäß einem zweiten Aspekt betrifft die Erfindung eine Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere zum Behandeln gebrauchter Lithium-Batterien, mit (a) einer Zerkleinerungseinheit zum Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) einer Inaktivierungsvorrichtung zum Inaktivieren des Zerkleinerungsguts und (c) einer Abfüllvorrichtung zum Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Aus der US 2005/0241943 A1 ist ein Verfahren zum Aufarbeiten gebrauchter Batterien bekannt, bei dem die Batterien vor einem Zerkleinerungsschritt erwärmt werden, sodass Kunststoffbestandteile der Batterien zerstört werden. Nachteilig an einem derartigen Vorgehen ist, dass die verbleibenden Komponenten der Batterien mit Abbauprodukten des Kunststoffs verunreinigt sein können.

Aus der DE 10 2012 024 876 A1 ist ein System zum Überführen transportkritischer Elektrolytzellen zunächst unter Inertgas zerkleinert und danach mit einem Desaktivierungspulver bestäubt werden, um eine Selbstentzündung des elektrochemisch aktiven Materials zu verhindern. Nachteilig daran ist, dass das entstehende Material dennoch ein vergleichsweise hohes Gefährdungspotential besitzt und dass das Bestäubungspulver selbst ein Explosionsrisiko darstellt und dass die Bildung einer entzündlichen und explosiven Atmosphäre in dem Transportbehälter nicht ausgeschlossen werden kann.

Aus der DE 10 2011 110 083 A1 ist ein Verfahren zum Wiedergewinnen von Aktivmaterial aus einer galvanischen Zelle bekannt, bei dem die galvanischen Zellen zunächst mechanisch zerkleinert, dann vorgetrocknet und nachfolgend gesichtet werden. Schließlich wird der Binder in einem Ofen zersetzt. Eine derartige Vorrichtung eignet sich sehr gut zum effizienten Recyceln größerer Mengen von galvanischen Elementen. Für den Teillast-Betrieb ist diese Anlage jedoch vergleichsweise komplex aufgebaut.

Der Erfindung liegt die Aufgabe zugrunde, Nachteile im Stand der Technik zu vermindern.

Die Erfindung löst das Problem durch ein gattungsgemäßes Verfahren, bei dem das Inaktivieren zumindest auch durch Trocknen des Zerkleinerungsguts erfolgt. Gemäß einem zweiten Aspekt löst die Erfindung das Problem durch eine gattungsgemäße Batterie-Verarbeitungsanlage, bei der die Aktivierungsvorrichtung eine Trocknungsvorrichtung umfasst.

Vorteilhaft an der Erfindung ist, dass durch das Trocknen dem Zerkleinerungsgut so viel Elektrolyt entzogen werden kann, dass eine elektrochemische Reaktion nicht mehr oder nur noch zu vernachlässigbar kleinem Ausmaß möglich ist. Zudem bildet sich keine entzündliche oder explosive Gasphase über den Batteriefragmenten, da diesen insbesondere die organischen Karbonate des Elektrolyten entzogen wurden. Das Zerkleinerungsgut ist daher weitgehend inert und kann, insbesondere dann, wenn es unter Vakuum verpackt wird, sicher transportiert werden.

Ein weiterer Vorteil ist es, dass zum Inaktivieren des Zerkleinerungsmaterials kein zusätzliches Material hinzugegeben werden muss. Das verringert den Aufwand beim Aufarbeiten der Batterien, verringert das Gewicht des inaktivierten Zerkleinerungsguts und erhöht die Reinheit in den folgenden Trenn- und Recyclingschritten. Insbesondere in potentiell folgenden hydrometallurgischen Aufarbeitungsschritten ist eine hohe Produktreinheit ohne Eintrag von Fremdionen

vorteilhaft.

Vorteilhaft ist zudem, dass ein transportsicheres Zerkleinerungsgut erhalten wird. So ist der Gehalt an organischen Carbonaten vorzugsweise so klein, dass die Bildung von Fluorophosphaten in relevanter Menge ausgeschlossen werden kann. Fluorophosphate sind oft starke Nervengifte, deren Bildung sicher verhindert werden muss. Zudem ist sichergestellt, dass es aufgrund des geringen Elektrolytgehalts nicht zu einer durch eine elektrochemische Reaktion ausgelöste, selbstverstärkend fortschreitende Wärmeentwicklung kommen kann.

Im Rahmen der vorliegenden Beschreibung wird unter dem Trocknen insbesondere das Entfernen zumindest eines Lösungsmittels des Leitsalzes verstanden. Insbesondere wird das Trocknen so durchgeführt, dass Kohlensäuredimethylester und/oder Kohlensäureethylmethylester entfernt wird.

Unter der Batterie wird insbesondere eine Lithium-Batterie verstanden. Eine Lithium-Batterie ist ein Akkumulator, bei dem die elektrochemische Reaktion Lithium und/oder Lithium-Ionen und/oder eine Lithium-Verbindung involviert.

Unter einer Batterie-Verarbeitungsanlage wird insbesondere auch eine Akku-Verarbeitungsanlage zum Verarbeiten von Akkumulatoren verstanden.

Unter dem Transportbehälter wird insbesondere auch eine oder eine Transportverpackung verstanden. Vorzugsweise wird die Transportverpackung durch Vakuumverschweißung verschlossen. Gut geeignet als Transportverpackung ist insbesondere Aluminium-Verbundfolie.

Günstig ist, wenn das Trocknen nach dem Zerkleinern der Batterien erfolgt. Zwar ist es möglich und stellt eine Ausführungsform der Erfindung dar, dass die Batterien im unzerkleinerten Zustand einem Vakuum ausgesetzt werden, sodass zumindest Teile des Elektrolyten verdampfen, wobei das entstehende Gas entweder durch ein Sicherheitsventil im Akkumulator entweicht oder die Batterie durch die Druckdifferenz zwischen der äußeren Umgebung und dem Innendruck

zerstört wird, sodass verdampfender Elektrolyt austreten kann. Da der Elektrolyt zumeist zwischen eng gewickelten oder gestapelten und verpressten Schichten aus Elektroden und Separator und in deren Poren vorliegt und mit anderen Bestandteilen der Batterien verbunden vorliegt, kann dieses Vorgehen jedoch zeitintensiv sein. Es ist daher oft günstiger und stellt eine bevorzugte Ausführungsform der Erfindung dar, dass die Batterien mechanisch zerkleinert werden, beispielsweise durch Schneiden, Scheren, Pralleinwirkung, Zertrennen und/oder Quetschen. So steht für den Stoffübergang in die Gasphase eine größere Grenzfläche zur Verfügung.

Das Trocknen kann beispielsweise durch Vakuumtrocknung, Kontakttrocknung, Konvektionstrocknung und/oder Strahlungstrocknung erfolgen. Günstig ist es, wenn das Trocknen unter Rühren und/oder Umwälzen des Zerkleinerungsguts erfolgt.

Vor dem Zerkleinern werden die gebrauchten Batterien vorzugsweise demontiert. Das heißt, dass größere Batteriesysteme in ihre kleineren Unterbausteine, die Module oder Stacks, demontiert werden oder sogar die Zellen, die das elektrochemisch wirksame Material enthalten, von der Ansteuerelektronik getrennt werden. Die Ansteuerelektronik umfasst beispielsweise Halbleiterbauelemente und/oder Sensoren und dient der Ladesteuerung der Batterien.

Gemäß einer bevorzugten Ausführungsform erfolgt das Trocknen unter Vakuum. Vorzugweise ist das Vakuum so groß gewählt, dass der Dampfdruck von Kohlensäuredimethylester bei 80°C, insbesondere bei 70°C, unterschritten wird. Günstig ist es, wenn das Trocknen unter einem Druck von höchstens 300 hPa, insbesondere höchstens 100 hPa erfolgt. Bei so niedrigen Drücken verdampfen wesentliche Bestandteile der meisten Elektrolyten, insbesondere Kohlensäuredimethylester und Kohlensäureethylmethylester, bereits bei Temperaturen von weniger als 80°C. Niedrige Temperaturen haben den Vorteil, dass die Bildung von Fluorwasserstoff behindert wird. Fluorwasserstoff stellt eine potentielle Gefährdung für die Batterie-Verarbeitungsanlage sowie die Umgebung dar. Es ist daher günstig, das Entstehen von Fluorwasserstoff zu verhindern.

Vorzugsweise erfolgt das Trocknen bei einer Temperatur, die unterhalb einer Zersetzungstemperatur liegt. Unter der Zersetzungstemperatur wird insbesondere die kleinste Temperatur verstanden, für die gilt, dass nach dem Halten des Zerkleinerungsguts auf dieser Temperatur für eine Stunde zumindest 80 Masseprozent des Binders sich in gasförmige Bestandteile zersetzt haben. Die Zersetzungstemperatur kann damit gemessen werden, indem die Temperatur des Zerkleinerungsguts sukzessive erhöht wird und erfasst wird, wann es zu einem Masseverlust, insbesondere durch Gasentwicklung aufgrund einer Zersetzung des Binders, kommt und das angegebene Kriterium erfüllt ist. Gegebenenfalls muss der Versuch mit jeweils einer neuen Probe an Zerkleinerungsgut bei jeweils erhöhter Temperatur mehrfach durchgeführt werden.
Günstig ist es, wenn das Trocknen unter einer Atmosphäre erfolgt, in der der Wasser-Partialdruck kleiner ist als 50 Pa, insbesondere kleiner als 10 Pa. Ein geringer Wasser-Partialdruck führt zu einer geringen Reaktionsrate von Lithium-Verbindungen zu Lithium-Hydroxid und damit zu nur geringer Wasserstoff-Entwicklung. Das verhindert die Bildung von zündfähigen Wasserstoff-Sauerstoff-Gemischen und trägt zur Anlagensicherheit bei.

Günstig ist es zudem, wenn der Sauerstoff-Partialdruck höchstens 10 Millibar, insbesondere höchstens 5 Millibar beträgt. Die Reaktion von Sauerstoff mit oxidierbaren Bestandteilen der Batterien wird so weitgehend unterdrückt. Es ist möglich, den niedrigen Sauerstoff-Partialdruck durch einen geringen Druck beim Trocknen zu erreichen. Alternativ oder zusätzlich kann das Trocknen in einer Inertgas-Atmosphäre erfolgen.

Bevorzugt ist ein Verfahren, bei dem das Trocknen des Zerkleinerungsguts erst beendet wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut mehr bilden kann und/oder wenn das Zerkleinerungsgut so trocken ist, dass sich im Transportbehälter oder während der Weiterverarbeitung ein entzündliches oder explosives Gasgemisch ergeben kann. Unter dem Merkmal, dass das Trocknen been-

det wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut bilden kann, wird insbesondere verstanden, dass sich in einem Transportbehälter in Form eines 50-Literbehälter, der zur Hälfte (bezogen auf sein Volumen) mit dem Zerkleinerungsgut gefüllt ist, innerhalb von einer Woche bei 50°C und 1013 hPa kein zündfähiges Gasgemisch bildet. Ob das Kriterium erfüllt ist, wird in Vorversuchen ermittelt. Bildet sich ein zündfähiges Gasgemisch, muss das Trocknen länger und/oder bei niedrigerem Druck durchgeführt werden. Die Vorversuche werden so lange wiederholt, bis eine Trocknungsdauer und/oder ein Trocknungsdruck ermittelt ist, bei der das Merkmal bei einem Testsatz von drei Transportbehälter für alle drei Transportbehälter erfüllt ist.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, bis ein Elektrolytgehalt im Zerkleinerungsgut so klein ist, dass eine elektrochemische Reaktion unmöglich ist. In anderen Worten ist der Elektrolytgehalt kleiner als ein Schwellenwert, wobei der Schwellenwert so gewählt ist, dass die Zellspannung bei Unterschreiten dieses Schwellenwerts auf höchstens ein Viertel abgesunken ist. Dieser Schwellenwert wird beispielsweise dadurch bestimmt, dass die Zellenspannung einer Batterie in Abhängigkeit vom Elektrolytgehalt bestimmt wird. Kurz vor dem Erreichen des Schwellenwerts bricht die Zellspannung zusammen, das heißt, dass sie um zumindest 75% absinkt. Ist der Schwellenwert unterschritten, enthält die Batterie so wenig Elektrolyt, dass in guter Näherung keine elektrochemische Reaktion mehr möglich ist.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, dass eine Menge von 50 kg an Zerkleinerungsmaterial, das kompaktiert in einem 50 Liter-Fass enthalten ist, keine oder eine nur so geringe Wärmeentwicklung zeigt, dass ein thermisches Durchgehen, also eine thermisch induzierte Kettenreaktion, für zumindest zwei Monate ausgeschlossen ist und dass eine etwaige Wasserstoff-Entwicklung zudem so gering ausgebildet ist, dass nach zwei Wochen kein Überdruck entsteht, wenn zu Beginn ein Unterdruck von 500 hPa herrscht.

Günstig ist es, wenn das Zerkleinerungsgut so lange getrocknet wird, bis der

Elektrolytgehalt an organischen Komponenten, die bei 80°C flüchtig sind, höchsten 3 Gew.-%, höchstens insbesondere 2 Gew.-%, besonders bevorzugt höchstens 1,5 Gew.-%, beträgt.

Vorzugsweise wird das Trocknen so lange durchgeführt, dass der akkumulierte Gehalt an organischen Karbonaten aus dem Elektrolyten, die bei 80°C flüchtig sind, in der Atmosphäre über dem Zerkleinerungsgut 3 Vol.-% unterschreitet.

Insbesondere wird das Trocknen so lange durchgeführt, bis der Gehalt an Kohlensäuredimethylester 4 Vol.-%, insbesondere 3 Vol.-%, unterschreitet und/oder der Gehalt an Cyclohexylbenzol 1 Vol.-%, insbesondere 0,5 Vol.-%, unterschreitet.

Vorzugsweise erfolgt das Trocknen unmittelbar nach dem Zerkleinern. Hierunter wird insbesondere verstanden, dass zwischen dem Beginn des Zerkleinerns der Batterien und dem Zeitpunkt, zu dem zumindest ein Teil des dadurch entstandenen Zerkleinerungsguts anfängt zu Trocknen, höchstens fünf Minuten beträgt, insbesondere höchstens eine Minute. Durch die schnelle Trocknung nach dem Zerkleinern wird die Masse an Material klein gehalten, die potentiell elektrochemisch reagieren könnte, zudem wird die elektrochemische Reaktionszeit potentieller exothermer Reaktionen klein gehalten. Das senkt das Risiko für die Anlage und die Umgebung.

Besonders günstig ist es, wenn das Vakuum mittels einer Strahlpumpe erzeugt wird. Strahlpumpen sind, insbesondere bei geeigneter Wahl des Strahlmediums, weitgehend unempfindlich gegen aggressive abzupumpende Gase. Günstig ist es, wenn das Strahlmedium, das eine Flüssigkeit ist, einen pH-Wert von zumindest 8, insbesondere von zumindest 9, beispielsweise zumindest 12, hat. In diesem Fall können sich unerwünschte Bestandteile des Gases, das abgepumpt wird, zersetzen oder zu weniger schädlichen Substanzen reagieren. So kann beispielsweise Kohlensäuredimethylester und/oder Kohlensäureethylmethylester durch eine Verseifungsreaktion abgebaut werden. Etwaig im Strahlenmedium

enthaltener Fluorwasserstoff kann im basischen Milieu in einer Säure-Base-Reaktion zu einem ungefährlichen Salz umgesetzt werden.

Vorzugsweise enthält das Strahlenmedium eine Fluor fällende Substanz. Beispielsweise kann das Strahlenmedium Natriumkarbonat oder Calziumkarbonat enthalten. Die bei der Reaktion mit einer Fluorverbindung, insbesondere Fluorwasserstoff, entstehenden Salze werden vorzugsweise abgetrennt, insbesondere abgefiltert oder aussedimentiert. Auf diese Weise wird zumindest weitgehend verhindert, dass Fluorwasserstoff oder andere giftige fluorhaltige Verbindungen in die Umgebung abgegeben werden können.

Vorzugsweise erfolgt das Trocknen bei einer Temperatur von höchstens 80°C. In diesem Fall entsteht kaum Fluorwasserstoff. Das erhöht die Lebensdauer der Batterie-Verarbeitungsanlage und verringert die Umweltgefährdung.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte des Kondensierens von Bestandteilen des Elektrolyten durch Abkühlen und/oder Druckerhöhen, sodass ein Elektrolytkondensat entsteht. Beispielsweise wird das Kondensieren an einer Stelle durchgeführt, die bezüglich des Gasflusses zwischen dem Trockner und der Vakuumpumpe liegt. Vom Trockner kommende Gase müssen daher in diesem Fall zunächst einen Kondensator passieren, bevor sie zur Vakuumpumpe gelangen können. Das führt dazu, dass gasförmiger Elektrolyt, der in dem Gas vorliegt, das beim Trocknen entsteht, zumindest zu einem überwiegenden Teil im Kondensator abgeschieden wird, bevor das verbleibende Gas zur Pumpe gelangt. So kann Elektrolyt zurückgewonnen werden. Zudem verringert sich der Gasstrom durch die Vakuumpumpe, was deren Lebensdauer verlängert und deren Energieverbrauch reduziert.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren alternativ den Schritt der Gasreinigung durch Adsorption der flüchtigen organischen Bestandteile an einem Aktivkohlfilter vor oder nach der Verdichtereinheit.

Vorzugsweise umfasst das erfindungsgemäße Verfahren alternativ oder zusätzlich den Schritt eines Reinigens des Gases, das beim Trocknen entsteht, bevor dieses die Vakuumpumpe erreicht. Das kann beispielsweise auch dadurch erfolgen, dass das Gas einen Aktivkohlefilter und/oder einen Filter passiert, in dem mit Fluorwasserstoff reagierende Substanzen enthalten sind, beispielsweise ein Calziumsalz wie Calziumkarbonat oder ein Kaliumsalz wie Kaliumkarbonat.

Vorzugsweise umfasst das erfindungsgemäße Verfahren den Schritt eines Trocknens bei einer Trocken-Temperatur und für eine Trocken-Dauer, die so gewählt sind, dass sich der Binder, der Aktivmaterial der Lithium-Batterie an einen Träger bindet, zumindest zum überwiegenden Teil zersetzt. Günstig ist es, wenn dieser Trocknungsschritt, der auch als Hochtemperatur-Trocknen bezeichnet werden kann, räumlich getrennt von einem ersten, oben beschriebenen Trocknungsschritt durchgeführt wird. Der letztgenannte Trocknungsschritt kann auch als Niedertemperatur-Trocknen bezeichnet werden.

Vorzugsweise wird das Hochtemperatur-Trocknen, bei dem sich der Binder zersetzt, so durchgeführt, dass die entstehenden Zersetzungsgase sich nicht mit den Gasen mischen, die beim Niedrigtemperatur-Trocknen entstehen. Es ist möglich, dass das Hochtemperatur-Trocknen und das Niedertemperatur-Trocknen bei unterschiedlichen Drücken erfolgt. Beispielsweise kann das Hochtemperatur-Trocknen bei Normaldruck erfolgen.

Unter dem Aktivmaterial wird der Stoff verstanden, der beim Betrieb der Batterien elektrochemisch reagiert. Unter dem Träger für das Aktivmaterial wird insbesondere eine Trägerfolie verstanden, auf der das Aktivmaterial in Form von Partikeln aufgebracht ist. Beispielsweise handelt es sich bei der Trägerfolie um eine Folie aus Aluminium oder eine Aluminiumlegierung. Der Binder ist der Stoff, der das Aktivmaterial mit dem Träger verbindet, beispielsweise enthält der Binder Polyvinylidenfluorid.

Günstig ist es, wenn beim Zerkleinern der Batterien flüssiger Stickstoff zugege-

ben wird. Das kühlt die Batterien, die Zerkleinerungsmaschine und das Zerkleinerungsgut und verdrängt zudem Sauerstoff und Wasserdampf aus der Atmosphäre.

Günstig ist es, wenn das Zerkleinern bei dem Wasserdampf-Partialdruck von höchstens 20 Pa und/oder einem Sauerstoff-Partial von höchstens 40 hPa, insbesondere höchstens 15 hPa erfolgt.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte eines Entnehmens des Zerkleinerungsguts aus dem Transportbehälter, eines Abtrennens von Hartteilen und/oder eines Separierens von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfaktion entstehen, und eines getrennten Verpackens von Aktivmaterialfraktion und Trägerfraktion in jeweilige Transportbehältnisse. Günstig ist es, wenn diese Transportbehältnisse luftdicht ausgebildet sind. Durch das Trennen von Aktivmaterialfraktion und Trägerfraktion ist ein Transport in der Regel ohne Genehmigungen möglich. Ein weiterer Vorteil ist, dass derartig getrennte Fraktionen nur eine geringe Gefährdung darstellen.

Das Entnehmen des Zerkleinerungsguts aus dem Transportbehälter erfolgt vorzugsweise unter Vakuum und/oder unter Schutzgas.

Es ist möglich, nicht aber notwendig, dass das Zerkleinerungsgut unter Vakuum in den Transportbehälter abgefüllt wird. Günstig ist es, wenn der Transportbehälter ein Vakuumbehälter ist, insbesondere ein evakuierter Vakuumbehälter, sodass im Transportbehälter nach dem Verschließen ein Unterdruck oder Vakuum herrscht. Alternativ kann der Transportbehälter mit einem Inertgas gefüllt werden.

In einer bevorzugten Batterie-Verarbeitungsanlage sind die Trennungseinheit und die Trockenvorrichtung in einem gemeinsamen Standard-Container angeordnet. Das hat den Vorteil, dass die Batterie-Verarbeitungsanlage besonders

einfach transportiert werden kann.

Die Trockenvorrichtung ist eingerichtet zum Trocknen des Zerkleinerungsguts für eine so lange Zeit, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist. Wird die Trockenvorrichtung im Chargenbetrieb betrieben, was eine mögliche Ausführungsform darstellt, so wird das Trocknen beispielsweise für eine vorgegebene Zeitspanne durchgeführt. Alternativ oder zusätzlich wird der Gehalt an organischen Substanzen, beispielsweise an organischen Carbonaten, in der Atmosphäre in der Trockenvorrichtung kontinuierlich gemessen und das Trocknen beendet, wenn eine vorgegebene Schwellen-Konzentration unterschritten wird.

Gemäß einer bevorzugten Ausführungsform umfasst die Batterie-Verarbeitungsanlage, insbesondere die Vakuumanlage, einen Kondensator, der eingerichtet ist zum Kondensieren von organischen Bestandteilen der Atmosphäre im Trockner, insbesondere von organischen Carbonaten, beispielsweise von Dimethylcarbonat, Ethylmethylcarbonat und/oder Ethylencarbonat. Vorzugsweise ist der Kondensator in Materialflussrichtung vor einer Vakuumpumpe angeordnet, mittels der der Trockner evakuiert wird. Günstig ist es, wenn der Kondensator gekühlt ist, vorzugsweise auf eine Temperatur von höchstens 90°C, vorzugsweise höchstens 80°C, insbesondere höchstens 70°C. Um den Kühlaufwand klein zu halten wird der Kondensator, sofern er gekühlt wird, auf eine Temperatur von zumindest -10°C, insbesondere zumindest 10 °C, gekühlt.

Günstig ist es, wenn die Trockenvorrichtung ein Rührwerkzeug aufweist, beispielsweise einen Ankerrührer oder einen Stabrührer, dessen Rührstäbe quer zu einer Rührwelle angebracht sein können. Alternativ oder zusätzlich ist das Rührwerkzeug ein externes Rührwerkzeug, das den Trockner als Ganzes bewegt.

Vorzugsweise besitzt die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung zum Erzeugen eines Vakuums in der Trockenvorrichtung verbunden ist. Besonders günstig ist es, wenn auch die Vakuumanlage im Standard-Container angeordnet ist. Bei dem Standard-Container handelt es sich

vorzugsweise um einen Container nach ISO-Norm 668, vorzugweise um einen 40-Fuß-Container oder einen 20-Fuß-Container.

Beispielweise umfasst die Vakuumanlage eine Strahlpumpe, bei der ein Strahlmedium zum Erzeugen des Unterdrucks verwendet wird.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionabtrennvorrichtung , eine Separiervorrichtung, insbesondere eine Klassiervorrichtung, zum Separieren von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht, und eine zweite Abfüllvorrichtung zum getrennten Abfüllen von Aktivmaterialfraktion und Trägerfraktion. Günstig ist es, wenn diese Abfüllvorrichtung zum Abfüllen unter Unterdruck und/oder unter Inertgas ausgebildet ist.

Unter einer Hartmetallabtrennvorrichtung wird insbesondere eine Vorrichtung zur Abtrennung von Fragmenten peripherer Bestandteile des Betriebssystems, der Batteriezellhülle und der Strom-Kontakte verstanden. Beispielsweise umfasst die Hartmetallabtrennvorrichtung eine Magnetseparationsvorrichtung und/oder einen Sichter, insbesondere einen Querstromsichter und/oder einen Zickzack-Sichter. Unter der Separiervorrichtung wird insbesondere eine Vorrichtung zum zur Abtrennen der Separatorfolie verstanden.

Die Leichtfraktionabtrennvorrichtung umfasst vorzugsweise einen Zickzack-Sichter und/oder einen Lufttrenner, wobei es günstig ist, wenn die Luft im Kreislauf geführt wird. Das reduziert die Staubexposition der Umwelt mit gesundheitsschädlichem Staub.

Vorzugsweise sind die zweite Abfüllvorrichtung und die Separiervorrichtungen in einem gemeinsamen Standard-Container angeordnet, beispielsweise im oben beschrieben ersten Standard-Container, oder einem zweiten Standard-Container. Günstig ist es, wenn der Container staubdicht gekapselt ist.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Schleuse zwischen der Zerkleinerungseinheit und der Inaktivierungsvorrichtung, insbesondere der Trocknungsvorrichtung. Beispielsweise handelt es sich um eine Zellenradschleuse. Durch die Schleuse wird der Sauerstoffeintrag in die Inaktivierungsvorrichtung, insbesondere die Trocknungsvorrichtung, vermindert.

Im Folgenden wird die Erfindung anhand der beigefügten Zeichnungen näher erläutert. Dabei zeigt

Figur 1 ein Ablaufdiagramm eines erfindungsgemäßen Verfahrens,

Figur 2 einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage und

Figur 3 einen Querschnitt durch weitere, optionale Bestandteile einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Figur 1 zeigt das Ablaufdiagramm eines erfindungsgemäßen Verfahrens. Zunächst werden Batterien 10.1, 10.2, ..., insbesondere Batteriesysteme bestehend aus mehreren Batteriemodulen oder Batteriestacks, welche wiederum aus mehreren Batteriezellen bestehen, in einer Entladeeinheit 12 entladen. Danach folgt an einer Demontagestation 14 die Demontage der Batterien 10, wenn diese erforderlich ist, weil die Batteriesysteme ansonsten aus geometrischen oder gravimetrischen Gründen nicht in die Zerkleinerungseinheit befördert werden können. Dazu werden die Batteriesysteme geöffnet und soweit demontiert, dass die Module/Stacks einzeln entfernt werden können. Gegebenenfalls können auch die Zellen von der Ansteuerelektronik getrennt werden. Die entstehenden Untereinheiten (Module/Stacks) und/oder Zellen 16.1, 16.2, ... werden einer Zerkleinerungseinheit 18 zugeführt, die beispielsweise eine Rotorschere mit einem Rotor und einen mit Statoren oder mehreren Rotoren, oder eine Schneidmühle mit einem Rotor und mehreren Rotoren umfasst.

Die Zerkleinerungseinheit 18 zerkleinert die Batterien 10 unter Schutzgas 20,

das beispielsweise einer Schutzgasflasche entnommen wird. Alternativ oder zusätzlich kann flüssiger Stickstoff aus einer Flüssigstickstoffquelle 19 zugedüst werden. Bei dem Schutzgas kann es sich beispielsweise um Stickstoff, ein Edelgas, Kohlendioxid, Lachgas oder ein sonstiges, vorzugweise nicht giftiges Gas handeln.

Beim Zerkleinern entsteht Zerkleinerungsgut 24, das einer Inaktivierungsvorrichtung in Form einer Trockenvorrichtung 26 zugeführt wird. Zwischen der Zerkleinerungseinheit 18 und der Trockenvorrichtung 26 ist eine Schleuse 28 angeordnet, die soweit gasdicht ist, dass die Druckvorrichtung 26 in guter Näherung von der Zerkleinerungseinheit 18 gasdicht getrennt ist.

Die Trockenvorrichtung 26 ist mit einer Vakuumanlage 29 verbunden, die eine Vakuumpumpe 30 aufweist und ein Vakuum erzeugt. In der Trockenvorrichtung 26 herrscht ein Druck p_{26} von $p_{26} \approx 100$ hPa, vorzugsweise 50 hPa. Es sei darauf hingewiesen, dass im Rahmen der vorliegenden Beschreibung unter einer Vakuumpumpe insbesondere ganz allgemein eine Vorrichtung verstanden wird, die ein Vakuum erzeugt. Es ist dabei möglich und bevorzugt, nicht aber notwendig, dass die Vakuumpumpe gleichzeitig als Verdichter wirkt, so dass von ihr Gas unter einem Druck abgegeben wird, der größer ist als der Umgebungsdruck.

In dem in Figur 1 gezeigten Fall handelt es sich bei der Vakuumpumpe um einen Verdichter, der Gas 31, das in der Trockenvorrichtung 26 vorliegt, ansaugt und verdichtet. Alternativ oder zusätzlich kann die Vakuumanlage 29 eine Strahlpumpe aufweisen, bei der ein Strahlmedium in Form einer Flüssigkeit mit hoher Geschwindigkeit durch Venturi-Düse geleitet wird. Das Strahlmedium ist alkalisch und hat einen pH-Wert von zumindest pH 13 und ist beispielsweise eine 10%ige Kaliumhydroxidlösung.

Die Vakuumanlage 29 umfasst eine Gasreinigungsvorrichtung 32, die zwischen der Trockenvorrichtung 26 und der Vakuumpumpe 30 angeordnet ist und die im

vorliegenden Fall einen Kondensator 34 und/oder einen Aktivkohlefilter 36 umfasst. Der Kondensator wird bei einer Temperatur von -10 °C betrieben, so dass Kohlensäuredimethylester und Kohlensäureethylmethylester kondensieren und in einen Kondensatbehälter 38 abgegeben werden können. Zudem wird etwaig vorhandenes Wasser ausgefroren. Ein Steuerventil 40 ist ausgebildet zum Öffnen, wenn der Druck p_{26} zu groß wird und zum Zufahren, wenn der Druck p_{26} zu klein wird, das heißt, dass ein vorgegebener Schwellenwert unterschritten wird.

Vorzugsweise ist das Trocknungsgut während der Trocknung bewegt. Dies kann beispielsweise durch Rühren mit einem Rührwerkzeug 41, wie einem Ankerrührer oder einem Stabrührer mit senkrecht zur Rührwelle angebrachten Stäben passieren. Alternativ durch einen bewegten Trocknungsbehälter.

Durch das Trocknen des Zerkleinerungsguts entsteht inaktiviertes Zerkleinerungsgut 42, das einer Abfüllvorrichtung 44 zugeführt wird. Dort wird das inaktivierte Zerkleinerungsgut 42 unter Vakuum und/oder unter Schutzgas in einen Transportbehälter 46 abgefüllt. Der Transportbehälter 46 ist vorzugsweise gasdicht. Es ist möglich, nicht aber notwendig, dass der Transportbehälter 46 vor dem Abtransport mit Inertgas aufgefüllt wird, so dass er unter Normaldruck steht. Alternativ ist es auch möglich, dass der Transportbehälter unter Vakuum verschlossen und transportiert wird. Es ist möglich, dass alternativ zum Transportbehälter eine vakuumverschweißte Folie, beispielsweise eine Aluminiumverbundfolie, gewählt wird.

Über eine Spülleitung 48 wird der Zerkleinerungseinheit 18 Schutzgas 20 von der Vakuumpumpe 30 zugeführt. Arbeitet die Vakuumpumpe 30 wie im vorliegenden Fall, der eine bevorzugte Ausführungsform darstellt, zudem als Kompressor, kann das Schutzgas auf eine Druckgasflasche 50 gezogen werden. Alternativ oder zusätzlich kann das Schutzgas 20, gegebenenfalls nach zusätzlicher Reinigung, in die Umgebung abgegeben werden.

Figur 2 zeigt schematisch einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage 52, die einen Standard-Container 54 aufweist, in dem

die Zerkleinerungseinheit 18, die Trockenvorrichtung 26 und die Abfüllvorrichtung 44 angeordnet sind. Hinter der Zerkleinerungseinheit 18 ist ein erster gasdichter Förderer 56 angeordnet, der beispielsweise eine Förderschnecken oder einen Rohrkettenförderer umfasst. Der erste Förderer 56 fördert das Zerkleinerungsgut 24 zur Trockenvorrichtung 26, die mit der in Figur 2 nicht sichtbaren Vakuum-Erzeugungsvorrichtung verbunden ist. In Materialstromrichtung hinter der Trockenvorrichtung 26 ist ein zweiter Förderer 58 angeordnet, der vorzugsweise ebenfalls gasdicht ausgebildet ist und eine Förderschnecke oder einen Rohrkettenförderer enthalten kann. Der zweite Förderer fördert das inaktivierte Zerkleinerungsgut 42 zur Abfüllvorrichtung 44.

Figur 3 zeigt optionale, in der vorliegenden Ausführungsform vorhandene Einheiten der erfindungsgemäßen Batterie-Verarbeitungsanlage 52, die einen Aufschlusszerkleinerer 60 sowie einen Sichter 62 umfasst. Der Aufschlusszerkleinerer 60 enthält eine Transportbehälter-Entleervorrichtung 64, mittels der aus dem Transportbehälter 46 inaktiviertes Zerkleinerungsgut 42 entnommen werden kann. Der Aufschlusszerkleinerer 60 erzeugt Aufschlussmaterial 66, das dem Sichter 62 zugeführt wird. Es kann sich bei dem Sichter beispielsweise um einen Zickzack-Sichter handeln.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage 52 einen Zerkleinerer, der vorzugsweise im Materialfluss vor der Klassiervorrichtung 74 steht und ein schnell laufendes Zerkleinerungswerkzeug enthält, wobei eine Rotorumfangsgeschwindigkeit größer ist als 1 m/s, vorzugsweise größer als 10 m/s. Dieser Zerkleinerer zerkleinert das Zerkleinerungsmaterial und beansprucht es mechanisch so, dass sich die elektrochemisch aktive Beschichtung mindestens teilweise vom Träger löst. Das Vorhandensein eines solchen Zerkleinerer ist ein allgemeines bevorzugtes Merkmal einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Im Sichter entstehen eine Leichtfraktion mit Separatorfolie und feinem Beschichtungsmaterial und eine Schwergutfraktion mit Trägerfolien (Aluminium- und Kupfer) mit größerem leicht anhaftender Beschichtung. Beide Fraktionen werden auf

Duesenfeld Ex. 2014-477

jeweils ein Sieb zur weiteren Trennung in Beschichtung und Separatorfolie beziehungsweise Beschichtung und Metallfolien gegeben. Die entstehenden Fraktionen werden getrennt weiter verarbeitet.

Das Aufschlussmaterial 66 wird dem Sichter 62 mittels einem dritten Förderer 68 zugeführt. Ein vierter Förderer 70 führt gesichtetes Material 72, insbesondere das Material der Leichtfraktion, das den Sichter 62 verlässt, einer Klassiervorrichtung 74 zu. Die Klassiervorrichtung 74 umfasst vorzugsweise ein Luftstrahlsieb, das gleichzeitig als Trennvorrichtung zum Trennen des Aktivmaterials von dem Träger fungiert. Durch das Trennen entsteht eine Aktivmaterialfraktion 76, die in ein Transportbehältnis 78 abgefüllt wird.

Es entsteht zudem eine Trägerfraktion 80, die in der vorliegenden Ausführungsform mit einem fünften Förderer 82 einer Abfülleinheit 84 zugeführt wird, die die Trägerfraktion 80 in einen Behälter 86 füllt. Die Abfülleinheit 84 ist zusammen mit einer zweiten Abfülleinheit 88 Teil einer zweiten Abfüllvorrichtung.

Bezugsleistenliste

10 Batterie
12 Entladeeinheit
14 Demotagesstation
16 Zelle
18 Zerkleinerungseinheit
19 Flüssigstickstoffquelle
20 Schutzgas
22 Schutzgasflasche
24 Zerkleinerungsgut
26 Trockenvorrichtung
28 Schleuse
29 Vakuumanlage
30 Vakuumpumpe
31 Gas
32 Gasreinigungsvorrichtung
34 Kondensator
36 Aktivkohlefilter
38 Kondensatbehälter
40 Steuerventil
41 Rührwerkzeug
42 inaktives Zerkleinerungsgut
44 Abfüllvorrichtung
46 Transportbehälter
48 Spülleitung
50 Druckgasflasche
52 Batterie-Verarbeitungsanlage
54 Standard-Container
56 erster Förderer
58 zweiter Förderer
60 Aufschlusszerkleinerer
62 Sichter
64 Transportbehälter-Entleervorrichtung
66 Aufschlussmaterial
68 dritter Förderer
70 vierter Förderer
72 gesichtetes Material
74 Klassiervorrichtung
76 Aktivmaterialfraktion
78 Transportbehältnis
80 Trägerfraktion
82 fünfter Förderer
84 Abfülleinheit
86 Behälter
88 zweite Abfülleinheit
p Druck

Patentansprüche.

1. Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten:
   (a) Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,
   (b) Inaktivieren des Zerkleinerungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und
   (c) Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehälter (46),
   dadurch gekennzeichnet, dass
   (d) das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass das Zerkleinerungsgut (24) solange getrocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

3. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Trocknen unter Vakuum erfolgt.

4. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass
   das Vakuum mittels einer Strahlpumpe erzeugt wird und
   ein Strahlmedium der Strahlpumpe
      einen pH-Wert von zumindest 8, insbesondere 9, hat und/oder
      eine Fluor fällende Substanz enthält.

5. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch die Schritte:
   (e) Entnehmen des Zerkleinerungsguts (24) aus dem Transportbehälter (46),
   (f) Abtrennen von Hartteilen und/oder
   Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion (80) entsteht, und
   (g) getrenntes Verpacken von Aktivmaterialfraktion (76) und Trägerfraktion (80) in Transportbehältnisse (78).

6. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Trocknen des Zerkleinerungsguts erst beendet wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut mehr bilden kann und/oder wenn das Zerkleinerungsgut so trocken ist, dass sich im Transportbehälter oder während der Weiterverarbeitung ein entzündliches oder explosives Gasgemisch ergeben kann.

7. Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere Lithium-Batterien, mit:
   (a) einer Zerkleinerungseinheit (18) zum Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,
   (b) einer Inaktivierungsvorrichtung (26) zum Inaktivieren des Zerkleinerungsguts (24) und
   (c) einer Abfüllvorrichtung (44) zum Abfüllen des inaktivierten Zerkleinerungsguts (24) in einen Transportbehälter (46),
   dadurch gekennzeichnet, dass
   (d) die Inaktivierungsvorrichtung eine Trockenvorrichtung (26) umfasst und
   (e) die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung (26) zum Erzeugen eines Vakuums in der Trockenvorrichtung (26) verbunden ist, aufweist.

8. Batterie-Verarbeitungsanlage nach Anspruch 7, dadurch gekennzeichnet, dass die Trockenvorrichtung (26) eingerichtet ist zum Trocknen des Zerkleinerungsguts (24) solange, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

9. Batterie-Verarbeitungsanlage nach Anspruch 7 oder 8, dadurch gekennzeichnet, dass
die Zerkleinerungseinheit und die Trockenvorrichtung (26) in einem Standard-Container (54) angeordnet sind.

10. Batterie-Verarbeitungsanlage nach einem der Ansprüche 7 bis 9, gekennzeichnet durch eine Vakuumanlage, die mit der Trockenvorrichtung (26) zum Erzeugen eines Vakuums in der Trockenvorrichtung (26) verbunden ist.

11. Batterie-Verarbeitungsanlage nach Anspruch 10, dadurch gekennzeichnet, dass
- die Vakuumanlage (29) eine Strahlpumpe mit einem Strahlmedium umfasst,
- das Strahlmedium im Kreis geführt ist und
- das Strahlmedium eine mit Fluorwasserstoff reagierende Substanz enthält.

12. Batterie-Verarbeitungsanlage nach einem der Ansprüche 7 bis 11, gekennzeichnet durch
- eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionsabtrennvorrichtung,
- eine Separiervorrichtung, insbesondere eine Klassiervorrichtung (74), zum Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben und/oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion entsteht, insbesondere Aluminium- und Kupferfolien, und

Ascend Elements EX1002 Page 482

Duesenfeld Ex. 2014-482

- eine zweite Abfüllvorrichtung (44) zum getrennten Abfüllen von Aktivmaterialfraktion (76) und Trägerfraktion (80), insbesondere Aluminium- und Kupferfolien.

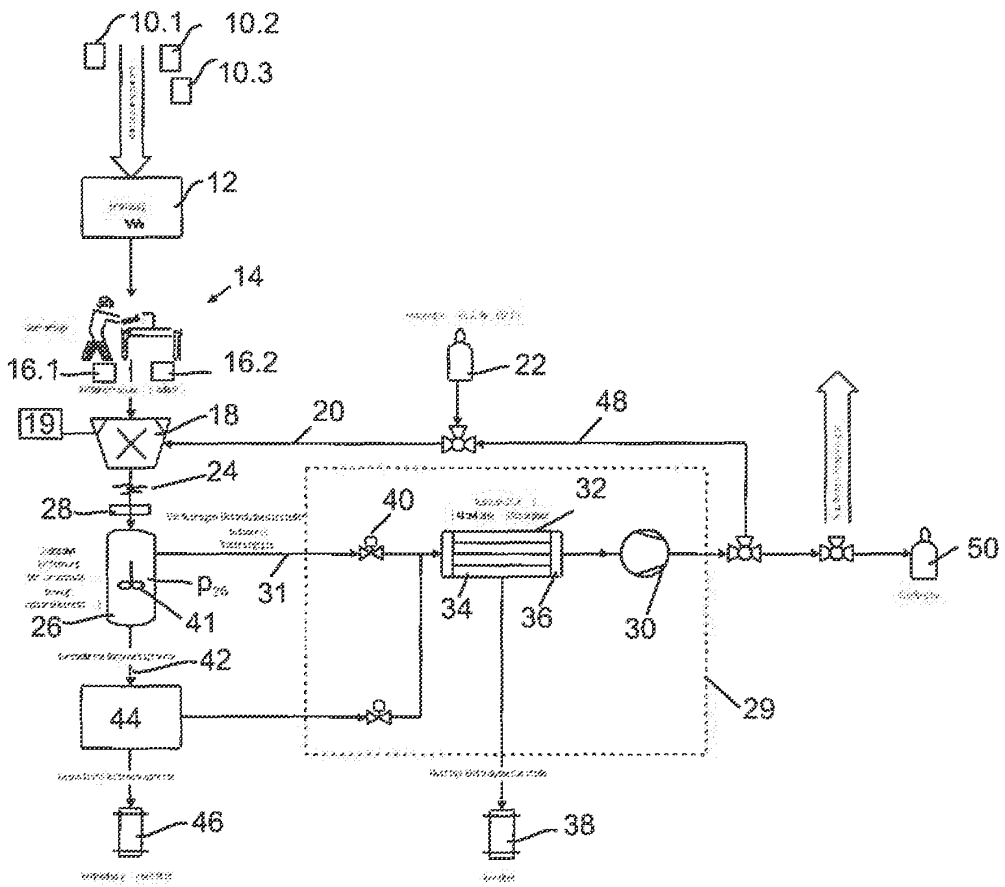
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Fig. 1

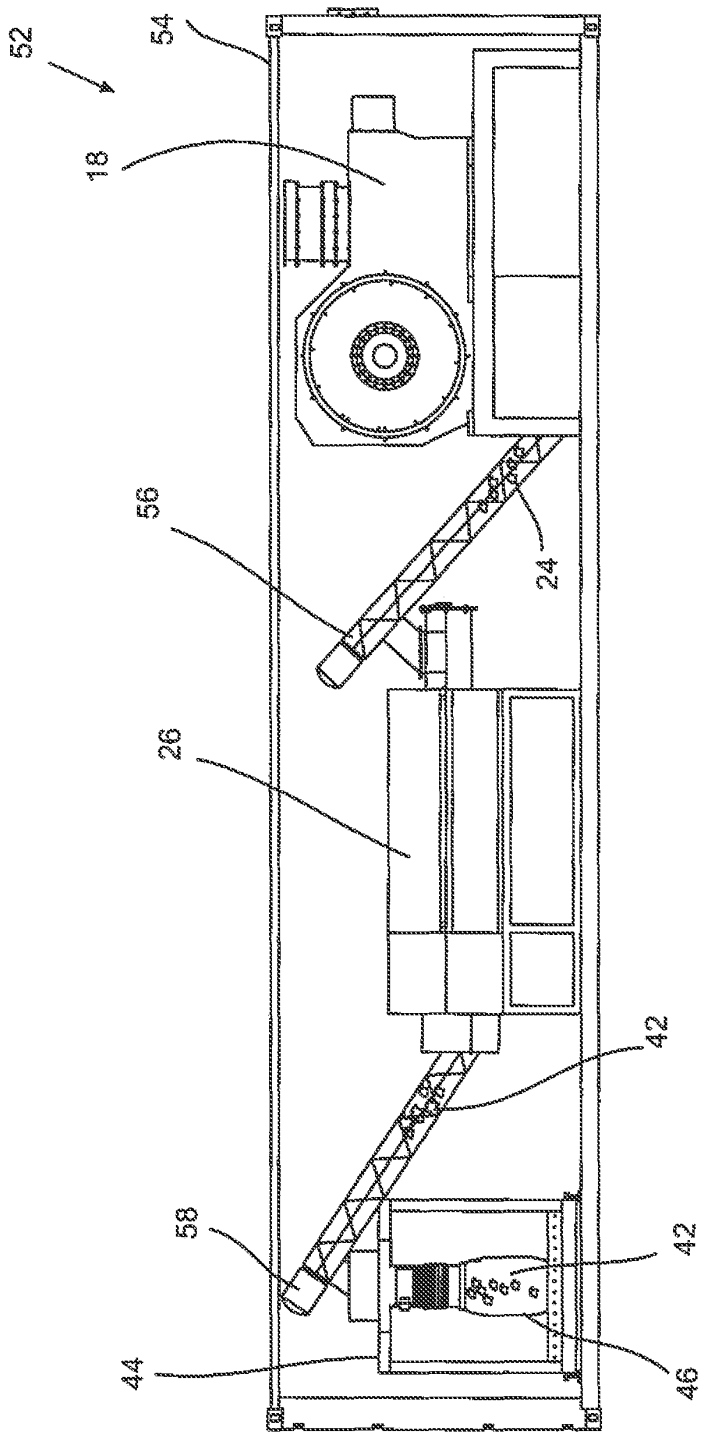

Fig. 2

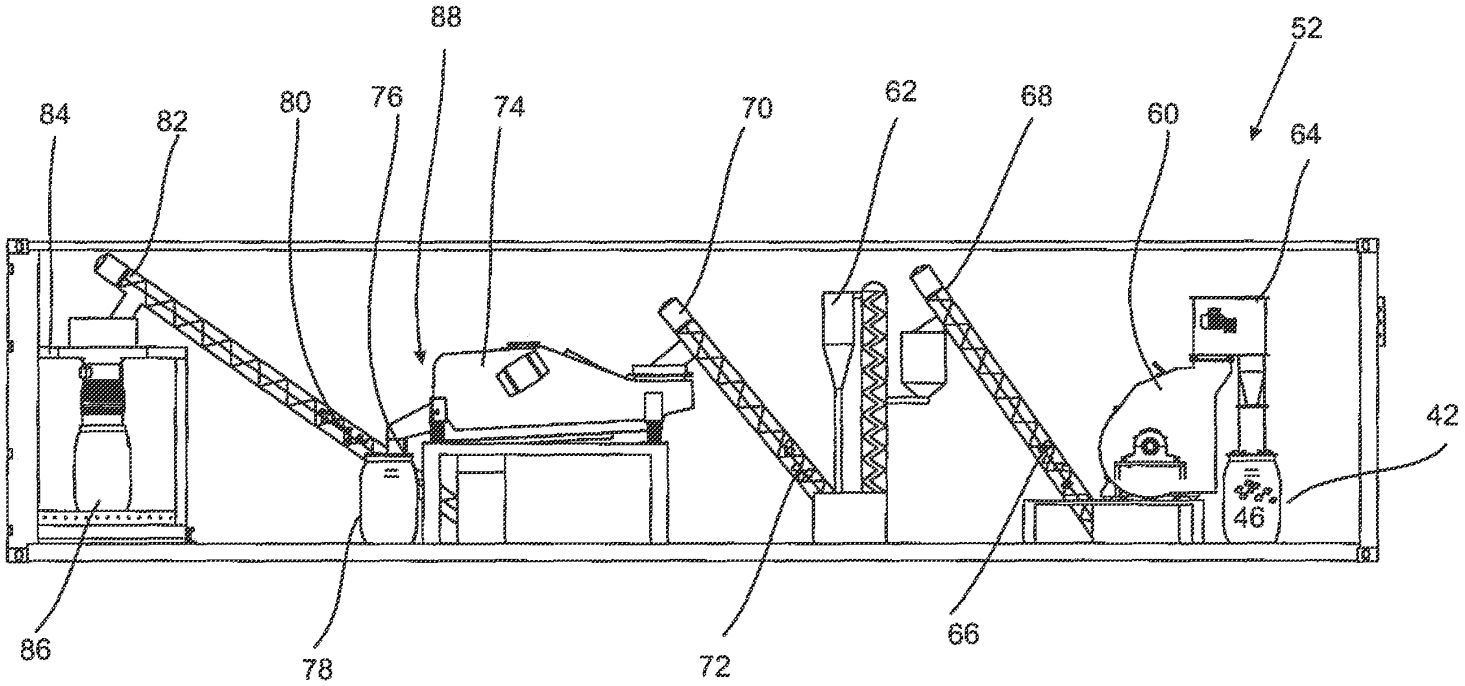

Fig. 3

INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
PCT/EP2016/059526

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
INV. H01M6/52 C22B13/00 H01M10/54
ADD.

Nach der Internationalen Patentklassifikation (IPC) oder nach der nationalen Klassifikation und der IPC

B. RECHERCHIERTE GEBIETE

Recherchierter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)
H01M C22B

Recherchierte, aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen

Während der internationalen Recherche konsultierte elektronische Datenbank (Name der Datenbank und evtl. verwendete Suchbegriffe)
EPO-Internal, WPI Data

C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
X	WO 2010/102377 A1 (ELLIOTT K W) 16. September 2010 (2010-09-16) Absätze [0015] - [0040]; Abbildungen 1, 3 -----	1-12
X	DE 10 2011 110083 A1 (TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA [DE]) 14. Februar 2013 (2013-02-14) in der Anmeldung erwähnt Absätze [0021], [0022], [0034] - [0039]; Abbildung 1 -----	1-3,5-8, 10,12

☐ Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen ☒ Siehe Anhang Patentfamilie

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Datum des Abschlusses der internationalen Recherche	Absendedatum des internationalen Recherchenberichts
5. Juli 2016	13/07/2016

Name und Postanschrift der internationalen Recherchenbehörde	Bevollmächtigter Bediensteter
Europäisches Patentamt, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Hofer, Astrid

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Formblatt PCT/ISA/210 (Blatt 2) (April 2005)

INTERNATIONALER RECHERCHENBERICHT

Angaben zu Veröffentlichungen, die zur selben Patentfamilie gehören

Internationales Aktenzeichen
PCT/EP2016/059526

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Publication No.: WO 2016/174156
PCT International Application No.: PCT/EP2016/059526

VERIFICATION OF A TRANSLATION

I, Megan Skinner B.A.

Freelance translator and interpreter for the language combinations German-English and Dutch-English hereby declare that I am knowledgeable in the German language, in which the international application with the above application number was filed. Furthermore, I hereby confirm that the English translation of the enclosed application WO 2016/174156 - is a true and complete translation to the best of my knowledge and belief.

I hereby declare that all the statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. Moreover, I hereby confirm that these statements were made in the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, pursuant to Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the subsequent patent application.

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Method for the treatment of used batteries, in particular rechargable batteries, and battery processing installation

The invention refers to a method for the treatment of used batteries, in particular used lithium batteries, such as lithium ion batteries, with the steps (a) comminuting the batteries such that comminuted material is obtained, (b) inactivating of the comminuted material such that an inactive comminuted material is obtained, and (c) filling a transport container with the inactive comminuted material.

According to a second aspect, the invention refers to a battery processing installation for the treatment of used batteries, in particular for the treatment of used lithium batteries with (a) a comminuting device for comminuting the batteries such that comminuted material is obtained, (b) an inactivation device for inactivating the comminuted material and (c) a filling device for filling a transport container with the inactivated comminuted material.

US 2005/0241943 A1 describes a method for processing used batteries in which the batteries are heated prior to a comminuting step, thereby destroying plastic components in the batteries. The disadvantage of this type of procedure is that the remaining components of the batteries may be contaminated with degradation products of the plastic.

DE 10 2012 024 876 A1 describes a system for transferring transport-critical electrolyte cells, in which they are initially comminuted under inert gas and then dusted with a deactivation powder so as to prevent the electrochemically active material from spontaneously combusting. The disadvantage of this is that the resulting material still poses a comparatively high hazard potential and that the dusting power itself poses a risk of explosion and that the formation of a flammable and explosive atmosphere in the transport container cannot be ruled out.

DE 10 2011 110 083 A1 describes a method for recovering active material from

a galvanic cell, in which the galvanic cells are initially mechanically comminuted, then pre-dried and subsequently sifted. Finally, the binder is broken down in an oven. This type of device is very well-suited to the efficient recycling of larger amounts of galvanic elements. However, for partial load operation, the construction of this installation is comparatively complex.

The invention aims to reduce disadvantages of the prior art.

The invention solves the problem by means of a method according to the preamble, in which the inactivation occurs at least also by way of drying the comminuted material. According to a second aspect, the invention solves the problem through a battery processing installation according to the preamble, the activation device of which comprises a drying device.

The advantage of the invention is that the amount of electrolyte that can be obtained from the comminuted material through drying is such that an electrochemical reaction is no longer possible, or only to a negligibly small extent. In addition, no flammable or explosive gas phase forms above the battery fragments, as the organic carbonates of the electrolyte have been removed [from the fragments]. The comminuted material is therefore largely inert and can be transported safely, especially if it is packed under vacuum.

A further advantage is that no additional material has to be added to inactivate the comminuted material. This decreases the complexity of the battery processing, reduces the weight of the inactivated comminuted material and increases the purity in the subsequent separation and recycling steps. In particular, in potential subsequent hydrometallurgical processing steps, a high degree of product purity that does not require the input of foreign ions is advantageous.

In addition, it is advantageous that a comminuted material is obtained that can be transported safely. The organic carbonate content is preferably so small that the formation of a significant amount of fluorophosphates can be ruled out.

Fluorophosphates are often strong neurotoxins, the formation of which must be reliably prevented. Furthermore, due to the low electrolyte content, it is guaranteed that a self-amplifying and intensifying build-up of heat triggered by an electrochemical reaction cannot occur.

Within the scope of the present description, the term drying should be understood particularly to mean the removal of at least one solvent in the conducting salt. In particular, the drying is executed such that dimethyl carbonate and/or ethyl methyl carbonate is removed.

The battery should be understood especially to mean a lithium battery. A lithium battery is a rechargeable battery whose electrochemical reaction involves lithium and/or lithium ions and/or a lithium compound.

A battery processing installation should also be understood particularly to mean a rechargeable battery processing installation for processing rechargeable batteries.

The transport container should also be understood particularly to mean transport packaging. The transport packaging is preferably sealed by way of a vacuum seal. Aluminium composite foil is especially well-suited as transport packaging.

It is beneficial if the drying occurs after the comminuting of the batteries. It is indeed possible and represents a preferred embodiment that the batteries are exposed to a vacuum when in an uncomminuted state such that at least parts of the electrolyte vaporise, wherein the resulting gas either escapes through a safety valve in the rechargeable battery or the battery is destroyed by the pressure difference between the external environment and the internal pressure, enabling a vaporising electrolyte to escape. However, since the electrolyte is predominantly located between tightly wound or stacked and pressed layers of electrodes and separators and in their pores, and it is connected to other components of the batteries, this procedure can be very time-consuming. It is thus often more beneficial and represents a preferred embodiment of the

invention for the batteries to be mechanically comminuted, for example through cutting, cropping, impact, separating and/or compressing. This means that a larger interface is available for the transition of materials into the gas phase.

The drying may occur by way of vacuum drying, contact drying, convection drying and/or infra-red drying. It is favourable if the drying occurs while the comminuted material is being agitated and/or circulated.

Prior to being comminuted, the used batteries are preferably dismantled. This means that larger battery systems are dismantled into their smaller subcomponents, the modules or stacks, or even that the cells which contain the electrochemically active material are separated from the control electronics. The control electronics comprise, for example, semiconductor elements and/or sensors and are responsible for the charge control of the batteries.

According to a preferred embodiment, the drying occurs under vacuum. The size of the vacuum is preferably selected such that the vapour pressure of dimethyl carbonate at 80°C, especially at 70°C, is not reached. It is beneficial if the drying occurs at a maximum pressure of 300 hPa, in particular a maximum of 100 hPa. At such low pressures, considerable parts of most electrolytes vaporise, especially dimethyl carbonate and ethyl methyl carbonate, and do so at temperatures of less than 80°C. The advantage of low temperatures is that the formation of hydrogen fluoride is hindered. Hydrogen fluoride poses a potential risk for the battery processing installation and the surroundings. It is therefore beneficial to prevent the development of hydrogen fluoride.

The drying preferably occurs at a temperature that is lower than a decomposition temperature. The decomposition temperature should be understood particularly to mean the lowest temperature at which at least 80 percent by mass of the binder has decomposed into gaseous components after keeping the comminuted material at this temperature for an hour. The decomposition temperature can be measured by successively increasing the temperature of the comminuted material and recording when a loss of mass

occurs, especially through the build-up of gas due to a decomposition of the binder, and the specified criteria is fulfilled. If necessary, the experiment must be conducted several times, each time using a new sample of comminuted material at an increased temperature.

It is favourable if the drying occurs under an atmosphere in which the partial pressure of the water vapor is lower than 50 Pa, in particular lower than 10 Pa. A low partial pressure of the water vapor leads to a low reaction rate of lithium compounds to lithium hydroxide and thus only to a low build-up of hydrogen. This prevents the formation of flammable hydrogen-oxygen mixtures and contributes to the safety of the installation.

In addition, it is favourable if the partial pressure of oxygen has a maximum value of 10 millibars, especially a maximum value of 5 millibars. This largely inhibits the reaction of oxygen with oxidisable components of the batteries. It is possible to achieve the low partial pressure of oxygen by means of drying at a low pressure. Alternatively or additionally, the drying may occur in an inert gas atmosphere.

A method is preferred in which the drying of the comminuted material is only completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material that has been filled [in the container] and/or when the comminuted material is so dry that a flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing. The property that the drying is completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material that has been filled [in the container] should be understood particularly to mean that, within the space of one week at 50°C and 1013 hPa, no flammable gas mixture forms in a transport container in the form of a 50 litre container that has been half-filled (relative to its volume) with the comminuted material. Pre-tests determine whether the criteria has been fulfilled. If a flammable gas mixture does form, the drying must be conducted for a longer time and/or at a lower pressure. The preliminary tests

are repeated until a drying time and/or drying pressure has been identified at which, in a set of tests of three transport containers, the requirements for the property have been fulfilled for all three transport containers.

The comminuted material is preferably dried until an electrolyte content in the comminuted material is so low that an electrochemical reaction is impossible. In other words, the electrolyte content is lower than a threshold value, the threshold value being selected such that, if this threshold value is not achieved, the cell voltage is reduced to a maximum of one quarter. This threshold value is determined, for example, by defining the cell voltage of a battery in relation to the electrolyte content. Shortly before achieving the threshold value, the cell voltage collapses, i.e. it decreases by at least 75%. If the threshold value is not achieved, the battery contains so little electrolyte that, to a good approximation, an electrochemical reaction is no longer possible.

The comminuted material is preferably dried for so long that a 50kg amount of comminuted material, which is contained in a compacted form in a 50 litre drum, does not experience a build-up of heat, or the build-up of heat is so low that a thermal runaway, i.e. a thermally induced chain reaction, is ruled out for at least two months, and that any build-up of hydrogen is also so low that after two weeks, no excess pressure occurs if a negative pressure of 500 hPa is present to begin with.

It is beneficial if the comminuted material is dried until the electrolyte content of organic components that are volatile at 80°C has a maximum value of 3% by weight, in particular a maximum of 2% by weight, especially preferably a maximum of 1.5% by weight.

The drying is preferably conducted for so long that the accumulated content of organic carbonates from the electrolyte that are volatile at 80°C falls short of 3% by volume in the atmosphere above the comminuted material.

In particular, the drying is conducted until the dimethyl carbonate content is

lower than 4% by volume, especially 3% by volume, and/or the cyclohexylbenzene content is lower than 1% by volume, in particular 0.5% by volume.

The drying preferably occurs immediately after comminution. This should be understood to mean that the time between the beginning of the comminution of the batteries and the point at which at least a part of the resulting comminuted material begins to dry is a maximum of five minutes, especially a maximum of one minute. The rapid drying after comminution means that the mass of material that may potentially experience an electrochemical reaction remains small; the electrochemical reaction time of potential exothermic reactions also remains small. This reduces the risk for the installation and the surroundings.

It is especially favourable if the vacuum is created by means of an injector, i.e. a venturi pump or an ejector-jet pump. Ejector-jet pumps are largely resistant to aggressive gases that are due to be pumped, particularly if an appropriate pump fluid, i.e. pump liquid, is selected. It is beneficial if the pump fluid, which is a liquid, has a pH value of at least 8, in particular of at least 9, for example at least 12. In this case, unwanted components of the gas that is being pumped can decompose or react to become less damaging substances. In this way, for example, dimethyl carbonates and/or ethyl methyl carbonates can be broken down by a saponification reaction. Any hydrogen fluoride contained in the pump fluid can be converted in the alkaline environment into a non-hazardous salt by way of an acid-base reaction.

The pump fluid preferably contains a substance that precipitates fluoride. For example, the pump fluid may contain sodium carbonate or calcium carbonate. The salts that result from the reaction with a fluorine compound, in particular hydrogen fluoride, are preferably separated, in particular filtered or removed by sedimentation. This at least largely prevents hydrogen fluoride or other poisonous fluorine compounds from being emitted into the surroundings.

The drying preferably occurs at a maximum temperature of 80°C: this produces

almost no hydrogen fluoride. This increases the service life of the battery processing installation and reduces the environmental risk.

According to a preferred embodiment, the method comprises the steps of condensing components of the electrolyte by cooling and/or increasing the pressure such that an electrolyte condensate occurs. For example, the condensation is conducted at a point that lies between the dryer and the vacuum pump relative to the flow of gas. In this case, gases coming from the dryer must initially pass through a condenser before reaching the vacuum pump. This causes the gaseous electrolyte in the gas, which is produced during the drying, to be at least largely separated in the condenser before the remaining gas reaches the pump. Electrolyte can be recovered in this way. In addition, the flow of gas through the vacuum pump decreases, which increases the vacuum pump's service life and reduces its energy consumption.

According to a preferred embodiment, the method alternatively comprises the step of purifying the gas through the adsorption of the volatile organic components of an activated carbon filter in front of or behind the compressor unit.

Alternatively or additionally, the method according to the invention preferably comprises the step of purifying the gas produced during the drying before it reaches the vacuum pump. This may also occur, for example, by the gas through passing an activated carbon filter and/or a filter that contains substances which react with hydrogen fluoride, such as a calcium salt like calcium carbonate or a potassium salt such as potassium carbonate.

The method according to the invention preferably comprises the step of drying at a drying temperature and for a drying time that have been selected such that the binder which binds the active material of the lithium battery to a carrier at least largely decomposes. It is favourable if this drying step, which can also be described as high temperature drying, occurs in a separate space from a first drying step, described above. The latter drying step can also be described as

low temperature drying.

The high temperature drying, during which the binder decomposes, is preferably conducted such that the resulting decomposition gases do not mix with the gases resulting from the low temperature drying. It is possible that the high temperature drying and the low temperature drying occur at different pressures. For example, the high temperature drying can be executed at normal pressure.

The active material should be understood to mean the material that reacts electrochemically during operation of the batteries. The carrier for the active material should be understood particularly to mean a carrier foil to which the active material is applied in the form of particles. For example, the carrier foil refers to a foil made of aluminium or an aluminium alloy. The binder is the material which binds the active material with the carrier; for example, the binder contains polyvinylidene fluoride.

It is beneficial if liquid nitrogen is added when comminuting the batteries. This cools the batteries, the comminuting machine and the comminuting material, and also drives oxygen and water vapour out of the atmosphere.

It is beneficial if the comminution occurs when the partial pressure of water vapor is a maximum of 20 Pa and/or the partial pressure of the oxygen is a maximum of 40 hPa, especially a maximum of 15 hPa.

According to a preferred embodiment, the method comprises the steps of removing the comminuted material from the transport container; detaching hard parts and/or separating active material from the carrier, particularly via a second comminuting stage and/or air jet sieving, thereby producing an active material fraction and a carrier fraction; and a separate packing of the active material fraction and carrier fraction in suitable transport containers. It is beneficial if these transport containers are designed to be airtight. By separating an active material fraction and a carrier fraction, transportation generally does not require any permits. An additional advantage is that fractions separated in this way only

pose a small risk.

The removal of the comminuted material from the transport container is preferably conducted under vacuum and/or shielding gas.

It is possible, but not necessary, for the the transport container to be filled with communited material under vacuum. It is beneficial if the transport container is a vacuum container, in particular an evacuated vacuum container, such that a negative pressure or vacuum occurs in the transport container once it has been sealed. Alternatively, the transport container may be filled with an inert gas.

In a preferred battery processing installation, the separation unit and the drying device are arranged in a joint standard container. The advantage of this is that it renders the battery processing installation especially easy to transport.

The drying device is configured to dry the comminuted material until an electrolyte content is so low that an electrochemical reaction is impossible. If the drying device is operated in batch mode, which represents a preferred embodiment, the drying shall be performed, for example, for a pre-determined period of time. Alternatively or additionally, the content of organic substances, such as organic carbonates, in the atmosphere in the drying device is continually measured and the drying stopped once the concentration is lower than a pre-determined threshold concentration.

According to a preferred embodiment, the battery processing installation, in particular the vacuum installation, comprises a condenser that is configured to condense organic components of the atmosphere in the dryer, especially organic carbonates such as dimethyl carbonate, ethyl methyl carbonate and/or ethylene carbonate. The condenser is preferably arranged in the direction of material flow in front of a vacuum pump, by means of which the dryer is evacuated. It is beneficial if the condenser is cooled, preferably to a maximum temperature of 90°C, preferably a maximum of 80°C, especially preferably a

maximum of 70°C. In order to keep the energy required for cooling low, the condenser, insofar as it is cooled, is cooled to a temperature of at least -10°C, in particular at least 10 °C.

It is beneficial if the drying device comprises an agitator, for example an anchor agitator or a rod agitator, whose stirring rods can be arranged transversely to an agitator shaft. Alternatively or additionally, the agitator is an external agitator that moves the dryer as a whole.

The battery processing installation preferably has a vacuum installation that is connected to the drying device for the purpose of generating a vacuum in the drying device. It is especially favourable if the vacuum installation is also arranged in the standard container. The standard container preferably refers to a container that conforms to ISO standard 668, preferably a 40 foot container or a 20 foot container.

For example, the vacuum installation comprises an injector or venturi pump, i.e. a jet pump with a pump liquid that is used to generate the negative pressure.

The battery processing installation preferably has a hard metal detachment device and/or a light fraction separation device; a separation device, especially a classification device, for separating active material from the carrier, in particular by means of a second comminution stage and/or air jet sieving, such that an active material fraction and a carrier fraction occur; and a second filling device for the separate filling of the active material fraction and the carrier fraction. It is beneficial if this filling device is designed for filling under negative pressure and/or inter gas.

A hard metal detachment device should be understood particularly to mean a device for detaching fragments of peripheral components of the operating system, the battery cell casing and the electrical contacts. For example, the hard metal detachment device has a magnet separation device and/or a separator, in particular a cross-flow separator and/or a zigzag separator. The separation

device should be understood particularly to mean a device for detaching the separator foil.

The light fraction separation device preferably has a zigzag separator and/or an air separator, wherein it is favourable if the air is conducted within a circuit. This reduces the exposure of the environment to harmful dust.

The second filling device and the separation devices are preferably arranged in a joint standard container, for example in the first standard container described above or a second standard container. It is beneficial if the container is sealed so as to be dust-tight.

The battery processing installation preferably has an airlock between the comminution unit and the inactivation device, especially the drying device. For example, this refers to a rotary airlock. The airlock reduces the amount of oxygen introduced into the inactivation device, especially the drying device.

In the following, the invention will be explained in more detail by way of the attached drawings. They show

Figure 1 a flow diagram of a method according to the invention,

Figure 2 a cross-section through a battery processing installation according to the invention and

Figure 3 a cross-section through further optional components of a battery processing installation according to the invention.

Figure 1 shows a flow diagram of a method according to the invention. Batteries 10.1, 10.2, ..., in particular battery systems made up of several battery modules or battery stacks, which are in turn made up of several battery cells, are initially discharged in a discharge unit 12. This is followed by the dismantling of the batteries 10 at a dismantling station 14, if this is necessary because the battery

systems cannot otherwise be delivered into the comminution unit for geometric or gravimetric reasons. In order to do this, the battery systems are opened and dismantled to the point at which the modules/stacks can be individually removed. If required, the cells can also be separated from the drive electronics. The resulting sub-units (modules/stacks) and/or cells 16.1, 16.2, ... are fed into a comminution unit 18, which comprises, for example, a rotary shear with a rotor and a stator or several rotors, or a cutting mill with a rotor and several rotors.

The comminution unit 18 comminutes the batteries 10 under shielding gas 20, which is extracted, for example, from a shielding gas cylinder. Alternatively or additionally, liquid nitrogen from a liquid nitrogen source 19 may be may be injected. The shielding gas may refer, for example, to nitrogen, a noble gas, carbon dioxide, nitrous oxide or another gas which is preferably not toxic.

Comminuted material 24 is produced during the comminuting; the material is fed into an inactivation device in the form of a drying device 26. An airlock 28 is arranged between the comminution unit 18 and the drying device 26, the airlock being so gas-tight that the pressure device 26 is - to a good approximation - separated from the comminution unit 18 so as to be gas-tight.

The drying device 26 is connected to a vacuum installation 29 that comprises a vacuum pump 30 and creates a vacuum. A pressure p_{26} from $p_{26} \approx 100$ hPa, preferably 50 hPa, is present in the drying device 26. It should be noted that, within the scope of the present description, the vacuum pump should be understood particularly generally to mean a device that creates a vacuum. It is possible and preferred, but not necessary, for the vacuum pump to simultaneously work as a compressor, such that gas is emitted from it under a pressure that is greater than the ambient pressure.

In the case depicted in figure 1, the vacuum pump is a compressor which sucks in and compresses gas 31 that is present in the drying device 26. Alternatively or additionally, the vacuum installation 29 may have a jet pump which uses a pump liquid in the form of a liquid that is conducted at a high speed through

Venturi nozzles. The pump liquid is alkaline and has a pH value of at least pH 1 and is, for example, a 10% potassium hydroxide solution.

The vacuum installation 29 comprises a gas purification device 32 that is arranged between the drying device 26 and the vacuum pump 30, and which has a condenser 34 and/or an activated carbon filter 36 in the present case. The condenser is operated at a temperature of -10°C so that dimethyl carbonate and ethyl methyl carbonate condense and can be dispensed into a condensate container 38. In addition, any water present is separated by freezing. A control valve 40 is designed to open if the pressure p_{26} becomes too great and to close if the pressure p_{26} becomes too small, i.e. when a pre-determined threshold value is not reached.

The drying material is preferably moved during drying. This may be achieved via agitating with an agitator 41, such as an anchor agitator or a rod agitator with rods arranged perpendicular to the agitator shaft. Alternatively, it can be achieved by way of a drying container that is moved.

The drying of the comminuted material results in inactivated comminuted material 42, which is fed into a filling device 44. A transport container 46 is then filled with the inactivated comminuted material 42 under vacuum and/or shielding gas. The transport container 46 is preferably gas-tight. It is possible, but not necessary, for the transport container 46 to be filled with inert gas prior to transportation such that it is under normal pressure. Alternatively, it is also possible for the transport container to be sealed under vacuum and transported. It is possible that, instead of the transport container, a vacuum-sealed foil is selected, such as an aluminium compound foil.

The comminution unit 18 is fed with shielding gas 20 from the vacuum pump 30 via a flushing line 48. If the vacuum pump 30 also functions as a compressor - as in the present case - which represents a preferred embodiment, the shielding gas can be stored in a pressurised gas cylinder 50. Alternatively or additionally, the shielding gas 20 can be given off into the surroundings, following additional

cleaning if necessary.

Figure 2 schematically depicts a cross-section through a battery processing installation 52 according to the invention, which comprises a standard container 54 in which the comminution unit 18, the drying device 26 and the filling device 44 are arranged. A first gas-tight conveyor 56 is arranged behind the comminution unit 18; the conveyor comprises, for example, a screw conveyor or a tube chain conveyor. The first conveyor 56 delivers the comminuted material 24 to the drying device 26, which is connected to the vacuum generation device, not depicted in figure 2. A second conveyor 58 is arranged behind the drying device 26 in the direction of material flow; preferably, the conveyor is also designed to be gas-tight and may include a screw conveyor or a tube chain conveyor. The second conveyor delivers the inactivated comminuted material 42 to the filling device 44.

Figure 3 depicts optional units - available in the present embodiment - of the battery processing installation 52 according to the invention which comprise a breakdown comminutor 60, as well as a separator 62. The breakdown comminutor 60 contains a transport container draining device 64, by means of which inactivated comminuted material 42 can be removed from the transport container 46. The breakdown comminutor 60 produces breakdown material 66, which is fed into the separator 62. The separator may refer, for example, to a zigzag separator.

The battery processing installation 52 preferably comprises a comminutor, which is preferably situated in the material flow in front of the classification device 74 and includes a rapid comminution tool, wherein a peripheral speed of the rotor is greater than 1 m/s, preferably greater than 10 m/s. This comminutor comminutes the comminuted material and subjects it to such mechanical stress that the electrochemically active coating at least partially detaches from the carrier. The presence of such a comminutor is a generally preferred feature of a battery processing installation according to the invention.

A light fraction with a separator foil and fine coating material, and a heavy material fraction with carrier foils (aluminium and copper) with bigger, weakly adhering coating occur in the separator. Both fractions are each placed on a sieve for further separation into coating and separator foil, or coating and metal foil. The further processing of the resulting fractions is conducted separately.

The breakdown material 66 is fed to the separator 62 by means of a third conveyor 68. A fourth conveyor 70 guides sifted material 72, in particular the material of the light fraction that leaves the separator 62, into a classification device 74. The classification device 74 preferably has an air jet sieve, which simultaneously functions as a separation device for separating the active material from the carrier. The separation results in an active material fraction 76, with which a transport container 78 is filled.

In addition, a carrier fraction 80 is produced, which - in the present embodiment - is fed into a filling unit 84 using a fifth conveyor 82; the filling unit fills a container 86 with the carrier fraction 80. The filling unit 84 comes together with a second filling unit 88 to form part of a second filling device.

Reference list

10 battery
12 discharge unit
14 dismantling station
16 cell
18 comminution unit
19 liquid nitrogen source
20 shielding gas
22 shielding gas cylinder
24 comminuted material
26 drying device
28 airlock
29 vacuum installation
30 vacuum pump
31 gas
32 gas purification device
34 condenser
36 activated charcoal filter
38 condensate container
40 control valve
41 agitator
42 inactive comminuted material
44 filling device
46 transport container
48 flushing line
50 pressurised gas cylinder
52 battery processing installation
54 standard container
56 first conveyor
58 second conveyor
60 breakdown comminutor
62 separator
64 transport container draining device
66 breakdown material
68 third conveyor
70 fourth conveyor
72 sifted material
74 classification device
76 active material fraction
78 transport container
80 carrier fraction
82 fifth conveyor
84 filling unit
86 container
88 second filling unit
p pressure

Patent claims:

1. Method for the treatment of used batteries (10), in particular lithium batteries, containing the steps:
   (a) comminuting the batteries (10) such that comminuted material (24) is obtained,
   (b) inactivating the comminuted material (24) such that an inactivated comminuted material (42) is obtained, and
   (c) filling a transport container (46) with the inactivated comminuted material (42),

   characterised by the fact that
   (d) the inactivation is performed by drying the comminuted material (24).

2. Method according to claim 1, characterised by the fact that the comminuted material (24) is dried until an electrolyte content is so low that an electrochemical reaction is impossible.

3. Method according to one of the above claims, characterised by the fact that the drying occurs under vacuum.

4. Method according to one of the above claims, characterised by the fact that the vacuum is created by means of a jet pump and
   a pump liquid of the jet pump
   has a pH value of at least 8, especially 9, and/or
   contains a substance that precipitates fluoride.

5. Method according to one of the above claims, characterised by the steps:
   (e) removal of the comminuted material (24) from the transport container (46),
   (f) detachment of hard parts and/or separation of active material from the carrier, particularly through air jet sieving or a second comminution stage such that an active material fraction (76) and a carrier fraction (80) are obtained, and
   (g) separate packing of the active material fraction (76) and carrier fraction (80) in transport containers (78).

6. Method according to one of the above claims, characterised by the fact that the drying of the comminuted material is only completed if, after the completion of the drying process, no flammable or explosive gas mixture can form above the comminuted material that has been filled in the container and/or if the comminuted material is so dry that no flammable or explosive gas mixture can emerge in the transport container or during the subsequent processing.

7. Battery processing installation for the treatment of used batteries, in particular lithium batteries, with:
   (a) a comminution unit (18) for comminuting the batteries (10) such that comminuted material (24) is obtained,
   (b) an inactivation device (26) for inactivating the comminuted material (24) and
   (c) a filling device (44) for filling a transport container (46) with the inactivated comminuted material (24),

   characterised by the fact that
   (d) the inactivation device comprises a drying device (26) and

(e) the battery processing installation has a vacuum installation that is connected to the drying device (26) for the purpose of generating a vacuum in the drying device (26).

8. Battery processing installation according to claim 7, characterised by the fact that the drying device (26) is configured to dry the comminuted material (24) until an electrolyte content is so low that an electrochemical reaction is impossible.

9. Battery processing installation according to claim 7 or 8, characterised by the fact that
the comminution unit and the drying device (26) are arranged in a standard container (54).

10. Battery processing installation according to one of the claims 7 to 9, characterised by a vacuum installation that is connected to the drying device (26) for the purpose of generating a vacuum in the drying device (26).

11. Battery processing installation according to claim 10, characterised by the fact that
- the vacuum installation (29) has a jet pump with a pump liquid,
- the pump liquid is conducted within a circuit and
- the pump liquid contains a substance that reacts with hydrogen fluoride.

12. Battery processing installation according to one of the claims 7 to 11, characterised by
- a hard metal detachment device and/or a light fraction detachment device,
- a separation device, especially a classification device (74), for separating active material from the carrier, in particular by means of air jet sieving and/or a second comminution stage such that an active material fraction

(76) and a carrier fraction occur, in particular aluminium and copper foils, and

- a second filling device (44) for the separate filling of the active material fraction (76) and the carrier fraction (80), in particular aluminium and copper foils.

Abstract

The invention relates to a method for the treatment of used batteries (10), in particular lithium batteries, containing the steps: comminuting the batteries (10) such that comminuted material (24) is obtained, inactivating the comminuted material (24) such that inactivated comminuted material (42) is obtained, and filling a transport container (46) with the inactivated comminuted material (42). It is provided according to the invention that the inactivation is performed by drying the comminuted material (24) and the comminuted material (24) is dried until an electrolyte content is so low that an electrochemical reaction is not possible.

Figure 1

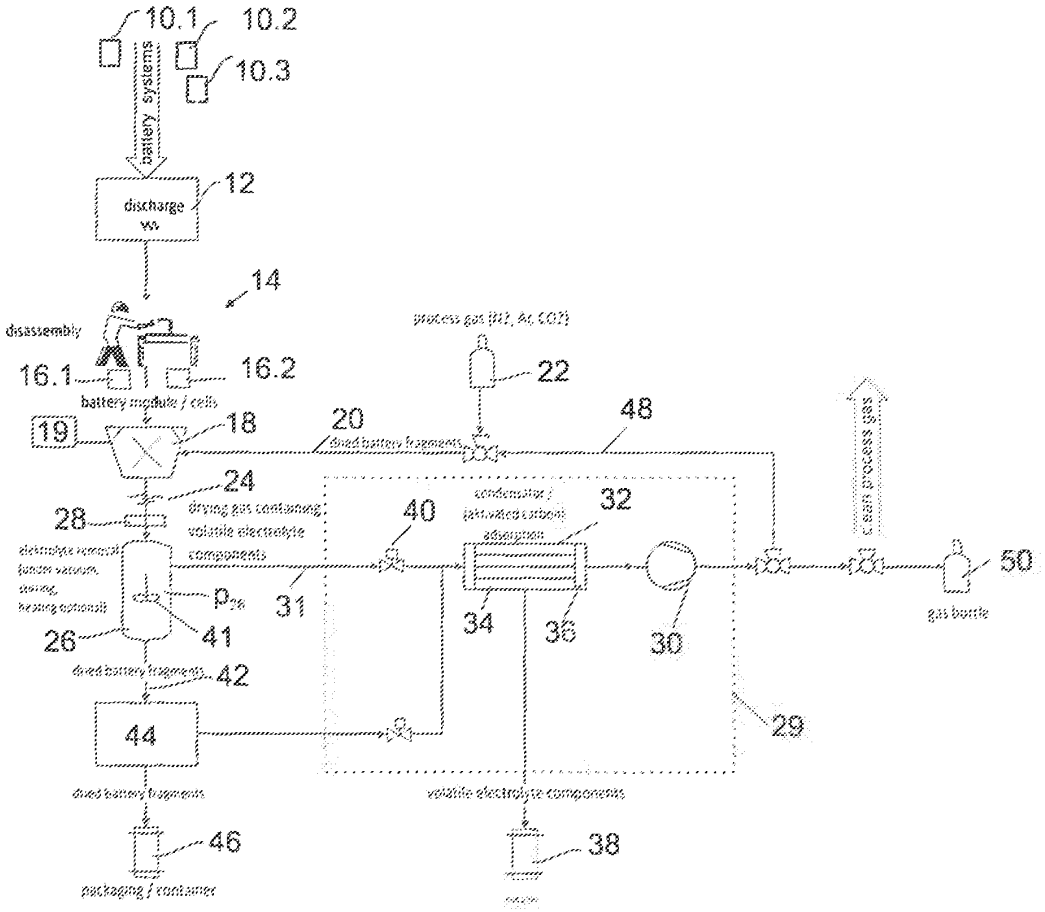
10.1
10.2
10.3
battery systems
discharge vs
12
14
disassembly
16.1
16.2
battery module / cells
19
18
20
dried battery fragments
process gas (N2, Ar, CO2)
22
48
clean process gas
24
28
drying gas containing volatile electrolyte components
40
condensator / (activated carbon) adsorption
32
50
gas bottle
31
34
36
30
26
41
dried battery fragments
42
44
29
dried battery fragments
volatile electrolyte components
46
packaging / container
38

Fig. 1

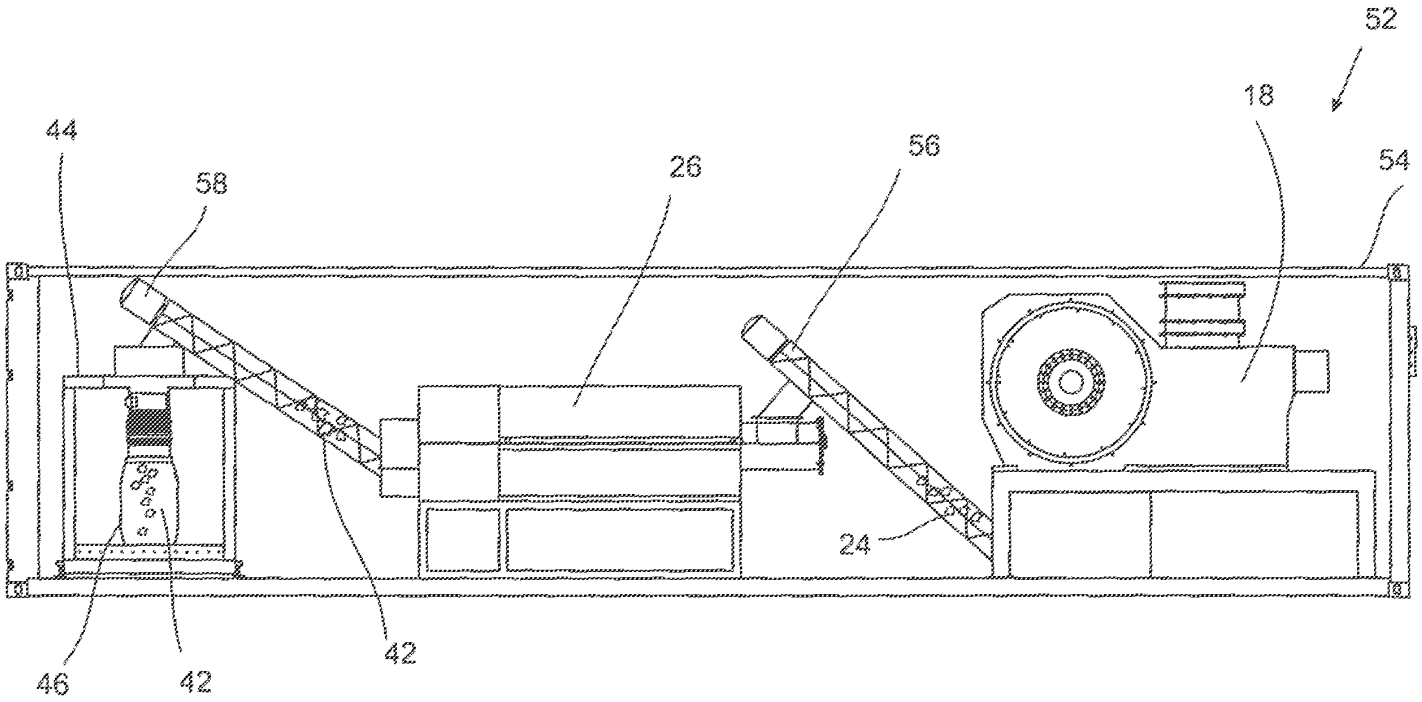

Fig. 2

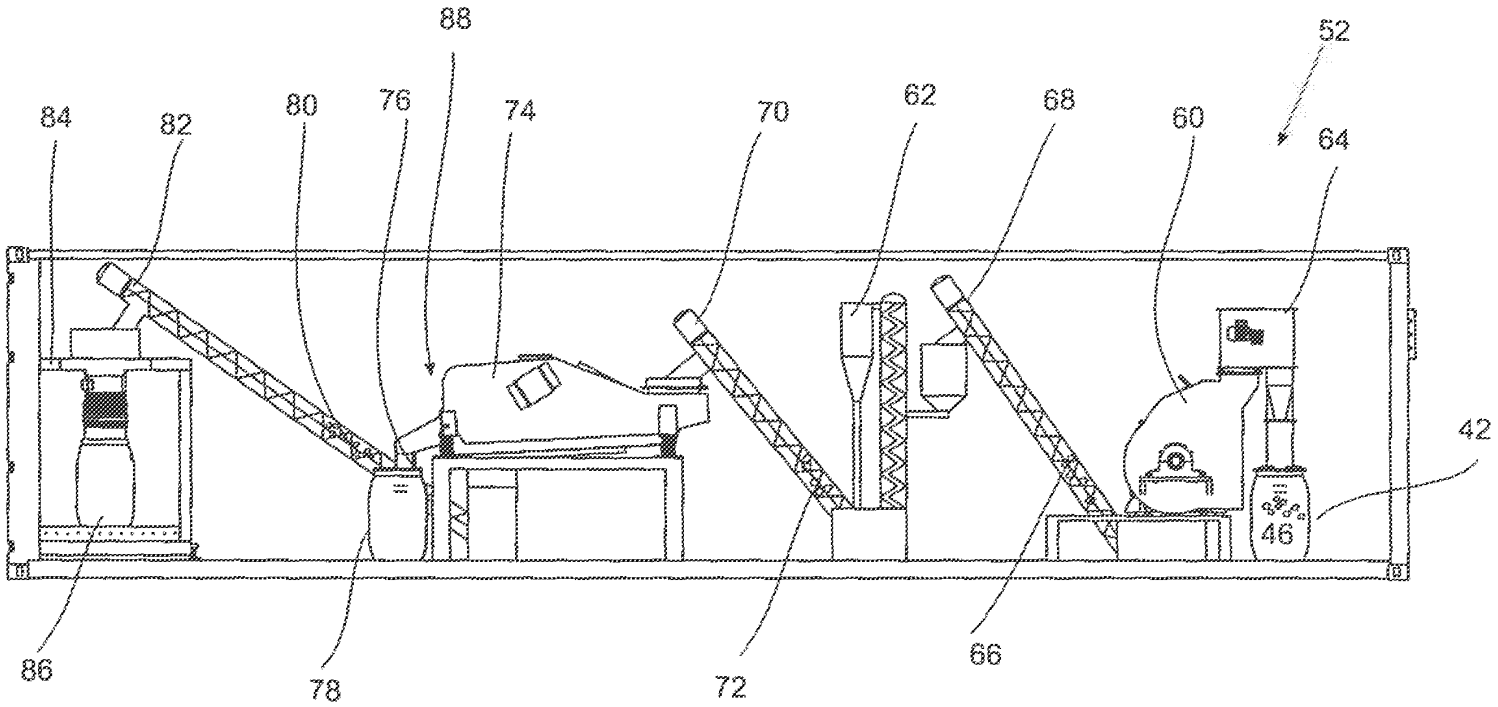

Fig. 3

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2016/059526

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M6/52 C22B13/00 H01M10/54
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
X	WO 2010/102377 A1 (ELLIOTT K W) 16 September 2010 (2010-09-16) paragraphs [0015] - [0040]; figures 1, 3 -----	1-12
X	DE 10 2011 110083 A1 (TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA [DE]) 14 February 2013 (2013-02-14) cited in the application paragraphs [0021], [0022], [0034] - [0039]; figure 1 -----	1-3,5-8, 10,12

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
5 July 2016	13/07/2016

Name and mailing address of the ISA/	Authorized officer
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2

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2016/059526

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2010102377 A1	16-09-2010	CA 2730320 A1	16-09-2010
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		EP 2742559 A1	18-06-2014
		US 2014290438 A1	02-10-2014
		WO 2013023640 A1	21-02-2013

PTO/AIA/14 (11-15)
Approved for use through 04/30/2017. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	03101160US
	Application Number	
Title of Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	03101160US
	Application Number	
Title of Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION	

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For further information see 37 CFR 1.33(a).

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	03101160US
	Application Number	
Title of Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION	

Application Information:

Title of the Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION		
Attorney Docket Number	03101160US	**Small Entity Status Claimed**	☐
Application Type	Nonprovisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)	3	**Suggested Figure for Publication (if any)**	

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

Publication Information:

☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)

☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application **has not and will not** be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32).
Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

Please Select One:	● Customer Number	US Patent Practitioner	○ Limited Recognition (37 CFR 11.9)
Customer Number	30743		

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Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.
When referring to the current application, please leave the "Application Number" field blank.

Prior Application Status	Pending		Remove
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)
	a 371 of international	PCT/EP2016/059526	2016-04-28

Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the **Add** button. Add

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)[i] the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Remove

Application Number	Country[i]	Filing Date (YYYY-MM-DD)	Access Code[i] (if applicable)
10 2015 207 843.4	DE	2015-04-28	

Additional Foreign Priority Data may be generated within this form by selecting the **Add** button. Add

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.
NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

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	Application Number	
Title of Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION	

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	03101160US
	Application Number	
Title of Invention	METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION	

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Applicant 1 Remove

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

Clear

● Assignee	Legal Representative under 35 U.S.C. 117	Joint Inventor
Person to whom the inventor is obligated to assign.	Person who shows sufficient proprietary interest	

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

Name of the Deceased or Legally Incapacitated Inventor:

If the Applicant is an Organization check here. ☒

Organization Name	DUESENFELD GMBH		
Mailing Address Information For Applicant:			
Address 1	Friedlandweg 5		
Address 2			
City	Braunschweig	**State/Province**	
Country	DE	Postal Code	38114
Phone Number		Fax Number	
Email Address			

Additional Applicant Data may be generated within this form by selecting the Add button. Add

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Assignee 1

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

Remove

If the Assignee or Non-Applicant Assignee is an Organization check here. ☐

Prefix	**Given Name**	Middle Name	**Family Name**	Suffix

Mailing Address Information For Assignee including Non-Applicant Assignee:

Address 1			
Address 2			
City		**State/Province**	
Country i		Postal Code	
Phone Number		Fax Number	
Email Address			

Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button. Add

Signature:

Remove

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). **However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).**

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

Signature	/Michael E. Whitham/			Date (YYYY-MM-DD)	2017-10-25
First Name	Michael	Last Name	Whitham	Registration Number	32635

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This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1 The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3 A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent CooperationTreaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99)**		
	Application Number	
	Filing Date	
	First Named Inventor	
	Art Unit	
	Examiner Name	
	Attorney Docket Number	

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	9780419	B2	2017-10-03	LION ENGINEERING GMBH	

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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20050241943	A1	2005-11-03	KAWASAKI JUKOGYO KABUSHIKI KAISHA	

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FOREIGN PATENT DOCUMENTS Remove

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	2010/102377	WO	A1	2010-09-16	STEVENS, WAYNE, C.		
	2	10 2011 110 083	DE	A1	2013-02-14	TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA		
	3	10 2012 024 876	DE	A1	2014-06-26	ELECTROCYCLING GMBH; VOLKSWAGEN AKTIENGESELLSCHAFT		

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	
	First Named Inventor	
	Art Unit	
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Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
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Examiner Signature		Date Considered	

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	
	First Named Inventor	
	Art Unit	
	Examiner Name	
	Attorney Docket Number	

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature	/Michael E. Whitham/	Date (YYYY-MM-DD)	2017-10-25
Name/Print	Michael E. Whitham	Registration Number	32635

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

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7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

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9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
16 September 2010 (16.09.2010)

PCT

(10) International Publication Number
WO 2010/102377 A1

(51) International Patent Classification:
H01M 6/52 (2006.01) *B02C 23/14* (2006.01)
A62D 3/30 (2007.01) *H01M 10/54* (2006.01)
B02C 23/08 (2006.01)

(21) International Application Number:
PCT/CA2009/001583

(22) International Filing Date:
3 November 2009 (03.11.2009)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
61/160,058 13 March 2009 (13.03.2009) US

(72) Inventor; and
(71) Applicant : **STEVENS, Wayne, C.** [CA/CA]; 9 Cherry Ridge Blvd., Fenwick, Ontario L0S 1C0 (CA).

(74) Agent: **KOCH, Mark**; 583 Main Street East, Hamilton, Ontario L8M 1J4 (CA).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

— *of inventorship (Rule 4.17(iv))*

Published:

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(54) Title: BATTERY RECYCLING

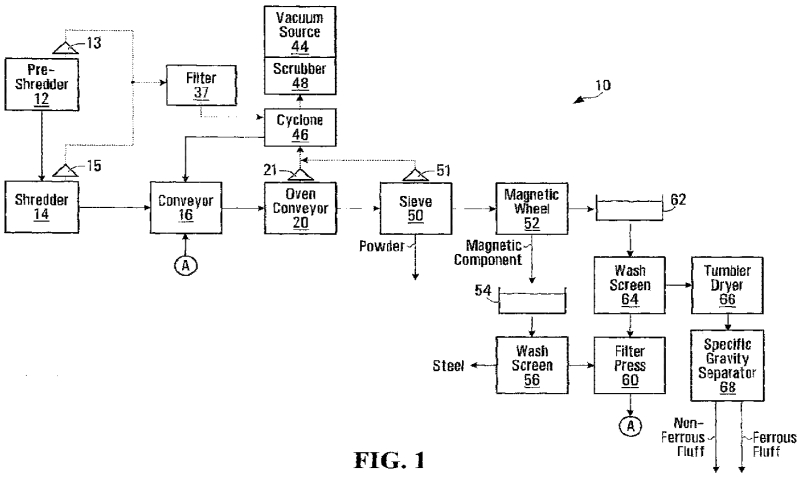


FIG. 1

(57) Abstract: In a method of recycling sealed batteries, the batteries are shredded to form a shredded feedstock. The shredded feedstock is heated above ambient temperature and rolled to form a dried material. The dried material is screen separated into a coarse fraction and a powder fraction and the powder fraction is output. A system for recycling sealed cell batteries comprises an oven with a first conveyor extending into the oven. A rotatable tunnel extends within the oven from an output of the first conveyor. The tunnel has a spiral vane depending from its inner surface which extends along a length of the tunnel. A second conveyor is positioned below an output of the rotatable tunnel.

WO 2010/102377 A1

BATTERY RECYCLING

BACKGROUND

[0001] The present invention relates to a system and process for recycling sealed cell batteries.

[0002] Ninety-five percent of portable batteries are household batteries. The vast majority of these are sealed cell alkaline batteries. Once spent, most of these batteries are simply discarded and find their way to landfill sites.

[0003] A known method for recycling alkaline batteries involves mechanically removing the casing from the battery cell and then using chemical processes to separate the solid materials of the cell. Major solid components of the cells are carbon, zinc, potassium, and manganese. There are several problems with chemical separation processes. Firstly, the component materials involved are of low value while chemical processing is expensive. Also, additional waste streams are created with the chemical processes. In view of these drawbacks, this recycling method has not found widespread use.

[0004] This invention seeks to overcome drawbacks of known sealed cell battery recycling processes.

SUMMARY

[0005] In a method of recycling sealed batteries, the batteries are shredded to form a shredded feedstock. The shredded feedstock is heated above ambient temperature and rolled to form a dried material. The dried material is screen separating into a coarse fraction and a powder fraction and the powder fraction is output.

Ascend Elements EX1002 Page 531

Duesenfeld Ex. 2014-531

[0006] A system for recycling sealed cell batteries comprises an oven with a first conveyor extending into the oven. A rotatable tunnel extends within the oven from an output of the first conveyor. The tunnel has a spiral vane depending from its inner surface which extends along a length of the tunnel. A second conveyor is positioned below an output of the rotatable tunnel.

[0007] Other features and advantages will be apparent from the following description in conjunction with the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] In the figures which illustrate an example embodiment of the invention,

[0009] **FIG. 1** is a schematic diagram of a system for battery recycling in accordance with this invention,

[0010] **FIG. 2** is a partially cut-away perspective view of a portion of the system of **FIG. 1**,

[0011] **FIG. 3** is a schematic diagram of another system for battery recycling in accordance with this invention, and

[0012] **FIG. 4** is a schematic diagram of a portion of another system for battery recycling in accordance with this invention.

DETAILED DESCRIPTION

[0013] Technically, a battery is a series of battery cells. Thus, a 9 V battery is a true battery, whereas AAA through D size batteries are cells. In this specification, the term battery is used to mean either true batteries or cells.

[0014] The central core of an alkaline cylindrical button cell is the anode which is a dispersion of zinc oxide powder in a gel containing a potassium hydroxide electrolyte. This core is surrounded by a separator which is a non-woven layer of

cellulose (paper) or a synthetic polymer (plastic). Surrounding the separator is an annular cathode which is a compressed paste of manganese dioxide with carbon (graphite) powder to increase conductivity. The anode, separator, and cathode are sealed in a drawn steel casing. Although some alkaline batteries are mercury-free, many spent alkaline batteries contain mercury, with an average overall concentration of 100 ppm.

[0015] Turning to **FIG. 1**, a system **10** for recycling sealed cell alkaline batteries has a pre-shredder **12** for pre-shredding the feedstock in order to rupture their sealed steel casings. Next the batteries may be further shredded in secondary shredder **14**, which may be a granulator, and are then conveyed downstream by conveyor **16**. The conveyor, which may be a chain conveyor or a belt conveyor with flights, assists in breaking up the feedstock into a more uniform material feed stream.

[0016] From the conveyor **16**, the feedstock passes to an oven conveyor **20**. The oven conveyor **20** is detailed in **FIG. 2**. Turning to **FIG. 2**, oven conveyor **20** has an input belt conveyor **22** with an overlying blade **24** which levels the feedstock. The input belt conveyor dispenses to an upstream screw tunnel **26**. The upstream screw tunnel **26** has a spiral vane depending from its inner surface that extends along the length of the tunnel. The upstream screw tunnel **26** rotates to roll the feedstock. The upstream screw tunnel **26** in turn dispenses to a middle belt conveyor **28** with an overlying blade **30** which levels the feedstock. The middle belt conveyor dispenses to a downstream screw tunnel **32** having a similar configuration as the upstream screw tunnel. The downstream screw tunnel dispenses to an output belt conveyor **34**. The output belt conveyor has an overlying blade **36** which levels the feedstock. An oven **40** surrounds all but the upstream end of the input belt conveyor and downstream end of the output belt conveyor.

[0017] Feedstock traveling on the input belt conveyor **22** is levelled by blade **24**. The material then drops into upstream screw tunnel **26**. This screw tunnel rotates

Ascend Elements EX1002 Page 533

Duesenfeld Ex. 2014-533

to roll the feedstock and feed it downstream. At the downstream end of the upstream screw tunnel, the feedstock drops to the middle belt conveyor **28** where it is levelled by blade **30** and conveyed into downstream screw tunnel **32**. The downstream screw tunnel rotates to roll the feedstock and convey it to output belt conveyor **34**, where the feedstock is levelled by blade **36** and then dispensed from the oven conveyor.

[0018] By repeatedly conveying, rolling and levelling the feedstock at elevated temperatures in the oven conveyor, the feedstock is dried and evaporation of mercury in the feedstock is promoted.

[0019] Returning to **FIG. 1**, there are air uptakes **13**, **15**, **21**, and **51** associated with each of pre-shredder **12**, shredder **14**, oven conveyer **20** and a sieve **50**, respectively. Each of the air uptakes feed ultimately to a cyclone **46**, with air uptakes **13** and **15** feeding to the cyclone through a particulate filter **37**. A vacuum source **44** draws air through the cyclone **46** to a scrubber **48** before the air leaves the system **10**. The cyclone swirls the moving air to drop out any powder or other solids entrained in the moving air. The scrubber sequesters vapours in the moving air which, with the alkaline battery feedstock, will be mercury vapours. The scrubber **48** may be a venturi scrubber with a pH control system that ensures the solubility of vapours. It may also be any other type known in the art to be effective in these conditions. With a venturi scrubber, the scrubber itself acts as the vacuum source. Filter **37** traps any paper and plastics drawn off by air intakes **13** and **15** so that the scrubber is not obstructed by this material and so that any fire that develops in the scrubber is not additionally fed by these paper and plastics.

[0020] While mercury has a boiling point of about 357°C (about 675 °F) , it is nevertheless volatile even at room temperature and therefore off gases at the various air uptakes as well as off gassing strongly in the oven conveyor.

[0021] Feedstock leaving the oven conveyor passes to a sieve **50** which may be a shaker table with a mesh size #30. This separates a coarser fraction of the

feedstock from a finer fraction. The mesh size is such that the finer fraction which drops through the sieve is a zinc oxide, manganese dioxide, and potassium hydroxide powder. This powder is recovered as a finished product of the process.

[0022] The coarser fraction, which comprise the shredded steel casing along with brass, cellulose (paper), graphite (carbon), and plastic, collectively known as fluff, pass to a magnetic separator **52**, which may be a magnetic wheel.

[0023] The separated steel (magnetic component) is immersed in a first water bath **54**. Water from the bath **54** drains through a wash screen **56**. Any remaining powder will pass through the screen and therefore remain in the water. Larger solids, namely the separated steel, are blocked by the screen and are washed off the screen to recover the steel as a second finished product of the process.

[0024] The water which passes through the screen **56** passes on to filter press **60**.

[0025] The non-magnetic component of the fluff which is separated by the magnetic separator is immersed in a second water bath **62**. Water from the bath **62** drains through a wash screen **64**. Any remaining powder in the water will pass through the screen with the water. Larger solids, namely the fluff, are blocked by the screen and are washed off the screen. This fluff passes to a tumbler dryer **66** where it is dried and then to a specific gravity separator **68**. The specific gravity separator may be of the forced air type known in the art. The specific gravity separator **68** separates the ferrous component of the fluff (graphite and brass) from the nonferrous component (paper and plastic). These two output streams are finished products of the process.

[0026] The water which passes through screen **64** passes on to filter press **60**.

[0027] The water passing to the filter press from screens **56** and **64** passes through the membranes of the filter press such that any powder which had been in

Ascend Elements EX1002 Page 535

Duesenfeld Ex. 2014-535

the water is recovered as a wet powder. This wet powder is fed back to the conveyor **16** in the system **10**. The water, which at this stage is normally caustic, then passes to a holding tank (not shown) wherein its pH is adjusted so that it may be reused in the water baths.

[0028] The recovered powder of zinc oxide, manganese dioxide, and potassium hydroxide may be used in fertilizer provided its mercury content has been sufficiently reduced. In this regard, typically, a powder with a mercury content of less than 75 ppm is suitable for use in fertilizer. With the subject system, mercury concentrations down to undetectable levels may be achieved.

[0029] The recovered steel may be used in the steel industry. The recovered paper and plastic from the fluff may be burned for energy (e.g., in oven **40**).

[0030] The same process described for alkaline batteries may also be used for any of zinc carbon, zinc chloride, and manganese batteries. The composition of the recovered powder will vary somewhat depending upon the type of battery which forms the feedstock. However, for all of these types of batteries, the recovered powder may be used in fertilizer.

[0031] The time during which the feedstock remains in the oven conveyor and the temperature of this conveyor are determined based on characteristics of the feedstock and desired properties of the powder output from the system. More specifically, a customer of the powder may specify a required dryness and mercury content for the powder. Characteristics of the feedstock which impact the required heating time and temperature are the size of the battery cells, the age of the cells (newer batteries need to be run slower at higher temperatures), mercury content, and the type of battery. Regardless of customer requirements, the feedstock must at least be sufficiently dry so that it separates at the screen separator sieve **50**.

[0032] While the temperature of the oven can be varied as required, typically, a temperature of between a low temperature of 300°F and a high temperature of

Ascend Elements EX1002 Page 536

Duesenfeld Ex. 2014-536

800°F is sufficient where the feedstock remains in the oven conveyor for 1 to 10 minutes. The speed of the conveyors determines the time in the oven conveyor. By way of example only one minute in the oven conveyor may correspond to a high speed whereas ten minutes in the oven conveyor may correspond to a low speed.

[0033] For example, for a mixed feedstock of alkaline, zinc carbon, zinc chloride, and manganese batteries and a 26 ft long oven conveyor, the speed through the oven conveyor may be 2.6 ft/min and the temperature may be set at 800° F.

[0034] The mesh size of the screen separator can be varied as required provided it is sufficiently small to separate the powder fraction of the feedstock. However, a #30 mesh size is suitable where the powder is to be used in the fertilizer industry and also allows the powder to pelletize well.

[0035] The described system may be modified for use with nickel metal hydride sealed cell batteries. More specifically, turning to **FIG. 3**, system **100** for use with a feedstock of nickel metal hydride batteries is with some exceptions the same as system **10** of **FIG. 1**, and like parts have been given like reference numerals. The differences are as follows. System **100** includes a tumbler dryer **76** at the output of the first wash screen **56** which feeds to a specific gravity separator **78**.

[0036] With nickel metal hydride batteries, the anode is nickel oxyhydroxide and the cathode is most commonly a lanthanoid mixture with nickel, cobalt, manganese, and/or aluminum. Some cathodes may include other metals, such as iron or chromium. The electrolyte is most commonly potassium hydroxide.

[0037] The processing of nickel metal hydride batteries in system **100** proceeds identically to the processing of alkaline batteries in system **10** of **FIG. 1** except as follows. Vapours generated from drying the feedstock are water and volatile organic compounds (VOCs). The speed of the oven conveyor is adjusted so that the feedstock dries sufficiently through the oven conveyor at the chosen oven

Ascend Elements EX1002 Page 537

Duesenfeld Ex. 2014-537

temperature. The scrubber (which may be a venturi scrubber as used in the system **10** of **FIG. 1**) sequesters the vapours from the drying process.

[0038] When the feedstock leaves the oven conveyor and reaches the sieve, the powder component which passes through the sieve comprises nickel oxyhydroxide powder, cobalt oxide, and lanthanoids. As when operating with alkaline batteries, sieve **50** may have a #30 mesh size. The recovered powder may be used in the coatings industry for pigmentation or the powder may be used in the stainless steel industry.

[0039] The solid component, which does not pass through the sieve, contains steel, fluff and nickel alloys. These nickel alloys include iron. The steel and nickel alloys form the magnetic component which is separated from the fluff by the magnetic wheel **52**. However, the nickel alloys of this magnetic component typically encapsulate some of the fluff as a consequence of the upstream shredding process. This encapsulated fluff remains with the magnetic component leaving the magnetic wheel. The magnetic component is directed through bath **54** to the first wash screen **56**. The solid component leaving the wash screen passes to tumbler dryer **76** where it is dried. The dried solid component then inputs specific gravity separator **78** to separate nonferrous fluff residues from the nickel alloys and to separate the nickel alloys from the steel. The steel and nickel alloys are finished products that may be used in the steel industry.

[0040] System **100** may be modified to operate with a feedstock of lithium button cells, namely lithium ion batteries, which are rechargeable, or lithium polymer batteries. With a lithium battery, the anode is a lithium metal or lithium compound. The cathode is, most commonly, manganese dioxide and the electrolyte is, most commonly, lithium perchlorate in propylene carbonate and dimethoxyethane.

[0041] **FIG. 4** illustrates a portion of a system **1000** for use with lithium button cells. System **1000** is identical to system **100** of **FIG. 3** except as follows. The scrubber **48** outputs to a water atomizer **84** and the atomizer **84** outputs to a tank

86. These modifications are employed because of the highly combustible nature of lithium and the volatile organic compounds (VOCs) in the batteries.

[0042] In this modified system, when the lithium batteries are shredded and conveyed by the conveyor, lithium, which gas off at any of the air pick up points and pass to the scrubber **48** where they are sequestered. Any lithium or VOCs which escape the scrubber are condensed by the atomized water from the atomizer **84** and the condensed lithium passes to the water tank **86**. The pH of the water in the atomizer and the tank is controlled with, for example, the addition of sodium hydroxide to a concentration of 50%, to ensure the gas remains condensed in the water. Any paper drawn off or drawn off is trapped by filter **70** before it reaches the scrubber so that it will not feed any fire that might occur at the scrubber or obstruct the scrubber. Because of the risk of combustion with lithium ion battery feedstock, the temperature in the oven conveyor is maintained below the combustion temperature of paper (454°F).

[0043] With lithium button cells, at the sieve **50**, the powder which drops out is mostly lithium cobalt oxide. This powder can be used in the coatings industry. As before, a mesh size #30 is suitable to limit contamination of this powder.

[0044] The magnetic component separated by the magnetic wheel includes steel and magnetic alloys as well as any aluminum and copper that is trapped by the steel and magnetic alloys. The specific gravity separator **78** (**FIG. 3**) separates out non-ferrous fluff encapsulated with this magnetic component and also separates the steel, magnetic alloys, aluminum, and copper from each other.

[0045] In general, with system **10** run with alkaline batteries, the oven conveyor is run at a low speed and high temperature (to promote evaporation of mercury), with system **1000** run with lithium batteries, the oven conveyor is run at a mid speed and temperature (to avoid combustion), and when the system **100** is run with nickel metal hydride batteries, the oven conveyor is run at high speeds and temperatures.

Ascend Elements EX1002 Page 539

Duesenfeld Ex. 2014-539

[0046] While three different systems have been described, they may be combined in a single system and components of that system switched in or out, or on or off, as appropriate, to adapt the system to the different kinds of feedstock.

[0047] Rather than providing a vacuum source to draw vapours into the scrubber, blowers could be used for this purpose.

[0048] There is a minimal amount of paper and light plastics released with the shredding of alkaline batteries. Accordingly, optionally, filter **37** may be omitted when running system **10**.

[0049] Other modifications will be apparent to those skilled in the art and, therefore, the invention is defined in the claims.

Ascend Elements EX1002 Page 540

Duesenfeld Ex. 2014-540

WHAT IS CLAIMED IS:

1. A method of recycling sealed batteries, comprising:

shredding said batteries to form a shredded feedstock;

heating said shredded feedstock above ambient temperature while rolling said shredded feedstock to form a dried material;

screen separating said dried material into a coarse fraction and a powder fraction; and

outputting said powder fraction.

2. The method of claim 1 further comprising levelling said shredded feedstock while heating said feedstock above ambient temperature.

3. The method of claim 1 wherein said batteries are alkaline batteries containing mercury, said heating comprises heating said shredded feedstock above ambient temperature and below the boiling point of mercury, said heating while rolling evaporates a portion of said mercury from said feedstock, and wherein said powder fraction comprises zinc oxide, manganese dioxide, and potassium hydroxide.

4. The method of claim 3 further comprising drawing air through said shredded material during said heating and rolling to draw off mercury vapour from said shredded feedstock.

5. The method of claim 4 further comprising sequestering said mercury in a scrubber.

6. The method of claim 5 further comprising swirling said air drawn from said shredded material to drop out any particulates from said air and returning said particulates to said shredded material.

7. The method of claim 1 further comprising magnetically separating said coarse fraction into a magnetic component and a non-magnetic component.

Ascend Elements EX1002 Page 541

Duesenfeld Ex. 2014-541

8. The method of claim 7 further comprising immersing said magnetic component in a first water bath and passing water from said first water bath through a screen to separate magnetic solids, and outputting said magnetic solids.

9. The method of claim 8 further comprising separating said magnetic solids based on their specific gravity prior to said outputting said magnetic solids and wherein said outputting said magnetic solids comprises outputting separated constituents of said magnetic solids.

10. The method of claim 8 further comprising immersing said non-magnetic component in a second water bath and passing water from said second water bath through a screen to separate fluff, and outputting said fluff.

11. The method of claim 10 further comprising separating said fluff based on specific gravity of constituents of said fluff prior to said outputting said fluff and wherein said outputting said fluff comprises outputting separated constituents of said fluff.

12. The method of claim 10 further comprising combining water from said first water bath and said second water bath as a water component and filtering said water component to recover a powder component.

13. The method of claim 12 further comprising adding said powder component to said shredded material.

14. A method of recycling sealed batteries containing mercury, comprising:

shredding said batteries to form a shredded feedstock;

heating said shredded feedstock above ambient temperature and below the boiling point of mercury while rolling said shredded feedstock to evaporate a portion of said mercury from said feedstock to form a mercury reduced dried material;

Ascend Elements EX1002 Page 542

Duesenfeld Ex. 2014-542

screen separating said dried material into a coarse fraction and a powder fraction; and

outputting said powder fraction.

15. A system for recycling sealed cell batteries, comprising:

an oven;

a first conveyor extending into said oven;

a rotatable tunnel with a spiral vane depending from an inner surface of said tunnel extending along a length of said tunnel, said rotatable tunnel disposed within said oven and extending from an output of said first conveyor;

a second conveyor below an output of said rotatable tunnel.

16. The system of claim 15 further comprising a sieve downstream of an output of said second conveyor.

17. The system of claim 16 further comprising a shredder upstream of an input of said first conveyor.

18. The system of claim 17 further comprising a leveller associated with said first conveyor.

19. The system of claim 15 further comprising a cyclone and a scrubber above said oven, and means for moving air upwardly through said oven, said cyclone and said scrubber.

20. The system of claim 19 further comprising at least one particulate filter between said oven and said scrubber.

Ascend Elements EX1002 Page 543

Duesenfeld Ex. 2014-543

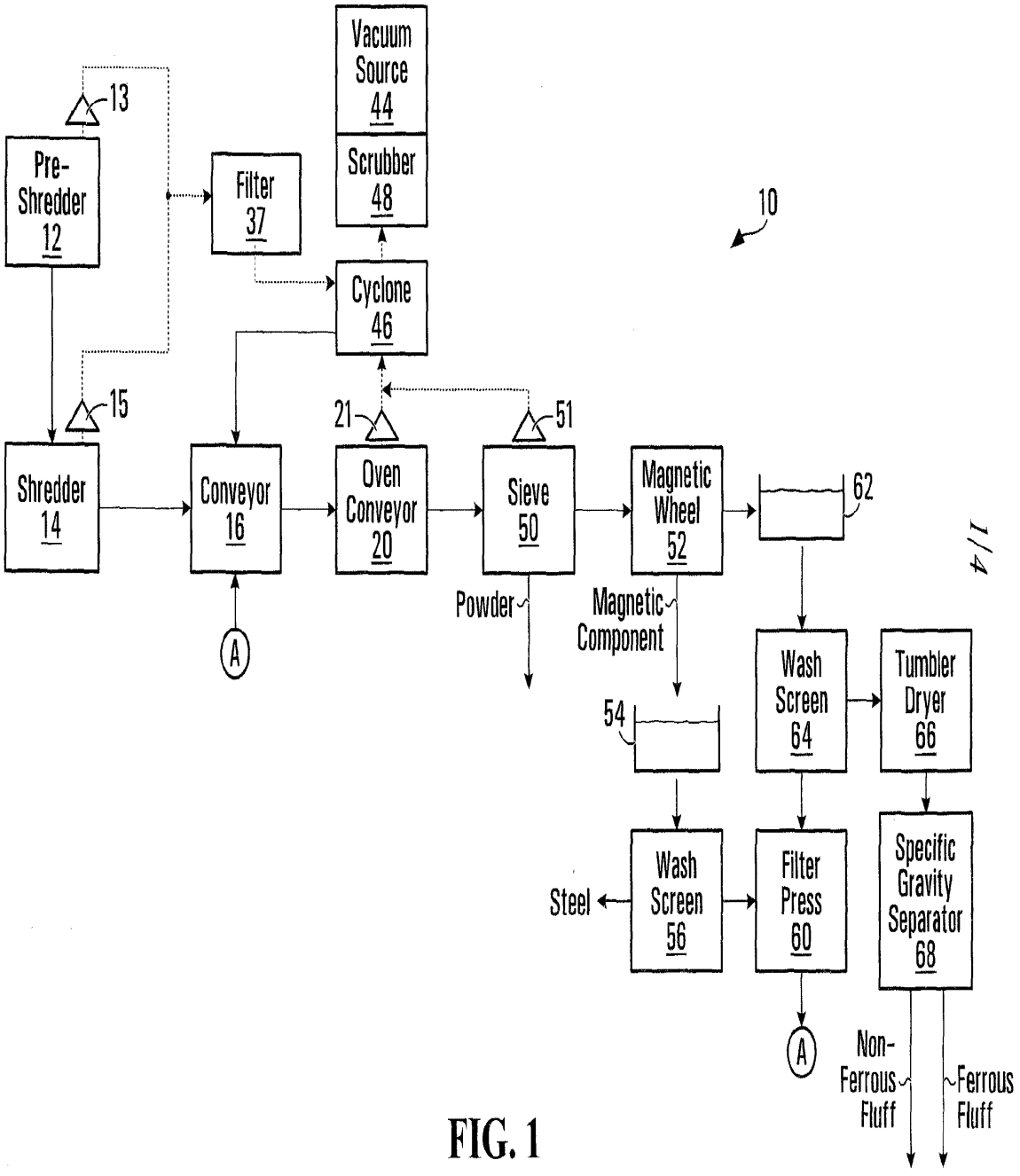


FIG. 1

PCT/CA2009/001583

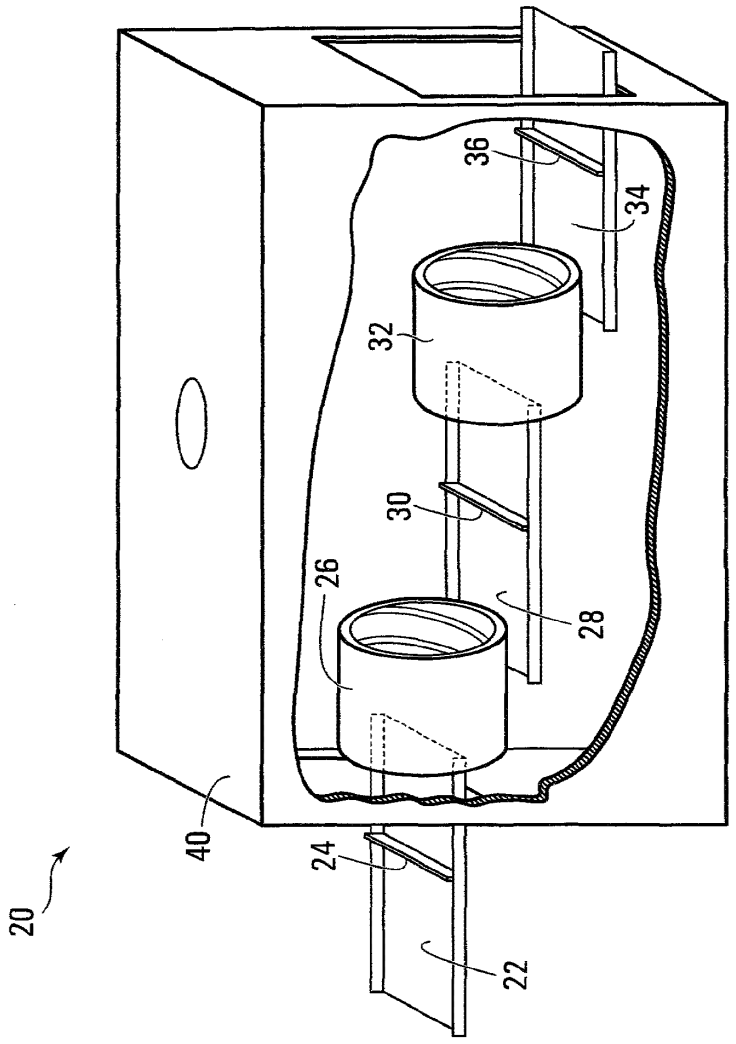
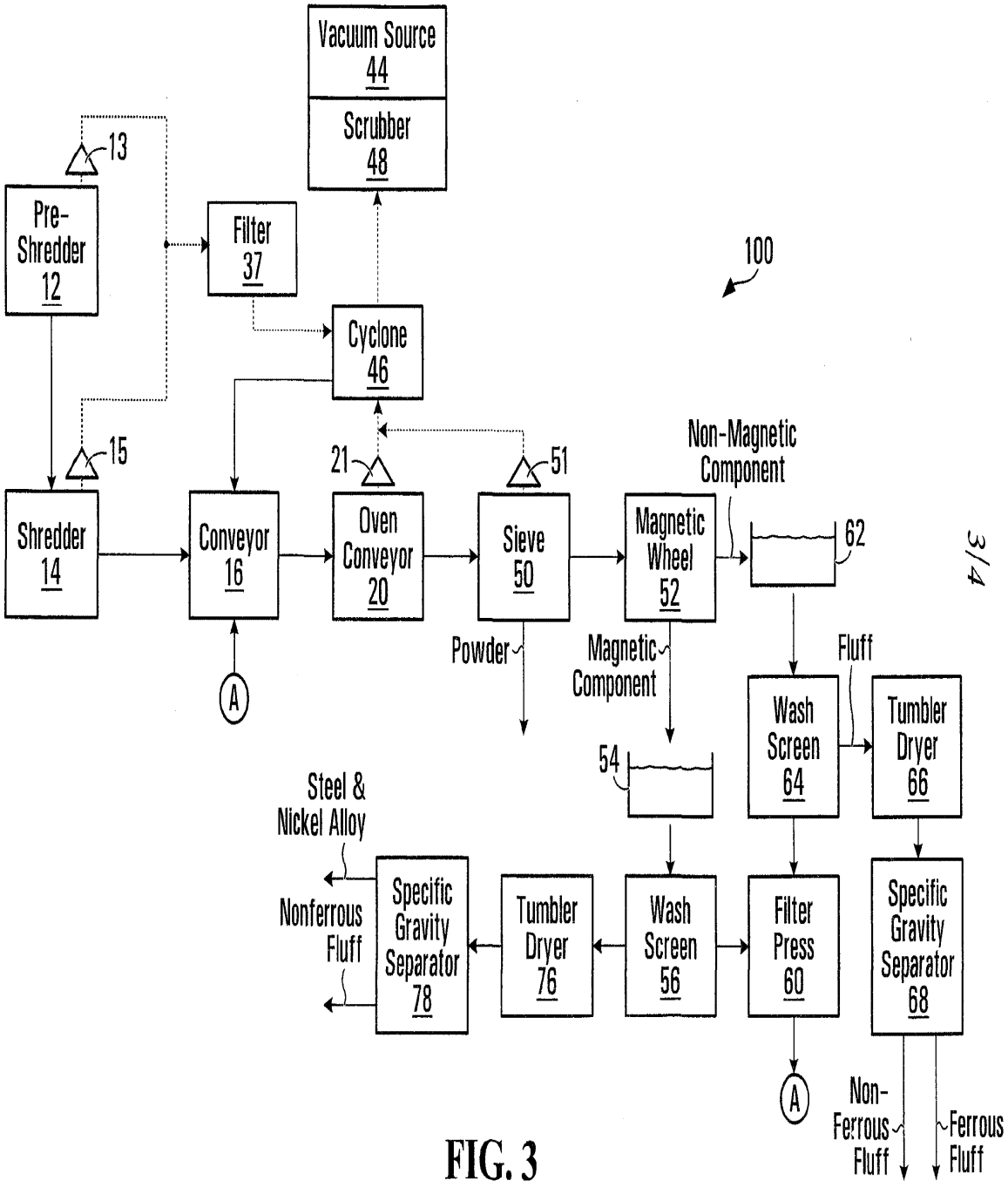


FIG. 2


Vacuum Source
44
Scrubber
48
13
Pre-
Shredder
12
Filter
37
100
Cyclone
46
15
21
51
Non-Magnetic
Component
Shredder
14
Conveyor
16
Oven
Conveyor
20
Sieve
50
Magnetic
Wheel
52
62
A
Powder
Magnetic
Component
Fluff
Wash
Screen
64
Tumbler
Dryer
66
54
Steel &
Nickel Alloy
Nonferrous
Fluff
Specific
Gravity
Separator
78
Tumbler
Dryer
76
Wash
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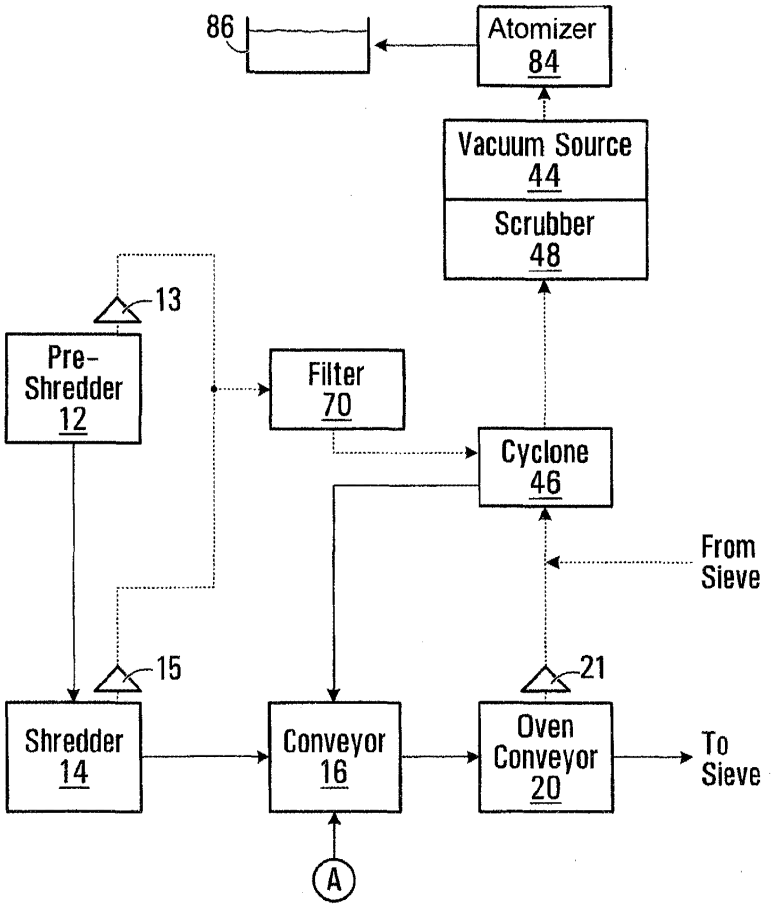
Ferrous
Fluff

FIG. 3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Atomizer
84
Vacuum Source
44
Scrubber
48
1000
13
Pre-
Shredder
12
Filter
70
Cyclone
46
From
Sieve
15
21
Shredder
14
Conveyor
16
Oven
Conveyor
20
To
Sieve
A

FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2009/001583

A. CLASSIFICATION OF SUBJECT MATTER
IPC: ***H01M 6/52*** (2006.01) , ***A62D 3/30*** (2007.01) , ***B02C 23/08*** (2006.01) , ***B02C 23/14*** (2006.01) , ***H01M 10/54*** (2006.01)
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
No classification restrictions

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
Canadian Patent Database, Intellect, Epoque, Delphion, Patent Abstracts of Japan

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,026,477 (Tremolada) 31 May 1977 (31.05.1977) col.1, lines 10-12, 32-45, Example, claim 1	1
Y		7
P, Y	US 2009/0095127 A1 (Petterson) 16 April 2009 (16.04.2009)	7
P, A	whole description and Figures 1-2	1-13
Y	WO 2009/028795 A2 (IM, Hyong Hag) 05 March 2009 (05.03.2009)	7
A	abstract, [12], [17] - [51] and claims	1-13
A	US 6,686,086 B1 (Martin et al) 03 February 2004 (03.02.2004) abstract, col.1, lines 18-39, col.2, lines 60-63, col.4, lines 35-37, col.6, line 5 - col.7, line 4 and claim 1	1-5, 14 and 15
A	CA 2,418,527 (BATREC INDUSTRIE AG, CH) 01 August 2003 (01.08.2003) abstract, page 1 to page 10, line 14	1

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents :
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
27 January 2010 (27-01-2010)	01 February 2010 (01-02-2010)

Name and mailing address of the ISA/CA	Authorized officer
Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 001-819-953-2476	Irina Kargina (819) 997-3059

Ascend Elements EX1002 Page 548

Duesenfeld Ex. 2014-548

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2009/001583

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CA 2,391,865 (AVESTOR) 21 March 2002 (21.03.2002) abstract, whole description	1

Ascend Elements EX1002 Page 549

Duesenfeld Ex. 2014-549

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CA2009/001583

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Ascend Elements EX1002 Page 550

Duesenfeld Ex. 2014-550

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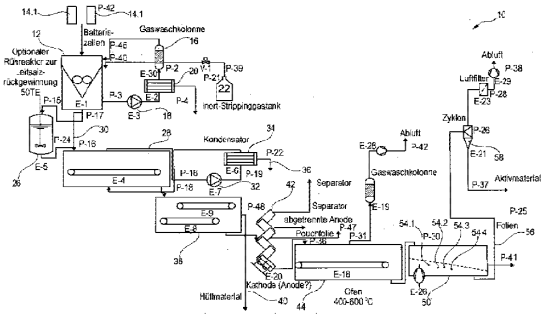
Prüfungsantrag gemäß § 44 PatG ist gestellt.

Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen

(54) Bezeichnung: **Verfahren zum Wiedergewinnen von Aktivmaterial aus einer galvanischen Zelle und Aktivmaterial-Separationsanlage, insbesondere Aktivmetall-Separationsanlage**

(57) Zusammenfassung: Verfahren zum Wiedergewinnen von Aktivmaterial einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial und einen Binder zum Verbinden des Aktivmaterials und des Trägers umfasst, mit den Schritten: Zerkleinern der Zellen, insbesondere unter Inertgas oder im Vakuum, so dass zumindest auch feste Zellenfragmente entstehen, Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur (T_Z), die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, vorzugsweise unter Intergas oder im Vakuum so dass wärmebehandelte Zellenfragmente entstehen, und Klassieren der wärmebehandelten Zellenfragmente, wobei das Klassieren ein Luftstrahlsieben umfasst.



Beschreibung

[0001] Die Erfindung betrifft ein Verfahren zum Wiedergewinnen von Aktivmaterial, insbesondere von Lithium oder Lithiumverbindungen, aus einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und gegebenenfalls einen Elektrolyten umfasst, mit den Schritten: (a) Zerkleinern der Zellen, insbesondere unter Inertgas, so dass zumindest auch feste Zellenfragmente entstehen, (b) Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur, die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, so dass wärmebehandelte Zellenfragmente entstehen, und (c) Klassieren der wärmebehandelten Zellenfragmente.

[0002] Gemäß einem zweiten Aspekt betrifft die Erfindung eine Aktivmetall-Separationsanlage, die ausgebildet ist zum Wiedergewinnen von Aktivmaterial, insbesondere Lithium, einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und einen Elektrolyten umfasst, mit (a) einer Zellenzerkleinerungsvorrichtung zum Zerkleinern der Zellen, wobei die Zellenzerkleinerungsvorrichtung gasdicht ist und eine Inertgas-Zuführvorrichtung umfasst, so dass die Zellen unter Inertgas zu Zellenfragmenten zerkleinerbar sind, und (b) einem Ofen, der zumindest mittelbar mit der Zellenzerkleinerungsvorrichtung verbunden und eingerichtet ist zum Erwärmen der Zellenfragmente auf eine Zersetzungstemperatur, die so hoch gewählt ist, dass der Binder sich zersetzt.

[0003] Beim Recycling von Aktivmaterial, insbesondere von Lithium aus Lithium-Akkumulatoren oder Batterien, besteht das Problem, dass die chemischen Prozesse relativ aufwendig sind. Der Grund dafür ist, dass die Trennung des Aktivmaterials von seinem Träger nur unzureichend gelingt.

[0004] Aus der US 200510241943 A1 ist ein Verfahren bekannt, bei dem die Zellen zunächst zerkleinert und dann einer Wärmebehandlung unterzogen werden. Danach werden die Zellen geschreddert und auf die Zersetzungstemperatur erwärmt. Anschließend werden die wärmebehandelten Zellenfragmente durch Vibrationssieben getrennt. Es hat sich jedoch gezeigt, dass dieses Verfahren aufwendig ist. Der Erfindung liegt die Aufgabe zugrunde, das Wiedergewinnen von Aktivmaterial, insbesondere von Lithium oder Lithiumverbindungen, aus alten Batterien oder Akkumulatoren zu verbessern.

[0005] Die Erfindung löst das Problem durch ein gattungsgemäßes Verfahren, bei dem das Klassieren ein Luftstrahlsieben umfasst. Gemäß einem zweiten Aspekt löst die Erfindung das Problem durch eine Aktivmetall-Separationsanlage, die eine Luftstrahlsiebvorrichtung besitzt, die zumindest unmittelbar mit dem Ofen verbunden und eingerichtet ist zum Klassieren von Aktivmaterial und Träger, insbesondere Trägerfolie.

[0006] Vorteilhaft an der Erfindung ist, dass das Aktivmaterial in hoher Reinheit gewonnen werden kann. Der Grund dafür ist, dass beim Luftstrahlsieben sehr kleine Maschenweiten einsetzbar sind, so dass auch kleine Verunreinigungspartikel ferngehalten werden.

[0007] Vorteilhafterweise kann auch das Trägermaterial in hoher Reinheit zurückgewonnen werden kann. Beispielsweise wird als Träger häufig Aluminiumfolie verwendet, die nur dann effizient recycelt werden kann, wenn sie gewisse Mindestanforderungen hinsichtlich der Reinheit erfüllt. Beispielsweise eignet sich eine Trägerfolie mit mehr als 0,03% Verunreinigungen nur wenig zum Recyceln. In der weiteren hydrometallurgischen Aufarbeitung neigen Übergangsmetallsalzlösungen aus einer sauren Lithiumlösung zur Agglomeration, wenn diese die bei der konventionellen Trennung üblichen ca. 1 Massen-% Aluminiumkontamination enthalten. Dieser Aspekt liegt darin begründet, dass sich Aluminiumhydroxid-Komplexe bilden, welche die Agglomerate fest verbinden. Diese festen Agglomerate lassen sich in einem herkömmlichen Prozess nicht vollständig vermahlen, was zu einem Materialverlust führt. Entsprechend hergestellte Aluminium-kontaminierte Übergangsmetallsalzlösungen müssten so vor der Vermahlung kostenintensiv aufgereinigt werden. Bei einer Reduktion des Aluminiumsgehalts auf unter 0,1% ist dieser Effekt nicht mehr zu beobachten. Mit dem erreichten Resultat einer Al-Kontamination von nur 0, 05–0,06 Massen-% lassen sich die Materialien demnach ökologisch und ökonomisch vorteilhaft recyceln. Dadurch, dass beim Aufprall auf das Sieb beim Luftstrahlsieben Aktivmaterial abgeklopft wird, lassen sich sehr hohe Reinheiten für den Träger, insbesondere die Trägerfolie, erreichen.

[0008] Der Erfindung liegt die Erkenntnis zugrunde, dass das Erwärmen der Zellenfragmente auf die Zersetzungstemperatur zwar theoretisch ausreichen müsste, um den Binder so weit zu zerstören, dass das Aktivmaterial freigesetzt wird, dass aber dennoch das Aktivmaterial nur schwer vom Träger zu lösen ist. Aufgrund welchen Effekts das Aktivmaterial auch ohne Binder am Träger anhaftet, ist bislang noch ungeklärt.

[0009] Beim Luftstrahlsieben kommt es zu einem heftigen Aufprall des Trägers mit noch anhaftendem Aktivmaterial auf das Sieb. Dieser Aufprall löst noch anhaftendes Aktivmaterial ab, so dass es wiedergewonnen werden kann.

Ascend Elements EX1002 Page 552

Duesenfeld Ex. 2014-552

[0010] Das Luftstrahlsieben ist bekannt als Laborverfahren zum Trennen von kleinen Partikeln von anderen kleinen Partikeln. Die wärmebehandelten Zellenfragmente haben jedoch einen um Größenordnungen größeren Durchmesser als die Partikel des Aktivmaterials, so dass die Fähigkeit des Luftstrahlsiebens zur Trennung von zwei Arten von Partikeln mit jeweils kleinen Durchmessern für das erfindungsgemäße Verfahren irrelevant ist. Im erfindungsgemäßen Verfahren wird das Luftstrahlsieben auch als zusätzlicher Trennschritt eingesetzt.

[0011] Im Rahmen der vorliegenden Beschreibung wird unter dem Zerkleinern der Zellen insbesondere ein Zerschneiden, Zerschlagen, Zerhäckseln oder Zerdrücken verstanden. Das kann bei Temperaturen zwischen –195,79°C–100°C erfolgen, so dass nur geringe Anforderungen hinsichtlich des Temperaturregimes bestehen.

[0012] Unter dem Erwärmen der festen Zellenfragmente wird insbesondere ein Erwärmen in einem gasdichten Behälter verstanden, so dass beispielsweise Sauerstoff am Zutritt gehindert wird. Insbesondere erfolgt das Erwärmen unter Inertgas oder im Vakuum.

[0013] Unter dem Aktivmaterial wird insbesondere eine Verbindung verstanden, welche Lithiumionen reversibel aufnehmen und abgeben kann. Auf der Kathodenseite beispielsweise Lithiumnickelmangankobaltoxid, Lithiumnickelaluminiumoxid und Lithiumeisenphosphat, auf der Anodenseite beispielsweise Graphit.

[0014] Unter dem Träger für das Aktivmaterial wird insbesondere eine Trägerfolie verstanden, auf der das Aktivmaterial in Form von Partikeln aufgebracht ist. Beispielsweise handelt es sich bei der Trägerfolie um Aluminiumfolie. Bei dem Binder handelt es sich beispielsweise um Polyvenylidenfluorid (PVDF).

[0015] Das Zerkleinern der Zellen und/oder das Erwärmen der festen Zellenfragmente erfolgt bevorzugt unter Inertgas, beispielsweise unter Stickstoff. Günstig ist es, wenn das Zerkleinern der Zellen unter Umgebungstemperatur, insbesondere unter bei höchstens 30°, erfolgt, wobei es vorteilhaft sein kann, wenn die Zerkleinerungsvorrichtung, mit der die Zellern zerkleinert werden, gekühlt wird, so dass der Wärmeeinfluss auf die Zellen gering bleibt.

[0016] Das erfindungsgemäße Verfahren wird vorzugsweise als kontinuierlicher Prozess durchgeführt, also nicht chargenweise.

[0017] Vorzugsweise beträgt der Durchsatz mehr als 100 Tonnen pro Jahr. Das Luftstrahlsieben wird bislang im Labormaßstab eingesetzt, wo es zur chargenweisen Bearbeitung von Proben genutzt wird. Diese Proben sind zudem sehr klein. Der Einsatz des Luftstrahlsiebens in einem Massenverfahren ist bislang unbekannt.

[0018] Gemäß einer bevorzugten Ausführungsform werden galvanische Zellen verwendet, deren Aktivmaterial aus Partikeln gebildet ist, wobei die Partikel eine Partikeldurchmesserverteilung haben und wobei diese Partikeldurchmesserverteilung einen 90%-Durchmesser besitzt, der demjenigen Durchmesser entspricht, der größer ist als der Durchmesser von 90% aller Partikel und der kleiner ist als der Durchmesser von 10% alter Partikel, wobei das Luftstrahlsieben mit einem Sieb erfolgt, dessen Maschenweite maximal dem 200-fachen des 90%-Durchmessers entspricht. Der angegebene Partikeldurchmesser ist derjenige, der durch Siebung erhalten wird.

[0019] Vorzugsweise beträgt die Maschenweite des Siebs höchstens 100 µm. Moderne Lithium-Ionen-Batterien besitzen ein Aktivmaterial, das aus Partikeln mit einem Durchmesser von weniger als 200 µm aufgebaut ist. Bei klassischen Siebverfahren, beispielsweise dem Vibrationssieben, muss eine deutlich größere Maschenweite gewählt werden, damit nicht die Siebzeit so groß wird, dass das Verfahren unwirtschaftlich wird. Eine größere Maschenweite jedoch hat zur Folge, dass mehr Schmutzpartikel durch das Sieb gelangen, so dass die Reinheit des gewonnen Aktivmaterials sinkt.

[0020] Vorzugsweise umfassen die galvanischen Zellen je zumindest eine Anode und zumindest eine Kathode, wobei die Anoden und Kathoden gemeinsam erwärmt und die entstehenden wärmebehandelten Zellenfragmente in einem gemeinsamen Luftstrahlsieb klassiert werden. Unter dem Merkmal, dass die Anoden und die Kathoden gemeinsam erwärmt werden, wird verstanden, dass ein Gemisch aus beiden in ein und derselben Vorrichtung erwärmt wird. Das ist möglich, da das Luftstrahlsieben zu einer hinreichend sortenreinen Trennung von Stromsammlerfolie und Beschichtungspartikeln führt.

[0021] Vorzugsweise umfasst das Verfahren den Schritt, dass vor dem Erwärmen der Zellenfragmente auf die Zersetzungstemperatur die Zellenfragmente bei einer Trocknungs-Temperatur getrocknet werden, so dass trockene Zellenfragmente und Trocknungsdämpfe entstehen, wobei zumindest ein Teil der Trocknungsdämpfe kondensiert wird. Das hat den Vorteil, dass das Elektrolytlöschungsmittel zurückgewonnen werden kann. Es ist dabei vorteilhaft, wenn die Trocknungs-Temperatur unter 100°C liegt, insbesondere unter 80°C.

[0022] Vorzugsweise wird das Erwärmen der Zellenfragmente auf die Zersetzungs-Temperatur unter Inertgas oder im Vakuum durchgeführt. Das hat den Vorteil, dass Kupfer dann nicht oxidiert, so dass so-

Ascend Elements EX1002 Page 553

Duesenfeld Ex. 2014-553

wohl die Anode als auch die Kathode gemeinsam verarbeitet werden können. Das wiederum führt zu einem besonders einfachen Trennverfahren, das dennoch hoch sortenreine Recyclingprodukte zur Verfügung stellt. Unter Inertgas wird ein Gas oder Gasgemisch verstanden, das unter den vorliegenden Umgebungsbedingungen weder mit Kupfer noch mit Aluminium (Stromsammelfolien) reagiert. Das Kupfer kann so als wertvolle Komponente in hoher Reinheit zurückgewonnen werden.

[0023] Vorzugsweise umfasst das Verfahren den Schritt eines Behandelns der trockenen Zellfragmente mittels Magnetabscheider, so dass eisenfreie Zellenfragmente entstehen. Günstig ist es, wenn diese eisenfreien Zellenfragmente gesichtet werden, beispielsweise durch einen Zickzacksichter. Die eisenfreien Zellenfragmente werden dann auf die Zersetzungs-Temperatur des Binders erhitzt.

[0024] Die Zersetzungs-Temperatur beträgt vorzugsweise zumindest 350°C und höchstens 800°C. Es hat sich herausgestellt, dass bei diesen Temperaturen der Binder sicher zersetzt wird.

[0025] Vorzugweise wird beim Zerkleinern der Zellen unter Inertgas zumindest auch das Elektrolytmittel abgezogen. Das steigert die Recyclingquote.

[0026] Gemäß einer bevorzugten Ausführungsform wird das Zerkleinern der Zellen so durchgeführt, dass der Median der längsten Durchmesser der resultierenden Folienfragmente 200 µm nicht unterschreitet. Gemäß einer bevorzugten Ausführungsform wird das Zerkleinern der Zellen so durchgeführt, dass die Folienfragmente zu zumindest 80% einen Durchmesser von über 200 µm haben, wobei der Durchmesser durch Siebung bestimmt wird

[0027] Die Aktivmaterial-Separationsanlage besitzt vorzugsweise einen Vortrockner, der im Materialfluss hinter der Zellenzerkleinerungsvorrichtung und vor dem Ofen angeordnet ist. Der Vortrockner ist eingerichtet, um die Zellenfragmente auf die Trocknungs-Temperatur zu erwärmen.

[0028] Vorzugsweise umfasst die Aktivmaterial-Separationsanlage zudem einen Magnetabscheider im Materialfluss hinter dem Vortrockner und vor dem Ofen.

[0029] Günstig ist es, wenn die Aktivmaterial-Separationsanlage einen Rührreaktor zur Leitsalzrückgewinnung aufweist, der mit der Zellenzerkleinerungsvorrichtung verbunden ist und aus der Zellenzerkleinerungsvorrichtung zumindest einen Teil der Flüssigfraktion abzieht.

[0030] Unter dem Verfahren zum Wiedergewinnen von Aktivmaterial der galvanischen Zelle wird insbesondere auch ein Verfahren verstanden, bei dem Elektroden aus der Elektrodenproduktion bearbeitet werden, ohne dass die Elektroden in eine galvanische Zelle eingebaut worden sind, jedoch in eine galvanische Zelle eingebaut werden könnten. In anderen Worten kann es sich bei dem Verfahren auch um ein Verfahren zum In-Prozess-Recycling handeln.

[0031] Erfindungsgemäß ist zudem die Verwendung eines Luftstrahlsiebs in einem Verfahren, insbesondere einem kontinuierlichen Verfahren, zum Wiedergewinnen von Aktivmaterial, insbesondere von Lithium und/oder Lithiumverbindungen, beispielsweise aus gebrauchten Batterien und/oder Akkumulatoren sowie aus Ausschuss bei der Produktion von Batterien und/oder Akkumulatoren.

[0032] Im Folgenden wird die Erfindung anhand der beigefügten Zeichnungen näher erläutert. Dabei zeigt

[0033] Fig. 1 ein Flussdiagramm einer erfindungsgemäßen Aktivmaterial-Separationsanlage zum Durchführen eines erfindungsgemäßen Verfahrens.

[0034] Fig. 1 zeigt ein Flussdiagramm einer erfindungsgemäßen Aktivmaterial-Separationsanlage **10**, die eine Zellenzerkleinerungsvorrichtung **12** besitzt, der galvanische Zellen **14.1**, **14.2**, ... zugeführt werden. Die Zellenzerkleinerungsvorrichtung **12** ist gasdicht und umfasst eine Gaswaschkolonne **16**, eine Umwälzpumpe **18** und einen Kondensator **20**, mittels denen ein Inertgas, beispielsweise Stickstoff, umgewälzt und gereinigt wird. Die Zellenzerkleinerungsvorrichtung umfasst zudem einen Inertgastank **22** zum Einstellen eines Inertgasdrucks in der Zellenzerkleinerungsvorrichtung **12**. Der Kondensator **20** besitzt einen Elektrolytabzug **24**, über den kondensierter Elektrolyt abgezogen werden kann.

[0035] In Materialstromrichtung hinter der Zellenzerkleinerungsvorrichtung **12** kann ein Rührreaktor **26** angeordnet sein, der zum Abziehen einer Flüssigfraktion aus der Zellenzerkleinerungsvorrichtung **12** und zum Rühren derselben angeordnet ist, so dass Leitsalz aus den galvanischen Zellen **14** (Bezugszeichen ohne Zählsuffix bezeichnen das Objekt als solches) zurückgewonnen werden kann.

[0036] In Materialflussrichtung hinter der Zellenzerkleinerungsvorrichtung **12** ist ein Vortrockner **28** angeordnet, der über eine Leitung **30** mit der Zellenzerkleinerungsvorrichtung **12** verbunden ist. Beim Zerkleinern der galvanischen Zellen **14** entstandene Zellenfragmente gelangen durch die Leitung **30** in den Vortrockner **28**. Dort herrscht eine Trocken-Temperatur T_t von vorzugsweise $T_t = 80°C$. Der Vortrockner **28** umfasst eine Umwälzpumpe **32** und einen Kondensator **34** zum Abkondensieren von Elektrolyt, der über eine Leitung **36** abgezogen werden kann. Der

Vortrockner **28** ist gasdicht ausgebildet und wird mit Inertgas durch die Umwälzpumpe **32** gespült.

[0037] In Materialstromrichtung hinter dem Vortrockner **28** ist ein Abscheider **38** angeordnet, der einen Magnetabscheider und gegebenenfalls zusätzlich einen Wirbelstromabscheider umfasst. Das magnetische Material wird über eine Ableitung **40** abgezogen. Das nicht magnetische Material gelangt in einen Sichter **42**, beispielsweise einen Zickzacksichter. Der Sichter **42** ist wie der Abscheider **38** optional und besonders dann vorteilhaft, wenn wie im vorliegenden Fall nur die Kathode der galvanischen Zellen weiterverwendet werden soll. Die Anoden werden ebenso wie gegebenenfalls vorliegende Pouchfolien abgezogen.

[0038] In Materialflussrichtung hinter dem Sichter **42** ist ein Ofen **44** angeordnet, in dem eine Zersetzungstemperatur T_z von 400 bis 600°C herrscht. Dadurch zersetzt sich der Binder, beispielsweise Polyvenylidenfluorid (PVDF). Das Zersetzungsprodukt wird über eine Ableitung **46** abgeleitet und in eine Gaswaschkolonne **48** geführt.

[0039] In Materialflussrichtung hinter dem Ofen **44** ist ein Luftstrahlsieb **50** angeordnet. Das Luftstrahlsieb **50** umfasst ein Siebelement **52**. Die aus dem Ofen **44** kommenden wärmebehandelten Zellenfragmente werden auf ein Luftstrahlsieb gefördert. Dieses ist dadurch charakterisiert, dass ober- oder unterhalb des feinmaschigen Siebelements (20–200 µm) ein Luftstrom zugeführt wird. Dieser wird unterhalb des Siebes abgezogen. Durch die Luftströmung werden die Elektrodenfragmente aufgewirbelt und dann mechanisch beansprucht. Diese Beanspruchung unterstützt die Separation von Trägerfolie und Beschichtung. Die Aktivmaterial-Partikel werden so im Luftstrom durch die Maschen abzogen und im Folgenden durch einen Zyklon aus dem Luftstrom abgetrennt. Die Folienfraktion (Aluminium und Kupfer) wird durch das Siebelement zurückgehalten und kann als Metallfraktion gewonnen und wieder verwendet werden.

Bezugszeichenliste

10 Aktivmaterial-Separationsanlage
12 Zellenzerkleinerungsvorrichtung
14 galvanische Zelle
16 Gaswaschkolonne
18 Umwälzpumpe
20 Kondensator
22 Inertgastank
24 Elektrolytabzug
26 Rührreaktor
28 Vortrockner
30 Leitung
32 Umwälzpumpe
34 Kondensator
36 Leitung
38 Abscheider
40 Ableitung
42 Sichter
44 Ofen
46 Ableitung
48 Gaswaschkolonne
50 Luftstrahlsieb
52 Siebelement
54 Partikel
56 Ableitung
58 Zyklon
T_t Trocken-Temperatur
T_z Zersetzungs-Temperatur

Ascend Elements EX1002 Page 555

Duesenfeld Ex. 2014-555

ZITATE ENTHALTEN IN DER BESCHREIBUNG

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Patentansprüche

1. Verfahren zum Wiedergewinnen von Aktivmaterial einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial und einen Binder zum Verbinden des Aktivmaterials und des Trägers umfasst, mit den Schritten:
(a) Zerkleinern der Zellen, insbesondere unter Inertgas oder im Vakuum, so dass zumindest auch feste Zellenfragmente entstehen,
(b) Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur (T_z), die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, vorzugsweise unter Intergas oder im Vakuum so dass wärmebehandelte Zellenfragmente entstehen, und
(c) Klassieren der wärmebehandelten Zellenfragmente,
dadurch gekennzeichnet, dass
(d) das Klassieren ein Luftstrahlsieben umfasst.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass
– galvanische Zellen (**14**) verwendet werden, deren Aktivmaterial aus Partikeln gebildet ist,
– die Partikel eine Partikeldurchmesserverteilung haben, wobei die Partikeldurchmesserverteilung einen 90%-Durchmesser besitzt, der demjenigen Durchmesser entspricht, der größer ist als der Durchmesser von 90% aller Partikel und der kleiner ist als der Durchmesser von 10% aller Partikel, und
– das Luftstrahlsieben mit einem Sieb erfolgt, dessen Maschenweite maximal dem 200-fachen des 90%-Durchmessers entspricht.

3. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die Maschenweite des Siebs höchstens 200 µm beträgt.

4. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die galvanischen Zellen (**14**) je zumindest eine Anode und zumindest eine Kathode umfassen und die Anoden und Kathoden gemeinsam erwärmt werden und die entstehenden wärmebehandelten Zellenfragmente in einem gemeinsamen Luftstrahlsieb (**50**) klassiert werden.

5. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch den Schritt:
(a2) vor dem Erwärmen der Zellenfragmente auf die eine Zersetzungs-Temperatur (T_z)
– Trocknen der Zellenfragmente bei einer Trocknungs-Temperatur (T_t), so dass trockene Zellenfragmente und Trocknungsdämpfe entstehen und
– Kondensieren zumindest eines Teils der Trocknungsdämpfe zum Zweck der Rückgewinnung der Elektrolytlösemittel.

6. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Erwärmen der Zellenfragmente auf die Zersetzungs-Temperatur (T_z) unter Inertgas oder in einem Vakuum durchgeführt wird.

7. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch die Schritte:
(a3) Behandeln der trockenen Zellenfragmente mittels Magnetabscheidung, so dass eisenfreie Zellenfragmente entstehen, und
(a4) Sichten der eisenfreien Zellenfragmente.

8. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass die galvanischen Zellen (**14**) einen Elektrolyten aufweisen und/oder beim Zerkleinern der Zellen unter Inertgas zumindest auch der Elektrolyt abgezogen wird.

9. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Zerkleinern der Zellen so durchgeführt wird, dass der Median der längsten Durchmesser der resultierenden Folienfragmente 200 µm nicht unterschreitet.

10. Aktivmaterial-Separationsanlage, insbesondere Aktivmetall-Separationsanlage (**10**), die ausgebildet ist zum Wiedergewinnen von Aktivmaterial, insbesondere Lithium, einer galvanischen Zelle (**14**), die ein Aktivmaterial, einen Träger für das Aktivmaterial, einen Binder zum Verbinden des Aktivmaterials und des Trägers und einen Elektrolyten umfasst, mit
(a) einer Zellenzerkleinerungsvorrichtung (**12**) zum Zerkleinern der Zellen, wobei die Zellenzerkleinerungsvorrichtung gasdicht ist und eine Inertgaszuführvorrichtung umfasst, so dass die Zellen unter Inertgas zu Zellenfragmenten zerkleinerbar sind, und
(b) einem Ofen (**44**), der zumindest mittelbar mit der Zellenzerkleinerungsvorrichtung (**12**) verbunden und eingerichtet ist zum Erwärmen der Zellenfragmente auf eine Zersetzungs-Temperatur (T_z), die so hoch gewählt ist, dass der Binder sich zersetzt,
gekennzeichnet durch
(c) eine Luftstrahlsiebvorrichtung, die zumindest mittelbar mit dem Ofen (**44**) verbunden und eingerichtet ist zum Klassieren von Aktivmaterial und Träger.

Es folgt ein Blatt Zeichnungen

Ascend Elements EX1002 Page 557

Duesenfeld Ex. 2014-557

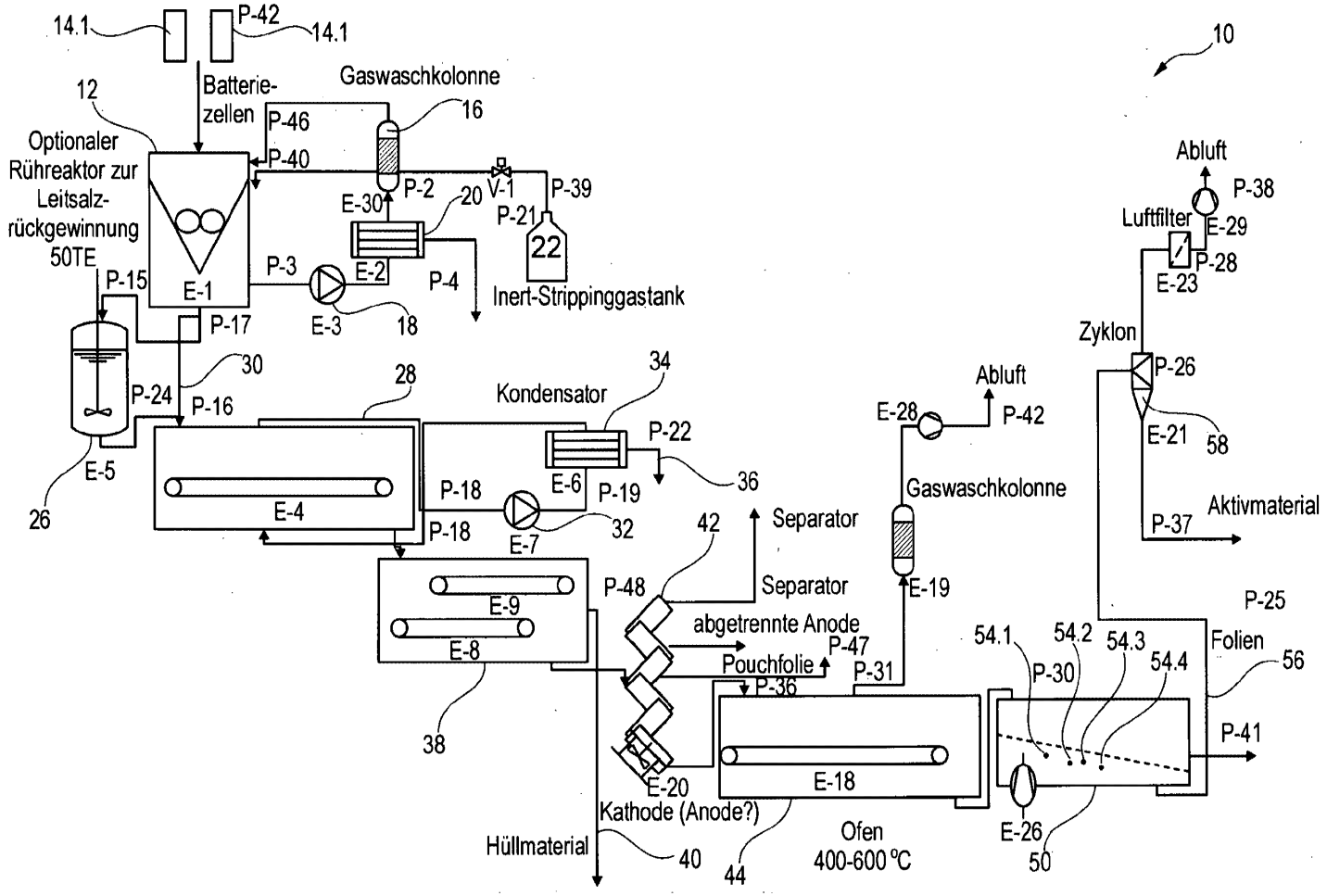


Fig. 1

DE 10 2011 110 083 A1 2013.02.14

Espacenet

Bibliographic data: DE102011110083 (A1) — 2013-02-14

Verfahren zum Wiedergewinnen von Aktivmaterial aus einer galvanischen Zelle und Aktivmaterial-Separationsanlage, insbesondere Aktivmetall-Separationsanlage

Inventor(s): HANISCH CHRISTIAN [DE]; HASELRIEDER WOLFGANG [DE]; KWADE ARNO [DE] ± (HANISCH, CHRISTIAN, ; HASELRIEDER, WOLFGANG, ; KWADE, ARNO, ; Hanisch, Christian, ; Haselrieder, Wolfgang, ; Kwade, Arno)

Applicant(s): TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA [DE] ± (TECHNISCHE UNIVERSITAET BRAUNSCHWEIG CAROLO-WILHELMINA, ; Technische Universität Braunschweig Carolo-Wilhelmina)

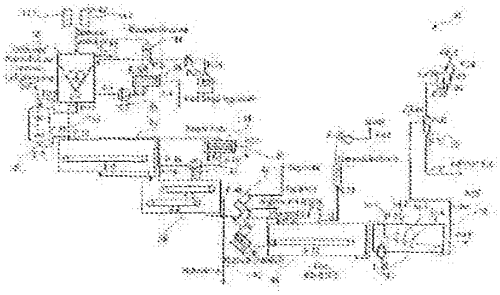
Classification: - international: ***H01M10/54; H01M6/52***
- cooperative: **B02C23/06**; **B02C23/08**; **B07B4/08**; **C22B26/12**; **H01M10/54**; B02C23/00; H01M10/052; Y02W30/84

Application number: DE201110110083 20110812

Priority number(s): DE201110110083 20110812

Also published as: DE102011110083 (B4) CN103959553 (A) CN103959553 (B) EP2742559 (A1) EP2742559 (B1) US2014290438 (A1) US9780419 (B2) WO2013023640 (A1) less

Abstract of DE102011110083 (A1)



Verfahren zum Wiedergewinnen von Aktivmaterial einer galvanischen Zelle, die ein Aktivmaterial, einen Träger für das Aktivmaterial und einen Binder zum Verbinden des Aktivmaterials und des Trägers umfasst, mit den Schritten: Zerkleinern der Zellen, insbesondere unter Inertgas oder im Vakuum, so dass zumindest auch feste Zellenfragmente entstehen, Erwärmen der festen Zellenfragmente auf eine Zersetzungs-Temperatur (Tz), die so hoch gewählt ist, dass der Binder sich zersetzt und/oder verdampft, vorzugsweise unter Intergas oder im Vakuum so dass wärmebehandelte Zellenfragmente entstehen, und Klassieren der wärmebehandelten Zellenfragmente, wobei das Klassieren ein Luftstrahlsieben umfasst.

Espacenet

Bibliographic data: US9780419 (B2) — 2017-10-03

Method for reclaiming active material from a galvanic cell, and an active material separation installation, particularly an active metal separation installation

Inventor(s): HANISCH CHRISTIAN [DE]; HASELRIEDER WOLFGANG [DE]; KWADE ARNO [DE] ± (HANISCH CHRISTIAN, ; HASELRIEDER WOLFGANG, ; KWADE ARNO, ; Hanisch Christian, ; Haselrieder Wolfgang, ; Kwade Arno)

Applicant(s): HANISCH CHRISTIAN [DE]; HASELRIEDER WOLFGANG [DE]; KWADE ARNO [DE]; LION ENG GMBH [DE] ± (HANISCH CHRISTIAN, ; HASELRIEDER WOLFGANG, ; KWADE ARNO, ; Hanisch Christian, ; Haselrieder Wolfgang, ; Kwade Arno, ; Lion Engineering GmbH)

Classification: - international: ***B02C23/00; B02C23/06; B02C23/08; B07B4/08; C22B26/12; H01M10/54;*** *H01M10/052*
- cooperative: **B02C23/06**; **B02C23/08**; **B07B4/08**; **C22B26/12**; **H01M10/54**; B02C23/00; H01M10/052; Y02W30/84

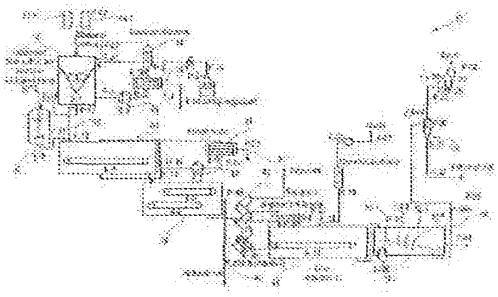
Application number: US201214238479 20120810 Global Dossier

Priority number(s): DE201110110083 20110812 ; WO2012DE00811 20120810

Also published as: CN103959553 (A) CN103959553 (B) DE102011110083 (A1) DE102011110083 (B4) EP2742559 (A1) more

Abstract of US9780419 (B2)

A method for retrieving active material from a galvanic cell is provided. The galvanic cell includes an active material, a support for the active material and a binder for bonding the active material and the support. The method includes the following steps: (a) crushing the cells, in particular under inert gas or in a vacuum, so that solid cell fragments are also formed, (b) heating the solid cell fragments up to the decomposition temperature (Tz), which is high enough to make the binder decompose so that it loses its binding properties, preferably under inert gas or in a vacuum, such that heat treated cell fragmented are formed, and (c) classifying the heat treated cell fragments, whereby (d) the classifying comprises air jet sieving and (e) the air jet sieving is carried out in such a way that the active material is separated from the support.



(19) Deutsches Patent- und Markenamt

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(12) **Offenlegungsschrift**

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(56) Ermittelter Stand der Technik:

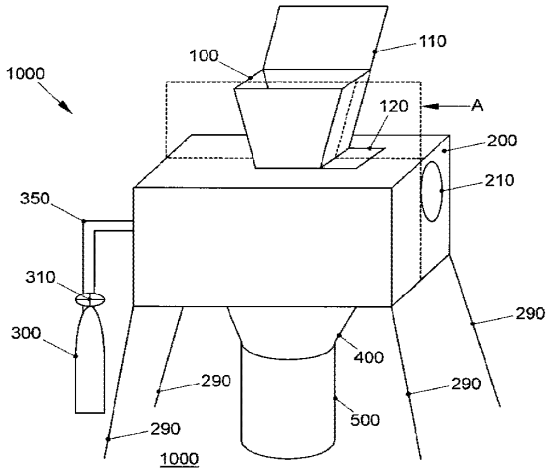
DE	**60 2005 001 937**	**T2**
US	**5 303 872**	**A**
EP	**1 333 522**	**A1**
WO	**2010/ 149 611**	**A1**

Rechercheantrag gemäß § 43 Abs. 1 Satz 1 PatG ist gestellt.

Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen

(54) Bezeichnung: **Vorrichtung und Verfahren zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand**

(57) Zusammenfassung: Die Erfindung betrifft ein System und ein Verfahren zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand.
Es ist vorgesehen, dass das System eine Vorrichtung zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand umfasst. Dabei umfasst die Vorrichtung eine Zerkleinerungsvorrichtung (200) zum Zerkleinern der Elektrolytzellen in einem von der Zerkleinerungsvorrichtung (200) umfassten Zerkleinerungsraum, eine Zuführvorrichtung (100) zum Zuführen von Elektrolytzellen in den Zerkleinerungsraum, eine Materialaustragvorrichtung (400) zum Austragen zerkleinerter Elektrolytzellen aus dem Zerkleinerungsraum, eine Inertgaszuführvorrichtung (300) zum Erzeugen einer im Wesentlichen Inertgas enthaltenden Atmosphäre zumindest im Zerkleinerungsraum und in der Materialaustragvorrichtung (400), sowie eine Gasabsaugvorrichtung (240) zum Absaugen der im Zerkleinerungsraum enthaltenden Atmosphäre. Die Gasabsaugvorrichtung (240) umfasst eine Gasreinigungsvorrichtung (220) und die Zuführvorrichtung (100) ist zum gasdichten Zuführen der zu zerkleinernden Elektrolytzellen in den Zerkleinerungsraum geeignet. Die Vorrichtung ist dadurch gekennzeichnet, dass die Materialaustragvorrichtung (400) eine Öffnung zur gasdichten Anbringung einer transportablen Auffangvorrichtung (500) zum Auffangen zerkleinerter Zellen umfasst.



Beschreibung

[0001] Die Erfindung betrifft eine Vorrichtung und ein Verfahren zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand.

[0002] Elektrolytzellen, beispielsweise Lithium-Ionen basierte Batteriezellen, finden in tragbaren Elektrogeräten oder in automobilen Anwendungen zunehmende Verwendung als verlässliche Energiespeicher. Werden Elektrolytzellen jedoch beschädigt, können sich aus dem Elektrolyt unter Zusammenwirken mit den Elektroden, dem Gehäuse und/oder der Umgebungsluft chemische Verbindungen mit hohen Umwelt- und Gesundheitsgefährdungspotential entwickeln. Vergleichbare Gefährdungen können von überalterten Elektrolytzellen ausgehen.

[0003] Daher dürfen selbst intakte Elektrolytzellen nur als Gefahrengut transportiert werden, sofern sie nicht in einem Kraftfahrzeug installiert sind. Defekte oder überalterte Elektrolytzellen dürfen nur nach Einzelfallprüfung durch einen technischen Fachmann und Oft nur mit Einzelausnahmegenehmigung unter Auflagen transportiert werden, sofern sie sich nicht in einem Kraftfahrzeug befinden. Ist nun das Kraftfahrzeug selbst defekt, ist auch bei im Kraftfahrzeug befindlichen Elektrolytzellen eine Einzelfallprüfung der Transportfähigkeit durch den technischen Fachmann notwendig. Defekte Elektrolytzellen, überalterte Elektrolytzellen und aus anderen Gründen nicht transportfähige Elektrolytzellen werden daher auch als transportkritische Elektrolytzellen bezeichnet.

[0004] Für das Recycling werden für den Regelbetrieb unbrauchbare Elektrolytzellen geschreddert, also zerkleinert. Bei der Zerkleinerung von Elektrolytzellen unter Normalatmosphäre kann sich das Elektrolyt beziehungsweise ein das Elektrolyt umfassendes Lösungsmittel durch freiwerdende elektrische Ladungen und einhergehende Lichtbögen leicht entzünden. Ein so entstandenes Feuer greift dann leicht auf andere brennbare Komponenten der Zelle, beispielsweise Kunststoffseparatoren, über. Bei Lithium-Ionen Zellen geht ein solches Feuer mit einer Exposition von Fluorwasserstoff einher.

[0005] In der europäischen Patentschrift 1 733 451 wird ein Verfahren beschrieben, das eine Trockenzerkleinerung eines Lithiumsystems bei Umgebungstemperatur und unter inerter Atmosphäre und eine Lithiumrückgewinnung durch wässrige Hydrolyse umfasst. Dabei sind ein Drehrotor und eine Drehschere in einem abgeschlossenen Behälter untergebracht, der mittels Gas inertisiert wird. Gase, die während der Zerkleinerung austreten, werden durch Spülen mit Wasser und Neutralisation mittels Natriumkarbonat behandelt. Die europäische Patentschrift 0 649 912 befasst sich mit einem Verfahren zur Rückgewinnung von verwertbaren Metallen aus einer Nickel-Wasserstoffzelle.

[0006] Der Erfindung liegt nun die Aufgabe zugrunde, eine Vorrichtung und ein Verfahren zur Verfügung zu stellen, welches eine Transportsicherheitsprüfung von transportkritischen Elektrolytzellen überflüssig macht.

[0007] Diese Aufgabe wird erfindungsgemäß durch die Bereitstellung einer Vorrichtung mit den Merkmalen des Anspruchs 1, einer transportablen Auffangvorrichtung mit den Merkmalen des Anspruchs 4, eines Systems mit den Merkmalen des Anspruchs 6 und eines Verfahrens mit den Merkmalen des Anspruchs 7 gelöst.

[0008] Die erfindungsgemäße Vorrichtung ist zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand geeignet und umfasst eine Zerkleinerungsvorrichtung zum Zerkleinern der Elektrolytzellen in einem von der Zerkleinerungsvorrichtung umfassten Zerkleinerungsraum, eine Zuführvorrichtung zum Zuführen von Elektrolytzellen in den Zerkleinerungsraum und eine Materialaustragvorrichtung zum Austragen zerkleinerter Elektrolytzellen aus dem Zerkleinerungsraum. Der Zerkleinerungsraum kann mittels einer Zuführschleuse gasdicht von der Zuführvorrichtung getrennt werden. Eine weiterhin von der Vorrichtung umfasste Materialaustragvorrichtung dient zum Austragen zerkleinerter Elektrolytzellen aus dem Zerkleinerungsraum, eine ebenfalls umfasste Inertgaszuführvorrichtung dient zum Erzeugen einer im Wesentlichen Inertgas enthaltenden Atmosphäre zumindest im Zerkleinerungsraum und in der Materialaustragvorrichtung, und eine ebenfalls umfasste Gasabsaugvorrichtung dient zum Absaugen der im Zerkleinerungsraum enthaltenden Atmosphäre, wobei die Gasabsaugvorrichtung eine Gasreinigungsvorrichtung umfasst. Die Vorrichtung ist dadurch gekennzeichnet, dass die Materialaustragvorrichtung eine Öffnung zur gasdichten Anbringung einer transportablen Auffangvorrichtung zum Auffangen zerkleinerter Zellen umfasst.

[0009] Die Vorrichtung ermöglicht, eine gasdichte Verbindung einer solchen Vorrichtung mit einer transportablen Auffangvorrichtung. Dann kann eine der Größe der Auffangvorrichtung angepasste Menge Elektrolytzellen unter Inertgasatmosphäre sicher zerkleinert und in die Auffangvorrichtung überführt werden. Zudem sind Wertstoffe in den zerkleinerten Elektrolytzellen einer Wiedergewinnung zugänglich.

[0010] In bevorzugter Ausgestaltung der Erfindung ist vorgesehen, dass die Materialaustragvorrichtung mindestens eine Bestäubungsvorrichtung zum Bestäuben der zerkleinerten Zellen mit einem Desaktivierungspulver umfasst.

Duesenfeld Ex. 2014-562

[0011] Dann gelangt nur desaktiviertes Elektrolytzellenmaterial in die Auffangvorrichtung, die dann nach Absaugen der Inertgasatmosphäre von der Vorrichtung gelöst, verschlossen und ohne spezielle Transportsicherheitsprüfung oder Ausnahmegenehmigung als Gefahrengut transportiert werden kann. Dies ermöglicht insbesondere auch Transporte über Ländergrenzen hinweg.

[0012] In dieser oder einer anderen Ausführungsform ist die Vorrichtung transportabel und umfasst eine Energieversorgungsvorrichtung, die eine standortunabhängige Energieversorgung ermöglicht.

[0013] Die erfindungsgemäße transportable Auffangvorrichtung ist dadurch gekennzeichnet, dass die Auffangvorrichtung zum Überführen transportkritischer Lithium-Ionen-Zellen in einen transportfähigen Zustand mit der erfindungsgemäßen Vorrichtung vorgesehen ist und eine vorbestimmte Menge Lithiumhydroxid-Lösung enthält, wobei die enthaltene Menge Lithiumhydroxid-Lösung und ein Volumen der Auffangvorrichtung so aufeinander abgestimmt sind, dass eine Menge Fluor, die in einer Menge in die Auffangvorrichtung passender zerkleinerter Lithium-Ionen-Zellen enthalten ist, durch die Menge Lithiumhydroxid-Lösung in der Auffangvorrichtung vollständig gebunden und/oder desaktiviert wird.

[0014] In einer bevorzugten Ausführungsform der erfindungsgemäßen transportablen Auffangvorrichtung ist das Volumen der Auffangvorrichtung zusätzlich auf ein Volumen der Zuführvorrichtung abgestimmt, so dass die Menge die Auffangvorrichtung passender zerkleinerter Lithium-Ionen-Zellen einer Menge unzerkleinert in die Zuführvorrichtung passender Lithium-Ionen-Zellen entspricht.

[0015] Das erfindungsgemäße System zum Überführen transportkritischer Lithium-Ionen-Zellen in einen transportfähigen Zustand umfasst die erfindungsgemäße Vorrichtung und die erfindungsgemäße transportable Auffangvorrichtung.

[0016] Das erfindungsgemäße Verfahren umfasst die Verwendung der erfindungsgemäßen Vorrichtung, wobei an die Öffnung der Materialaustragvorrichtung eine transportable Auffangvorrichtung gasdicht angebracht ist. Das Verfahren umfasst weiterhin die folgenden Schritte:
Erzeugen der im Wesentlichen inertgashaltigen Atmosphäre im Zerkleinerungsraum, in der Materialaustragvorrichtung und in der Auffangvorrichtung, Befüllen der Zuführvorrichtung mit einer Charge Elektrolytzellen, gasdichtes Schließen der Zuführvorrichtung, Zuführen der Elektrolytzellen in den Zerkleinerungsraum, Zerkleinern der Zellen unter der inertgashaltigen Atmosphäre und Austragen der zerkleinerten Zellen in die Auffangvorrichtung unter der inertgashaltigen Atmosphäre, Absaugen der inertgashaltigen Atmosphäre, Abtrennen der Auffangvorrichtung von der Öffnung und Verschließen der Auffangvorrichtung.

[0017] In einer bevorzugten Ausführungsform ist vorgesehen, dass das Verfahren weiterhin folgende Schritte umfasst, die vor der Erzeugung der im Wesentlichen Inertgas enthaltenden Atmosphäre ausgeführt werden:
Befüllen der Auffangvorrichtung mit einer zur Desaktivierung und/oder Bindung eines Elektrolyts der Charge geeigneten Lösung, wobei ein Volumen der Auffangvorrichtung und ein Volumen der Zuführvorrichtung so aufeinander abgestimmt, dass eine Menge in die Zuführvorrichtung passender Elektrolytzellen nach Zerkleinern in das Volumen der Auffangvorrichtung passt, und wobei die zum Befüllen verwendete Menge an Lösung zur Desaktivierung und/oder Bindung einer in der Charge enthaltenen Elektrolytmenge ausreichend ist, und gasdichtes Anbringen der transportablen Auffangvorrichtung an der Öffnung.

[0018] In einer anderen bevorzugten Ausführungsform ist vorgesehen, dass das Verfahren weiterhin folgenden Schritt umfasst, der unter der im Wesentlichen Inertgas enthaltenden Atmosphäre ausgeführt wird:
Bestäuben der zerkleinerten Zellen mit einem Desaktivierungspulver, wobei die zum Bestäuben verwendete Menge an Desaktivierungspulver zur Desaktivierung einer in der Charge enthaltenen Elektrolytmenge ausreichend ist.

[0019] Bei dem Inertgas kann es sich um ein einziges reaktionsträges oder nicht reaktives Gas oder ein Gemisch solcher Gase handeln. In einer Ausführungsform der Erfindung wird Stickstoff als Inertgas verwendet.

[0020] In einer Ausführungsform ist die Auffangvorrichtung ein Kunststoffass, welches mittels Dichtflansch und Schnellwechselvorrichtung gasdicht an die Öffnung angebracht ist.

[0021] Wenn der Zerkleinerungsraum eine Messvorrichtung zum Messen einer Sauerstoffkonzentration im Zerkleinerungsraum umfasst, kann zum Beispiel während dem Erzeugen der im Wesentlichen Inertgas enthaltenden Atmosphäre eine Sauerstoffkonzentration im Zerkleinerungsraum wiederkehrend gemessen und mit einem Grenzwert verglichen werden. Dann kann bei einem Unterschreiten des Grenzwertes durch die gemessene Sauerstoffkonzentration das Erzeugen der im Wesentlichen inertgashaltigen Atmosphäre beendet werden. Der Grenzwert für die Sauerstoffkonzentration kann beispielsweise bei 6% liegen.

Ascend Elements EX1002 Page 563

Duesenfeld Ex. 2014-563

[0022] Weitere bevorzugte Ausgestaltungen der Erfindung ergeben sich aus den übrigen, in den Unteransprüchen genannten Merkmalen.

[0023] Die Erfindung wird nachfolgend in Ausführungsbeispielen anhand der zugehörigen Zeichnungen erläutert. Es zeigen:

[0024] **Fig.** 1 eine schematische Ansicht eines ersten Ausführungsbeispiels des erfindungsgemäßen Systems,

[0025] **Fig.** 2 eine schematische Ansicht eines Aufschnitts des ersten Ausführungsbeispiels des erfindungsgemäßen Systems,

[0026] **Fig.** 3 eine schematische Ansicht eines Aufschnitts eines zweiten Ausführungsbeispiels des erfindungsgemäßen Systems, und

[0027] **Fig.** 4 ein schematisches Flussdiagramm einer beispielhaften Ausführungsform des erfindungsgemäßen Verfahrens.

[0028] In der in **Fig.** 1 dargestellten schematischen Ansicht eines ersten Ausführungsbeispiels des erfindungsgemäßen Systems **1000** befindet sich eine Zuführvorrichtung **100** in Form eines rechteckigen Trichters über einer Zerkleinerungsvorrichtung **200**. Ein Deckel **110** erlaubt die Zuführvorrichtung **100** von der Außenwelt gasdicht zu trennen. Weiterhin erlaubt eine zwischen Zuführvorrichtung **100** und Zerkleinerungsvorrichtung **200** angeordnete, als Absperrschieber dargestellte Zuführschleuse **120** einen Füllraum der Zuführvorrichtung **100** von einem Zerkleinerungsraum der Zerkleinerungsvorrichtung **200** gasdicht zu trennen beziehungsweise die beiden Räume gasflussfähig zu verbinden. Die Zerkleinerungsvorrichtung **200** weist eine Abgasöffnung **210** und einen nicht dargestellten Gaseinlass auf, an den über eine Gasleitung **350** eine Gasquelle **300** für Inertgas oder Inertgasgemisch angeschlossen ist. Die Zerkleinerungsvorrichtung **200** steht auf Beinen **290**. Unter der Zerkleinerungsvorrichtung **200** ist eine Materialaustragvorrichtung **400** angeordnet, die den in **Fig.** 1 nicht sichtbaren Zerkleinerungsraum innerhalb der Zerkleinerungsvorrichtung **200** mit einem Füllraum einer Auffangvorrichtung **500** gasflussfähig verbindet und dabei gasdicht gegen die Außenwelt abschließt.

[0029] **Fig.** 2 zeigt das erste Ausführungsbeispiel aufgeschnitten entlang der Ebene A in **Fig.** 1. Wie dargestellt befindet sich der Füllraum des Einfülltrichters **100** oberhalb des in **Fig.** 2 sichtbaren Zerkleinerungsraums. Aus Gründen der Übersichtlichkeit ist die Zuführschleuse **120** in **Fig.** 2, anders als in **Fig.** 1, schematisch zu einer anderen Seite hin verschiebbar dargestellt. Im Zerkleinerungsraum sind schematisch dargestellte Schneidscheiben **230** angeordnet. Wie dargestellt setzt sich die Gasleitung **350** in der Zerkleinerungsvorrichtung fort und verbindet so die Inertgasquelle **300** gasflussfähig mit dem Zerkleinerungsraum. Die in **Fig.** 2 nicht sichtbare Abgasöffnung ist über einen Aktivkohlefilter **220** mit dem Zerkleinerungsraum verbunden. Ein steuerbares Ventil **310** erlaubt die Kontrolle über die Menge einströmenden Inertgases. Das steuerbare Ventil **310** kann so ausgelegt sein, dass es anstelle des Zustroms von Inertgas einen Zustrom von Umgebungsluft erlaubt. Ein weiteres zur Steuersignalverbindung ausgestaltetes Ventil **250** kann zur Kontrolle der Menge über die Abgasöffnung abströmenden Gases oder Gasgemisches angesteuert werden. Im Ausführungsbeispiel misst eine Sauerstoffkonzentrationsmessvorrichtung **270** eine Sauerstoffkonzentration im Zerkleinerungsraum und leitet den Messwert an eine Steuervorrichtung **260** mit einem Komparator weiter, der den Messwert mit einem Grenzwert von beispielsweise 10%, vorzugsweise 6% oder weniger, vergleicht. Dabei erzeugt die Steuervorrichtung **260** bei unterschreiten des Grenzwerts ein Steuersignal zur Schließung des mit der Steuervorrichtung **260** verbundenen Ventils **350** und steuert das Ventil **350** entsprechend an. Die Steuervorrichtung **260** kann insbesondere so ausgelegt sein, dass sie die vorbeschriebene Steuerung in einem ersten und eine weitere Steuerung in einem zweiten Betriebsmodus realisiert, wobei die Steuerungsvorrichtung **260** im zweiten Betriebsmodus den Messwert mit einem anderen Grenzwert von beispielsweise 20% oder 21% vergleicht. Dabei erzeugt die Steuervorrichtung **260** im zweiten Betriebsmodus bei Unterschreiten des anderen Grenzwerts ein Steuersignal zur Öffnung des mit der Steuervorrichtung **260** verbundenen Ventils **350** zur Außenwelt und steuert das Ventil **350** entsprechend an. Bei Erreichen oder Überschreiten des anderen Grenzwertes im zweiten Betriebsmodus wird das Ventil **350** wieder geschlossen.

[0030] Im ersten Ausführungsbeispiel befindet sich in der fassförmig dargestellten Auffangvorrichtung **500** eine Lösung zur Bindung freier Ionen. Unterschiedliche Lösungen können dabei an unterschiedliche Elektrolyte angepasst sein. Beispielsweise lassen sich bei der Zerkleinerung von Lithium-Ionen-Zellen freiwerdende Fluor-Ionen in einer wässrigen Lithiumhydroxid-Lösung binden.

[0031] In einem zweiten Ausführungsbeispiel befinden sich in der Materialaustragvorrichtung Düsen **450**. Die Düsen **450** dienen zum Bestäuben oder Besprühen der Zellbruchstücke, die aus dem Schneidwerk fallen, wobei das Schneidwerk die Schneidscheiben **230** umfasst. Dabei werden die Zellbruchstücke mit einem desaktivierenden Pulver oder Aerosol besprüht oder bestäubt, das Bindungspartnerionen für die aus dem Elektrolyt der Zellen freiwerdenden Ionen bietet. Dies ist in **Fig.** 3 schematisch dargestellt.

Ascend Elements EX1002 Page 564

Duesenfeld Ex. 2014-564

[0032] In einer beispielhaften Ausführungsform des Verfahrens werden defekte und/oder beschädigte Lithium-Ionen Batteriezellen in einen Einfülltrichter **100** eingefüllt. Der Einfülltrichter **100** dieser beispielhaften Ausführungsform ist gegen die Außenwelt gasdicht verschließbar, beispielsweise mittels eines Gasdruckdämpfers unterstützen Deckels **110**, der im geöffneten Zustand offen bleibt und so ein leichtes Beschicken ermöglicht. Die gasdichte Abdichtung des Einfülltrichters **100** bei geschlossenem Deckel **110** erfolgt dabei beispielsweise mittels eines Dichtflansches. Eine Zuführschleuse **120** verschließt einen Füllraum des Einfülltrichters **100** gasdicht gegenüber einem Zerkleinerungsraum eines Schredders **200**. Dabei ist bei erfindungsgemäß vorgesehener Aufstellung der Füllraum über dem Zerkleinerungsraum angeordnet, so dass das Öffnen der Zuführschleuse **120** ein Hinabfallen der im Füllraum befindlichen Zellen in den Zerkleinerungsraum bewirkt.

[0033] Der Schredder **200** wird in einer beispielhaften Ausführungsform von zwei getrennten Elektromotoren mittels Zahnriemen und einem mehrstufigen Stirnradgetriebe angetrieben, die von einem Kraftstoff betriebenen transportablen Generator mit Spannung versorgt werden. Das mehrstufige Stirnradgetriebe kann etwa dreistufig sein und zur Verringerung des Wartungsbedarfs in einem Ölbad laufen. Die Elektromotoren treiben gemeinsam über das Stirnradgetriebe das Nebenschneidwerk und das Hauptschneidwerk an. Jedes der Schneidwerke umfasst dabei ein Paar Schneidscheiben **230**. In den Zerkleinerungsraum von oben aus dem Füllraum gefallende Zellen werden bei laufenden Schneidwerken von diesen zerkleinert. Die Zerkleinerungsprodukte, also die Zellfragmente, fallen unten aus den Schneidwerken heraus und durch eine Öffnung.

[0034] An dieser Öffnung ist im Ausführungsbeispiel ein Kunststofffass **500** mittels Dichtflansch und Schnellwechselvorrichtung gasdicht gegen die Außenwelt angebracht. In dieser Ausführungsform enthält das Kunststofffass eine vorbestimmte Menge LiOH-Lösung **510**, die zur Desaktivierung einer entsprechenden Menge von aus Leitsalz freiwerdendem Fluor durch Bindung als Lithiumfluorid geeignet ist. Der entsprechenden Menge Fluor entspricht eine Höchstanzahl von Zellen, die in geschredderter Form von der LiOH-Lösung desaktiviert werden können. Dabei ist eine Wässrigkeit der LiOH-Lösung so gewählt, dass eine Sättigungsgrenze des Wassers für Lösungsmittel des Elektrolyts nicht überschritten wird.

[0035] In einer anderen Ausführungsform fallen aus den Schneidwerken heraus fallende Zellfragmente durch einen Bereich, in dem seitlich Bestäubungsdüsen **450** angebracht sind, die die fallenden Zellfragmente mit einem zur Desaktivierung des Fluor geeigneten Pulvers bestäuben, ehe sie durch die Öffnung in das Kunststofffass **500** fallen, welches in dieser anderen Ausführungsform keine LiOH-Lösung enthalten muss.

[0036] Es ist möglich, ein Volumen des Füllraums des Einfülltrichters **100** so zu wählen, dass eine in den Füllraum passende Menge von Zellen in geschredderter Form in das Kunststofffass **500** passt. Bei Desaktivierung mittels LiOH-Lösung kann das Volumen des Füllraums zudem der Menge von Zellen angepasst sein, deren Fluorgehalt sicher mittels der LiOH-Lösung gebunden werden kann. Diese Anpassung kann zelltypbezogen sein. In einer beispielhaften Ausführungsform ist die Auffangvorrichtung in verschiedenen Dimensionierungen und Füllmengen so ausgebildet, dass für Batteriezellen unterschiedlicher Hybrid- oder Elektromobilbatterietypen passende Auffangvorrichtungen vorbereitet zur Verfügung stehen, die beispielsweise auch nur für Fragmente genau einer Batterie dieses Typs dimensioniert sind. Einfülltrichter und Schredder können dann entsprechend ausgelegt sein, so dass die Vorrichtung etwa an Verkehrsunfallstellen Sekundärbatterien von verunfallten Elektro- oder Hybridfahrzeugen einzeln typgerecht im transportfähigen und recyclingfähigen Zustand überführt.

[0037] Der Zerkleinerungsraum des Schredders und mit ihm im Gasaustausch stehende Räume wie etwa das Kunststofffass lassen sich nach gasdichtem Abschluss gegenüber der Außenwelt mittels einer Begasungsanlage beispielsweise mit Stickstoff aus einer Stickstoff (N_2) gefüllten Druckflasche befüllen, so dass im Zerkleinerungsraum eine inerte, also eine reaktionsschwache oder nicht reaktive Atmosphäre entsteht. Zuvor in den zu begasenden Räumen vorhandenes Gas oder Gasgemisch, also beispielsweise Luft, wird dabei durch das einströmende Inertgas verdrängt. Eine Absaugvorrichtung kann die Verdrängung unterstützen.

[0038] Fasst das Kunststofffass mehr zerkleinerte Zellen als unzerkleinert in den Einfülltrichter passen, so ermöglicht die Zuführschleuse zwischen Einfülltrichter und Zerkleinerungsraum, dass eine bereits vorhandene inerte Atmosphäre im Zerkleinerungsraum weitgehend erhalten werden kann, wenn erst der Einfülltrichter mit Zellen gefüllt und dann gasdicht gegen die Außenwelt abgeschlossen wird, ehe die Zuführschleuse geöffnet wird.

[0039] Der Einfülltrichter kann beispielsweise druckfest und in Edelstahl ausgeführt sein.

[0040] Wie in **Fig.** 4 dargestellt, beginnt eine beispielhafte Ausführungsform des erfindungsgemäßen Verfahrens mit Schritt S1, dem gegenüber der Außenwelt gasdichten Anschließen des Auffangfasses an die Zerkleinerungsvorrichtung. Dann wird bei geschlossener Zuführschleuse die Steuerungsvorrich-

Ascend Elements EX1002 Page 565

Duesenfeld Ex. 2014-565

tung des Systems etwa durch händische Bedienung in einen ersten Betriebszustand versetzt, in dem die Steuerungsvorrichtung in Abhängigkeit von der Messsonde in Schritt S2 gemessenen Sauerstoffkonzentration ein Ventil ansteuert, mit dem ein Zustrom von Inertgas kontrolliert werden kann. Dazu vergleicht eine Steuervorrichtung des Systems in Schritt S3 den aktuellen Messwert mit einem Grenzwert und bestimmt, ob der Grenzwert unterschritten ist. Ist der Grenzwert nicht unterschritten, steuert die Steuerungsvorrichtung das Ventil in Schritt S4 so an, dass es sich öffnet beziehungsweise geöffnet bleibt. Die Schritte S2, S3 und S4 werden dann solange wiederholt, bis in Schritt S3 eine Grenzwertunterschreitung festgestellt wird. Es folgt Schritt S5, das Öffnen des Deckels des Einfülltrichters bei gleichzeitig geschlossener Zuführschleuse und Einfüllen von zu zerkleinernden Elektrolytzellen. Dann wird in Schritt S6 der Deckel geschlossen bevor in Schritt S7 die Zuführschleuse geöffnet wird. Nach Hinabfallen der zu zerkleinernden Elektrolytzellen in den Zerkleinerungsraum wird die Zuführschleuse in Schritt S8 wieder geschlossen. Dann schließt sich Schritt S9 an, in dem die im Zerkleinerungsraum befindlichen Zellen zerkleinert werden, wobei die Zerkleinerungsprodukte in das Auffangfass fallen. In einem optionalen Schritt S10 werden sie mit einem Pulver bestäubt oder mit einer Flüssigkeit eingenebelt, die freie Ionen bindet und desaktiviert. Bei Verwendung eines Pulvers kann insbesondere auch Flüssigkeit gebunden werden. In einem Schritt S11 wird geprüft, ob das Fass vollständig gefüllt ist. Wird im Schritt S11 festgestellt, dass das Auffangfass noch nicht vollständig gefüllt ist, werden die Schritte S1 bis S9 beziehungsweise bis S10 wiederholt. Wird hingegen in Schritt S11 festgestellt, dass das Auffangfass vollständig gefüllt ist, geht die Steuerungsvorrichtung des Systems in einen zweiten Betriebszustand, in dem die Steuerungsvorrichtung in Abhängigkeit von der Messsonde in Schritt S12 gemessenen Sauerstoffkonzentration, ein Umgebungsluftventil und eine Absaugvorrichtung ansteuert, mit dem ein Absaugen von Gas aus dem Zerkleinerungsraum und ein Zuströmen von Umgebungsluft kontrolliert werden kann. Dazu vergleicht eine Steuervorrichtung des Systems in Schritt S13 den aktuellen Messwert mit einem anderen, höheren Grenzwert und bestimmt, ob der andere, höhere Grenzwert unterschritten ist. Ist der höhere Grenzwert unterschritten, steuert die Steuerungsvorrichtung das Umgebungsluftventil in Schritt S14 so an, dass es sich öffnet beziehungsweise geöffnet bleibt. Gleichzeitig wird in Schritt S15 die Absaugvorrichtung so angesteuert, dass das Gasgemisch im Zerkleinerungsraum abgesaugt und über einen Aktivkohlefilter gereinigt in die Umgebungsatmosphäre abgegeben wird. Die Schritte S12, S13, S14 und S15 werden dann solange wiederholt, bis in Schritt S13 eine Grenzwerterreichung oder -überschreitung des höheren Grenzwertes festgestellt wird. Dann schließt sich Schritt S16 an, in dem das Fass vom System getrennt und mit einem Deckel und einer Schnellspannschelle verschlossen wird.

Bezugszeichenliste

100	Zuführvorrichtung/Einfülltrichter
110	Deckel
120	Zuführschleuse
200	Zerkleinerungsvorrichtung/Schredder
210	Abgasöffnung
220	Aktivkohlefilter
230	Schneidscheiben
240	Gasabsaugvorrichtung
250	Steuersignalverbindung
260	Steuervorrichtung
270	Sauerstoffkonzentrationssensor
300	Inertgasquelle
310	Gasströmungsverbindung zur Atmosphäre
350	Gasleitung
400	Materialaustragvorrichtung
450	Bestäubungsdüsen
500	Auffangvorrichtung/Auffangfass
510	LiOH-Lösung
1000	System
S1	gasdichtes Anschließen des Auffangfasses an die Zerkleinerungsvorrichtung
S2	Messen der Sauerstoffkonzentration
S3	Vergleichen von Messwert und Grenzwert
S4	Öffnen beziehungsweise Offenhalten des Ventils für Inertgaszustrom
S5	Öffnen des Deckels des Einfülltrichters bei gleichzeitig geschlossener Zuführschleuse
S6	Schließen des Deckels des Einfülltrichters
S7	Öffnen der Zuführschleuse
S8	Schließen der Zuführschleuse
S9	Zerkleinern der Zellen
S10	Besprühen oder Bestäuben mit desaktivierendem Pulver oder Nebel
S11	Vergleichen von Füllkapazität des Auffangfasses mit Menge bereits eingefüllter Zellen
S12	Messen der Sauerstoffkonzentration
S13	Vergleichen von Messwert und dem anderen, höheren Grenzwert
S14	Öffnen beziehungsweise Offenhalten des Ventils für Umgebungsluftzustrom
S15	Einschalten beziehungsweise Eingeschaltet halten der Absaugvorrichtung
S16	Trennen des Auffangfasses von der Zerkleinerungsvorrichtung und Schließen des Auffangfasses

Ascend Elements EX1002 Page 566

Duesenfeld Ex. 2014-566

ZITATE ENTHALTEN IN DER BESCHREIBUNG

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Zitierte Patentliteratur

- EP 1733451 [0005]
- EP 0649912 [0005]

Ascend Elements EX1002 Page 567

Duesenfeld Ex. 2014-567

Patentansprüche

1. Vorrichtung zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand, wobei die Vorrichtung eine Zerkleinerungsvorrichtung (**200**) zum Zerkleinern der Elektrolytzellen in einem von der Zerkleinerungsvorrichtung (**200**) umfassten Zerkleinerungsraum ist, eine Zuführvorrichtung (**100**) zum Zuführen von Elektrolytzellen in den Zerkleinerungsraum, wobei der Zerkleinerungsraum mittels einer Zuführschleuse (**120**) gasdicht von der die Zuführvorrichtung (**100**) getrennt werden kann, eine Materialaustragvorrichtung (**400**) zum Austragen zerkleinerter Elektrolytzellen aus dem Zerkleinerungsraum, eine Inertgaszuführvorrichtung (**300**, **350**) zum Erzeugen einer im Wesentlichen Inertgas enthaltenden Atmosphäre zumindest im Zerkleinerungsraum und in der Materialaustragvorrichtung (**400**), sowie eine Gasabsaugvorrichtung (**240**) zum Absaugen der im Zerkleinerungsraum enthaltenden Atmosphäre umfasst, wobei die Gasabsaugvorrichtung (**240**) eine Gasreinigungsvorrichtung (**220**) umfasst, **dadurch gekennzeichnet,** dass die Materialaustragvorrichtung (**400**) eine Öffnung zur gasdichten Anbringung einer transportablen Auffangvorrichtung (**500**) zum Auffangen zerkleinerter Zellen umfasst.

2. Vorrichtung nach Anspruch 1, **dadurch gekennzeichnet,** dass Materialaustragvorrichtung mindestens eine Bestäubungsvorrichtung (**450**) zum Bestäuben der zerkleinerten Zellen mit einem Desaktivierungspulver umfasst.

3. Vorrichtung nach Anspruch 1 oder 2, **dadurch gekennzeichnet,** dass die die Vorrichtung transportabel ist und eine Energieversorgungsvorrichtung umfasst, die eine standortunabhängige Energieversorgung ermöglicht.

4. Transportable Auffangvorrichtung (**500**) **dadurch gekennzeichnet,** dass die Auffangvorrichtung zum Überführen transportkritischer Lithium-Ionen-Zellen in einen transportfähigen Zustand mit einer Vorrichtung nach einem der vorangehenden Ansprüche vorgesehen ist und eine vorbestimmte Menge Lithiumhydroxid-Lösung (**510**) enthält, wobei die enthaltene Menge Lithiumhydroxid-Lösung und ein Volumen der Auffangvorrichtung so aufeinander abgestimmt ist, dass eine Menge Fluor, die in einer Menge die Auffangvorrichtung passender zerkleinerter Lithium-Ionen-Zellen enthalten ist, durch die Menge Lithiumhydroxid-Lösung in der Auffangvorrichtung vollständig gebunden und/oder desaktiviert wird.

5. Transportable Auffangvorrichtung nach Anspruch 4, **dadurch gekennzeichnet,** dass das Volumen der Auffangvorrichtung zusätzlich auf ein Volumen der Zuführvorrichtung abgestimmt ist, so dass die Menge die Auffangvorrichtung passender zerkleinerter Lithium-Ionen-Zellen einer Menge unzerkleinert in die Zuführvorrichtung passender Lithium-Ionen-Zellen entspricht.

6. System (**1000**) zum Überführen transportkritischer Lithium-Ionen-Zellen in einen transportfähigen Zustand mit einer Vorrichtung nach einem der Ansprüche 1 bis 3 und einer transportablen Auffangvorrichtung nach Anspruch 4 oder 5.

7. Verfahren zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand **dadurch gekennzeichnet,** dass das Verfahren die Verwendung einer Vorrichtung nach einem der Ansprüche 1 bis 3 umfasst, wobei an die Öffnung der Materialaustragvorrichtung eine transportable Auffangvorrichtung gasdicht angebracht ist und wobei das Verfahren die folgenden Schritte umfasst: Erzeugen (S2, S3, S4) der im Wesentlichen inertgashaltigen Atmosphäre im Zerkleinerungsraum, in der Materialaustragvorrichtung und in der Auffangvorrichtung, Befüllen (S5) der Zuführvorrichtung mit einer Charge Elektrolytzellen, gasdichtes Schließen (S6) der Zuführvorrichtung, Zuführen (S7) der Elektrolytzellen in den Zerkleinerungsraum, Zerkleinern (S9) der Zellen unter der inertgashaltigen Atmosphäre und Austragen der zerkleinerten Zellen in die Auffangvorrichtung unter der inertgashaltigen Atmosphäre, Absaugen (S12, S13, S14, S15) der inertgashaltigen Atmosphäre, Abtrennen (S16) der Auffangvorrichtung von der Öffnung und Verschließen der Auffangvorrichtung.

8. Verfahren nach Anspruch 7 **dadurch gekennzeichnet,** dass das Verfahren weiterhin folgende Schritte umfasst, die vor der Erzeugung der im Wesentlichen Inertgas enthaltenden Atmosphäre ausgeführt werden: Befüllen der Auffangvorrichtung mit einer zur Desaktivierung und/oder Bindung eines Elektrolyts der Charge geeigneten Lösung, wobei ein Volumen der Auffangvorrichtung und ein Volumen der Zuführvorrichtung so aufeinander abgestimmt, dass eine Menge in die Zuführvorrichtung passender Elektrolytzellen nach Zerkleinern in das Volumen der Auffangvorrichtung passt, und wobei die zum Befüllen verwendete Menge an Lösung zur Desaktivierung und/oder Bindung einer in der Charge enthaltenen Elektrolytmenge ausreichend ist, und gasdichtes Anbringen der transportablen Auffangvorrichtung an der Öffnung.

9. Verfahren nach Anspruch 7 **dadurch gekennzeichnet,** dass das Verfahren weiterhin folgenden Schritt umfasst, der unter der im Wesentlichen Inertgas enthaltenden Atmosphäre ausgeführt wird: Bestäuben (S10) der zerkleinerten Zellen mit einem Desaktivierungspulver, wobei die zum Bestäuben verwendete Menge an Desaktivierungspulver zur Desaktivierung einer in der Charge enthaltenen Elektrolytmenge ausreichend ist.

Ascend Elements EX1002 Page 568

Duesenfeld Ex. 2014-568

10. Verfahren nach einem der Ansprüche 7 bis 9 **dadurch gekennzeichnet,** dass während dem Erzeugen der im Wesentlichen Inertgas enthaltenden Atmosphäre eine Sauerstoffkonzentration im Zerkleinerungsraum gemessen (S2) und mit einem Grenzwert verglichen (S3) wird, wobei bei einem Unterschreiten des Grenzwertes durch die gemessene Sauerstoffkonzentration das Erzeugen der im Wesentlichen inertgashaltige Atmosphäre beendet wird.

Es folgen 4 Seiten Zeichnungen

Ascend Elements EX1002 Page 569

Duesenfeld Ex. 2014-569

Anhängende Zeichnungen

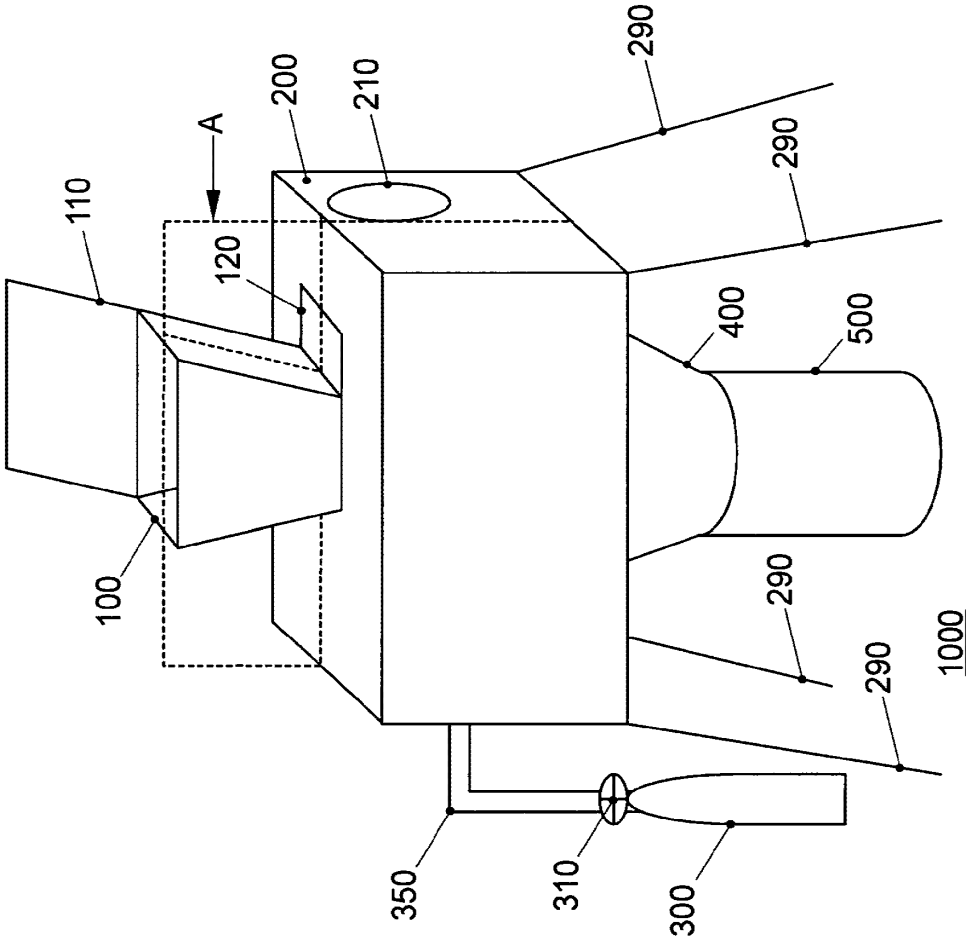

FIG. 1

Ascend Elements EX1002 Page 570

Duesenfeld Ex. 2014-570

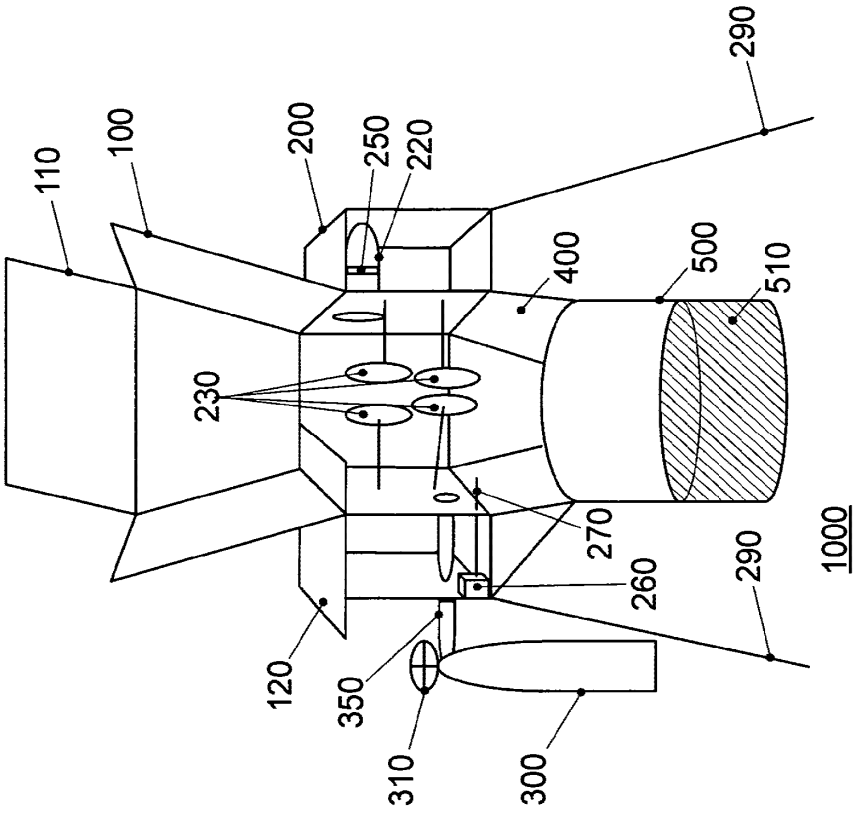

FIG. 2

Ascend Elements EX1002 Page 571

Duesenfeld Ex. 2014-571

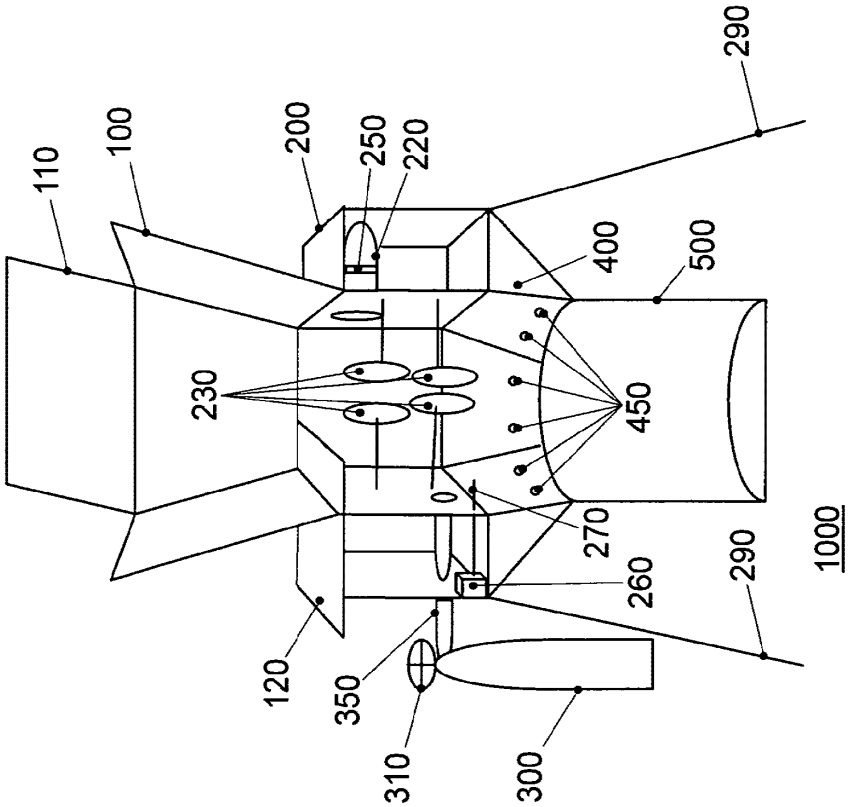

FIG. 3

Ascend Elements EX1002 Page 572

Duesenfeld Ex. 2014-572

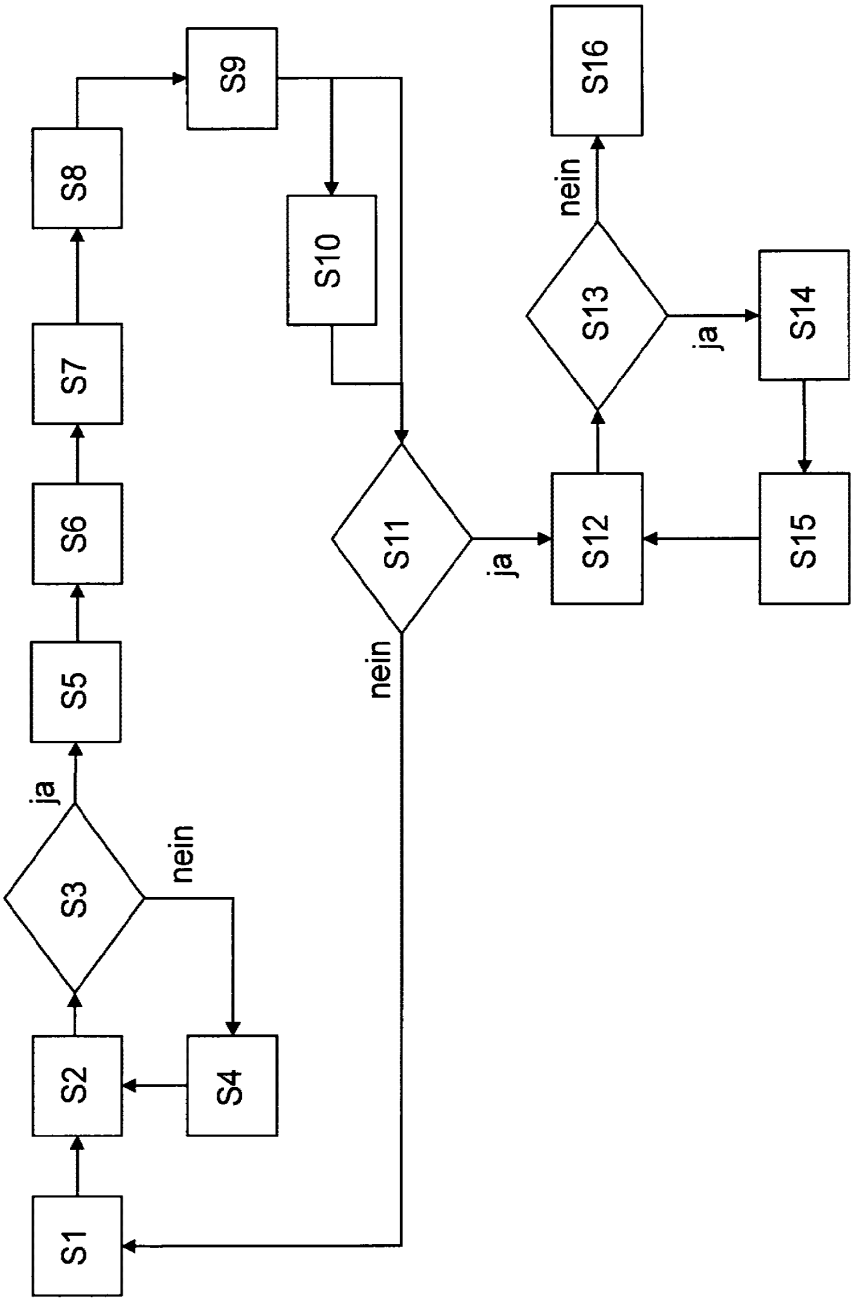

FIG. 4

Ascend Elements EX1002 Page 573

Duesenfeld Ex. 2014-573

Espacenet

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Apparatus for transferring transportable critical electrolytic cells into transportable state, has material discharge device with opening for gas-tight attachment of portable collection device for collecting chopped cells

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- cooperative: **H01M10/0525**; **H01M10/54**; Y02W30/84

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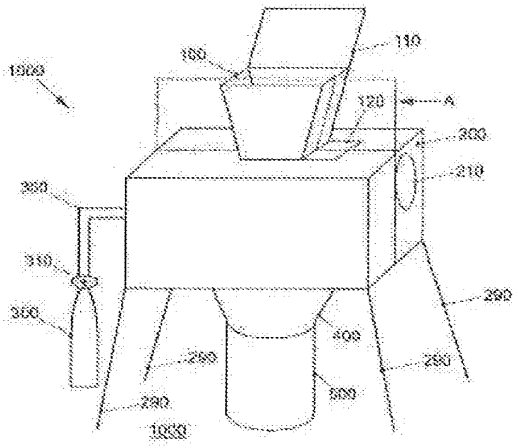
Priority number(s): DE20121024876 20121219

Abstract of DE102012024876 (A1)

The apparatus has a feed device (100) for supplying electrolytic cells in a milling chamber of a comminution device (200). A material discharge device (400) discharges crushed electrolyte cells from the milling chamber. The inert gas supply portions (300,350) generate inert gas atmosphere in the milling chamber and material discharge device. A gas suction device comprising a gas purification apparatus extracts the inert gas. The material discharge device has an opening for gas-tight attachment of portable collection device (500) for collecting chopped cells. Independent claims are included for the following: (1) a portable collection device; (2) a system for transferring transportable critical electrolytic cells into transportable state; and (3) a method for transferring transportable critical electrolytic cells into transportable state.



From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92*bis*.1 and Administrative Instructions, Section 422)

To:

PLÖGER, Jan
Gramm, Lins & Partner Patent- und Rechtsanwälte PartGmbB
Theodor-Heuss-Strasse 1
38122 Braunschweig
ALLEMAGNE

Date of mailing *(day/month/year)*
11 October 2017 (11.10.2017)

Applicant's or agent's file reference
5569-002 PCT-1

IMPORTANT NOTIFICATION

International application No.
PCT/EP2016/059526

International filing date *(day/month/year)*
28 April 2016 (28.04.2016)

1. The following indications appeared on record concerning:

☒ the applicant ☐ the inventor ☐ the agent ☐ the common representative

Name and Address
LION ENGINEERING GMBH
Rebenring 33
38106 Braunschweig
Germany

State of Nationality: DE
State of Residence: DE
Telephone No.:
Facsimile No.:
E-mail address:

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☒ the person ☐ the name ☐ the address ☐ the nationality ☐ the residence

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Friedlandweg 5
38114 Braunschweig
Germany

State of Nationality: DE
State of Residence: DE
Telephone No.:
Facsimile No.:
E-mail address:
☐ Notifications by e-mail authorized

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

☒ the receiving Office
☒ the International Searching Authority
☐ the Authority(ies) specified for supplementary search
☐ the International Preliminary Examining Authority
☒ the designated Offices concerned
☐ the elected Offices concerned
☐ other:

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Ascend Elements EX1002 Page 575

WO/2017/186FKYX3H0

Duesenfeld Ex. 2014-575

PCT-ANTRAG

Ausdruck (Original in elektronischem Format)

0	**Vom Anmeldeamt auszufüllen**	
0-1	Internationales Aktenzeichen	**PCT/EP2016/059526**
0-2	Internationales Anmeldedatum	**28 APR 2016 (28.04.2016)**
0-3	Name des Anmeldeamts und "PCT International Application"	**RO/EP**

0-4	**Formular PCT/RO/101 PCT-Antrag**	
0-4-1	Erstellt mit	PCT Online Filing Version 3.5.000.244e MT/FOP 20141031/0.20.5.20
0-5	**Antragsersuchen** Der Unterzeichnete beantragt, daß die vorliegende internationale Anmeldung nach dem Vertrag über die internationale Zusammenarbeit auf dem Gebiet des Patentwesens behandelt wird	
0-6	**(Vom Anmelder gewähltes) Anmeldeamt**	Europäisches Patentamt (EPA) (RO/EP)
0-7	**Aktenzeichen des Anmelders oder Anwalts**	5569-002 PCT-1
I	**Bezeichnung der Erfindung**	Verfahren zum Behandeln gebrauchter Batterien, insbesondere wieder aufladbarer Batterien und Batterie-Verarbeitungsanlage
II	**Anmelder**	
II-1	Diese Person ist	Nur Anmelder
II-2	Anmelder für	Alle Bestimmungsstaaten
II-4	Name	LION ENGINEERING GMBH
II-5	Anschrift	Rebenring 33 38106 Braunschweig Deutschland
II-6	Staatsangehörigkeit (Staat)	DE
II-7	Sitz/Wohnsitz (Staat)	DE
II-11	Registrierungsnummer des Anmelders beim Amt	li
III-1	**Anmelder und/oder Erfinder**	
III-1-1	Diese Person ist	Erfinder nur
III-1-3	Erfinder für	Alle Bestimmungsstaaten
III-1-4	Name (FAMILIENNAME, Vorname)	HANISCH, Christian
III-1-5	Anschrift	Sophienstraße 14 38118 Braunschweig Deutschland

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III-2	**Anmelder und/oder Erfinder**	
III-2-1	Diese Person ist	Erfinder nur
III-2-3	Erfinder für	Alle Bestimmungsstaaten
III-2-4	Name (FAMILIENNAME, Vorname)	WESTPHAL, Bastian
III-2-5	Anschrift	Karlstraße 92 38106 Braunschweig Deutschland
III-3	**Anmelder und/oder Erfinder**	
III-3-1	Diese Person ist	Erfinder nur
III-3-3	Erfinder für	Alle Bestimmungsstaaten
III-3-4	Name (FAMILIENNAME, Vorname)	HASELRIEDER, Wolfgang
III-3-5	Anschrift	Görgesstraße 3 38118 Braunschweig Deutschland
III-4	**Anmelder und/oder Erfinder**	
III-4-1	Diese Person ist	Erfinder nur
III-4-3	Erfinder für	Alle Bestimmungsstaaten
III-4-4	Name (FAMILIENNAME, Vorname)	SCHOENITZ, Martin
III-4-5	Anschrift	Wabestraße 31 38106 Braunschweig Deutschland
IV-1	**Anwalt oder gemeinsamer Vertreter oder besondere Zustellanschrift** Die unten bezeichnete Person ist/wird hiermit bestellt, um für den (die) Anmelder vor den zuständigen internationalen Behörden zu handeln, und zwar in folgender Eigenschaft:	Anwalt
IV-1-1	Name (FAMILIENNAME, Vorname)	PLÖGER, Dr.-Ing., Jan
IV-1-2	Anschrift	Gramm, Lins & Partner Patent- und Rechtsanwälte PartGmbB Theodor-Heuss-Strasse 1 38122 Braunschweig Deutschland
IV-1-3	Telefonnr.	0531-28140-0
IV-1-4	Telefaxnr.	0531-28140-28
IV-1-5	E-Mail	braunschweig@grammpatent.de
V	**BESTIMMUNGEN**	
V-1	**Die Einreichung dieses Antrags umfaßt gemäß Regel 4.9 Absatz a die Bestimmung aller Vertragsstaaten, für die der PCT am internationalen Anmeldedatum verbindlich ist, und, insoweit verfügbar, für jede Art von Schutzrecht und sowohl für ein regionales als auch für ein nationales Patent**	

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VI-1	**Priorität einer früheren nationalen Anmeldung beansprucht**		
VI-1-1	Anmeldedatum	28. April 2015 (28.04.2015)	
VI-1-2	Nummer	10 2015 207 843.4	
VI-1-3	Staat oder Mitglied der WTO	DE	
VI-2	**Einbeziehung durch Verweis :** Ist ein in Artikel 11 Absatz 1 Ziffer iii Buchstabe d oder e genannter Bestandteil der internationalen Anmeldung oder ein in Regel 20.5 Absatz a genannter Teil der Beschreibung, der Ansprüche oder der Zeichnungen nicht anderswo in der internationalen Anmeldung, aber vollständig in einer früheren Anmeldung enthalten, deren Priorität, zu dem Datum, an dem ein oder mehrere in Artikel 11 Absatz 1 Ziffer iii genannte Bestandteile zuerst beim Anmeldeamt eingegangen sind, beansprucht wird, so wird dieser Bestandteil oder Teil, vorbehaltlich der Bestätigung nach Regel 20.6, für die Zwecke der Regel 20.6 in diese internationale Anmeldung einbezogen.		
VII-1	**Gewählte Internationale Recherchenbehörde**	Europäisches Patentamt (EPA) (ISA/EP)	
VIII	**Erklärungen**	Anzahl der Erklärungen	
VIII-1	Erklärung hinsichtlich der Identität des Erfinders	–	
VIII-2	Erklärung hinsichtlich der Berechtigung des Anmelders, zum Zeitpunkt des internationalen Anmeldedatums, ein Patent zu beantragen und zu erhalten	1	
VIII-3	Erklärung hinsichtlich der Berechtigung des Anmelders, zum Zeitpunkt des internationalen Anmeldedatums, die Priorität einer früheren Anmeldung zu beanspruchen	–	
VIII-4	Erfindererklärung (nur im Hinblick auf die Bestimmung der Vereinigten Staaten von Amerika)	–	
VIII-5	Erklärung hinsichtlich unschädlicher Offenbarungen oder Ausnahmen von der Neuheitsschädlichkeit	–	

VIII-2-1	**Erklärung: Berechtigung, ein Patent zu beantragen und zu erhalten** Erklärung hinsichtlich der Berechtigung des Anmelders, zum Zeitpunkt des internationalen Anmeldedatums, ein Patent zu beantragen und zu erhalten (Regeln 4.17 Ziffer ii und 51bis.1 Absatz a Ziffer ii), für den Fall, daß eine Erklärung nach Regel 4.17 Ziffer iv nicht einschlägig ist: Name (FAMILIENNAME, Vorname)	**In bezug auf diese internationale Anmeldung** **LION ENGINEERING GMBH ist kraft des nachfolgend Aufgeführten berechtigt, ein Patent zu beantragen und zu erhalten:**
VIII-2-1(ii)		**LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, HASELRIEDER, Wolfgang**
VIII-2-1(ii)		**LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, SCHOENITZ, Martin**
VIII-2-1(ii)		**LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, HANISCH, Christian**
VIII-2-1(ii)		**LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, WESTPHAL, Bastian**

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Ausdruck (Original in elektronischem Format)

IX	Kontrolliste	Anzahl der Blätter	Elektronische Datei(en) beigefügt
IX-1	Antrag (inklusive Erklärungsblätter)	5	✓
IX-2	Beschreibung	18	✓
IX-3	Ansprüche	4	✓
IX-4	Zusammenfassung	1	✓
IX-5	Zeichnung(en)	3	✓
IX-7	INSGESAMT	31	
	Beigefügte Unterlagen	Unterlage(n) in Papierform beigefügt	Elektronische Datei(en) beigefügt
IX-8	Blatt für die Gebührenberechnung	–	✓
IX-20	**Nr. der Abb. der Zeichn., die mit der Zusammenf. veröffentlicht werden soll**	2	
IX-21	**Sprache der int. Anmeldung**	Deutsch	
X-1	**Unterschrift des Anmelders, des Anwalts oder des Gemeinsamen Vertreters**	(PKCS7 digitale Unterschrift)	
X-1-1	Name (FAMILIENNAME, Vorname)	PLÖGER, Dr.-Ing., Jan	
X-1-3	Eigenschaft (sofern sich dies nicht eindeutig aus dem Antrag ergibt)	(Vertreter)	

VOM ANMELDEAMT AUSZUFÜLLEN

10-1	**Datum des tatsächlichen Eingangs dieser internationalen Anmeldung**	**28 APR 2016 (28.04.2016)**
10-2	**Zeichnung(en):**	
10-2-1	Eingegangen X	
10-2-2	Nicht eingegangen	
10-3	**Geändertes Eingangsdatum aufgrund nachträglich, jedoch fristgerecht eingeg. Unterlage(n) oder Zeichnung(en) zur Vervollständigung dieser int. Anmeldung**	
10-4	**Datum des fristgerechten Eingangs der Berichtigung nach PCT Artikel 11(2)**	
10-5	**Internationale Recherchenbehörde**	ISA/EP
10-6	**Übermittlung des Recherchenexemplars bis zur Zahlung der Recherchengebühr aufgeschoben**	

VOM INTERNATIONALEN BÜRO AUSZUFÜLLEN

11-1	**Datum des Eingangs des Aktenexemplars beim Internationalen Büro**	

Informationen zur Recherchenstrategie
Pilotphase (vgl. ABl. EPA 2015, A86)
Die Art der auf diesem Blatt enthaltenen Informationen kann sich während des Pilotprojekts zur Verbesserung des Nutzens dieses neuen Dienstes ändern.

Anmeldenummer
PCT/EP2016/059526

ERFINDUNGSBEZEICHNUNG: VERFAHREN ZUM BEHANDELN GEBRAUCHTER BATTERIEN, INSBESONDERE WIEDER AUFLADBARER BATTERIEN UND BATTERIE-VERARBEITUNGSANLAGE

ANMELDER: LION ENGINEERING GMBH

IPC-KLASSIFIKATION: H01M6/52, C22B13/00, H01M10/54

PRÜFER: Hofer, Astrid

KONSULTIERTE DATENBANKEN: EPODOC, WPI

KLASSIFIKATIONSSYMBOLE, DIE DEN RECHERCHENUMFANG DEFINIEREN:

IPC:

CPC: H01M6/52, C22B13/00, H01M10/54

FI/F-TERMS:

SCHLAGWÖRTER ODER SONSTIGE ELEMENTE, IN DENEN DIE ERFINDUNG VORKOMMT:
Die Anmeldung offenbart eine Methode zum Recycling gebrauchter Batterien basierend auf den Schritten Zerkleinern, Inaktivieren und Abfüllen der Batterieteile, wobei die Batterien durch Verdampfung des Elektrolyts mittels Trocknung unter Vakkum inaktiviert werden. Zusätzlich werden Zersetzungsgase durch das Strahlmedium einer Strahlpumpe ausgefällt.

EPO FORM P04A44

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VIII-2-1	**Erklärung: Berechtigung, ein Patent zu beantragen und zu erhalten** Erklärung hinsichtlich der Berechtigung des Anmelders, zum Zeitpunkt des internationalen Anmeldedatums, ein Patent zu beantragen und zu erhalten (Regeln 4.17 Ziffer ii und 51bis.1 Absatz a Ziffer ii), für den Fall, daß eine Erklärung nach Regel 4.17 Ziffer iv nicht einschlägig ist: Name (FAMILIENNAME, Vorname)	In bezug auf diese internationale Anmeldung LION ENGINEERING GMBH ist kraft des nachfolgend Aufgeführten berechtigt, ein Patent zu beantragen und zu erhalten:
VIII-2-1(ii)		LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, HASELRIEDER, Wolfgang
VIII-2-1(ii)		LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, SCHOENITZ, Martin
VIII-2-1(ii)		LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, HANISCH, Christian
VIII-2-1(ii)		LION ENGINEERING GMBH ist berechtigt, als Arbeitgeber des Erfinders, WESTPHAL, Bastian

BUNDESREPUBLIK DEUTSCHLAND

Prioritätsbescheinigung
DE 10 2015 207 843.4
über die Einreichung einer Patentanmeldung

Aktenzeichen:	10 2015 207 843.4
Anmeldetag:	28. April 2015
Anmelder/Inhaber:	Lion Engineering GmbH, 38106 Braunschweig, DE
Bezeichnung:	Verfahren zum Behandeln gebrauchter Batterien, insbesondere wieder aufladbarer Batterien und Batterie-Verarbeitungsanlage
IPC:	H01M 10/54; H01M 6/52

Die angehefteten Stücke sind eine richtige und genaue Wiedergabe der Teile der am 28. April 2015 eingereichten elektronischen Dokumente dieser Patentanmeldung unabhängig von gegebenenfalls durch das Druckverfahren bedingten Farbabweichungen.

München, den 29. April 2016
Deutsches Patent- und Markenamt
Die Präsidentin
Im Auftrag

Weiss

A 9161(E)/1.12

Gramm, Lins & Partner
Patent- und Rechtsanwaltssozietät GbR

Lion Engineering GmbH
Rebenring 33
38106 Braunschweig
Deutschland

Anwaltsakte:
5569-002 DE-1
Datum:
28. April 2015

Verfahren zum Behandeln gebrauchter Batterien, insbesondere wieder aufladbarer Batterien und Batterie-Verarbeitungsanlage

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien, insbesondere gebrauchter Lithium-Batterien, beispielsweise Lithium-Ionen-Batterien, mit den Schritten (a) Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) Inaktivieren des Zerkleinerungsguts, sodass ein inaktiviertes Zerkleinerungsgut erhalten wird, und (c) Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Gemäß einem zweiten Aspekt betrifft die Erfindung eine Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere zum Behandeln gebrauchter Lithium-Batterien, mit (a) einer Zerkleinerungseinheit zum Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) einer Inaktivierungsvorrichtung zum Inaktivieren des Zerkleinerungsguts und (c) einer Abfüllvorrichtung zum Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Aus der US 2005/0241943 A1 ist ein Verfahren zum Aufarbeiten gebrauchter Batterien bekannt, bei dem die Batterien vor einem Zerkleinerungsschritt erwärmt werden, sodass Kunststoffbestandteile der Batterien zerstört werden. Nachteilig an einem derartigen Vorgehen ist, dass die verbleibenden Komponenten der Batterien mit Abbauprodukten des Kunststoffs verunreinigt sein können.

Aus der DE 10 2012 024 876 A1 ist ein System zum Überführen transportkritischer Elektrolytzellen in einen transportfähigen Zustand bekannt, bei dem die Elektro-

lytzellen zunächst unter Inertgas zerkleinert und danach mit einem Desaktivierungspulver bestäubt werden, um eine Selbstentzündung des elektrochemisch aktiven Materials zu verhindern. Nachteilig daran ist, dass das entstehende Material dennoch ein vergleichsweise hohes Gefährdungspotential besitzt und dass das Bestäubungspulver selbst ein Explosionsrisiko darstellt und dass die Bildung einer entzündlichen und explosiven Atmosphäre in dem Transportbehälter nicht ausgeschlossen werden kann.

Der Erfindung liegt die Aufgabe zugrunde, Nachteile im Stand der Technik zu vermindern.

Die Erfindung löst das Problem durch ein gattungsgemäßes Verfahren, bei dem das Inaktivieren zumindest auch durch Trocknen des Zerkleinerungsguts erfolgt. Gemäß einem zweiten Aspekt löst die Erfindung das Problem durch eine gattungsgemäße Batterie-Verarbeitungsanlage, bei der die Aktivierungsvorrichtung eine Trocknungsvorrichtung umfasst.

Vorteilhaft an der Erfindung ist, dass durch das Trocknen dem Zerkleinerungsgut so viel Elektrolyt entzogen werden kann, dass eine elektrochemische Reaktion nicht mehr oder nur noch zu vernachlässigbar kleinem Ausmaß möglich ist. Zudem bildet sich keine entzündliche oder explosive Gasphase über den Batteriefragmenten, da diesen insbesondere die organischen Karbonate des Elektrolyten entzogen wurden. Das Zerkleinerungsgut ist daher weitgehend inert und kann, insbesondere dann, wenn es unter Vakuum verpackt wird, sicher transportiert werden.

Ein weiterer Vorteil ist es, dass zum Inaktivieren des Zerkleinerungsmaterials kein zusätzliches Material hinzugegeben werden muss. Das verringert den Aufwand beim Aufarbeiten der Batterien, verringert das Gewicht des inaktivierten Zerkleinerungsguts und erhöht die Reinheit in den folgenden Trenn- und Recyclingschrit-

ten. Insbesondere in potentiell folgenden hydrometallurgischen Aufarbeitungsschritten ist eine hohe Produktreinheit ohne Eintrag von Fremdionen vorteilhaft.

Im Rahmen der vorliegenden Beschreibung wird unter dem Trocknen insbesondere das Entfernen zumindest eines Lösungsmittels des Leitsalzes verstanden. Insbesondere wird das Trocknen so durchgeführt, dass Kohlensäuredimethylester und/oder Kohlensäureethylmethylester entfernt wird.

Unter der Batterie wird insbesondere eine Lithium-Batterie verstanden. Eine Lithium-Batterie ist ein Akkumulator, bei dem die elektrochemische Reaktion Lithium und/oder Lithium-Ionen und/oder eine Lithium-Verbindung involviert.

Unter einer Batterie-Verarbeitungsanlage wird insbesondere auch eine Akku-Verarbeitungsanlage zum Verarbeiten von Akkumulatoren verstanden.

Unter dem Transportbehälter wird insbesondere auch eine oder eine Transportverpackung verstanden. Vorzugsweise wird die Transportverpackung durch Vakuumverschweißung verschlossen. Gut geeignet als Transportverpackung ist insbesondere Aluminium-Verbundfolie.

Günstig ist, wenn das Trocknen nach dem Zerkleinern der Batterien erfolgt. Zwar ist es möglich und stellt eine Ausführungsform der Erfindung dar, dass die Batterien im unzerkleinerten Zustand einem Vakuum ausgesetzt werden, sodass zumindest Teile des Elektrolyten verdampfen, wobei das entstehende Gas entweder durch ein Sicherheitsventil im Akkumulator entweicht oder die Batterie durch die Druckdifferenz zwischen der äußeren Umgebung und dem Innendruck zerstört wird, sodass verdampfender Elektrolyt austreten kann. Da der Elektrolyt zumeist zwischen eng gewickelten oder gestapelten und verpressten Schichten aus Elektroden und Separator und in deren Poren vorliegt und mit anderen Bestandteilen der Batterien verbunden vorliegt, kann dieses Vorgehen jedoch zeitintensiv sein.

Es ist daher oft günstiger und stellt eine bevorzugte Ausführungsform der Erfindung dar, dass die Batterien mechanisch zerkleinert werden, beispielsweise durch Schneiden, Scheren, Pralleinwirkung, Zertrennen und/oder Quetschen. So steht für den Stoffübergang in die Gasphase eine größere Grenzfläche zur Verfügung.

Das Trocknen kann beispielsweise durch Vakuumtrocknung, Kontakttrocknung, Konvektionstrocknung und/oder Strahlungstrocknung erfolgen. Günstig ist es, wenn das Trocknen unter Rühren und/oder Umwälzen des Zerkleinerungsguts erfolgt.

Vor dem Zerkleinern werden die gebrauchten Batterien vorzugsweise demontiert. Das heißt, dass größere Batteriesysteme in ihre kleineren Unterbausteine, die Module oder Stacks, demontiert werden oder sogar die Zellen, die das elektrochemisch wirksame Material enthalten, von der Ansteuerelektronik getrennt werden. Die Ansteuerelektronik umfasst beispielsweise Halbleiterbauelemente und/oder Sensoren und dient der Ladesteuerung der Batterien.

Gemäß einer bevorzugten Ausführungsform erfolgt das Trocknen unter Vakuum. Vorzugweise ist das Vakuum so groß gewählt, dass der Dampfdruck von Kohlensäuredimethylester bei 80°C, insbesondere bei 70°C, unterschritten wird. Günstig ist es, wenn das Trocknen unter einem Druck von höchstens 300 hPa, insbesondere höchstens 100 hPa erfolgt. Bei so niedrigen Drücken verdampfen wesentliche Bestandteile der meisten Elektrolyten, insbesondere Kohlensäuredimethylester und Kohlensäureethylmethylester, bereits bei Temperaturen von weniger als 80°. Niedrige Temperaturen haben den Vorteil, dass die Bildung von Fluorwasserstoff behindert wird. Fluorwasserstoff stellt eine potentielle Gefährdung für die Batterie-Verarbeitungsanlage sowie die Umgebung dar. Es ist daher günstig, das Entstehen von Fluorwasserstoff zu verhindern.

Günstig ist es, wenn das Trocknen unter einer Atmosphäre erfolgt, in der der

Wasser-Partialdruck kleiner ist als 50 Pa, insbesondere kleiner als 10 Pa. Ein geringer Wasser-Partialdruck führt zu einer geringen Reaktionsrate von Lithium-Verbindungen zu Lithium-Hydroxid und damit zu nur geringer Wasserstoff-Entwicklung. Das verhindert die Bildung von zündfähigen Wasserstoff-Sauerstoff-Gemischen und trägt zur Anlagensicherheit bei.

Günstig ist es zudem, wenn der Sauerstoff-Partialdruck höchstens 10 Millibar, insbesondere höchstens 5 Millibar beträgt. Die Reaktion von Sauerstoff mit oxidierbaren Bestandteilen der Batterien wird so weitgehend unterdrückt. Es ist möglich, den niedrigen Sauerstoff-Partialdruck durch einen geringen Druck beim Trocknen zu erreichen. Alternativ oder zusätzlich kann das Trocknen in einer Inertgas-Atmosphäre erfolgen.

Bevorzugt ist ein Verfahren, bei dem das Trocknen des Zerkleinerungsguts beendet wird, bevor sich ein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut bildet und/oder bevor das Zerkleinerungsgut so trocken ist, dass sich während der Weiterverarbeitung ein entzündliches oder explosives Gasgemisch ergibt.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, bis ein Elektrolytgehalt im Zerkleinerungsgut so klein ist, dass eine elektrochemische Reaktion unmöglich ist. In anderen Worten ist der Elektrolytgehalt kleiner als ein Schwellenwert, wobei der Schwellenwert so gewählt ist, dass die Zellspannung bei Unterschreiten dieses Schwellenwerts auf höchstens ein Viertel abgesunken ist. Dieser Schwellenwert wird beispielsweise dadurch bestimmt, dass die Zellenspannung einer Batterie in Abhängigkeit vom Elektrolytgehalt bestimmt wird. Kurz vor dem Erreichen des Schwellenwerts bricht die Zellspannung zusammen, das heißt, dass sie um zumindest 75% absinkt. Ist der Schwellenwert unterschritten, enthält die Batterie so wenig Elektrolyt, dass in guter Näherung keine elektrochemische Reaktion mehr möglich ist.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, dass eine Menge von 50 kg an Zerkleinerungsmaterial, das kompaktiert in einem 50 Liter-Fass enthalten ist, keine oder eine nur so geringe Wärmeentwicklung zeigt, dass ein thermisches Durchgehen, also eine thermisch induzierte Kettenreaktion, für zumindest zwei Monate ausgeschlossen ist und dass eine etwaige Wasserstoff-Entwicklung zudem so gering ausgebildet ist, dass nach zwei Wochen kein Überdruck entsteht, wenn zu Beginn ein Unterdruck von 500 hPa herrscht.

Günstig ist es, wenn das Zerkleinerungsgut so lange getrocknet wird, bis der Elektrolytgehalt an organischen Komponenten, die bei 80°C flüchtig sind, höchstens 3 Gew.-%, höchstens insbesondere 2 Gew.-% beträgt.

Vorzugsweise wird das Trocknen so lange durchgeführt, dass der akkumulierte Gehalt an organischen Karbonaten aus dem Elektrolyten, die bei 80°C flüchtig sind, in der Atmosphäre über dem Zerkleinerungsgut 3 Vol.-% unterschreitet.

Insbesondere wird das Trocknen so lange durchgeführt, bis der Gehalt an Kohlensäuredimethylester 4 Vol.-%, insbesondere 3 Vol.-%, unterschreitet und/oder der Gehalt an Cyclohexylbenzol 1 Vol.-%, insbesondere 0,5 Vol.-%, unterschreitet.

Vorzugsweise erfolgt das Trocknen unmittelbar nach dem Zerkleinern. Hierunter wird insbesondere verstanden, dass zwischen dem Beginn des Zerkleinerns der Batterien und dem Zeitpunkt, zu dem zumindest ein Teil des dadurch entstandenen Zerkleinerungsguts anfängt zu Trocknen, höchstens fünf Minuten beträgt, insbesondere höchstens eine Minute. Durch die schnelle Trocknung nach dem Zerkleinern wird die Masse an Material klein gehalten, die potentiell elektrochemisch reagieren könnte, zudem wird die elektrochemische Reaktionszeit potentieller exothermer Reaktionen klein gehalten. Das senkt das Risiko für die Anlage und die Umgebung.

Besonders günstig ist es, wenn das Vakuum mittels einer Strahlpumpe erzeugt wird. Strahlpumpen sind, insbesondere bei geeigneter Wahl des Strahlmediums, weitgehend unempfindlich gegen aggressive abzupumpende Gase. Günstig ist es, wenn das Strahlmedium, das eine Flüssigkeit ist, einen pH-Wert von zumindest 8, insbesondere von zumindest 9, beispielsweise zumindest 12, hat. In diesem Fall können sich unerwünschte Bestandteile des Gases, das abgepumpt wird, zersetzen oder zu weniger schädlichen Substanzen reagieren. So kann beispielsweise Kohlensäuredimethylester und/oder Kohlensäureethylmethylester durch eine Verseifungsreaktion abgebaut werden. Etwaig im Strahlenmedium enthaltener Fluorwasserstoff kann im basischen Milieu in einer Säure-Base-Reaktion zu einem ungefährlichen Salz umgesetzt werden.

Vorzugsweise enthält das Strahlenmedium eine Fluor fällende Substanz. Beispielsweise kann das Strahlenmedium Natriumkarbonat oder Kalziumkarbonat enthalten. Die bei der Reaktion mit einer Fluorverbindung, insbesondere Fluorwasserstoff, entstehenden Salze werden vorzugsweise abgetrennt, insbesondere abgefiltert oder aussedimentiert. Auf diese Weise wird zumindest weitgehend verhindert, dass Fluorwasserstoff oder andere giftige fluorhaltige Verbindungen in die Umgebung abgegeben werden können.

Vorzugsweise erfolgt das Trocknen bei einer Temperatur von höchstens 80°. In diesem Fall entsteht kaum Fluorwasserstoff. Das erhöht die Lebensdauer der Batterie-Verarbeitungsanlage und verringert die Umweltgefährdung.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte des Kondensierens von Bestandteilen des Elektrolyten durch Abkühlen und/oder Druckerhöhen, sodass ein Elektrolytkondensat entsteht. Beispielsweise wird das Kondensieren an einer Stelle durchgeführt, die bezüglich des Gasflusses zwischen dem Trockner und der Vakuumpumpe liegt. Vom Trockner kommende Gase müssen daher in diesem Fall zunächst einen Kondensator passieren, bevor

sie zur Vakuumpumpe gelangen können. Das führt dazu, dass gasförmiger Elektrolyt, der in dem Gas vorliegt, das beim Trocknen entsteht, zumindest zu einem überwiegenden Teil im Kondensator abgeschieden wird, bevor das verbleibende Gas zur Pumpe gelangt. So kann Elektrolyt zurückgewonnen werden. Zudem verringert sich der Gasstrom durch die Vakuumpumpe, was deren Lebensdauer verlängert und deren Energieverbrauch reduziert.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren alternativ den Schritt der Gasreinigung durch Adsoption der flüchtigen organischen Bestandteile an einem Aktivkohlfilter vor oder nach der Verdichtereinheit.

Vorzugsweise umfasst das erfindungsgemäße Verfahren alternativ oder zusätzlich den Schritt eines Reinigens des Gases, das beim Trocknen entsteht, bevor dieses die Vakuumpumpe erreicht. Das kann beispielsweise auch dadurch erfolgen, dass das Gas einen Aktivkohlefilter und/oder einen Filter passiert, in dem mit Fluorwasserstoff reagierende Substanzen enthalten sind, beispielsweise ein Kalziumsalz wie Kalziumkarbonat.

Vorzugsweise umfasst das erfindungsgemäße Verfahren den Schritt eines Trocknens bei einer Trocken-Temperatur und für eine Trocken-Dauer, die so gewählt sind, dass sich der Binder, der Aktivmaterial der Lithium-Batterie an einen Träger bindet, zumindest zum überwiegenden Teil zersetzt. Günstig ist es, wenn dieser Trocknungsschritt, der auch als Hochtemperatur-Trocknen bezeichnet werden kann, räumlich getrennt von einem ersten, oben beschriebenen Trocknungsschritt durchgeführt wird. Der letztgenannte Trocknungsschritt kann auch als Niedertemperatur-Trocknen bezeichnet werden.

Vorzugsweise wird das Hochtemperatur-Trocknen, bei dem sich der Binder zersetzt, so durchgeführt, dass die entstehenden Zersetzungsgase sich nicht mit den Gasen mischen, die beim Niedrigtemperatur-Trocknen entstehen. Es ist möglich,

dass das Hochtemperatur-Trocknen und das Niedertemperatur-Trocknen bei unterschiedlichen Drücken erfolgt. Beispielsweise kann das Hochtemperatur-Trocknen bei Normaldruck erfolgen.

Unter dem Aktivmaterial wird der Stoff verstanden, der beim Betrieb der Batterien elektrochemisch reagiert. Unter dem Träger für das Aktivmaterial wird insbesondere eine Trägerfolie verstanden, auf der das Aktivmaterial in Form von Partikeln aufgebracht ist. Beispielsweise handelt es sich bei der Trägerfolie um eine Folie aus Aluminium oder eine Aluminiumlegierung. Der Binder ist der Stoff, der das Aktivmaterial mit dem Träger verbindet, beispielsweise enthält der Binder Polyvinylidenfluorid.

Günstig ist es, wenn beim Zerkleinern der Batterien flüssiger Stickstoff zugegeben wird. Das kühlt die Batterien, die Zerkleinerungsmaschine und das Zerkleinerungsgut und verdrängt zudem Sauerstoff und Wasserdampf aus der Atmosphäre.

Günstig ist es, wenn das Zerkleinern bei dem Wasserdampf-Partialdruck von höchstens 20 Pa und/oder einem Sauerstoff-Partial von höchstens 40 hPa, insbesondere höchstens 15 hPa erfolgt.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte eines Entnehmens des Zerkleinerungsguts aus dem Transportbehälter, eines Abtrennens von Hartteilen und/oder eines Separierens von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfaktion entstehen, und eines getrennten Verpackens von Aktivmaterialfraktion und Trägerfraktion in jeweilige Transportbehältnisse. Günstig ist es, wenn diese Transportbehältnisse luftdicht ausgebildet sind. Durch das Trennen von Aktivmaterialfraktion und Trägerfraktion ist ein Transport in der Regel ohne Genehmigungen möglich. Ein weiterer Vorteil ist, dass derartig getrennte Fraktionen nur eine geringe Gefährdung darstellen.

In einer bevorzugten Batterie-Verarbeitungsanlage sind die Trennungseinheit und die Trockenvorrichtung in einem gemeinsamen Standard-Container angeordnet. Das hat den Vorteil, dass die Batterie-Verarbeitungsanlage besonders einfach transportiert werden kann.

Günstig ist es, wenn die Trockenvorrichtung ein Rührwerkzeug aufweist, beispielsweise einen Ankerrührer oder einen Stabrührer, dessen Rührstäbe quer zu einer Rührwelle angebracht sein können. Alternativ oder zusätzlich ist das Rührwerkzeug ein externes Rührwerkzeug, das den Trockner als Ganzes bewegt.

Vorzugsweise besitzt die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung zum Erzeugen eines Vakuums in der Trockenvorrichtung verbunden ist. Besonders günstig ist es, wenn auch die Vakuumanlage im Standard-Container angeordnet ist. Bei dem Standard-Container handelt es sich vorzugweise um einen 40-Fuß-Container. Beispielweise umfasst die Vakuumanlage eine Strahlpumpe, bei der ein Strahlmedium zum Erzeugen des Unterdrucks verwendet wird.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionabtrennvorrichtung , eine Separiervorrichtung, insbesondere eine Klassiervorrichtung, zum Separieren von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht, und eine zweite Abfüllvorrichtung zum getrennten Abfüllen von Aktivmaterialfraktion und Trägerfraktion. Günstig ist es, wenn diese Abfüllvorrichtung zum Abfüllen unter Unterdruck und/oder unter Inertgas ausgebildet ist.

Unter einer Hartmetallabtrennvorrichtung wird insbesondere eine Vorrichtung zur Abtrennung von Fragmenten peripherer Bestandteile des Betriebssystems, der Batteriezellhülle und der Strom-Kontakte verstanden. Beispielsweise umfasst die

Hartmetallabtrennvorrichtung eine Magnetseparationsvorrichtung und/oder einen Sichter, insbesondere einen Querstromsichter und/oder einen Zickzack-Sichter. Unter der Separiervorrichtung wird insbesondere eine Vorrichtung zum zur Abtrennen der Separatorfolie verstanden.

Die Leichtfraktionabtrennvorrichtung umfasst vorzugsweise einen Zickzack-Sichter und/oder einen Lufttrenner, wobei es günstig ist, wenn die Luft im Kreislauf geführt wird. Das reduziert die Staubexposition der Umwelt mit gesundheitsschädlichem Staub.

Vorzugsweise sind die zweite Abfüllvorrichtung und die Separiervorrichtungen in einem gemeinsamen Standard-Container angeordnet, beispielsweise im oben beschrieben ersten Standard-Container, oder einem zweiten Standard-Container. Günstig ist es, wenn der Container staubdicht gekapselt ist.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Schleuse zwischen der Zerkleinerungseinheit und der Inaktivierungsvorrichtung. Beispielsweise handelt es sich um eine Zellenradschleuse.

Im Folgenden wird die Erfindung anhand der beigefügten Zeichnungen näher erläutert. Dabei zeigt

Figur 1 ein Ablaufdiagramm eines erfindungsgemäßen Verfahrens,

Figur 2 einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage und

Figur 3 einen Querschnitt durch weitere, optionale Bestandteile einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Figur 1 zeigt das Ablaufdiagramm eines erfindungsgemäßen Verfahrens. Zunächst werden Batterien 10.1, 10.2, ..., insbesondere Batteriesysteme bestehend aus mehreren Batteriemodulen oder Batteriestacks, welche wiederum aus mehreren Batteriezellen bestehen, in einer Entladeeinheit 12 entladen. Danach folgt an einer Demontagestation 14 die Demontage der Batterien 10, wenn diese erforderliche ist, weil die Batteriesysteme ansonsten aus geometrischen oder gravimetrischen Gründen nicht in die Zerkleinerungseinheit befördert werden können. Dazu werden die Batteriesysteme geöffnet und soweit demontiert, dass die Module/Stacks einzeln entfernt werden können. Gegebenenfalls können auch die Zellen von der Ansteuerelektronik getrennt werden. Die entstehenden Untereinheiten (Module/Stacks) und/oder Zellen 16.1, 16.2, ... werden einer Zerkleinerungseinheit 18 zugeführt, die beispielsweise eine Rotorschere mit einem Rotor und einen mit Statoren oder mehreren Rotoren, oder eine Schneidmühle mit einem Rotor und mehreren Rotoren umfasst.

Die Zerkleinerungseinheit 18 zerkleinert die Batterien 10 unter Schutzgas 20, das beispielsweise einer Schutzgasflasche entnommen wird. Alternativ oder zusätzlich kann flüssiger Stickstoff aus einer Flüssigstickstoffquelle 19 zugedüst werden. Bei dem Schutzgas kann es sich beispielsweise um Stickstoff, ein Edelgas, Kohlendioxid, Lachgas oder ein sonstiges, vorzugweise nicht giftiges Gas handeln.

Beim Zerkleinern entsteht Zerkleinerungsgut 24, das einer Inaktivierungsvorrichtung in Form einer Trockenvorrichtung 26 zugeführt wird. Zwischen der Zerkleinerungseinheit 18 und der Trockenvorrichtung 26 ist eine Schleuse 28 angeordnet, die soweit gasdicht ist, dass die Druckvorrichtung 26 in guter Näherung von der Zerkleinerungseinheit 18 gasdicht getrennt ist.

Die Trockenvorrichtung 26 ist mit einer Vakuumanlage 29 verbunden, die eine Vakuumpumpe 30 aufweist und ein Vakuum erzeugt. In der Trockenvorrichtung 26 herrscht ein Druck p_{26} von $p_{26} \approx 100$ hPa, vorzugsweise 50 hPa. Es sei darauf

hingewiesen, dass im Rahmen der vorliegenden Beschreibung unter einer Vakuumpumpe insbesondere ganz allgemein eine Vorrichtung verstanden wird, die ein Vakuum erzeugt. Es ist dabei möglich und bevorzugt, nicht aber notwendig, dass die Vakuumpumpe gleichzeitig als Verdichter wirkt, so dass von ihr Gas unter einem Druck abgegeben wird, der größer ist als der Umgebungsdruck.

In dem in Figur 1 gezeigten Fall handelt es sich bei der Vakuumpumpe um einen Verdichter, der Gas 31, das in der Trockenvorrichtung 26 vorliegt, ansaugt und verdichtet. Alternativ oder zusätzlich kann die Vakuumanlage 29 eine Strahlpumpe aufweisen, bei der ein Strahlmedium in Form einer Flüssigkeit mit hoher Geschwindigkeit durch Venturi-Düse geleitet wird. Das Strahlmedium ist alkalisch und hat einen pH-Wert von zumindest pH 13 und ist beispielsweise eine 10%ige Kaliumhydroxidlösung.

Die Vakuumanlage 29 umfasst eine Gasreinigungsvorrichtung 32, die zwischen der Trockenvorrichtung 26 und der Vakuumpumpe 30 angeordnet ist und die im vorliegenden Fall einen Kondensator 34 und/oder einen Aktivkohlefilter 36 umfasst. Der Kondensator wird bei einer Temperatur von -10 °C betrieben, so dass Kohlensäuredimethylester und Kohlensäureethylmethylester kondensieren und in einen Kondensatbehälter 38 abgegeben werden können. Zudem wird etwaig vorhandenes Wasser ausgefroren. Ein Steuerventil 40 ist ausgebildet zum Öffnen, wenn der Druck p_{26} zu groß wird und zum Zufahren, wenn der Druck p_{26} zu klein wird, das heißt, dass ein vorgegebener Schwellenwert unterschritten wird.

Vorzugsweise ist das Trocknungsgut während der Trocknung bewegt. Dies kann beispielsweise durch langsames Rühren mit einem Rührwerkzeug 41, wie einem Ankerrührer oder einem Stabrührer mit senkrecht zur Rührwelle angebrachten Stäben passieren. Alternativ durch einen bewegten Trocknungsbehälter.

Durch das Trocknen des Zerkleinerungsguts entsteht inaktiviertes Zerkleine-

rungsgut 42, das einer Abfüllvorrichtung 44 zugeführt wird. Dort wird das inaktivierte Zerkleinerungsgut 42 unter Vakuum und/oder unter Schutzgas in einen Transportbehälter 46 abgefüllt. Der Transportbehälter 46 ist vorzugsweise gasdicht. Es ist möglich, nicht aber notwendig, dass der Transportbehälter 46 vor dem Abtransport mit Inertgas aufgefüllt wird, so dass er unter Normaldruck steht. Alternativ ist es auch möglich, dass der Transportbehälter unter Vakuum verschlossen und transportiert wird. Es ist möglich, dass alternativ zum Transportbehälter eine vakuumverschweißte Folie, beispielsweise eine Aluminiumverbundfolie, gewählt wird.

Über eine Spülleitung 48 wird der Zerkleinerungseinheit 18 Schutzgas 20 von der Vakuumpumpe 30 zugeführt. Arbeitet die Vakuumpumpe 30 wie im vorliegenden Fall, der eine bevorzugte Ausführungsform darstellt, zudem als Kompressor, kann das Schutzgas auf eine Druckgasflasche 50 gezogen werden. Alternativ oder zusätzlich kann das Schutzgas 20, gegebenenfalls nach zusätzlicher Reinigung, in die Umgebung abgegeben werden.

Figur 2 zeigt schematisch einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage 52, die einen Standard-Container 54 aufweist, in dem die Zerkleinerungseinheit 18, die Trockenvorrichtung 26 und die Abfüllvorrichtung 44 angeordnet sind. Hinter der Zerkleinerungseinheit 18 ist ein erster gasdichter Förderer 56 angeordnet, der beispielsweise eine Förderschnecken oder einen Rohrkettenförderer umfasst. Der erste Förderer 56 fördert das Zerkleinerungsgut 24 zur Trockenvorrichtung 26, die mit der in Figur 2 nicht sichtbaren Vakuum-Erzeugungsvorrichtung verbunden ist. In Materialstromrichtung hinter der Trockenvorrichtung 26 ist ein zweiter Förderer 58 angeordnet, der vorzugsweise ebenfalls gasdicht ausgebildet ist und eine Förderschnecke oder einen Rohrkettenförderer enthalten kann. Der zweite Förderer fördert das inaktivierte Zerkleinerungsgut 42 zur Abfüllvorrichtung 44.

Figur 3 zeigt optionale, in der vorliegenden Ausführungsform vorhandene Einheiten der erfindungsgemäßen Batterie-Verarbeitungsanlage 52, die einen Aufschlusszerkleinerer 60 sowie einen Sichter 62 umfasst. Der Aufschlusszerkleinerer 60 enthält eine Transportbehälter-Entleervorrichtung 64, mittels der aus dem Transportbehälter 46 inaktiviertes Zerkleinerungsgut 42 entnommen werden kann. Der Aufschlusszerkleinerer 60 erzeugt Aufschlussmaterial 66, das dem Sichter 62 zugeführt wird. Es kann sich bei dem Sichter beispielsweise um einen Zickzack-Sichter handeln.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage 52 einen Zerkleinerer, der vorzugsweise im Materialfluss vor der Klassiervorrichtung 74 steht und ein schnell laufendes Zerkleinerungswerkzeug enthält, wobei eine Rotorumfangsgeschwindigkeit größer ist als 1 m/s, vorzugsweise größer als 10 m/s. Dieser Zerkleinerer zerkleinert das Zerkleinerungsmaterial und beansprucht es mechanisch so, dass sich die elektrochemisch aktive Beschichtung mindestens teilweise vom Träger löst. Das Vorhandensein eines solchen Zerkleinerer ist ein allgemeines bevorzugtes Merkmal einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Im Sichter entstehen eine Leichtfraktion mit Separatorfolie und feinem Beschichtungsmaterial und eine Schwergutfraktion mit Trägerfolien (Aluminium- und Kupfer) mit größerem leicht anhaftender Beschichtung. Beide Fraktionen werden auf jeweils ein Sieb zur weiteren Trennung in Beschichtung und Separatorfolie beziehungsweise Beschichtung und Metallfolien gegeben. Die entstehenden Fraktionen werden getrennt weiter verarbeitet.

Das Aufschlussmaterial 66 wird dem Sichter 62 mittels einem dritten Förderer 68 zugeführt. Ein vierter Förderer 70 führt gesichtetes Material 72, insbesondere das Material der Leichtfraktion, das den Sichter 62 verlässt, einer Klassiervorrichtung 74 zu. Die Klassiervorrichtung 74 umfasst vorzugsweise ein Luftstrahlsieb, das gleichzeitig als Trennvorrichtung zum Trennen des Aktivmaterials von dem Träger fungiert. Durch das Trennen entsteht eine Aktivmaterialfraktion 76, die in ein

Transportbehältnis 78 abgefüllt wird.

Es entsteht zudem eine Trägerfraktion 80, die in der vorliegenden Ausführungsform mit einem fünften Förderer 82 einer Abfülleinheit 84 zugeführt wird, die die Trägerfraktion 80 in einen Behälter 86 füllt. Die Abfülleinheit 84 ist zusammen mit einer zweiten Abfülleinheit 88 Teil einer zweiten Abfüllvorrichtung.

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Bezugsleistenliste

10	Batterie
12	Entladeeinheit
14	Demotagesstation
16	Zelle
18	Zerkleinerungseinheit
19	Flüssigstickstoffquelle
20	Schutzgas
22	Schutzgasflasche
24	Zerkleinerungsgut
26	Trockenvorrichtung
28	Schleuse
29	Vakuumanlage
30	Vakuumpumpe
31	Gas
32	Gasreinigungsvorrichtung
34	Kondensator
36	Aktivkohlefilter
38	Kondensatbehälter
40	Steuerventil
41	Rührwerkzeug
42	inaktives Zerkleinerungsgut
44	Abfüllvorrichtung
46	Transportbehälter
48	Spülleitung
50	Druckgasflasche
52	Batterie-Verarbeitungsanlage
54	Standard-Container
56	erster Förderer
58	zweiter Förderer
60	Aufschlusszerkleinerer
62	Sichter
64	Transportbehälter-Entleervorrichtung
66	Aufschlussmaterial
68	dritter Förderer
70	vierter Förderer
72	gesichtetes Material
74	Klassiervorrichtung
76	Aktivmaterialfraktion
78	Transportbehältnis
80	Trägerfraktion
82	fünfter Förderer
84	Abfülleinheit
86	Behälter
88	zweite Abfülleinheit
p	Druck

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Patentansprüche:

1. Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten:
   (a) Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,
   (b) Inaktivieren des Zerkleinerungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und
   (c) Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehälter (46),
   dadurch gekennzeichnet, dass
   (d) das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass das Trocknen unter Vakuum erfolgt.

3. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Zerkleinerungsgut (24) solange getrocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

4. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass
   das Vakuum mittels einer Strahlpumpe erzeugt wird und
   ein Strahlmedium der Strahlpumpe
   einen pH-Wert von zumindest 8, insbesondere 9, hat und/oder
   eine Fluor fällende Substanz enthält.

5. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch die Schritte:

(e) Entnehmen des Zerkleinerungsguts (24) aus dem Transportbehälter (46),

(f) Abtrennen von Hartteilen und/oder
Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion (80) entsteht, und

(g) getrenntes Verpacken von Aktivmaterialfraktion (76) und Trägerfraktion (80) in Transportbehältnisse (78).

6. Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere Lithium-Batterien, mit:

(a) einer Zerkleinerungseinheit (18) zum Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,

(b) einer Inaktivierungsvorrichtung (26) zum Inaktivieren des Zerkleinerungsguts (24) und

(c) einer Abfüllvorrichtung (44) zum Abfüllen des inaktivierten Zerkleinerungsguts (24) in einen Transportbehälter (46),

dadurch gekennzeichnet, dass

(d) die Inaktivierungsvorrichtung eine Trockenvorrichtung (26) umfasst.

7. Batterie-Verarbeitungsanlage nach Anspruch 6, dadurch gekennzeichnet, dass
die Zerkleinerungseinheit und die Trockenvorrichtung (26) in einem Standard-Container (54) angeordnet sind.

8. Batterie-Verarbeitungsanlage nach Anspruch 6 oder 7, gekennzeichnet durch eine Vakuumanlage, die mit der Trockenvorrichtung (26) zum Erzeugen eines Vakuums in der Trockenvorrichtung (26) verbunden ist.

Gramm, Lins & Partner
Patent- und Rechtsanwaltssozietät GbR

9. Batterie-Verarbeitungsanlage nach Anspruch 8, dadurch gekennzeichnet, dass
   - die Vakuumanlage (29) eine Strahlpumpe mit einem Strahlmedium umfasst,
   - das Strahlmedium im Kreis geführt ist und
   - das Strahlmedium eine mit Fluorwasserstoff reagierende Substanz enthält

10. Batterie-Verarbeitungsanlage nach einem der Ansprüche 6 bis 9, gekennzeichnet durch
   - eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionsabtrennvorrichtung,
   - eine Separiervorrichtung, insbesondere eine Klassiervorrichtung (74), zum Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben und/oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion entsteht, insbesondere Aluminium- und Kupferfolien, und
   - eine zweite Abfüllvorrichtung (44) zum getrennten Abfüllen von Aktivmaterialfraktion (76) und Trägerfraktion (80), insbesondere Aluminium- und Kupferfolien.

PL/be

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5569-002 DE-1

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28. April 2015

Zusammenfassung

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten: Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird, Inaktivieren des Zerkleinerungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehälter (46). Erfindungsgemäß ist vorgesehen, dass das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt.

Figur 1

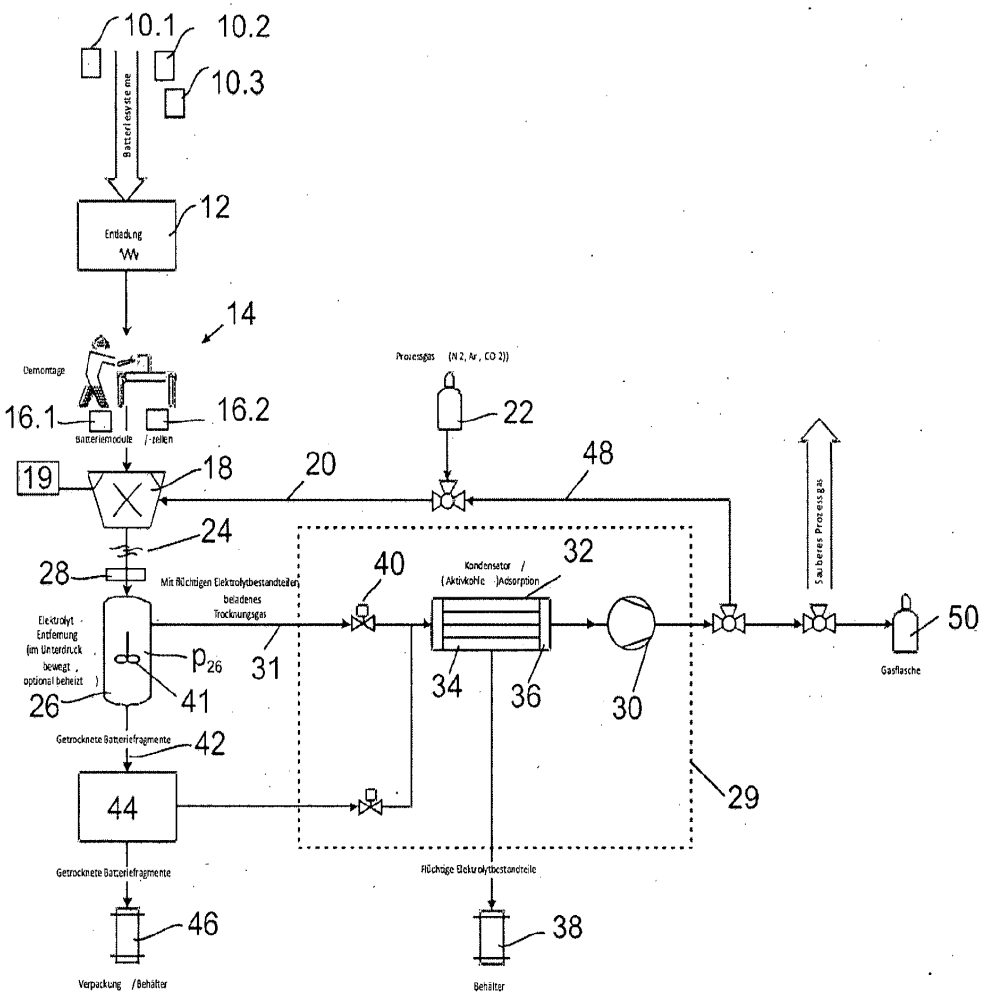
10.1
10.2
10.3
Batteriesysteme
12
Entladung
14
Demontage
16.1
16.2
Batteriemodule
/-zellen
19
18
20
Prozessgas (N 2, Ar, CO 2))
22
48
24
28
Mit flüchtigen Elektrolytbestandteilen beladenes Trocknungsgas
40
Kondensator / (Aktivkohle-)Adsorption
32
Sauberes Prozessgas
50
Gasflasche
Elektrolyt Entfernung (im Unterdruck bewegt, optional beheizt)
p_{26}
31
34
36
30
26
41
Getrocknete Batteriefragmente
42
44
29
Getrocknete Batteriefragmente
Flüchtige Elektrolytbestandteile
46
38
Verpackung /Behälter
Behälter

Fig. 1

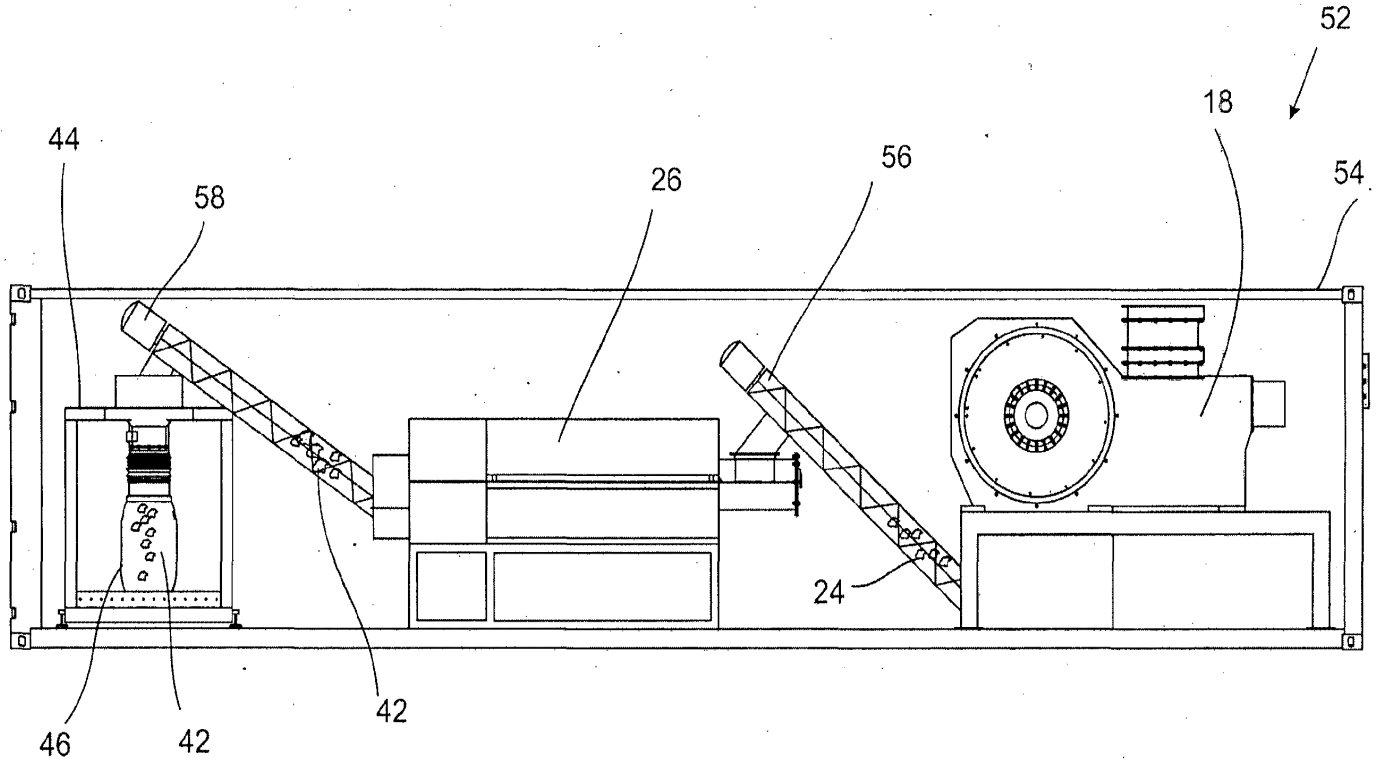

Fig. 2

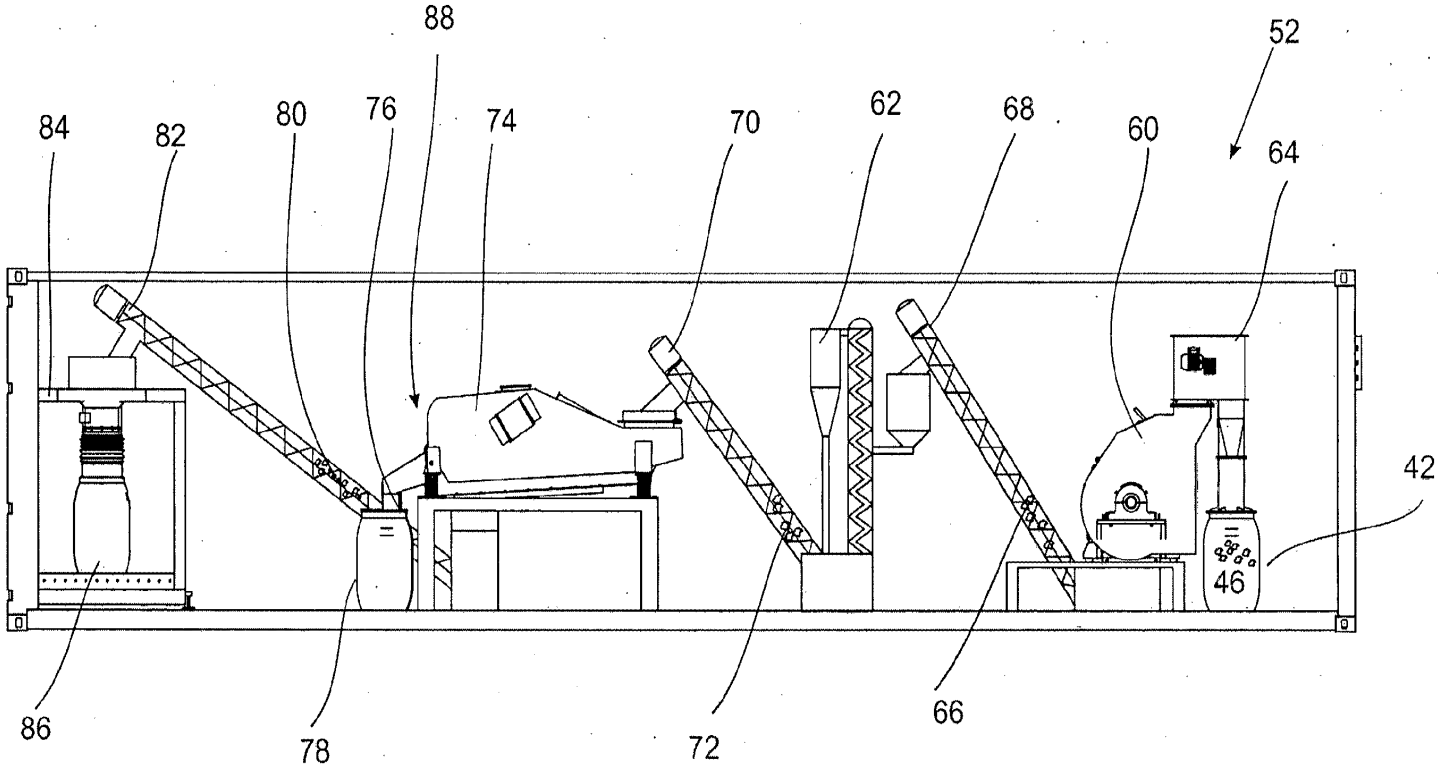

Fig. 3

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Erklärungen gemäß Regel 4.17:

— *hinsichtlich der Berechtigung des Anmelders, ein Patent zu beantragen und zu erhalten (Regel 4.17 Ziffer ii)*

[Fortsetzung auf der nächsten Seite]

(54) **Title**: METHOD FOR THE TREATMENT OF USED BATTERIES, IN PARTICULAR RECHARGEABLE BATTERIES, AND BATTERY PROCESSING INSTALLATION

(54) **Bezeichnung** : VERFAHREN ZUM BEHANDELN GEBRAUCHTER BATTERIEN, INSBESONDERE WIEDER AUFLADBARER BATTERIEN UND BATTERIE-VERARBEITUNGSANLAGE

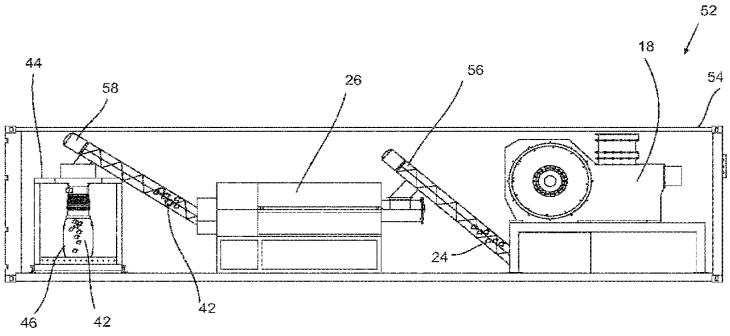

Fig. 2

(57) **Abstract**: The invention relates to a method for the treatment of used batteries (10), in particular lithium batteries, having the steps: comminuting the batteries (10) such that comminuted material (24) is obtained, inactivating the comminuted material (24) such that inactivated comminuted material (42) is obtained, and filling a transport container (46) with the inactivated comminuted material (42). It is provided according to the invention that the inactivation is performed by drying the comminuted material (24), and the comminuted material (24) is dried until an electrolyte content is so low that an electrochemical reaction is not possible.

(57) **Zusammenfassung**:

[Fortsetzung auf der nächsten Seite]

WO 2016/174156 A1

Veröffentlicht:

— *mit internationalem Recherchenbericht (Artikel 21 Absatz 3)*

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten: Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird, Inaktivieren des Zerkleine- rungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehäl- ter (46). Erfindungsgemäß ist vorgesehen, dass das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt und das Zerkleinerungsgut (24) solange ge- trocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

Verfahren zum Behandeln gebrauchter Batterien, insbesondere wieder aufladbarer Batterien und Batterie-Verarbeitungsanlage

Die Erfindung betrifft ein Verfahren zum Behandeln gebrauchter Batterien, insbesondere gebrauchter Lithium-Batterien, beispielsweise Lithium-Ionen-Batterien, mit den Schritten (a) Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) Inaktivieren des Zerkleinerungsguts, sodass ein inaktiviertes Zerkleinerungsgut erhalten wird, und (c) Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Gemäß einem zweiten Aspekt betrifft die Erfindung eine Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere zum Behandeln gebrauchter Lithium-Batterien, mit (a) einer Zerkleinerungseinheit zum Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird, (b) einer Inaktivierungsvorrichtung zum Inaktivieren des Zerkleinerungsguts und (c) einer Abfüllvorrichtung zum Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter.

Aus der US 2005/0241943 A1 ist ein Verfahren zum Aufarbeiten gebrauchter Batterien bekannt, bei dem die Batterien vor einem Zerkleinerungsschritt erwärmt werden, sodass Kunststoffbestandteile der Batterien zerstört werden. Nachteilig an einem derartigen Vorgehen ist, dass die verbleibenden Komponenten der Batterien mit Abbauprodukten des Kunststoffs verunreinigt sein können.

Aus der DE 10 2012 024 876 A1 ist ein System zum Überführen transportkritischer Elektrolytzellen zunächst unter Inertgas zerkleinert und danach mit einem Desaktivierungspulver bestäubt werden, um eine Selbstentzündung des elektrochemisch aktiven Materials zu verhindern. Nachteilig daran ist, dass das entstehende Material dennoch ein vergleichsweise hohes Gefährdungspotential besitzt und dass das Bestäubungspulver selbst ein Explosionsrisiko darstellt und dass die Bildung einer entzündlichen und explosiven Atmosphäre in dem Transportbehälter nicht ausgeschlossen werden kann.

Aus der DE 10 2011 110 083 A1 ist ein Verfahren zum Wiedergewinnen von Aktivmaterial aus einer galvanischen Zelle bekannt, bei dem die galvanischen Zellen zunächst mechanisch zerkleinert, dann vorgetrocknet und nachfolgend gesichtet werden. Schließlich wird der Binder in einem Ofen zersetzt. Eine derartige Vorrichtung eignet sich sehr gut zum effizienten Recyceln größerer Mengen von galvanischen Elementen. Für den Teillast-Betrieb ist diese Anlage jedoch vergleichsweise komplex aufgebaut.

Der Erfindung liegt die Aufgabe zugrunde, Nachteile im Stand der Technik zu vermindern.

Die Erfindung löst das Problem durch ein gattungsgemäßes Verfahren, bei dem das Inaktivieren zumindest auch durch Trocknen des Zerkleinerungsguts erfolgt. Gemäß einem zweiten Aspekt löst die Erfindung das Problem durch eine gattungsgemäße Batterie-Verarbeitungsanlage, bei der die Aktivierungsvorrichtung eine Trocknungsvorrichtung umfasst.

Vorteilhaft an der Erfindung ist, dass durch das Trocknen dem Zerkleinerungsgut so viel Elektrolyt entzogen werden kann, dass eine elektrochemische Reaktion nicht mehr oder nur noch zu vernachlässigbar kleinem Ausmaß möglich ist. Zudem bildet sich keine entzündliche oder explosive Gasphase über den Batteriefragmenten, da diesen insbesondere die organischen Karbonate des Elektrolyten entzogen wurden. Das Zerkleinerungsgut ist daher weitgehend inert und kann, insbesondere dann, wenn es unter Vakuum verpackt wird, sicher transportiert werden.

Ein weiterer Vorteil ist es, dass zum Inaktivieren des Zerkleinerungsmaterials kein zusätzliches Material hinzugegeben werden muss. Das verringert den Aufwand beim Aufarbeiten der Batterien, verringert das Gewicht des inaktivierten Zerkleinerungsguts und erhöht die Reinheit in den folgenden Trenn- und Recyclingschritten. Insbesondere in potentiell folgenden hydrometallurgischen Aufarbeitungsschritten ist eine hohe Produktreinheit ohne Eintrag von Fremdionen

vorteilhaft.

Vorteilhaft ist zudem, dass ein transportsicheres Zerkleinerungsgut erhalten wird. So ist der Gehalt an organischen Carbonaten vorzugsweise so klein, dass die Bildung von Fluorophosphaten in relevanter Menge ausgeschlossen werden kann. Fluorophosphate sind oft starke Nervengifte, deren Bildung sicher verhindert werden muss. Zudem ist sichergestellt, dass es aufgrund des geringen Elektrolytgehalts nicht zu einer durch eine elektrochemische Reaktion ausgelöste, selbstverstärkend fortschreitende Wärmeentwicklung kommen kann.

Im Rahmen der vorliegenden Beschreibung wird unter dem Trocknen insbesondere das Entfernen zumindest eines Lösungsmittels des Leitsalzes verstanden. Insbesondere wird das Trocknen so durchgeführt, dass Kohlensäuredimethylester und/oder Kohlensäureethylmethylester entfernt wird.

Unter der Batterie wird insbesondere eine Lithium-Batterie verstanden. Eine Lithium-Batterie ist ein Akkumulator, bei dem die elektrochemische Reaktion Lithium und/oder Lithium-Ionen und/oder eine Lithium-Verbindung involviert.

Unter einer Batterie-Verarbeitungsanlage wird insbesondere auch eine Akku-Verarbeitungsanlage zum Verarbeiten von Akkumulatoren verstanden.

Unter dem Transportbehälter wird insbesondere auch eine oder eine Transportverpackung verstanden. Vorzugsweise wird die Transportverpackung durch Vakuumverschweißung verschlossen. Gut geeignet als Transportverpackung ist insbesondere Aluminium-Verbundfolie.

Günstig ist, wenn das Trocknen nach dem Zerkleinern der Batterien erfolgt. Zwar ist es möglich und stellt eine Ausführungsform der Erfindung dar, dass die Batterien im unzerkleinerten Zustand einem Vakuum ausgesetzt werden, sodass zumindest Teile des Elektrolyten verdampfen, wobei das entstehende Gas entweder durch ein Sicherheitsventil im Akkumulator entweicht oder die Batterie durch die Druckdifferenz zwischen der äußeren Umgebung und dem Innendruck

zerstört wird, sodass verdampfender Elektrolyt austreten kann. Da der Elektrolyt zumeist zwischen eng gewickelten oder gestapelten und verpressten Schichten aus Elektroden und Separator und in deren Poren vorliegt und mit anderen Bestandteilen der Batterien verbunden vorliegt, kann dieses Vorgehen jedoch zeitintensiv sein. Es ist daher oft günstiger und stellt eine bevorzugte Ausführungsform der Erfindung dar, dass die Batterien mechanisch zerkleinert werden, beispielsweise durch Schneiden, Scheren, Pralleinwirkung, Zertrennen und/oder Quetschen. So steht für den Stoffübergang in die Gasphase eine größere Grenzfläche zur Verfügung.

Das Trocknen kann beispielsweise durch Vakuumtrocknung, Kontakttrocknung, Konvektionstrocknung und/oder Strahlungstrocknung erfolgen. Günstig ist es, wenn das Trocknen unter Rühren und/oder Umwälzen des Zerkleinerungsguts erfolgt.

Vor dem Zerkleinern werden die gebrauchten Batterien vorzugsweise demontiert. Das heißt, dass größere Batteriesysteme in ihre kleineren Unterbausteine, die Module oder Stacks, demontiert werden oder sogar die Zellen, die das elektrochemisch wirksame Material enthalten, von der Ansteuerelektronik getrennt werden. Die Ansteuerelektronik umfasst beispielsweise Halbleiterbauelemente und/oder Sensoren und dient der Ladesteuerung der Batterien.

Gemäß einer bevorzugten Ausführungsform erfolgt das Trocknen unter Vakuum. Vorzugweise ist das Vakuum so groß gewählt, dass der Dampfdruck von Kohlensäuredimethylester bei 80°C, insbesondere bei 70°C, unterschritten wird. Günstig ist es, wenn das Trocknen unter einem Druck von höchstens 300 hPa, insbesondere höchstens 100 hPa erfolgt. Bei so niedrigen Drücken verdampfen wesentliche Bestandteile der meisten Elektrolyten, insbesondere Kohlensäuredimethylester und Kohlensäureethylmethylester, bereits bei Temperaturen von weniger als 80°C. Niedrige Temperaturen haben den Vorteil, dass die Bildung von Fluorwasserstoff behindert wird. Fluorwasserstoff stellt eine potentielle Gefährdung für die Batterie-Verarbeitungsanlage sowie die Umgebung dar. Es ist daher günstig, das Entstehen von Fluorwasserstoff zu verhindern.

Vorzugsweise erfolgt das Trocknen bei einer Temperatur, die unterhalb einer Zersetzungstemperatur liegt. Unter der Zersetzungstemperatur wird insbesondere die kleinste Temperatur verstanden, für die gilt, dass nach dem Halten des Zerkleinerungsguts auf dieser Temperatur für eine Stunde zumindest 80 Masseprozent des Binders sich in gasförmige Bestandteile zersetzt haben. Die Zersetzungstemperatur kann damit gemessen werden, indem die Temperatur des Zerkleinerungsguts sukzessive erhöht wird und erfasst wird, wann es zu einem Masseverlust, insbesondere durch Gasentwicklung aufgrund einer Zersetzung des Binders, kommt und das angegebene Kriterium erfüllt ist. Gegebenenfalls muss der Versuch mit jeweils einer neuen Probe an Zerkleinerungsgut bei jeweils erhöhter Temperatur mehrfach durchgeführt werden.
Günstig ist es, wenn das Trocknen unter einer Atmosphäre erfolgt, in der der Wasser-Partialdruck kleiner ist als 50 Pa, insbesondere kleiner als 10 Pa. Ein geringer Wasser-Partialdruck führt zu einer geringen Reaktionsrate von Lithium-Verbindungen zu Lithium-Hydroxid und damit zu nur geringer Wasserstoff-Entwicklung. Das verhindert die Bildung von zündfähigen Wasserstoff-Sauerstoff-Gemischen und trägt zur Anlagensicherheit bei.

Günstig ist es zudem, wenn der Sauerstoff-Partialdruck höchstens 10 Millibar, insbesondere höchstens 5 Millibar beträgt. Die Reaktion von Sauerstoff mit oxidierbaren Bestandteilen der Batterien wird so weitgehend unterdrückt. Es ist möglich, den niedrigen Sauerstoff-Partialdruck durch einen geringen Druck beim Trocknen zu erreichen. Alternativ oder zusätzlich kann das Trocknen in einer Inertgas-Atmosphäre erfolgen.

Bevorzugt ist ein Verfahren, bei dem das Trocknen des Zerkleinerungsguts erst beendet wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut mehr bilden kann und/oder wenn das Zerkleinerungsgut so trocken ist, dass sich im Transportbehälter oder während der Weiterverarbeitung ein entzündliches oder explosives Gasgemisch ergeben kann. Unter dem Merkmal, dass das Trocknen been-

det wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut bilden kann, wird insbesondere verstanden, dass sich in einem Transportbehälter in Form eines 50-Literbehälter, der zur Hälfte (bezogen auf sein Volumen) mit dem Zerkleinerungsgut gefüllt ist, innerhalb von einer Woche bei 50°C und 1013 hPa kein zündfähiges Gasgemisch bildet. Ob das Kriterium erfüllt ist, wird in Vorversuchen ermittelt. Bildet sich ein zündfähiges Gasgemisch, muss das Trocknen länger und/oder bei niedrigerem Druck durchgeführt werden. Die Vorversuche werden so lange wiederholt, bis eine Trocknungsdauer und/oder ein Trocknungsdruck ermittelt ist, bei der das Merkmal bei einem Testsatz von drei Transportbehälter für alle drei Transportbehälter erfüllt ist.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, bis ein Elektrolytgehalt im Zerkleinerungsgut so klein ist, dass eine elektrochemische Reaktion unmöglich ist. In anderen Worten ist der Elektrolytgehalt kleiner als ein Schwellenwert, wobei der Schwellenwert so gewählt ist, dass die Zellspannung bei Unterschreiten dieses Schwellenwerts auf höchstens ein Viertel abgesunken ist. Dieser Schwellenwert wird beispielsweise dadurch bestimmt, dass die Zellenspannung einer Batterie in Abhängigkeit vom Elektrolytgehalt bestimmt wird. Kurz vor dem Erreichen des Schwellenwerts bricht die Zellspannung zusammen, das heißt, dass sie um zumindest 75% absinkt. Ist der Schwellenwert unterschritten, enthält die Batterie so wenig Elektrolyt, dass in guter Näherung keine elektrochemische Reaktion mehr möglich ist.

Vorzugsweise wird das Zerkleinerungsgut so lange getrocknet, dass eine Menge von 50 kg an Zerkleinerungsmaterial, das kompaktiert in einem 50 Liter-Fass enthalten ist, keine oder eine nur so geringe Wärmeentwicklung zeigt, dass ein thermisches Durchgehen, also eine thermisch induzierte Kettenreaktion, für zumindest zwei Monate ausgeschlossen ist und dass eine etwaige Wasserstoff-Entwicklung zudem so gering ausgebildet ist, dass nach zwei Wochen kein Überdruck entsteht, wenn zu Beginn ein Unterdruck von 500 hPa herrscht.

Günstig ist es, wenn das Zerkleinerungsgut so lange getrocknet wird, bis der

Elektrolytgehalt an organischen Komponenten, die bei 80°C flüchtig sind, höchsten 3 Gew.-%, höchstens insbesondere 2 Gew.-%, besonders bevorzugt höchstens 1,5 Gew.-%, beträgt.

Vorzugsweise wird das Trocknen so lange durchgeführt, dass der akkumulierte Gehalt an organischen Karbonaten aus dem Elektrolyten, die bei 80°C flüchtig sind, in der Atmosphäre über dem Zerkleinerungsgut 3 Vol.-% unterschreitet.

Insbesondere wird das Trocknen so lange durchgeführt, bis der Gehalt an Kohlensäuredimethylester 4 Vol.-%, insbesondere 3 Vol.-%, unterschreitet und/oder der Gehalt an Cyclohexylbenzol 1 Vol.-%, insbesondere 0,5 Vol.-%, unterschreitet.

Vorzugsweise erfolgt das Trocknen unmittelbar nach dem Zerkleinern. Hierunter wird insbesondere verstanden, dass zwischen dem Beginn des Zerkleinerns der Batterien und dem Zeitpunkt, zu dem zumindest ein Teil des dadurch entstandenen Zerkleinerungsguts anfängt zu Trocknen, höchstens fünf Minuten beträgt, insbesondere höchstens eine Minute. Durch die schnelle Trocknung nach dem Zerkleinern wird die Masse an Material klein gehalten, die potentiell elektrochemisch reagieren könnte, zudem wird die elektrochemische Reaktionszeit potentieller exothermer Reaktionen klein gehalten. Das senkt das Risiko für die Anlage und die Umgebung.

Besonders günstig ist es, wenn das Vakuum mittels einer Strahlpumpe erzeugt wird. Strahlpumpen sind, insbesondere bei geeigneter Wahl des Strahlmediums, weitgehend unempfindlich gegen aggressive abzupumpende Gase. Günstig ist es, wenn das Strahlmedium, das eine Flüssigkeit ist, einen pH-Wert von zumindest 8, insbesondere von zumindest 9, beispielsweise zumindest 12, hat. In diesem Fall können sich unerwünschte Bestandteile des Gases, das abgepumpt wird, zersetzen oder zu weniger schädlichen Substanzen reagieren. So kann beispielsweise Kohlensäuredimethylester und/oder Kohlensäureethylmethylester durch eine Verseifungsreaktion abgebaut werden. Etwaig im Strahlenmedium

enthaltener Fluorwasserstoff kann im basischen Milieu in einer Säure-Base-Reaktion zu einem ungefährlichen Salz umgesetzt werden.

Vorzugsweise enthält das Strahlenmedium eine Fluor fällende Substanz. Beispielsweise kann das Strahlenmedium Natriumkarbonat oder Calziumkarbonat enthalten. Die bei der Reaktion mit einer Fluorverbindung, insbesondere Fluorwasserstoff, entstehenden Salze werden vorzugsweise abgetrennt, insbesondere abgefiltert oder aussedimentiert. Auf diese Weise wird zumindest weitgehend verhindert, dass Fluorwasserstoff oder andere giftige fluorhaltige Verbindungen in die Umgebung abgegeben werden können.

Vorzugsweise erfolgt das Trocknen bei einer Temperatur von höchstens 80°C. In diesem Fall entsteht kaum Fluorwasserstoff. Das erhöht die Lebensdauer der Batterie-Verarbeitungsanlage und verringert die Umweltgefährdung.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte des Kondensierens von Bestandteilen des Elektrolyten durch Abkühlen und/oder Druckerhöhen, sodass ein Elektrolytkondensat entsteht. Beispielsweise wird das Kondensieren an einer Stelle durchgeführt, die bezüglich des Gasflusses zwischen dem Trockner und der Vakuumpumpe liegt. Vom Trockner kommende Gase müssen daher in diesem Fall zunächst einen Kondensator passieren, bevor sie zur Vakuumpumpe gelangen können. Das führt dazu, dass gasförmiger Elektrolyt, der in dem Gas vorliegt, das beim Trocknen entsteht, zumindest zu einem überwiegenden Teil im Kondensator abgeschieden wird, bevor das verbleibende Gas zur Pumpe gelangt. So kann Elektrolyt zurückgewonnen werden. Zudem verringert sich der Gasstrom durch die Vakuumpumpe, was deren Lebensdauer verlängert und deren Energieverbrauch reduziert.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren alternativ den Schritt der Gasreinigung durch Adsorption der flüchtigen organischen Bestandteile an einem Aktivkohlfilter vor oder nach der Verdichtereinheit.

Vorzugsweise umfasst das erfindungsgemäße Verfahren alternativ oder zusätzlich den Schritt eines Reinigens des Gases, das beim Trocknen entsteht, bevor dieses die Vakuumpumpe erreicht. Das kann beispielsweise auch dadurch erfolgen, dass das Gas einen Aktivkohlefilter und/oder einen Filter passiert, in dem mit Fluorwasserstoff reagierende Substanzen enthalten sind, beispielsweise ein Calziumsalz wie Calziumkarbonat oder ein Kaliumsalz wie Kaliumkarbonat.

Vorzugsweise umfasst das erfindungsgemäße Verfahren den Schritt eines Trocknens bei einer Trocken-Temperatur und für eine Trocken-Dauer, die so gewählt sind, dass sich der Binder, der Aktivmaterial der Lithium-Batterie an einen Träger bindet, zumindest zum überwiegenden Teil zersetzt. Günstig ist es, wenn dieser Trocknungsschritt, der auch als Hochtemperatur-Trocknen bezeichnet werden kann, räumlich getrennt von einem ersten, oben beschriebenen Trocknungsschritt durchgeführt wird. Der letztgenannte Trocknungsschritt kann auch als Niedertemperatur-Trocknen bezeichnet werden.

Vorzugsweise wird das Hochtemperatur-Trocknen, bei dem sich der Binder zersetzt, so durchgeführt, dass die entstehenden Zersetzungsgase sich nicht mit den Gasen mischen, die beim Niedrigtemperatur-Trocknen entstehen. Es ist möglich, dass das Hochtemperatur-Trocknen und das Niedertemperatur-Trocknen bei unterschiedlichen Drücken erfolgt. Beispielsweise kann das Hochtemperatur-Trocknen bei Normaldruck erfolgen.

Unter dem Aktivmaterial wird der Stoff verstanden, der beim Betrieb der Batterien elektrochemisch reagiert. Unter dem Träger für das Aktivmaterial wird insbesondere eine Trägerfolie verstanden, auf der das Aktivmaterial in Form von Partikeln aufgebracht ist. Beispielsweise handelt es sich bei der Trägerfolie um eine Folie aus Aluminium oder eine Aluminiumlegierung. Der Binder ist der Stoff, der das Aktivmaterial mit dem Träger verbindet, beispielsweise enthält der Binder Polyvinylidenfluorid.

Günstig ist es, wenn beim Zerkleinern der Batterien flüssiger Stickstoff zugege-

ben wird. Das kühlt die Batterien, die Zerkleinerungsmaschine und das Zerkleinerungsgut und verdrängt zudem Sauerstoff und Wasserdampf aus der Atmosphäre.

Günstig ist es, wenn das Zerkleinern bei dem Wasserdampf-Partialdruck von höchstens 20 Pa und/oder einem Sauerstoff-Partial von höchstens 40 hPa, insbesondere höchstens 15 hPa erfolgt.

Gemäß einer bevorzugten Ausführungsform umfasst das Verfahren die Schritte eines Entnehmens des Zerkleinerungsguts aus dem Transportbehälter, eines Abtrennens von Hartteilen und/oder eines Separierens von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfaktion entstehen, und eines getrennten Verpackens von Aktivmaterialfraktion und Trägerfraktion in jeweilige Transportbehältnisse. Günstig ist es, wenn diese Transportbehältnisse luftdicht ausgebildet sind. Durch das Trennen von Aktivmaterialfraktion und Trägerfraktion ist ein Transport in der Regel ohne Genehmigungen möglich. Ein weiterer Vorteil ist, dass derartig getrennte Fraktionen nur eine geringe Gefährdung darstellen.

Das Entnehmen des Zerkleinerungsguts aus dem Transportbehälter erfolgt vorzugsweise unter Vakuum und/oder unter Schutzgas.

Es ist möglich, nicht aber notwendig, dass das Zerkleinerungsgut unter Vakuum in den Transportbehälter abgefüllt wird. Günstig ist es, wenn der Transportbehälter ein Vakuumbehälter ist, insbesondere ein evakuierter Vakuumbehälter, sodass im Transportbehälter nach dem Verschließen ein Unterdruck oder Vakuum herrscht. Alternativ kann der Transportbehälter mit einem Inertgas gefüllt werden.

In einer bevorzugten Batterie-Verarbeitungsanlage sind die Trennungseinheit und die Trockenvorrichtung in einem gemeinsamen Standard-Container angeordnet. Das hat den Vorteil, dass die Batterie-Verarbeitungsanlage besonders

einfach transportiert werden kann.

Die Trockenvorrichtung ist eingerichtet zum Trocknen des Zerkleinerungsguts für eine so lange Zeit, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist. Wird die Trockenvorrichtung im Chargenbetrieb betrieben, was eine mögliche Ausführungsform darstellt, so wird das Trocknen beispielsweise für eine vorgegebene Zeitspanne durchgeführt. Alternativ oder zusätzlich wird der Gehalt an organischen Substanzen, beispielsweise an organischen Carbonaten, in der Atmosphäre in der Trockenvorrichtung kontinuierlich gemessen und das Trocknen beendet, wenn eine vorgegebene Schwellen-Konzentration unterschritten wird.

Gemäß einer bevorzugten Ausführungsform umfasst die Batterie-Verarbeitungsanlage, insbesondere die Vakuumanlage, einen Kondensator, der eingerichtet ist zum Kondensieren von organischen Bestandteilen der Atmosphäre im Trockner, insbesondere von organischen Carbonaten, beispielsweise von Dimethylcarbonat, Ethylmethylcarbonat und/oder Ethylencarbonat. Vorzugsweise ist der Kondensator in Materialflussrichtung vor einer Vakuumpumpe angeordnet, mittels der der Trockner evakuiert wird. Günstig ist es, wenn der Kondensator gekühlt ist, vorzugsweise auf eine Temperatur von höchstens 90°C, vorzugsweise höchstens 80°C, insbesondere höchstens 70°C. Um den Kühlaufwand klein zu halten wird der Kondensator, sofern er gekühlt wird, auf eine Temperatur von zumindest -10°C, insbesondere zumindest 10 °C, gekühlt.

Günstig ist es, wenn die Trockenvorrichtung ein Rührwerkzeug aufweist, beispielsweise einen Ankerrührer oder einen Stabrührer, dessen Rührstäbe quer zu einer Rührwelle angebracht sein können. Alternativ oder zusätzlich ist das Rührwerkzeug ein externes Rührwerkzeug, das den Trockner als Ganzes bewegt.

Vorzugsweise besitzt die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung zum Erzeugen eines Vakuums in der Trockenvorrichtung verbunden ist. Besonders günstig ist es, wenn auch die Vakuumanlage im Standard-Container angeordnet ist. Bei dem Standard-Container handelt es sich

vorzugsweise um einen Container nach ISO-Norm 668, vorzugweise um einen 40-Fuß-Container oder einen 20-Fuß-Container.

Beispielweise umfasst die Vakuumanlage eine Strahlpumpe, bei der ein Strahlmedium zum Erzeugen des Unterdrucks verwendet wird.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionabtrennvorrichtung , eine Separiervorrichtung, insbesondere eine Klassiervorrichtung, zum Separieren von Aktivmaterial vom Träger, insbesondere durch eine zweite Zerkleinerungsstufe und/oder Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht, und eine zweite Abfüllvorrichtung zum getrennten Abfüllen von Aktivmaterialfraktion und Trägerfraktion. Günstig ist es, wenn diese Abfüllvorrichtung zum Abfüllen unter Unterdruck und/oder unter Inertgas ausgebildet ist.

Unter einer Hartmetallabtrennvorrichtung wird insbesondere eine Vorrichtung zur Abtrennung von Fragmenten peripherer Bestandteile des Betriebssystems, der Batteriezellhülle und der Strom-Kontakte verstanden. Beispielsweise umfasst die Hartmetallabtrennvorrichtung eine Magnetseparationsvorrichtung und/oder einen Sichter, insbesondere einen Querstromsichter und/oder einen Zickzack-Sichter. Unter der Separiervorrichtung wird insbesondere eine Vorrichtung zum zur Abtrennen der Separatorfolie verstanden.

Die Leichtfraktionabtrennvorrichtung umfasst vorzugsweise einen Zickzack-Sichter und/oder einen Lufttrenner, wobei es günstig ist, wenn die Luft im Kreislauf geführt wird. Das reduziert die Staubexposition der Umwelt mit gesundheitsschädlichem Staub.

Vorzugsweise sind die zweite Abfüllvorrichtung und die Separiervorrichtungen in einem gemeinsamen Standard-Container angeordnet, beispielsweise im oben beschrieben ersten Standard-Container, oder einem zweiten Standard-Container. Günstig ist es, wenn der Container staubdicht gekapselt ist.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage eine Schleuse zwischen der Zerkleinerungseinheit und der Inaktivierungsvorrichtung, insbesondere der Trocknungsvorrichtung. Beispielsweise handelt es sich um eine Zellenradschleuse. Durch die Schleuse wird der Sauerstoffeintrag in die Inaktivierungsvorrichtung, insbesondere die Trocknungsvorrichtung, vermindert.

Im Folgenden wird die Erfindung anhand der beigefügten Zeichnungen näher erläutert. Dabei zeigt

Figur 1 ein Ablaufdiagramm eines erfindungsgemäßen Verfahrens,

Figur 2 einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage und

Figur 3 einen Querschnitt durch weitere, optionale Bestandteile einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Figur 1 zeigt das Ablaufdiagramm eines erfindungsgemäßen Verfahrens. Zunächst werden Batterien 10.1, 10.2, ..., insbesondere Batteriesysteme bestehend aus mehreren Batteriemodulen oder Batteriestacks, welche wiederum aus mehreren Batteriezellen bestehen, in einer Entladeeinheit 12 entladen. Danach folgt an einer Demontagestation 14 die Demontage der Batterien 10, wenn diese erforderlich ist, weil die Batteriesysteme ansonsten aus geometrischen oder gravimetrischen Gründen nicht in die Zerkleinerungseinheit befördert werden können. Dazu werden die Batteriesysteme geöffnet und soweit demontiert, dass die Module/Stacks einzeln entfernt werden können. Gegebenenfalls können auch die Zellen von der Ansteuerelektronik getrennt werden. Die entstehenden Untereinheiten (Module/Stacks) und/oder Zellen 16.1, 16.2, ... werden einer Zerkleinerungseinheit 18 zugeführt, die beispielsweise eine Rotorschere mit einem Rotor und einen mit Statoren oder mehreren Rotoren, oder eine Schneidmühle mit einem Rotor und mehreren Rotoren umfasst.

Die Zerkleinerungseinheit 18 zerkleinert die Batterien 10 unter Schutzgas 20,

das beispielsweise einer Schutzgasflasche entnommen wird. Alternativ oder zusätzlich kann flüssiger Stickstoff aus einer Flüssigstickstoffquelle 19 zugedüst werden. Bei dem Schutzgas kann es sich beispielsweise um Stickstoff, ein Edelgas, Kohlendioxid, Lachgas oder ein sonstiges, vorzugweise nicht giftiges Gas handeln.

Beim Zerkleinern entsteht Zerkleinerungsgut 24, das einer Inaktivierungsvorrichtung in Form einer Trockenvorrichtung 26 zugeführt wird. Zwischen der Zerkleinerungseinheit 18 und der Trockenvorrichtung 26 ist eine Schleuse 28 angeordnet, die soweit gasdicht ist, dass die Druckvorrichtung 26 in guter Näherung von der Zerkleinerungseinheit 18 gasdicht getrennt ist.

Die Trockenvorrichtung 26 ist mit einer Vakuumanlage 29 verbunden, die eine Vakuumpumpe 30 aufweist und ein Vakuum erzeugt. In der Trockenvorrichtung 26 herrscht ein Druck p_{26} von $p_{26} \approx 100$ hPa, vorzugsweise 50 hPa. Es sei darauf hingewiesen, dass im Rahmen der vorliegenden Beschreibung unter einer Vakuumpumpe insbesondere ganz allgemein eine Vorrichtung verstanden wird, die ein Vakuum erzeugt. Es ist dabei möglich und bevorzugt, nicht aber notwendig, dass die Vakuumpumpe gleichzeitig als Verdichter wirkt, so dass von ihr Gas unter einem Druck abgegeben wird, der größer ist als der Umgebungsdruck.

In dem in Figur 1 gezeigten Fall handelt es sich bei der Vakuumpumpe um einen Verdichter, der Gas 31, das in der Trockenvorrichtung 26 vorliegt, ansaugt und verdichtet. Alternativ oder zusätzlich kann die Vakuumanlage 29 eine Strahlpumpe aufweisen, bei der ein Strahlmedium in Form einer Flüssigkeit mit hoher Geschwindigkeit durch Venturi-Düse geleitet wird. Das Strahlmedium ist alkalisch und hat einen pH-Wert von zumindest pH 13 und ist beispielsweise eine 10%ige Kaliumhydroxidlösung.

Die Vakuumanlage 29 umfasst eine Gasreinigungsvorrichtung 32, die zwischen der Trockenvorrichtung 26 und der Vakuumpumpe 30 angeordnet ist und die im

vorliegenden Fall einen Kondensator 34 und/oder einen Aktivkohlefilter 36 umfasst. Der Kondensator wird bei einer Temperatur von -10 °C betrieben, so dass Kohlensäuredimethylester und Kohlensäureethylmethylester kondensieren und in einen Kondensatbehälter 38 abgegeben werden können. Zudem wird etwaig vorhandenes Wasser ausgefroren. Ein Steuerventil 40 ist ausgebildet zum Öffnen, wenn der Druck p_{26} zu groß wird und zum Zufahren, wenn der Druck p_{26} zu klein wird, das heißt, dass ein vorgegebener Schwellenwert unterschritten wird.

Vorzugsweise ist das Trocknungsgut während der Trocknung bewegt. Dies kann beispielsweise durch Rühren mit einem Rührwerkzeug 41, wie einem Ankerrührer oder einem Stabrührer mit senkrecht zur Rührwelle angebrachten Stäben passieren. Alternativ durch einen bewegten Trocknungsbehälter.

Durch das Trocknen des Zerkleinerungsguts entsteht inaktiviertes Zerkleinerungsgut 42, das einer Abfüllvorrichtung 44 zugeführt wird. Dort wird das inaktivierte Zerkleinerungsgut 42 unter Vakuum und/oder unter Schutzgas in einen Transportbehälter 46 abgefüllt. Der Transportbehälter 46 ist vorzugsweise gasdicht. Es ist möglich, nicht aber notwendig, dass der Transportbehälter 46 vor dem Abtransport mit Inertgas aufgefüllt wird, so dass er unter Normaldruck steht. Alternativ ist es auch möglich, dass der Transportbehälter unter Vakuum verschlossen und transportiert wird. Es ist möglich, dass alternativ zum Transportbehälter eine vakuumverschweißte Folie, beispielsweise eine Aluminiumverbundfolie, gewählt wird.

Über eine Spülleitung 48 wird der Zerkleinerungseinheit 18 Schutzgas 20 von der Vakuumpumpe 30 zugeführt. Arbeitet die Vakuumpumpe 30 wie im vorliegenden Fall, der eine bevorzugte Ausführungsform darstellt, zudem als Kompressor, kann das Schutzgas auf eine Druckgasflasche 50 gezogen werden. Alternativ oder zusätzlich kann das Schutzgas 20, gegebenenfalls nach zusätzlicher Reinigung, in die Umgebung abgegeben werden.

Figur 2 zeigt schematisch einen Querschnitt durch eine erfindungsgemäße Batterie-Verarbeitungsanlage 52, die einen Standard-Container 54 aufweist, in dem

die Zerkleinerungseinheit 18, die Trockenvorrichtung 26 und die Abfüllvorrichtung 44 angeordnet sind. Hinter der Zerkleinerungseinheit 18 ist ein erster gasdichter Förderer 56 angeordnet, der beispielsweise eine Förderschnecken oder einen Rohrkettenförderer umfasst. Der erste Förderer 56 fördert das Zerkleinerungsgut 24 zur Trockenvorrichtung 26, die mit der in Figur 2 nicht sichtbaren Vakuum-Erzeugungsvorrichtung verbunden ist. In Materialstromrichtung hinter der Trockenvorrichtung 26 ist ein zweiter Förderer 58 angeordnet, der vorzugsweise ebenfalls gasdicht ausgebildet ist und eine Förderschnecke oder einen Rohrkettenförderer enthalten kann. Der zweite Förderer fördert das inaktivierte Zerkleinerungsgut 42 zur Abfüllvorrichtung 44.

Figur 3 zeigt optionale, in der vorliegenden Ausführungsform vorhandene Einheiten der erfindungsgemäßen Batterie-Verarbeitungsanlage 52, die einen Aufschlusszerkleinerer 60 sowie einen Sichter 62 umfasst. Der Aufschlusszerkleinerer 60 enthält eine Transportbehälter-Entleervorrichtung 64, mittels der aus dem Transportbehälter 46 inaktiviertes Zerkleinerungsgut 42 entnommen werden kann. Der Aufschlusszerkleinerer 60 erzeugt Aufschlussmaterial 66, das dem Sichter 62 zugeführt wird. Es kann sich bei dem Sichter beispielsweise um einen Zickzack-Sichter handeln.

Vorzugsweise umfasst die Batterie-Verarbeitungsanlage 52 einen Zerkleinerer, der vorzugsweise im Materialfluss vor der Klassiervorrichtung 74 steht und ein schnell laufendes Zerkleinerungswerkzeug enthält, wobei eine Rotorumfangsgeschwindigkeit größer ist als 1 m/s, vorzugsweise größer als 10 m/s. Dieser Zerkleinerer zerkleinert das Zerkleinerungsmaterial und beansprucht es mechanisch so, dass sich die elektrochemisch aktive Beschichtung mindestens teilweise vom Träger löst. Das Vorhandensein eines solchen Zerkleinerer ist ein allgemeines bevorzugtes Merkmal einer erfindungsgemäßen Batterie-Verarbeitungsanlage.

Im Sichter entstehen eine Leichtfraktion mit Separatorfolie und feinem Beschichtungsmaterial und eine Schwergutfraktion mit Trägerfolien (Aluminium- und Kupfer) mit größerem leicht anhaftender Beschichtung. Beide Fraktionen werden auf

jeweils ein Sieb zur weiteren Trennung in Beschichtung und Separatorfolie beziehungsweise Beschichtung und Metallfolien gegeben. Die entstehenden Fraktionen werden getrennt weiter verarbeitet.

Das Aufschlussmaterial 66 wird dem Sichter 62 mittels einem dritten Förderer 68 zugeführt. Ein vierter Förderer 70 führt gesichtetes Material 72, insbesondere das Material der Leichtfraktion, das den Sichter 62 verlässt, einer Klassiervorrichtung 74 zu. Die Klassiervorrichtung 74 umfasst vorzugsweise ein Luftstrahlsieb, das gleichzeitig als Trennvorrichtung zum Trennen des Aktivmaterials von dem Träger fungiert. Durch das Trennen entsteht eine Aktivmaterialfraktion 76, die in ein Transportbehältnis 78 abgefüllt wird.

Es entsteht zudem eine Trägerfraktion 80, die in der vorliegenden Ausführungsform mit einem fünften Förderer 82 einer Abfülleinheit 84 zugeführt wird, die die Trägerfraktion 80 in einen Behälter 86 füllt. Die Abfülleinheit 84 ist zusammen mit einer zweiten Abfülleinheit 88 Teil einer zweiten Abfüllvorrichtung.

Bezugsleistenliste

10 Batterie
12 Entladeeinheit
14 Demotagesstation
16 Zelle
18 Zerkleinerungseinheit
19 Flüssigstickstoffquelle
20 Schutzgas
22 Schutzgasflasche
24 Zerkleinerungsgut
26 Trockenvorrichtung
28 Schleuse
29 Vakuumanlage
30 Vakuumpumpe
31 Gas
32 Gasreinigungsvorrichtung
34 Kondensator
36 Aktivkohlefilter
38 Kondensatbehälter
40 Steuerventil
41 Rührwerkzeug
42 inaktives Zerkleinerungsgut
44 Abfüllvorrichtung
46 Transportbehälter
48 Spülleitung
50 Druckgasflasche
52 Batterie-Verarbeitungsanlage
54 Standard-Container
56 erster Förderer
58 zweiter Förderer
60 Aufschlusszerkleinerer
62 Sichter
64 Transportbehälter-Entleervorrichtung
66 Aufschlussmaterial
68 dritter Förderer
70 vierter Förderer
72 gesichtetes Material
74 Klassiervorrichtung
76 Aktivmaterialfraktion
78 Transportbehältnis
80 Trägerfraktion
82 fünfter Förderer
84 Abfülleinheit
86 Behälter
88 zweite Abfülleinheit
p Druck

Duesenfeld Ex. 2014-628

Patentansprüche.

1. Verfahren zum Behandeln gebrauchter Batterien (10), insbesondere Lithium-Batterien, mit den Schritten:
   (a) Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,
   (b) Inaktivieren des Zerkleinerungsguts (24), sodass ein inaktiviertes Zerkleinerungsgut (42) erhalten wird, und
   (c) Abfüllen des inaktivierten Zerkleinerungsguts (42) in einen Transportbehälter (46),
   dadurch gekennzeichnet, dass
   (d) das Inaktivieren durch Trocknen des Zerkleinerungsguts (24) erfolgt.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass das Zerkleinerungsgut (24) solange getrocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

3. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Trocknen unter Vakuum erfolgt.

4. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass
   das Vakuum mittels einer Strahlpumpe erzeugt wird und
   ein Strahlmedium der Strahlpumpe
   einen pH-Wert von zumindest 8, insbesondere 9, hat und/oder
   eine Fluor fällende Substanz enthält.

5. Verfahren nach einem der vorstehenden Ansprüche, gekennzeichnet durch die Schritte:
   (e) Entnehmen des Zerkleinerungsguts (24) aus dem Transportbehälter (46),
   (f) Abtrennen von Hartteilen und/oder
   Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion (80) entsteht, und
   (g) getrenntes Verpacken von Aktivmaterialfraktion (76) und Trägerfraktion (80) in Transportbehältnisse (78).

6. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das Trocknen des Zerkleinerungsguts erst beendet wird, wenn sich nach Beenden des Trocknens kein entzündliches oder explosives Gasgemisch über dem abgefüllten Zerkleinerungsgut mehr bilden kann und/oder wenn das Zerkleinerungsgut so trocken ist, dass sich im Transportbehälter oder während der Weiterverarbeitung ein entzündliches oder explosives Gasgemisch ergeben kann.

7. Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien, insbesondere Lithium-Batterien, mit:
   (a) einer Zerkleinerungseinheit (18) zum Zerkleinern der Batterien (10), sodass Zerkleinerungsgut (24) erhalten wird,
   (b) einer Inaktivierungsvorrichtung (26) zum Inaktivieren des Zerkleinerungsguts (24) und
   (c) einer Abfüllvorrichtung (44) zum Abfüllen des inaktivierten Zerkleinerungsguts (24) in einen Transportbehälter (46),
   dadurch gekennzeichnet, dass
   (d) die Inaktivierungsvorrichtung eine Trockenvorrichtung (26) umfasst und
   (e) die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung (26) zum Erzeugen eines Vakuums in der Trockenvorrichtung (26) verbunden ist, aufweist.

8. Batterie-Verarbeitungsanlage nach Anspruch 7, dadurch gekennzeichnet, dass die Trockenvorrichtung (26) eingerichtet ist zum Trocknen des Zerkleinerungsguts (24) solange, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist.

9. Batterie-Verarbeitungsanlage nach Anspruch 7 oder 8, dadurch gekennzeichnet, dass
die Zerkleinerungseinheit und die Trockenvorrichtung (26) in einem Standard-Container (54) angeordnet sind.

10. Batterie-Verarbeitungsanlage nach einem der Ansprüche 7 bis 9, gekennzeichnet durch eine Vakuumanlage, die mit der Trockenvorrichtung (26) zum Erzeugen eines Vakuums in der Trockenvorrichtung (26) verbunden ist.

11. Batterie-Verarbeitungsanlage nach Anspruch 10, dadurch gekennzeichnet, dass
- die Vakuumanlage (29) eine Strahlpumpe mit einem Strahlmedium umfasst,
- das Strahlmedium im Kreis geführt ist und
- das Strahlmedium eine mit Fluorwasserstoff reagierende Substanz enthält.

12. Batterie-Verarbeitungsanlage nach einem der Ansprüche 7 bis 11, gekennzeichnet durch
- eine Hartmetallabtrennvorrichtung und/oder eine Leichtfraktionsabtrennvorrichtung,
- eine Separiervorrichtung, insbesondere eine Klassiervorrichtung (74), zum Separieren von Aktivmaterial vom Träger, insbesondere durch Luftstrahlsieben und/oder durch eine zweite Zerkleinerungsstufe, sodass eine Aktivmaterialfraktion (76) und eine Trägerfraktion entsteht, insbesondere Aluminium- und Kupferfolien, und

Ascend Elements EX1002 Page 631

Duesenfeld Ex. 2014-631

- eine zweite Abfüllvorrichtung (44) zum getrennten Abfüllen von Aktivmaterialfraktion (76) und Trägerfraktion (80), insbesondere Aluminium- und Kupferfolien.

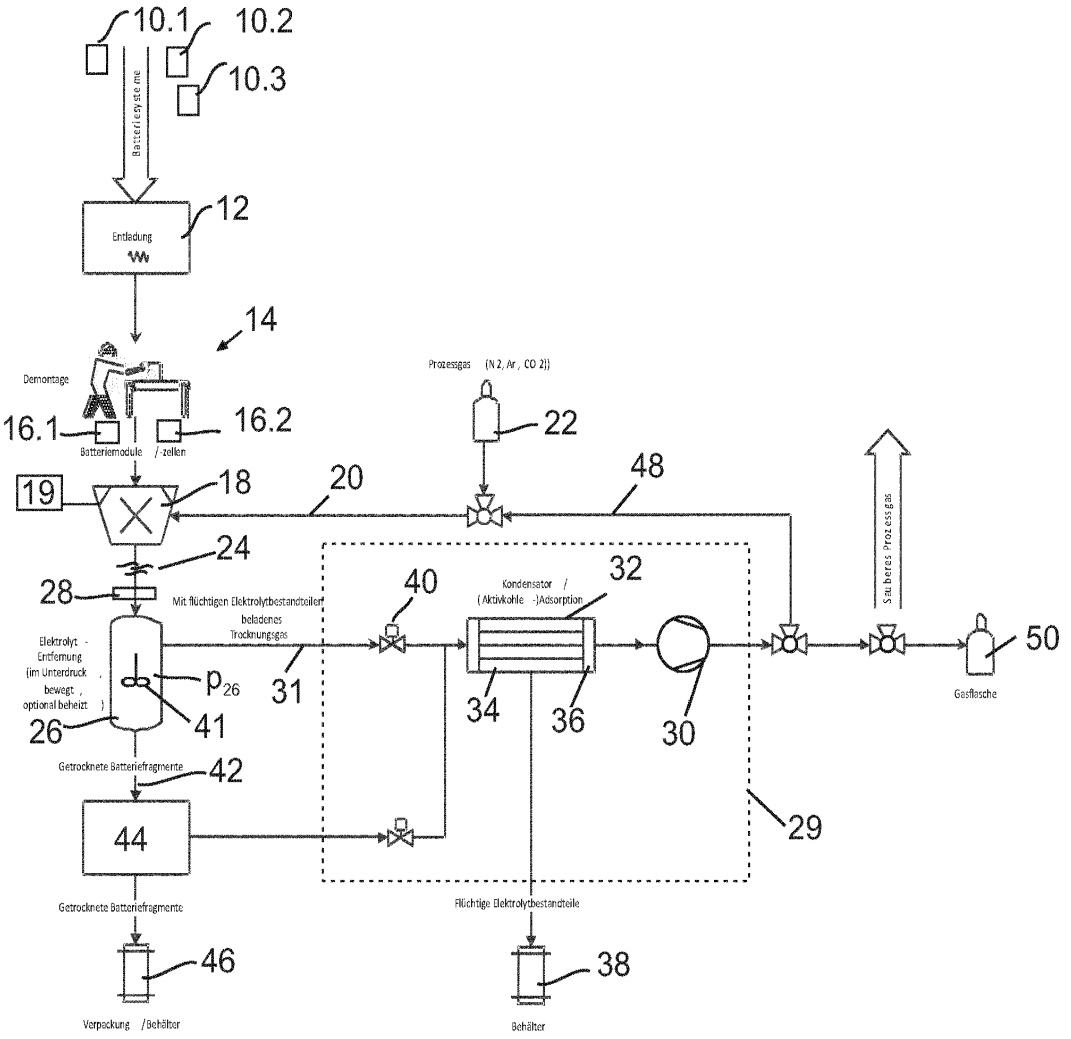
10.1
10.2
10.3
Batteriesysteme
Entladung
12
14
Demontage
16.1
16.2
Batteriemodule / -zellen
19
18
20
Prozessgas (N2, Ar, CO2)
22
48
Sauberes Prozessgas
24
28
Mit flüchtigen Elektrolytbestandteilen beladenes Trocknungsgas
40
Kondensator / (Aktivkohle -)Adsorption
32
50
Gesflasche
Elektrolyt - Entfernung (im Unterdruck , bewegt , optional beheizt)
p_{26}
31
34
36
30
26
41
Getrocknete Batteriefragmente
42
44
29
Getrocknete Batteriefragmente
Flüchtige Elektrolytbestandteile
46
38
Verpackung / Behälter
Behälter

Fig. 1

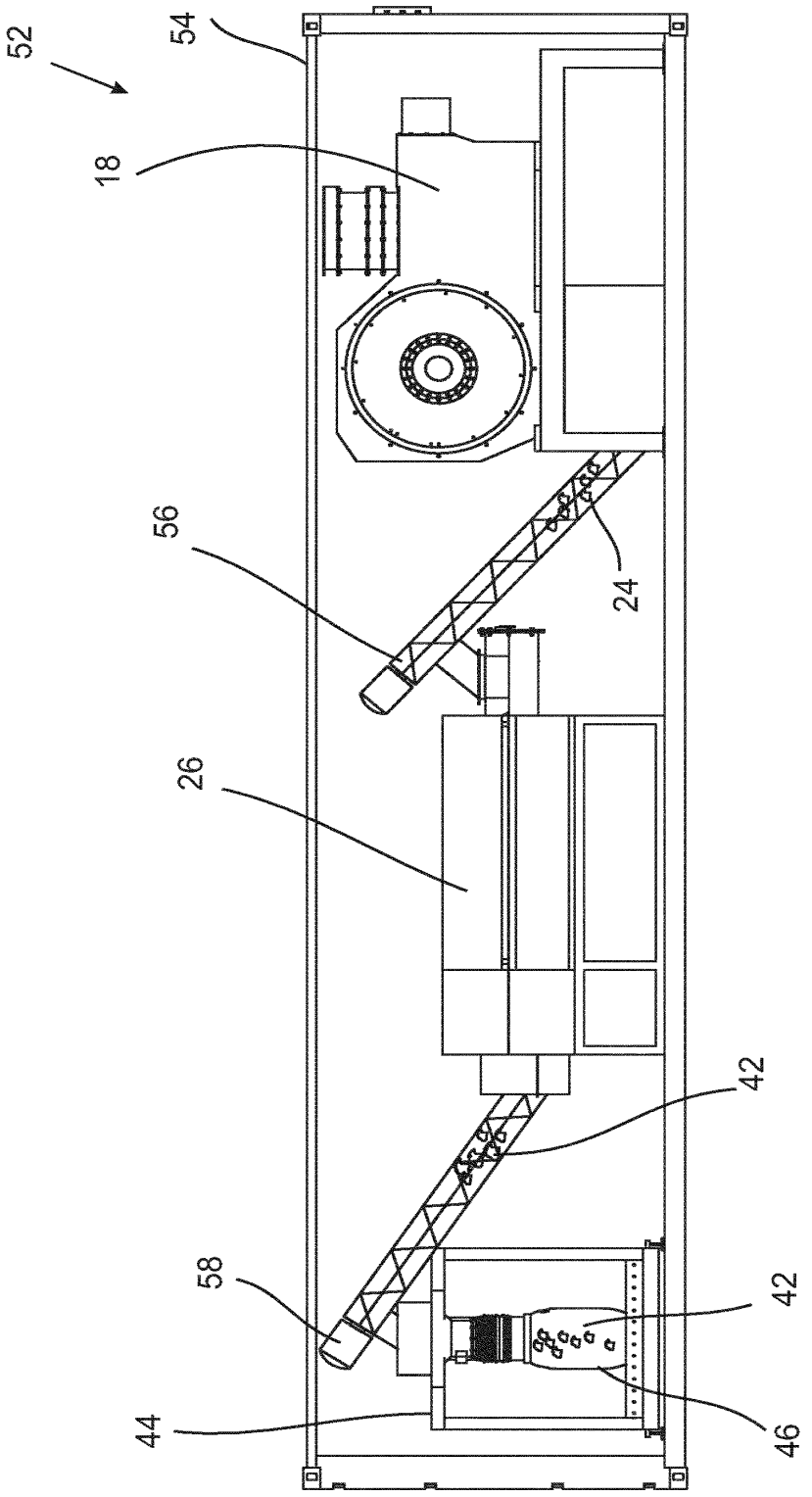

Fig. 2

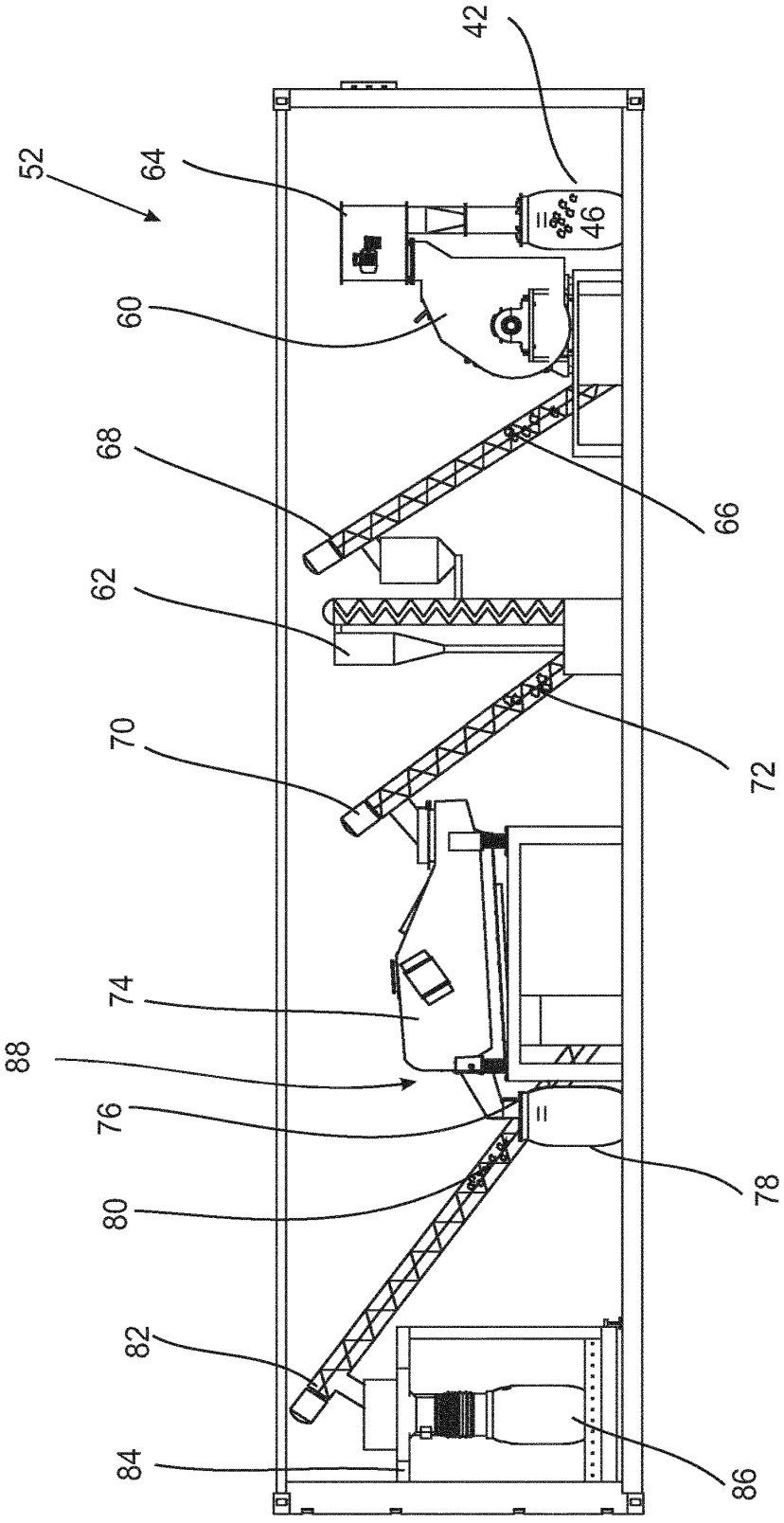

Fig. 3

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44*bis*)

Applicant's or agent's file reference 5569-002 PCT-1	**FOR FURTHER ACTION**	See item 4 below
International application No. PCT/EP2016/059526	International filing date *(day/month/year)* 28 April 2016 (28.04.2016)	Priority date *(day/month/year)* 28 April 2015 (28.04.2015)
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237		
Applicant DUESENFELD GMBH		

1. This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 *bis*.1(a).

2. This REPORT consists of a total of 8 sheets, including this cover sheet.

   In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.

3. This report contains indications relating to the following items:

☒	Box No. I	Basis of the report
☐	Box No. II	Priority
☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☐	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☒	Box No. VIII	Certain observations on the international application

4. The International Bureau will communicate this report to designated Offices in accordance with Rules 44*bis*.3(c) and 93*bis*.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44*bis* .2).

	Date of issuance of this report 31 October 2017 (31.10.2017)
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. +41 22 338 82 70	Authorized officer Nora Lindner e-mail: pct.team5@wipo.int

Form PCT/IB/373 (January 2004)

PATENT COOPERATION TREATY **TRANSLATION**

From the INTERNATIONAL SEARCHING AUTHORITY

To:

PCT

WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY

(PCT Rule 43*bis*.1)

Date of mailing *(day/month/year)*	**13 July 2016**
Applicant's or agent's file reference **5569-002 PCT-1**	**FOR FURTHER ACTION** See paragraph 2 below

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/EP2016/059526	**28 April 2016**	**28 April 2015**

International Patent Classification (IPC) or both national classification and IPC
H01M 6/52(2006.01)i; ***C22B 13/00***(2006.01)i; ***H01M 10/54***(2006.01)i

Applicant
LION ENGINEERING GMBH

1. This opinion contains indications relating to the following items:
   - ☑ Box No. I Basis of the opinion
   - ☐ Box No. II Priority
   - ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - ☐ Box No. IV Lack of unity of invention
   - ☑ Box No. V Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
   - ☐ Box No. VI Certain documents cited
   - ☐ Box No. VII Certain defects in the international application
   - ☑ Box No. VIII Certain observations on the international application

2. **FURTHER ACTION**

   If a demand for international preliminary examination is made, this opinion will be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1*bis*(b) that written opinions of this International Searching Authority will not be so considered.

   If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

   For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA/	Date of completion of this opinion	Authorized officer

TRANSLATION

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/EP2016/059526

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:
   - [✓] the international application in the language in which it was filed.
   - [ ] a translation of the international application into _____ which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b)).

2. [ ] This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43*bis*.1(a)).

3. [ ] With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:
   - a. [ ] forming part of the international application as filed:
     - [ ] in the form of an Annex C/ST.25 text file.
     - [ ] on paper or in the form of an image file.
   - b. [ ] furnished together with the international application under PCT Rule 13*ter*.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.
   - c. [ ] furnished subsequent to the international filing date for the purposes of international search only:
     - [ ] in the form of an Annex C/ST.25 text file (Rule 13*ter*.1(a)).
     - [ ] on paper or in the form of an image file (Rule 13*ter*.1(b) and Administrative Instructions, Section 713).

4. [ ] In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

5. Additional comments:

Ascend Elements EX1002 Page 638

Duesenfeld Ex. 2014-638

TRANSLATION

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/EP2016/059526

Box No. V **Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	4, 9, 11	YES
	Claims	1-3, 5-8, 10, 12	NO
Inventive step (IS)	Claims		YES
	Claims	1-12	NO
Industrial applicability (IA)	Claims	1-12	YES
	Claims		NO

2. Citations and explanations :

See Supplemental Box

Ascend Elements EX1002 Page 639

Duesenfeld Ex. 2014-639

TRANSLATION

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/EP2016/059526

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

See Supplemental Box

Ascend Elements EX1002 Page 640

Duesenfeld Ex. 2014-640

TRANSLATION

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/EP2016/059526

Supplemental Box

In case **the space in any of the preceding boxes is not sufficient.**
Continuation of:

Box V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

D1	WO 2010/102377 A1 (ELLIOTT K W) 16 September 2010 (2010-09-16)
D2	DE 10 2011 110083 A1 (TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA [DE]) 14 February 2013 (2013-02-14), cited in the application

[1] The present application fails to satisfy the requirements of PCT Article 33(2) because the subject matter of claims 1-3, 5-8, 10 and 12 is not novel.

[1.1] D1 discloses, in figures 1 and 2 and paragraphs 15-40, a method for the handling of used batteries, having the steps:
(a) comminuting the batteries such that comminuted material is obtained (paragraph 15),
(b) inactivating the comminuted material such that an inactivated comminuted material is obtained (paragraph 16) and
(c) filling the inactivated comminuted material into a transport container, characterized in that
(d) the inactivation is performed by drying the comminuted material, wherein

- the comminuted material is dried until an electrolyte content is so low that an electrochemical reaction is impossible (paragraph 37),

- the drying is performed in a vacuum (paragraph 19) and

(f) separating off hard parts and/or separating active material from the carrier, such that an active material fraction and a carrier fraction are formed (paragraph 25).

D1 does not explicitly disclose that the comminuted material and the active material fraction and carrier fraction are filled or packed into transport containers. Since the products of the process in D1 are referred to as end products for industrial recycling, it can be assumed that such products are packed into a container, and are therefore implicitly disclosed by D1.

Claims 2 and 6 are defined in terms of the result to be achieved (in this regard, see also Box VII). The same result is also disclosed in D1 in paragraph 37 by the expression "sufficiently dried".

The subject matter of claims 1-3, 5 and 6 is therefore not novel (PCT Article 33(2)).

[1.2] Furthermore, D1 discloses, in figures 1 and 2 and paragraphs 15-40, a battery processing installation for the handling of used batteries, having:
(a) a comminution unit for comminuting the batteries such that comminuted material is obtained (paragraph 15),
(b) an inactivation device for inactivating the comminuted material (paragraph 16) and
(c) a filling device for filling the inactivated comminuted material into a transport container, characterized in that
(d) the inactivation device comprises a drying device (see reference line 20 in figure 1) and
(e) the battery processing installation has a vacuum installation which is connected to the drying device for the purposes of generating a vacuum in the drying device (see reference sign 48 in figure 1), and wherein the comminuted material is dried until an electrolyte content is so low that an electrochemical reaction is impossible (paragraph 37) and

- a hard-metal separating-off device and a light-fraction separating-off device,
- a separation device for separating active material from the carrier, such that an active material fraction and a carrier fraction are formed (paragraph 25).

Ascend Elements EX1002 Page 641

Duesenfeld Ex. 2014-641

Supplemental Box

The filling with the products is considered to be implicitly disclosed in D1 (see section 1.1). The result to be achieved is disclosed in paragraph 37 (see section 1.1 and Box VII).

The subject matter of claims 7, 8, 10 and 12 is therefore not novel (PCT Article 33(2)).

[1.3] D2 has been cited as prior art by the applicant in the description, with the disadvantage of the complexity of the installation. Since D2 however has all of the features of the present invention defined in the claims, D2 is considered to be prejudicial to novelty.

[1.4] D2, in paragraphs 21, 22 and 34-39 and figure 1, discloses a method for the handling of used batteries, in particular lithium batteries, having the steps:
(a) comminuting the batteries such that comminuted material is obtained (reference sign 12 in figure 1),
(b) inactivating the comminuted material such that an inactivated comminuted material is obtained, and
(c) filling the inactivated comminuted material into a transport container, characterized in that
(d) the inactivation is performed by drying the comminuted material in a vacuum (paragraphs 21 and 22) for the purposes of removing the electrolyte, and
(c) separating off hard parts and/or separating active material from the carrier, such that an active material fraction and a carrier fraction are formed (paragraph 39).

As already stated in paragraph 1.1, the filling or packing of the fractions is regarded as being implicitly disclosed.

The subject matter of claims 1-3, 5 and 6 is therefore not novel (PCT Article 33(2)).

[1.5] Furthermore, D2, in 21, 22 and 34-39 and figure 1, discloses a battery processing installation for the handling of used batteries, having:
(a) a comminution unit for comminuting the batteries such that comminuted material is obtained (reference sign 12 in figure 1),
(b) an inactivation device for inactivating the comminuted material (28) and
(c) a filling device for filling the inactivated comminuted material into a transport container (30), characterized in that
(d) the inactivation device comprises a drying device (28), and
(e) the battery processing installation has a vacuum installation (32), which is connected to the drying device for the purposes of generating a vacuum in the drying device, and
- a hard-metal separating-off device and a light-fraction separating-off device,
- a separating device for separating active material from the carrier by air jet sieving, such that an act of material fraction and a carrier fraction are formed (paragraph 39).

The subject matter of claims 7, 8, 10 and 12 is therefore not novel (PCT Article 33(2)).

[2] The present application does not meet the requirements of PCT Article 33(1) because the subject matter of claims 4, 9 and 11 does not involve an inventive step (PCT Article 33(3)).

[2.1] D1, in paragraph 19, discloses that the jet pump has a pH monitoring system which ensures the solubility of the vapours. With regard to paragraph 42, in which D1 discloses that the volatile substances react with sodium hydroxide, a person skilled in the art would set the pH value of the medium to be alkaline, which thus reacts with hydrogen fluoride. The subject matter of claim 4 is therefore not inventive.

[2.2] Furthermore, dependent claim 11 defines a minor structural detail which is not mentioned in D1. Circulating the jet medium of a jet pump is however an alternative of the kind that a person skilled in the art would normally implement as a matter of routine, especially since the resulting advantages are readily foreseeable. The subject matter of claim 11 is therefore also not inventive in relation to D1.

[2.3] The feature of the arrangement of the installation in a standard container is merely one of several obvious possibilities from which a person skilled in the art would choose, according to the circumstances, without exercising inventive skill. The subject matter of claim 9 is therefore not inventive.

Box VIII

Certain observations on the international application

[3] Claims 2 and 6 do not meet the requirements of PCT Article 6 because the subject matter for which protection is sought is not clearly defined. The claims attempt to define the subject matter in terms of the result to be

Supplemental Box

achieved, but in so doing merely state the problem to be solved, without indicating the technical features necessary for achieving this result.

[4] Although claims 7 and 10 have been drafted as separate, independent claims, they in fact appear to relate to one and the same subject matter. Hence, the claims are not concise and therefore do not meet the requirements of PCT Article 6.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44*bis*)

Applicant's or agent's file reference 5569-002 PCT-1	**FOR FURTHER ACTION**	See item 4 below
International application No. PCT/EP2016/059526	International filing date *(day/month/year)* 28 April 2016 (28.04.2016)	Priority date *(day/month/year)* 28 April 2015 (28.04.2015)
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237		
Applicant DUESENFELD GMBH		

1. This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 *bis*.1(a).

2. This REPORT consists of a total of 8 sheets, including this cover sheet.

   In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.

3. This report contains indications relating to the following items:

☒	Box No. I	Basis of the report
☐	Box No. II	Priority
☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☐	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☒	Box No. VIII	Certain observations on the international application

4. The International Bureau will communicate this report to designated Offices in accordance with Rules 44*bis*.3(c) and 93*bis*.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44*bis* .2).

	Date of issuance of this report 31 October 2017 (31.10.2017)
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. +41 22 338 82 70	Authorized officer Nora Lindner e-mail: pct.team5@wipo.int

Form PCT/IB/373 (January 2004)

Duesenfeld Ex. 2014-644

VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS

Absender: INTERNATIONALE RECHERCHENBEHÖRDE

An:

siehe Formular PCT/ISA/220

PCT

SCHRIFTLICHER BESCHEID DER INTERNATIONALEN RECHERCHENBEHÖRDE

(Regel 43*bis*.1 PCT)

Absendedatum *(Tag/Monat/Jahr)*	siehe Formular PCT/ISA/210 (Blatt 2)
Aktenzeichen des Anmelders oder Anwalts: siehe Formular PCT/ISA/220	**WEITERES VORGEHEN** siehe Punkt 2 unten

Internationales Aktenzeichen	Internationales Anmeldedatum *(Tag/Monat/Jahr)*	Prioritätsdatum *(Tag/Monat/Jahr)*
PCT/EP2016/059526	28.04.2016	28.04.2015

Internationale Patentklassifikation (IPC) oder nationale Klassifikation und IPC
INV. H01M6/52 C22B13/00 H01M10/54

Anmelder
LION ENGINEERING GMBH

1. Dieser Bescheid enthält Angaben zu folgenden Punkten:

- ☒ Feld Nr. I Grundlage des Bescheids
- ☐ Feld Nr. II Priorität
- ☐ Feld Nr. III Keine Erstellung eines Gutachtens über Neuheit, erfinderische Tätigkeit und gewerbliche Anwendbarkeit
- ☐ Feld Nr. IV Mangelnde Einheitlichkeit der Erfindung
- ☒ Feld Nr. V Begründete Feststellung nach Regel 43*bis*.1 a) i) hinsichtlich der Neuheit, der erfinderischen Tätigkeit und der gewerblichen Anwendbarkeit; Unterlagen und Erklärungen zur Stützung dieser Feststellung
- ☐ Feld Nr. VI Bestimmte angeführte Unterlagen
- ☐ Feld Nr. VII Bestimmte Mängel der internationalen Anmeldung
- ☒ Feld Nr. VIII Bestimmte Bemerkungen zur internationalen Anmeldung

2. **WEITERES VORGEHEN**

Wird ein Antrag auf internationale vorläufige Prüfung gestellt, so gilt dieser Bescheid als schriftlicher Bescheid der mit der internationalen vorläufigen Prüfung beauftragten Behörde ("IPEA"); dies trifft nicht zu, wenn der Anmelder eine andere Behörde als diese als IPEA wählt und die gewählte IPEA dem Internationale Büro nach Regel 66.1*bis* b) mitgeteilt hat, dass schriftliche Bescheide dieser Internationalen Recherchenbehörde nicht anerkannt werden.

Wenn dieser Bescheid wie oben vorgesehen als schriftlicher Bescheid der IPEA gilt, so ist der Anmelder aufgefordert, bei der IPEA vor Ablauf von 3 Monaten ab dem Tag, an dem das Formblatt PCT/ISA/220 abgesandt wurde oder vor Ablauf von 22 Monaten ab dem Prioritätsdatum, je nachdem, welche Frist später abläuft, eine schriftliche Stellungnahme und, wo dies angebracht ist, Änderungen einzureichen.

Weitere Optionen siehe Formblatt PCT/ISA/220.

Name und Postanschrift der Internationalen Recherchenbehörde	Datum der Fertigstellung dieses Bescheids	Bevollmächtigter Bediensteter
Europäisches Patentamt P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016	siehe Formular PCT/ISA/210	Hofer, Astrid Tel. +31 70 340-0

Feld Nr. I Grundlage des Bescheids

1. Hinsichtlich der **Sprache** beruht der Bescheid auf

   ☒ der internationalen Anmeldung in der Sprache, in der sie eingereicht wurde.

   ☐ einer Übersetzung der internationalen Anmeldung in die folgende Sprache , bei der es sich um die Sprache der Übersetzung handelt, die für die Zwecke der internationalen Recherche eingereicht worden ist (Regeln 12.3 a) und 23.1 b)).

2. ☐ Dieser Bescheid wurde erstellt unter Berücksichtigung der **Berichtigung eines offensichtlichen Fehlers**, die nach Regel 91 von dieser Behörde genehmigt wurde bzw. dieser Behörde mitgeteilt wurde (Regel 43*bis*.1 a)).

3. ☐ Hinsichtlich der **Nucleotid- und/oder Aminosäuresequenz**, die in der internationalen Anmeldung offenbart wurde, ist der Bescheid auf der Grundlage eines Sequenzprotokolls erstellt worden, das

   a) ☐ im Anmeldezeitpunkt Bestandteil der internationalen Anmeldung war und

      ☐ in Form einer Textdatei gemäß Anhang C/ST.25 vorlag.

      ☐ in Papierform oder in Form einer Bilddatei vorlag.

   b) ☐ zusammen mit der internationalen Anmeldung gemäß Regel 13*ter*.1 a) PCT nur für die Zwecke der internationalen Recherche in Form einer Textdatei gemäß Anhang C/ST.25 eingereicht wurde.

   c) ☐ nach dem internationalen Anmeldedatum nur für die Zwecke der internationalen Recherche eingereicht wurde, und zwar

      ☐ in Form einer Textdatei gemäß Anhang C/ST.25 (Regel 13*ter*.1 a)).

      ☐ in Papierform oder in Form einer Bilddatei (Regel 13*ter*.1 b) und Abschnitt 713 der Verwaltungsvorschriften).

4. ☐ In dem Fall, dass mehr als eine Version oder Kopie eines Sequenzprotokolls eingereicht wurde, wurden zusätzlich die erforderlichen Erklärungen eingereicht, dass die Informationen in den nachgereichten oder zusätzlichen Kopien denen entsprechen, die im Anmeldezeitpunkt Bestandteil der Anmeldung waren, bzw. dass sie nicht über den Offenbarungsgehalt der Anmeldung im Anmeldezeitpunkt hinausgehen.

5. Zusätzliche Bemerkungen:

Feld Nr. V Begründete Feststellung nach Regel 43*bis*.1 a) i) hinsichtlich der Neuheit, der erfinderischen Tätigkeit und der gewerblichen Anwendbarkeit; Unterlagen und Erklärungen zur Stützung dieser Feststellung

1. Feststellung

Neuheit	Ja:	Ansprüche	4, 9, 11
	Nein:	Ansprüche	1-3, 5-8, 10, 12
Erfinderische Tätigkeit	Ja:	Ansprüche	
	Nein:	Ansprüche	1-12
Gewerbliche Anwendbarkeit	Ja:	Ansprüche:	1-12
	Nein:	Ansprüche:	

2. Unterlagen und Erklärungen:

siehe Beiblatt

Feld Nr. VIII Bestimmte Bemerkungen zur internationalen Anmeldung

Zur Klarheit der Patentansprüche, der Beschreibung und der Zeichnungen oder zu der Frage, ob die Ansprüche in vollem Umfang durch die Beschreibung gestützt werden, ist folgendes zu bemerken:

siehe Beiblatt

Zu Punkt V

Begründete Feststellung hinsichtlich der Neuheit, der erfinderischen Tätigkeit und der gewerblichen Anwendbarkeit; Unterlagen und Erklärungen zur Stützung dieser Feststellung

Es wird auf die folgenden Dokumente verwiesen:

D1 WO 2010/102377 A1 (ELLIOTT K W) 16. September 2010 (2010-09-16)

D2 DE 10 2011 110083 A1 (TECH UNI BRAUNSCHWEIG CAROLO WILHELMINA [DE]) 14. Februar 2013 (2013-02-14)in der Anmeldung erwähnt

1 Die vorliegende Anmeldung erfüllt nicht die Erfordernisse des Artikels 33 (2) PCT, weil der Gegenstand des der Ansprüche 1-3, 5-8, 10, 12 nicht neu ist.

1.1 D1 offenbart in Abbildungen 1 und 2 und Absätze 15-40 ein Verfahren zum Behandeln gebrauchter Batterien, mit den Schritten:
(a) Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird (Absatz 15),
(b) Inaktivieren des Zerkleinerungsguts, sodass ein inaktiviertes Zerkleinerungsgut erhalten wird (Absatz 16), und
(c) Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter, dadurch gekennzeichnet, dass
(d) das Inaktivieren durch Trocknen des Zerkleinerungsguts erfolgt wobei

- das Zerkleinerungsgut solange getrocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist (Absatz 37),

- das Trocknen unter Vakuum erfolgt (Absatz 19) und

(f) Abtrennen von Hartteilen und oder Separieren von Aktivmaterial vom Träger, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht (Absatz 25).

D1 offenbart nicht explizit, dass das Zerkleinerungsgut sowie die die Aktivmaterialfraktion und Trägerfraktion in Transportbehältnisse abgefüllt bzw. verpackt wird. Da die Produkte des Prozesses in D1 als Endprodukte zur industriellen Wiederverwertung genannt werden, kann davon ausgegangen werden, dass diese Produkte in einem Behältnis verpackt werden und daher von D1 implizit offenbart werden.

Ansprüche 2 und 6 sind durch das zu erreichende Ergebnis definiert (siehe dazu auch Punkt VII). Das selbe Ergebnis wird auch in D1 in Absatz 37 durch den Ausdruck "genügend getrocknet" offenbart.

Der Gegenstand der Ansprüche 1-3, 5, 6 ist somit nicht neu im Sinne des Artikels 33 (2) PCT.

1.2 Weiters offenbart D1 in Abbildungen 1 und 2 und Absätze 15-40 eine Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien mit:
(a) einer Zerkleinerungseinheit zum Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird (Absatz 15),
(b) einer Inaktivierungsvorrichtung zum Inaktivieren des Zerkleinerungsguts (Absatz 16) und
(c) einer Abfüllvorrichtung zum Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter, dadurch gekennzeichnet, dass
(d) die Inaktivierungsvorrichtung eine Trockenvorrichtung (siehe Referenzzeichen 20 in Abbildung 1) umfasst und
(e) die Batterie-Verarbeitungsanlage eine Vakuumanlage, die mit der Trockenvorrichtung zum Erzeugen eines Vakuums in der Trockenvorrichtung verbunden ist, aufweist (siehe Referenzzeichen 48 in Abbildung 1) und wobei das Zerkleinerungsgut solange getrocknet wird, bis ein Elektrolytgehalt so klein ist, dass eine elektrochemische Reaktion unmöglich ist (Absatz 37) sowie

- eine Hartmetallabtrennvorrichtung und eine Leichtfraktionsabtrennvorrichtung,
- eine Separiervorrichtung zum Separieren von Aktivmaterial vom Träger, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht (Absatz 25).

Das Abfüllen der Produkte wird als implizit von D1 offenbart betrachtet (siehe Abschnitt 1.1). Das zu erreichende Ergebnis wird in Absatz 37 offenbart (siehe Abschnitt 1.1 und Punkt VII).

Der Gegenstand der Ansprüche 7, 8, 10, 12 ist somit nicht neu im Sinne des Artikels 33 (2) PCT.

1.3 D2 wurde vom Anmelder als Stand der Technik in der Beschreibung genannt mit dem Nachteil der Komplexität der Anlage. da D2 jedoch alle Merkmale der in den Ansprüchen definierten gegenwärtigen Erfindung aufweist, wird D2 als neuheitsschädlich betrachtet.

1.4 D2 offenbart in Absätzen 21, 22, 34-39 und Abbildung 1 ein Verfahren zum Behandeln gebrauchter Batterien, insbesondere Lithium-Batterien, mit den Schritten:
(a) Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird (Referenzzeichen 12 in Abbildung 1),
(b) Inaktivieren des Zerkleinerungsguts, sodass ein inaktiviertes Zerkleinerungsgut erhalten wird, und
(c) Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter, dadurch gekennzeichnet, dass
(d) das Inaktivieren durch Trocknen des Zerkleinerungsguts unter Vakuum (Absätze 21, 22) zum Entfernen des Elektrolyts und
(c) Abtrennen von Hartteilen und oder Separieren von Aktivmaterial vom Träger, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht (Absatz 39).

Wie bereits in Absatz 1.1 ausgeführt, wird die Abfüllung bzw. Verpackung der Fraktionen als implizit offenbart angesehen.

Der Gegenstand der Ansprüche 1-3, 5, 6 ist somit nicht neu im Sinne des Artikels 33 (2) PCT.

1.5 Weiters offenbart D2 in 21, 22, 34-39 und Abbildung 1 eine Batterie-Verarbeitungsanlage zum Behandeln gebrauchter Batterien mit:
(a) einer Zerkleinerungseinheit zum Zerkleinern der Batterien, sodass Zerkleinerungsgut erhalten wird (Referenzzeichen 12 in Abbildung 1),
(b) einer Inaktivierungsvorrichtung zum Inaktivieren des Zerkleinerungsguts (28) und
(c) einer Abfüllvorrichtung zum Abfüllen des inaktivierten Zerkleinerungsguts in einen Transportbehälter (30), dadurch gekennzeichnet, dass
(d) die Inaktivierungsvorrichtung eine Trockenvorrichtung (28) umfasst und
(e) die Batterie-Verarbeitungsanlage eine Vakuumanlage (32), die mit der Trockenvorrichtung zum Erzeugen eines Vakuums in der Trockenvorrichtung verbunden ist, aufweist sowie
- eine Hartmetallabtrennvorrichtung und eine Leichtfraktionsabtrennvorrichtung,
- eine Separiervorrichtung zum Separieren von Aktivmaterial vom Träger durch Luftstrahlsieben, sodass eine Aktivmaterialfraktion und eine Trägerfraktion entsteht (Absatz 39).

Der Gegenstand der Ansprüche 7, 8, 10, 12 ist somit nicht neu im Sinne des Artikels 33 (2) PCT.

2 Die vorliegende Anmeldung erfüllt nicht die Erfordernisse des Artikels 33 (1) PCT, weil der Gegenstand der Ansprüche 4, 9, 11 nicht auf einer erfinderischen Tätigkeit im Sinne des Artikels 33 (3) PCT beruht.

2.1 D1 offenbart in Absatz 19 dass die Strahlpumpe ein pH-Kontrollsystem aufweist, das die Löslichkeit der Dämpfe sicherstellt. Mit Hinblick auf Absatz 42, in dem D1 offenbart dass die flüchtigen Substanzen mit Natriumhydroxid reagieren, würde die Fachperson den pH-Wert des Mediums alkalisch einstellen welches somit mit Fluorwasserstoff reagiert. Der Gegenstand des Anspruchs 4 ist somit nicht erfinderisch.

2.2 Des weiteren wird im abhängigen Anspruch 11 ist eine geringfügiges bauliches Detail definiert, welches nicht in D1 erwähnt wird. Die Kreisführung des Strahlmediums einer Strahlpumpe liegt jedoch innerhalb dessen, was eine Fachperson im Rahmen der üblichen Praxis zu tun pflegt, zumal die damit erreichten Vorteile ohne Weiteres im Voraus abzusehen sind. Folglich ist auch der Gegenstand des Anspruchs 11 nicht erfinderisch über D1.

2.3 Beim Merkmal der Anordnung der Anlage in einem Standard-Container handelt es sich nur um eine von mehreren naheliegenden Möglichkeiten, aus denen der Fachmann ohne erfinderisches Zutun den Umständen entsprechend eine wählen würde. Der Gegenstand des Anspruchs 9 ist somit nicht erfinderisch.

Zu Punkt VIII

Bestimmte Bemerkungen zur internationalen Anmeldung

3 Die Ansprüche 2 und 6 entsprechen nicht den Erfordernissen des Artikels 6 PCT, da der Gegenstand des Schutzbegehrens nicht klar definiert ist. In den Ansprüchen wird versucht, den Gegenstand durch das zu erreichende Ergebnis zu definieren; damit wird aber lediglich die zu lösende Aufgabe angegeben, ohne die für die Erzielung dieses Ergebnisses notwendigen technischen Merkmale zu nennen.

4 Die Ansprüche 7 und 10 wurden zwar als separate Ansprüche abgefasst, scheinen sich aber de facto auf ein und denselben Gegenstand zu beziehen. Aus diesem Grund sind die Ansprüche nicht knapp gefasst und erfüllen somit nicht die Erfordernisse des Artikels 6 PCT.