

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797	13529797 - GAU: 1723
	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

/E.C./	1	European Patent Office, International Preliminary Report on Patentability, PCT Application No. PCT/US2012/043534, September 24, 2013	☐
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EXAMINER SIGNATURE			
Examiner Signature	/Eric Connelly/	Date Considered	06/23/2014
*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.			
<small> 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. </small>			

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number	13529797	13529797 - GAU: 1723
	Filing Date	2012-06-21	
	First Named Inventor	Clive P. Bosnyak	
	Art Unit	1726	
	Examiner Name	Unknown	
	Attorney Docket Number	DENA-0008 (137124.41)	

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	/T. Ling Chwang/	Date (YYYY-MM-DD)	2013-11-19
Name/Print	T. Ling Chwang	Registration Number	33590

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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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.
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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.

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	2623	H01M4/587.cpc.	US-PGPUB; USPAT	OR	ON	2014/07/09 12:16
L2	612	H01M4/587.cpc. and (graphene OR nanotube OR CNT OR ALCT)	US-PGPUB; USPAT	OR	ON	2014/07/09 12:16
S2	2	(lithium with ion with carbon with nanotube with oxidation with aspect).clm.	US-PGPUB; USPAT	OR	ON	2014/06/20 14:54
S3	994	(carbon adj nanotube) with lithium	US-PGPUB; USPAT	OR	ON	2014/06/20 16:41
S4	156	"s1" and (aspect adj ration)	US-PGPUB; USPAT	OR	ON	2014/06/20 16:41
S8	238	S3 and (aspect adj ratio)	US-PGPUB; USPAT	OR	ON	2014/06/20 16:41
S11	9501	(carbon adj nanotube) and lithium	US-PGPUB; USPAT	OR	ON	2014/06/20 16:43
S12	1906	S11 and (aspect adj ratio)	US-PGPUB; USPAT	OR	ON	2014/06/20 16:43
S13	8	S12 and (oxidation adj level)	US-PGPUB; USPAT	OR	ON	2014/06/20 16:44
S14	2	("20110104551" "20110311876" "20110311876").PN.	US-PGPUB; USPAT	OR	ON	2014/06/23 18:32
S16	2	wo-2009155267\$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2014/06/23 18:33
S17	2	wo-2012083358\$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2014/06/23 18:34
S18	2	wo-2013011516\$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2014/06/23 18:34
S19	5	ep-1665446\$.did.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2014/06/23 18:34
S20	3	wo-2010117392\$.did.	US-PGPUB; USPAT; USOCR; FPRS;	OR	ON	2014/06/23 18:37

			EPO; JPO; DERWENT; IBM_TDB			
S21	780	graphene same lithium	US-PGPUB; USPAT	OR	ON	2014/06/24 10:46
S22	1505	graphene same lithium "0.1%-10%"	US-PGPUB; USPAT	OR	ON	2014/06/24 10:47
S23	5	graphene and lithium and "0.1%-10%"	US-PGPUB; USPAT	OR	ON	2014/06/24 10:47
S24	14	graphene and lithium and "0.1-10%"	US-PGPUB; USPAT	OR	ON	2014/06/24 10:48
S25	70	graphene and lithium and 5% WITH mass	US-PGPUB; USPAT	OR	ON	2014/06/24 10:49
S26	1	graphene with lithium with 5% WITH mass	US-PGPUB; USPAT	OR	ON	2014/06/24 10:50
S27	51	(graphene OR nanotube OR CNT OR ALCT) with (lithium or material or active) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH mass	US-PGPUB; USPAT	OR	ON	2014/06/24 10:52
S28	5	(graphene OR nanotube OR CNT OR ALCT) with (lithium or active adj material) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH mass	US-PGPUB; USPAT	OR	ON	2014/06/24 10:54
S29	47	(graphene OR nanotube OR CNT OR ALCT) with (lithium or active adj material) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH (mass or weight)	US-PGPUB; USPAT	OR	ON	2014/06/24 10:58
S30	16	(graphene OR nanotube OR CNT OR ALCT) with (lithium or active adj material) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH (mass or weight) and CVD	US-PGPUB; USPAT	OR	ON	2014/06/24 10:59
S35	0	(graphene OR nanotube OR CNT OR ALCT) with (lithium or active adj material) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH (mass or weight) same CVD and "429"	US-PGPUB; USPAT	OR	ON	2014/06/24 11:03
S36	12	(graphene OR nanotube OR CNT OR ALCT) with (lithium or active adj material) with (1% or 2% or 3% or 4% or 5% or 6% or 7% or 8% or 9% or 10%) WITH (mass or weight) and CVD and "429".clas.	US-PGPUB; USPAT	OR	ON	2014/06/24 11:03
S37	528	(graphene OR nanotube OR CNT OR ALCT) and "429/231.8".cls.	US-PGPUB; USPAT	OR	ON	2014/06/24 11:07
S38	662	(graphene OR nanotube OR CNT OR ALCT) and "429/231.8"	US-PGPUB; USPAT	OR	ON	2014/06/24 11:07
S39	366	(graphene OR nanotube OR CNT OR ALCT) and "429/231.8" and "%" with (mass or weight)	US-PGPUB; USPAT	OR	ON	2014/06/24 11:07
S40	73	"429/231.8" and "%" with (mass or weight) with (graphene OR nanotube OR CNT OR ALCT)	US-PGPUB; USPAT	OR	ON	2014/06/24 11:08

S41	26	"429/231.8" and "%" with (mass or weight) with (graphene OR nanotube OR CNT OR ALCT) with (lithium or active ADJ material)	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:08
S42	4	"429/231.8" and "%" with (mass or weight) with (graphene OR nanotube OR CNT OR ALCT) with (lithium or active ADJ material) and CVD	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:10
S43	0	"429/231.8" and "%" with (mass or weight) with (graphene OR nanotube OR CNT OR ALCT) with nanocoating	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:12
S44	0	"429/231.8" and (graphene OR nanotube OR CNT OR ALCT) with nanocoating	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:12
S45	6	"429/231.8" and nanocoating	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:12
S46	719	977/948	US-PGPUB; USPAT	OR	ON	2014/06/24: 11:14
S47	2	"20050042450"	US-PGPUB; USPAT	OR	ON	2014/06/25: 15:36
S48	8	("DESIGNED NANOTUBES" or SWOGGER OR BOSNYAK OR "MOLECULAR REBAR DESIGN").as.	US-PGPUB; USPAT	OR	ON	2014/06/25: 16:38

7/9/2014 12:19:00 PM

C:\Users\econnelly\Documents\EAST\Workspaces\13529797.wsp

<i>Index of Claims</i> 	Application/Control No. 13529797	Applicant(s)/Patent Under Reexamination BOSNYAK ET AL.
	Examiner ERIC CONNELLY	Art Unit 1723

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

☐ Claims renumbered in the same order as presented by applicant			☐ CPA			☐ T.D.			☐ R.1.47		
CLAIM		DATE									
Final	Original	06/25/2014									
	1	✓									
	2	✓									
	3	✓									
	4	✓									
	5	✓									
	6	✓									
	7	✓									
	8	N									
	9	N									
	10	✓									
	11	✓									
	12	✓									
	13	✓									

Search Notes 	Application/Control No. 13529797	Applicant(s)/Patent Under Reexamination BOSNYAK ET AL.
	Examiner ERIC CONNELLY	Art Unit 1723

CPC- SEARCHED		
Symbol	Date	Examiner
H01M 4/587	07/09/2014	EMC

CPC COMBINATION SETS - SEARCHED		
Symbol	Date	Examiner

US CLASSIFICATION SEARCHED			
Class	Subclass	Date	Examiner
429	231.8	06/24/2014	EMC

SEARCH NOTES		
Search Notes	Date	Examiner
429 Consultation with Nicholas D'Aniello	06/24/2014	EMC
429 Consultation with Brian Talbot	07/09/2014	EMC
Inventor Search in PALM	06/24/2014	EMC
Assignee Search in EAST	06/24/2014	EMC
Application 14/128392 History and Search Reviewed	06/24/2014	EMC
EAST Keyword Search	06/24/2014	EMC

INTERFERENCE SEARCH			
US Class/ CPC Symbol	US Subclass / CPC Group	Date	Examiner

/ERIC CONNELLY/ Examiner.Art Unit 1723	
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Doc code: IDS

Doc description: Information Disclosure Statement (IDS) Filed

13529797 - GALL: 1723

Approved for use through 07/31/2012. OMB 0851-0031

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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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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		
/E.C./	1	20110104551		2011-05-05	Yang, et al.			
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Examiner Initial*	Cite No	Foreign Document Number ³	Country Code ² j	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
/E.C./	1	2010/117392	WO		2010-10-14	Bosnyak, et al.		☐
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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	

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<small> 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. </small>			

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Signature	/Sara K. Borrelli/	Date (YYYY-MM-DD)	2012-10-17
Name/Print	Sara K. Borrelli	Registration Number	50,253

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/E.C./	1	20110311876		2011-12-22	Sturgeon et al.			
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/E.C./	1	2009/155267	WO		2009-12-23	Mysticmd, Inc.		☐
/E.C./	2	2012/083358	WO		2012-06-28	Commonwealth Scientific and Ind. Res. Organisation		☐
/E.C./	3	2013/011516	WO		2013-01-24	Vulcan Automotive Industries Ltd. et al.		☐

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Attorney Docket Number		DENA-0008 (137124.41)		

/E.C./	4	1665446	EP	B1	2012-03-21	Commonwealth Scientific and Ind. Res. Organisation	☐
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Examiner Signature	/Eric Connelly/	Date Considered	06/23/2014

*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	13529797	13529797 - GAU: 1723
	Filing Date	2012-06-21	
	First Named Inventor	Clive P. Bosnyak	
	Art Unit	1726	
	Examiner Name		
	Attorney Docket Number	DENA-0008 (137124.41)	

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

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☐ 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	/Sara K. Borrelli/	Date (YYYY-MM-DD)	2013-10-11
Name/Print	Sara K. Borrelli	Registration Number	50,253

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

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Doc code: IDS

Doc description: Information Disclosure Statement (IDS) Filed

13529797 - GALL: 1723

Approved for use through 07/31/2012. OMB 0851-0031

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	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

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	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

/E.C./	1	European Patent Office, Response to Written Opinion and Demand, PCT Application No. PCT/US2012/043534, April 23, 2013	☐
/E.C./	2	European Patent Office, Second Written Opinion, PCT Application No. PCT/US2012/043534, June 19, 2013	☐
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Examiner Signature	/Eric Connelly/		Date Considered 06/23/2014
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<small> 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. </small>			

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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	/Amy Ulfers Dunstan/	Date (YYYY-MM-DD)	2013-07-16
Name/Print	Amy Ulfers Dunstan	Registration Number	61,069

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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CONFIRMATION NO. 1004

SERIAL NUMBER 13/529,797	FILING or 371(c) DATE 06/21/2012 RULE	CLASS 429	GROUP ART UNIT 1723	ATTORNEY DOCKET NO. DENA-0008 (137124.00041)		
APPLICANTS INVENTORS Clive P. Bosnyak, Dripping Springs, TX; Kurt W. Swogger, Austin, TX; ** CONTINUING DATA ***** This appln claims benefit of 61/500,560 06/23/2011 ** FOREIGN APPLICATIONS ***** ** IF REQUIRED, FOREIGN FILING LICENSE GRANTED ** ** SMALL ENTITY ** 07/20/2012						
Foreign Priority claimed ☐ Yes ☒ No 35 USC 119(a-d) conditions met ☐ Yes ☐ No Verified and /ERIC M CONNELLY/ Acknowledged Examiner's Signature		☐ Met after Allowance Initials	STATE OR COUNTRY TX	SHEETS DRAWINGS 3	TOTAL CLAIMS 13	INDEPENDENT CLAIMS 6
ADDRESS Molecular Rebar Design, LLC Jackson Walker L.L.P. 901 Main Street, Suite 6000 Dallas, TX 75202 UNITED STATES						
TITLE LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM						
FILING FEE RECEIVED 970	FEES: Authority has been given in Paper No. _____ to charge/credit DEPOSIT ACCOUNT No. _____ for following:			☐ All Fees ☐ 1.16 Fees (Filing) ☐ 1.17 Fees (Processing Ext. of time) ☐ 1.18 Fees (Issue) ☐ Other _____ ☐ Credit		

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	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

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1	European Patent Office, International Preliminary Report on Patentability, PCT Application No. PCT/US2012/043534, September 24, 2013	☐
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Signature	/T. Ling Chwang/	Date (YYYY-MM-DD)	2013-11-19
Name/Print	T. Ling Chwang	Registration Number	33590

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PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

Vereenigde
Johan De Wittlaan 7
2517 JR Den Haag
PAYS-BAS

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(PCT Rule 71.1)

Date of mailing
(day/month/year)

24.09.2013

Applicant's or agent's file reference
P100028PC00

IMPORTANT NOTIFICATION

International application No.
PCT/US2012/043534

International filing date (day/month/year)
21.06.2012

Priority date (day/month/year)
23.06.2011

Applicant
Molecular Rebar Design, LLC

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary report on patentability and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary report on patentability. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international
preliminary examining authority:



European Patent Office P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Pays Bas
Tel. +31 70 340 - 2040
Fax: +31 70 340 - 3016

Authorized Officer

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Form PCT/PEA/416 (January 2004)

PATENT COOPERATION TREATY
PCT
INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter II of the Patent Cooperation Treaty)
(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P100028PC00	FOR FURTHER ACTION		See Form PCT/PEA416
International application No. PCT/US2012/043534	International filing date (<i>day/month/year</i>) 21.06.2012	Priority date (<i>day/month/year</i>) 23.06.2011	
International Patent Classification (IPC) or national classification and IPC INV. H01M4/62			
Applicant Molecular Rebar Design, LLC			
<p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>6</u> sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p style="margin-left: 20px;">a. ☒ <i>sent to the applicant and to the International Bureau</i> a total of <u>6</u> sheets, as follows:</p> <p style="margin-left: 40px;">☒ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).</p> <p style="margin-left: 40px;">☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.</p> <p style="margin-left: 20px;">b. ☐ (<i>sent to the International Bureau only</i>) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see paragraph 3bis of Annex C of the Administrative Instructions).</p>			
<p>4. This report contains indications relating to the following items:</p> <p>☒ Box No. I Basis of the report</p> <p>☐ Box No. II Priority</p> <p>☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p>☐ Box No. IV Lack of unity of invention</p> <p>☒ Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>☐ Box No. VI Certain documents cited</p> <p>☐ Box No. VII Certain defects in the international application</p> <p>☐ Box No. VIII Certain observations on the international application</p>			
Date of submission of the demand 23.04.2013		Date of completion of this report 24.09.2013	
Name and mailing address of the international preliminary examining authority:  European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016		Authorized officer Kelly, Michael Telephone No. +31 70 340-8973 	

Form PCT/PEA/409 (Cover Sheet) (July 2010)

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/US2012/043534

Box No. I Basis of the report

1. With regard to the **language**, this report is based on
- ☒ 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 (under Rules 12.3(a) and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4(a))
 - ☐ international preliminary examination (under Rules 55.2(a) and/or 55.3(a))
2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report):*

Description, Pages

1-12 as originally filed

Claims, Numbers

1-12 filed with telefax on 23-04-2013

Drawings, Sheets

1/3-3/3 as originally filed

☐ a sequence listing - see Supplemental Box Relating to Sequence Listing.

3. ☒ The amendments have resulted in the cancellation of:
- ☐ the description, pages
 - ☒ the claims, Nos. 13
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
 - ☐ any table(s) related to sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since either they are considered to go beyond the disclosure as filed, or they were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in the Supplemental Box (Rules 70.2(c) and (c-bis)):
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
5. ☐ 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 70.2 (e)).
6. ☐ Supplementary international search report(s) from Authority(ies) have been received and taken into account in drawing up this report (Rule 45bis.8(b) and (c)).

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/US2012/043534

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-12</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	<u>1-12</u>
	No: Claims	
Industrial applicability (IA)	Yes: Claims	<u>1-12</u>
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Re Item 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 2010/117392 A1 (BOSNYAK CLIVE P [US]; SWOGGER KURT W [US])
14 October 2010 (2010-10-14)

D2 US 2011/104551 A1 (YANG JUNBING [US] ET AL) 5 May 2011 (2011-05-05)

Novelty

A composition useful for lithium ion batteries comprising: discrete carbon nanotubes, the discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface wherein the discrete carbon nanotubes have an aspect ratio of 10 to 500 and oxidation levels from 1 % to 15% by weight of the carbon nanotube is not disclosed in the cited prior art.

The subject matter of independent product claim 3 and independent method claims 1 and 2 are therefore new.

Claims 4-12 are dependent on claim 3. The present application therefore meets the criteria of Article 33(2) PCT, because the subject-matter of claims 1-12 is new.

Inventive step

The present application meets the criteria of Article 33(3) PCT, because the subject-matter of claims 1-12 involves an inventive step.

D1 is regarded as being the prior art closest to the subject-matter of independent product claim 3 and discloses: a composition useful for lithium ion batteries comprising: discrete carbon nanotubes (D1: page 15, paragraph 0061), the discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface (D1: page 21, paragraph 0082 to page 22, paragraph 0083), wherein the discrete carbon nanotubes have an aspect ratio of 100 to 1000 (D1: page 10, paragraph 0042) and the carbon nanotubes are oxidised (D1: page 6, paragraph 0032).

The document D1 does not disclose carbon nanotubes with oxidation levels of 1% to 15% percent by weight.

The problem to be solved by the subject matter of claim 3 is to provide intimate electron transfer and enhanced mechanical strength of the active material.

Taking into account later-filed evidence provided by the applicant and supporting an inventive step, it is considered not obvious to the skilled person to select carbon nanotubes with oxidation levels of 1% to 15% percent by weight in order to provide intimate electron transfer and enhanced mechanical strength of the active material as it is not disclosed nor suggested in the cited prior art nor is it common knowledge in the technical field to do so.

Therefore the subject matter of claim 3 meets the requirements of the PCT in respect of inventive step.

Dependent claims 4-12 are dependent on claim 3 and as such also meet the requirements of the PCT in respect of inventive step.

D1 is regarded as being the prior art closest to the subject-matter of independent method claims 1 and 2, and discloses a process for the production of the discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, wherein the discrete carbon nanotubes have an aspect ratio of 100 to 1000 (D1: page 10, paragraph 0042) and the carbon nanotubes are oxidised (D1: page 6, paragraph 0032), comprising the steps of: mixing the carbon nanotubes with a liquid, reagents to synthesize a lithium salt and a surfactant, reacting the mixture at a temperature to form the lithium salt while sonicating the mixture, separating the solid discrete carbon nanotube salts from the liquid; and drying solid discrete carbon nanotube salts (D1: page 11, paragraph 0049 to page 12, paragraph 0052, page 21, paragraph 0082 to page 22, paragraph 0083 and page 28-29, example 9)

The subject-matter of claims 1 and 2 therefore differ from this known method in that the carbon nanotubes have oxidation levels of 1% to 15% percent by weight and the solid discrete carbon nanotube salts are annealed under an inert atmosphere at sufficient temperature to obtain an olivine crystal structure.

The problem to be solved by the present invention may therefore be regarded as providing a method to produce improved electrode material for lithium ion batteries.

The solution proposed in claims 1 and 2 of the present application can be considered as involving an inventive step (Article 33(3) PCT) for the following reasons: It is not obvious to the skilled person starting out from D1 to provide a method to produce improved electrode material for lithium ion batteries by selecting carbon nanotubes with oxidation levels of 1% to 15% percent by weight and producing discrete carbon nanotubes having crystals or layers of olivine lithium ion active material attached to

their surface, by annealing under an inert atmosphere at sufficient temperature to obtain the olivine crystal structure, as it is not disclosed nor suggested in the cited prior art nor is it common knowledge to do so.

Industrial applicability

The subject matter described in claims 1-12 can find use in industry and is therefore industrially applicable(Article 33(4) PCT).

Electronic Acknowledgement Receipt	
EFS ID:	17444929
Application Number:	13529797
International Application Number:	
Confirmation Number:	1004
Title of Invention:	LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM
First Named Inventor/Applicant Name:	Clive P. Bosnyak
Customer Number:	117364
Filer:	T. Ling Chwang/Debra Pepper
Filer Authorized By:	T. Ling Chwang
Attorney Docket Number:	DENA-0008 (137124.00041)
Receipt Date:	19-NOV-2013
Filing Date:	21-JUN-2012
Time Stamp:	16:08:48
Application Type:	Utility under 35 USC 111(a)

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	Information Disclosure Statement (IDS) Form (SB08)	DENA0008IDS4.pdf	611722 <small>125a925041f71d178b52cc5351fd605465338998</small>	no	4
Warnings:					
Information:					

A U.S. Patent Number Citation or a U.S. Publication Number Citation is required in the Information Disclosure Statement (IDS) form for autoloading of data into USPTO systems. You may remove the form to add the required data in order to correct the Informational Message if you are citing U.S. References. If you chose not to include U.S. References, the image of the form will be processed and be made available within the Image File Wrapper (IFW) system. However, no data will be extracted from this form. Any additional data such as Foreign Patent Documents or Non Patent Literature will be manually reviewed and keyed into USPTO systems.

2	Other Reference-Patent/App/Search documents	DENA0008IPRP.pdf	543490	no	7
			eabf18780e3c978a76e1e888524a5649e641882b		

Warnings:

Information:

Total Files Size (in bytes):	1155212
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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.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797	
	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name			
	Attorney Docket Number	DENA-0008 (137124.41)		

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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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	20110311876		2011-12-22	Sturgeon et al.			
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Examiner Initial*	Cite No	Foreign Document Number ³	Country Code ² j	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	2009/155267	WO		2009-12-23	Mysticmd, Inc.		☐
	2	2012/083358	WO		2012-06-28	Commonwealth Scientific and Ind. Res. Organisation		☐
	3	2013/011516	WO		2013-01-24	Vulcan Automotive Industries Ltd. et al.		☐

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797	
	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name			
	Attorney Docket Number	DENA-0008 (137124.41)		

	4	1665446	EP	B1	2012-03-21	Commonwealth Scientific and Ind. Res. Organisation		☐
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NON-PATENT LITERATURE DOCUMENTS

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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
	1		☐

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EXAMINER SIGNATURE

Examiner Signature		Date Considered	
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*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		13529797
	Filing Date		2012-06-21
	First Named Inventor	Clive P. Bosnyak	
	Art Unit		1726
	Examiner Name		
	Attorney Docket Number		DENA-0008 (137124.41)

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	/Sara K. Borrelli/	Date (YYYY-MM-DD)	2013-10-11
Name/Print	Sara K. Borrelli	Registration Number	50,253

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.**

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.

Electronic Acknowledgement Receipt	
EFS ID:	17104093
Application Number:	13529797
International Application Number:	
Confirmation Number:	1004
Title of Invention:	LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM
First Named Inventor/Applicant Name:	Clive P. Bosnyak
Customer Number:	117364
Filer:	Sara Kay Borrelli/Debra Pepper
Filer Authorized By:	Sara Kay Borrelli
Attorney Docket Number:	DENA-0008 (137124.00041)
Receipt Date:	11-OCT-2013
Filing Date:	21-JUN-2012
Time Stamp:	12:36:08
Application Type:	Utility under 35 USC 111(a)

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	Information Disclosure Statement (IDS) Form (SB08)	DENA0008IDS3.pdf	611966 f8a9613211c00fca305e0cb90bd528808aa32b85	no	4
Warnings:					
Information:					

2	Foreign Reference	MOREWO2009155267.pdf	1580403	no	35
			efffe458129c8f2763c56f914d3a34125ab7bea0		
Warnings:					
Information:					
3	Foreign Reference	MOREWO2012083358.pdf	2053251	no	45
			ccc6807128498dcd3da45fc57de11613078df131		
Warnings:					
Information:					
4	Foreign Reference	MOREWO2013011516.pdf	2729988	no	38
			ff1f18022f71e86a16a8c39621e4dbc2ba8e5e0d		
Warnings:					
Information:					
5	Foreign Reference	MOREEP1665446.pdf	1247302	no	21
			c8955545fc7409da61da61b6eb6a358e4efb7daa		
Warnings:					
Information:					
Total Files Size (in bytes):			8222910		
<p>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.</p> <p><u>New Applications Under 35 U.S.C. 111</u> 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.</p> <p><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.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> 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.</p>					



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APPLICATION NUMBER	PATENT NUMBER	GROUP ART UNIT	FILE WRAPPER LOCATION
13/529,797		1723	



Correspondence Address/Fee Address Change

The following fields have been set to Customer Number 117364 on 08/27/2013

- Correspondence Address
- Maintenance Fee Address

The address of record for Customer Number 117364 is:

117364
Molecular Rebar Design, LLC
Jackson Walker L.L.P.
901 Main Street, Suite 6000
Dallas, TX 75202

PART 1 - ATTORNEY/APPLICANT COPY
page 1 of 1

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797	
	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

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							
If you wish to add additional U.S. Patent citation information please click the Add button.							Add	
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		
	1							
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Examiner Initial*	Cite No	Foreign Document Number ³	Country Code ² j	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							☐
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

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797
	Filing Date		2012-06-21
	First Named Inventor	Clive P. Bosnyak	
	Art Unit	1726	
	Examiner Name	Unknown	
	Attorney Docket Number	DENA-0008 (137124.41)	

1	European Patent Office, Response to Written Opinion and Demand, PCT Application No. PCT/US2012/043534, April 23, 2013	☐
2	European Patent Office, Second Written Opinion, PCT Application No. PCT/US2012/043534, June 19, 2013	☐
If you wish to add additional non-patent literature document citation information please click the Add button Add		
EXAMINER SIGNATURE		
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.		
<small> 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. </small>		

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797
	Filing Date		2012-06-21
	First Named Inventor	Clive P. Bosnyak	
	Art Unit		1726
	Examiner Name	Unknown	
	Attorney Docket Number		DENA-0008 (137124.41)

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	/Amy Ulfers Dunstan/	Date (YYYY-MM-DD)	2013-07-16
Name/Print	Amy Ulfers Dunstan	Registration Number	61,069

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.**

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.

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

Vereenigde
Johan De Wittlaan 7
2517 JR Den Haag
PAYS-BAS

PCT

WRITTEN OPINION OF THE INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY (PCT Rule 66)

Date of mailing (day/month/year) 19.06.2013	
Applicant's or agent's file reference P100028PC00	REPLY DUE within <u>2</u> month(s) from the above date of mailing
International application No. PCT/US2012/043534	International filing date (day/month/year) 21.06.2012
Priority date (day/month/year) 23.06.2011	
International Patent Classification (IPC) or both national classification and IPC INV. H01M4/62	
Applicant Designed Nanotubes, LLC	

1.	☒ The written opinion established by the International Searching Authority: <div style="margin-left: 20px;"> ☒ is ☐ is not considered to be a written opinion of the International Preliminary Examining Authority. </div>
2.	This <u>second</u> opinion contains indications relating to the following items: <div style="margin-left: 20px;"> ☒ 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 66.2(a)(ii) 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 </div>
3.	The applicant is hereby invited to reply to this opinion. When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(e). How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 55.3 and 66.8. Also: For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4bis. For an informal communication with the examiner, see Rule 66.6. For an additional opportunity to submit amendments, see Rule 66.4. If no reply is filed , the international preliminary examination report will be established on the basis of this opinion.
4.	The final date by which the international preliminary report on patentability (Chapter II of the PCT) must be established according to Rule 69.2 is: <u>23.10.2013</u>

Name and mailing address of the international preliminary examining authority: <div style="display: flex; align-items: center;"> <div> European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016 </div> </div>	Authorized Officer Kelly, Michael Telephone No. +31 70 340-8973
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**WRITTEN OPINION OF THE INTERNATIONAL
PRELIMINARY EXAMINING AUTHORITY**

International application No.
PCT/US2012/043534

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 (under Rules 12.3(a) and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4(a))
 - ☐ international preliminary examination (under Rules 55.2(a) and/or 55.3(a) and (b))

2. With regard to the elements of the international application, this opinion has been established on the basis of
(*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article
14 are referred to in this opinion as "originally filed"*):

Description, Pages

1-12 as originally filed

Claims, Numbers

1-12 filed with telefax on 23-04-2013

Drawings, Sheets

1/3-3/3 as originally filed

- ☐ a sequence listing - see Supplemental Box Relating to Sequence Listing.

3. ☒ The amendments have resulted in the cancellation of:

- ☐ the description, pages
- ☒ the claims, Nos. 13
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):

4. ☐ This opinion has been established as if (some of) the amendments listed below had not been made, since
either they are considered to go beyond the disclosure as filed, or they were not accompanied by a
letter indicating the basis for the amendments in the application as filed, as indicated in the Supplemental
Box (Rules 70.2(c) and (c-bis)):

- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):

5. ☐ 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 66.1(d-bis)).
- ☐ without taking into account the rectification of an obvious mistake authorized by or notified to this
Authority under Rule 91 (Rule 66.4bis).

**WRITTEN OPINION OF THE INTERNATIONAL
PRELIMINARY EXAMINING AUTHORITY**

International application No.
PCT/US2012/043534

6. ☐ Supplementary international search report(s) from Authority(ies) have been received and taken into account in drawing up this opinion (Rule 45bis.8(b) and (c)).

Box No. V Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	No:	Claims
Inventive step (IS)	No:	Claims 3-12
Industrial applicability (IA)	No:	Claims

2. Citations and explanations:

see separate sheet

Re Item 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 2010/117392 A1 (BOSNYAK CLIVE P [US]; SWOGGER KURT W [US])
14 October 2010 (2010-10-14)

D2 US 2011/104551 A1 (YANG JUNBING [US] ET AL) 5 May 2011 (2011-05-05)

Novelty

The present application meets the criteria of Article 33(2) PCT, because the subject-matter of claims 1-12 is new.

Inventive step

The present application meets the criteria of Article 33(3) PCT, because the subject-matter of claims 1-2 involves an inventive step.

D1 is regarded as being the prior art closest to the subject-matter of claims 1 and 2, and discloses a process for the production of the discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, comprising the steps of: mixing the carbon nanotubes with a liquid, reagents to synthesize a lithium salt and a surfactant, reacting the mixture at a temperature to form the lithium salt while sonicating the mixture, separating the solid discrete carbon nanotube salts from the liquid; and drying solid discrete carbon nanotube salts (D1: page 11, paragraph 0049 to page 12, paragraph 0052, page 21, paragraph 0082 to page 22, paragraph 0083 and page 28-29, example 9)

The subject-matter of claims 1 and 2 therefore differ from this known method in that the solid discrete carbon nanotube salts are annealed under an inert atmosphere at sufficient temperature to obtain an olivine crystal structure.

The problem to be solved by the present invention may therefore be regarded as providing a method to produce improved electrode material for lithium ion batteries.

The solution proposed in claims 1 and 2 of the present application can be considered as involving an inventive step (Article 33(3) PCT) for the following reasons: It is not obvious to the skilled person starting out from D1 to provide a method to produce improved electrode material for lithium ion batteries by producing discrete carbon

nanotubes having crystals or layers of olivine lithium ion active material attached to their surface, by annealing under an inert atmosphere at sufficient temperature to obtain the olivine crystal structure, as it is not disclosed nor suggested in the cited prior art nor is it common knowledge to do so.

The present application does not meet the criteria of Article 33(3) PCT, because the subject-matter of claims 3-12 does not involve an inventive step.

D1 and D2 disclose the subject matter of claim 1: a composition useful for lithium ion batteries comprising: discrete carbon nanotubes (D1: page 15, paragraph 0061; D2: page 1, paragraph 0009, Figures 1, 4), the discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface (D1: page 21, paragraph 0082 to page 22, paragraph 0083; D2: Figures 2-3, abstract, page 1, paragraphs 0007-0009), the discrete carbon nanotubes of aspect ratio 10 to 500 (D1: page 10, paragraph 0042; D2: page 4, paragraph 0042) and the carbon nanotubes are oxidised (D1: page 6, paragraph 0032; D2: page 3, paragraph 0039).

The documents D1 and D2 do not explicitly disclose the oxidation level, however oxidation levels of 1% to 15% percent by weight is not considered to involve an inventive step as such levels would be considered obvious to the skilled person in order to provide an improved electrode material.

Furthermore there is no surprising technical effect attributed to these oxidation levels. There is no evidence in the application to show that the oxidation levels of the carbon nanotubes provide the desired advantages listed in paragraphs 0006 to 0008 of the description. There are no comparative examples in the application which can show an effect associated with the selection of such an oxidation level. Therefore the subject matter of claim 3 is not inventive.

Dependent claims 4-12 do not contain any features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT in respect of inventive step:

D1 discloses that the lithium ion active material can be "for example SnO_2 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$, silicon nanotubes, silicon nanoparticles and various combinations thereof" (D1: page 22, paragraph 0082). D2 discloses that "the lithium-alloying material preferably is selected from Si, Sn, Pb, Al, Au, Pt, Zn, Cd, Ag, Mg, and a combination of two or more of the foregoing" (D2: page 1, paragraph 0007). The subject matter of claims 4 and 6 are therefore not inventive.

That the lithium metal salt has an olivine crystal structure is a well known alternative to a lithium metal salt such as $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as disclosed in D1 (see D1: page 22, paragraph 0082; D2: page 1, paragraph 0004). The subject matter of claim 5 is therefore not inventive.

D2 discloses that the carbon nanotubes comprise graphene (D2: page 4, paragraph 0042) and that the weight ratio of graphene to carbon nanotube is 0.1:99.9 to 99.9:0.1 is considered implicit. The subject matter of claims 7-8 is not inventive.

That the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10% is not considered to involve an inventive step. This feature is merely a straightforward possibility from which the skilled person would select, in accordance with circumstances, without the exercise of inventive skill, in order to provide an improved battery electrode composition; there is no surprising technical effect attributed to this feature and therefore the subject matter of independent claim 9 is not inventive.

It is clear from D1 and D2 that the nanotubes are uniformly dispersible and therefore the subject matter of claims 10-11 is not inventive.

D1 discloses that the active materials are chemically attached to the carbon nanotubes and the subject matter of claim 12 is not inventive (D1: pages 21-22, paragraph 0082, "bound").

Industrial applicability

The subject matter described in claims 1-12 can find use in industry and is therefore industrially applicable (Article 33 (1) and (4) PCT).

Electronic Acknowledgement Receipt	
EFS ID:	16324122
Application Number:	13529797
International Application Number:	
Confirmation Number:	1004
Title of Invention:	LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM
First Named Inventor/Applicant Name:	Clive P. Bosnyak
Customer Number:	25555
Filer:	Amy L. Ulfers/Debra Pepper
Filer Authorized By:	Amy L. Ulfers
Attorney Docket Number:	DENA-0008 (137124.00041)
Receipt Date:	16-JUL-2013
Filing Date:	21-JUN-2012
Time Stamp:	08:27:40
Application Type:	Utility under 35 USC 111(a)

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	Information Disclosure Statement (IDS) Form (SB08)	DENA0008IDS2.pdf	611755 b1169c0a4dc2463963274ec4f95601f3ff6320fc	no	4
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A U.S. Patent Number Citation or a U.S. Publication Number Citation is required in the Information Disclosure Statement (IDS) form for autoloading of data into USPTO systems. You may remove the form to add the required data in order to correct the Informational Message if you are citing U.S. References. If you chose not to include U.S. References, the image of the form will be processed and be made available within the Image File Wrapper (IFW) system. However, no data will be extracted from this form. Any additional data such as Foreign Patent Documents or Non Patent Literature will be manually reviewed and keyed into USPTO systems.

2	Other Reference-Patent/App/Search documents	DENA0008RESPONSETOWO.pdf	848717	no	11
			87fbc2c9e3cfb36dccc3922eca3034bf95c5110d5		

Warnings:

Information:

3	Other Reference-Patent/App/Search documents	DENA0008SECONDWO.pdf	481617	no	6
			352525e9d1dabbcd71d4acd41b32de38beb277f5		

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Information:

Total Files Size (in bytes):			1942089
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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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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/529,797	06/21/2012	Clive P. Bosnyak	DENA-0008 (137124.00041)

CONFIRMATION NO. 1004

25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

PUBLICATION NOTICE



OC000000058355705

Title:LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM

Publication No.US-2012-0328946-A1

Publication Date:12/27/2012

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.

The publication may be accessed through the USPTO's publicly available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently <http://www.uspto.gov/patft/>.

The publication process established by the Office does not provide for mailing a copy of the publication to applicant. A copy of the publication may be obtained from the Office upon payment of the appropriate fee set forth in 37 CFR 1.19(a)(1). Orders for copies of patent application publications are handled by the USPTO's Office of Public Records. The Office of Public Records can be reached by telephone at (703) 308-9726 or (800) 972-6382, by facsimile at (703) 305-8759, by mail addressed to the United States Patent and Trademark Office, Office of Public Records, Alexandria, VA 22313-1450 or via the Internet.

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 <http://pair.uspto.gov/>. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

Further assistance in electronically accessing the publication, or about PAIR, is available by calling the Patent Electronic Business Center at 1-866-217-9197.

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)	Application Number		13529797	
	Filing Date		2012-06-21	
	First Named Inventor	Clive P. Bosnyak		
	Art Unit	1726		
	Examiner Name	Unknown		
	Attorney Docket Number	DENA-0008 (137124.41)		

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	20110104551		2011-05-05	Yang, et al.			
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	1	2010/117392	WO		2010-10-14	Bosnyak, et al.		☐
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	Art Unit	1726	
	Examiner Name	Unknown	
	Attorney Docket Number	DENA-0008 (137124.41)	

1	European Patent Office, International Search Report and Written Opinion, PCT Application No. PCT/US2012/043534, October 5, 2012	☐
If you wish to add additional non-patent literature document citation information please click the Add button Add		
EXAMINER SIGNATURE		
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.		
<small> 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. </small>		

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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	/Sara K. Borrelli/	Date (YYYY-MM-DD)	2012-10-17
Name/Print	Sara K. Borrelli	Registration Number	50,253

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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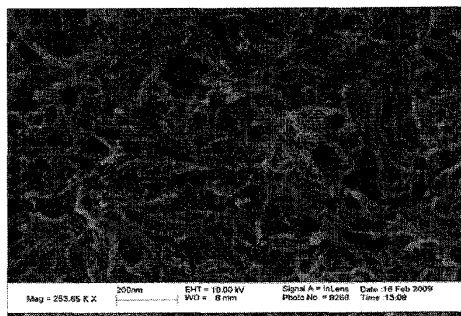


FIGURE 8

(57) **Abstract:** In various embodiments, exfoliated carbon nanotubes are described in the present disclosure. The carbon nanotubes maintain their exfoliated state, even when not dispersed in a medium such as a polymer or a liquid solution. Methods for making the exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a nanocrystalline material, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes. Nanocrystalline materials may include nanorods, hydroxyapatite and various hydroxyapatite derivatives. In some embodiments, methods for making exfoliated carbon nanotubes include preparing a solution of carbon nanotubes in an acid and filtering the solution through a filter to collect exfoliated carbon nanotubes on the filter. In some embodiments, a concentration of carbon nanotubes in the acid is below the percolation threshold. In other various embodiments, energy storage devices and polymer composites containing exfoliated carbon nanotubes are described herein. The energy storage device may be a battery containing at least two electrodes and an electrolyte in contact with the at least two electrodes. At least one of the electrodes in the energy storage device advantageously contains exfoliated carbon nanotubes. The polymer composites are prepared by mixing exfoliated carbon nanotubes with a polymer material. After being mixed in the polymer material, the carbon nanotubes maintain their exfoliated state.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
PATENT APPLICATION

**EXFOLIATED CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF
AND PRODUCTS OBTAINED THEREFROM**

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EXFOLIATED CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND
PRODUCTS OBTAINED THEREFROM

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to United States provisional patent applications 61/138,551, filed December 18, 2008, and 61/139,050, filed December 19, 2008, each of which is incorporated by reference herein in its entirety.

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH

[0002] Not applicable

BACKGROUND

[0003] Carbon nanotubes in their solid state are currently produced as agglomerated nanotube bundles in a mixture of chiral forms. Current technologies cannot fully exfoliate bundles of carbon nanotubes to produce individualized carbon nanotubes in the solid state without significant chemical and physical property modifications taking place to the carbon nanotubes. Additionally, there are currently no effective methods to separate carbon nanotubes on a bulk scale by length, diameter, chirality, or a combination thereof.

[0004] Various methods have been developed to debundle carbon nanotubes in solution. For example, carbon nanotubes may be shortened by oxidative means and then dispersed as individual nanotubes in solution. Carbon nanotubes may also be dispersed in solution as individuals by sonication in the presence of a surfactant. Illustrative surfactants used for dispersing carbon nanotubes in solution include, for example, sodium dodecyl sulfate and PLURONICS. In some instances, solutions of individualized carbon nanotubes may be prepared from polymer-wrapped carbon nanotubes. Individualized single-wall carbon nanotube solutions have also been prepared using polysaccharides, polypeptides, water-soluble polymers, nucleic acids, DNA, polynucleotides, polyimides, and polyvinylpyrrolidone.

[0005] A number of uses for carbon nanotubes have been proposed including, for example, energy storage devices (e.g., ultracapacitors, supercapacitors and batteries), field emitters, conductive films, conductive wires and membrane filters. Use of carbon nanotubes as a

reinforcing agent in polymer composites is another area in which carbon nanotubes are predicted to have significant utility. However, utilization of carbon nanotubes in these applications has been hampered due to the general inability to reliably produce individualized carbon nanotubes. For example, load transfer to carbon nanotubes in polymer composites is typically less than would be expected than if the carbon nanotubes were fully exfoliated as individual nanotubes.

[0006] Likewise, in applications involving electrical conduction, conductivity is lower than anticipated due to reduced access to the carbon nanotube's surface when the carbon nanotubes are agglomerated as opposed to being dispersed as individuals. Furthermore, when mixtures of conducting and non-conducting or semiconducting carbon nanotubes (i.e., carbon nanotubes having a mixture of chiralities) are used in applications involving electrical conduction, conductivity is less than could be achieved were all the carbon nanotubes electrical conductors. As noted above, current methods for producing exfoliated carbon nanotubes usually results in shortening or functionalization of the nanotubes. Such shortening or functionalization also generally results in reduced conductivity, which is also disadvantageous for applications where high electrical conductivity is beneficial.

[0007] In view of the foregoing, solid exfoliated carbon nanotubes and methods for efficiently exfoliating carbon nanotubes without nanotube damage are of considerable interest in the art. Such exfoliated carbon nanotubes are likely to exhibit considerably improved properties in applications including, for example, energy storage devices and polymer composites. Further separation of the exfoliated carbon nanotubes by chirality, length, diameter, or a combination thereof would also be of considerable interest in the art to further take advantage of their properties.

SUMMARY

[0008] In various embodiments, compositions of exfoliated carbon nanotubes are disclosed herein. The exfoliated carbon nanotubes are dispersed in the solid state such as, for example, a mat of dispersed carbon nanotubes. The exfoliated carbon nanotubes are maintained in an exfoliated state without being dispersed in a continuous matrix such as, for example, a polymer matrix dispersant or a solution.

[0009] In other various embodiments, methods for preparing exfoliated carbon nanotubes are disclosed herein.

[0010] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a first quantity of a nanocrystalline material, precipitating a first quantity of exfoliated carbon nanotubes from the solution and isolating the first quantity of exfoliated carbon nanotubes.

[0011] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing hydroxyapatite, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes.

[0012] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a nanorod material, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes.

[0013] In some embodiments, the methods for preparing exfoliated carbon nanotubes include preparing a solution of carbon nanotubes in an acid and filtering the solution through a filter to collect exfoliated carbon nanotubes on the filter.

[0014] In still other various embodiments, energy storage devices containing exfoliated carbon nanotubes are disclosed herein. In some embodiments, the energy storage device is a battery containing at least two electrodes and an electrolyte in contact with the at least two electrodes. At least one of the electrodes contains exfoliated carbon nanotubes.

[0015] In yet additional various embodiments, methods for making a polymer composite are disclosed herein. The methods include a) providing exfoliated carbon nanotubes and b) mixing the exfoliated carbon nanotubes in a polymer material to form a polymer composite. The exfoliated carbon nanotubes remain in an exfoliated state after being mixed in the polymer material.

[0016] The foregoing has outlined rather broadly various features of the present disclosure in order that the detailed description that follows may be better understood. Additional features and advantages of the disclosure will be described hereinafter, which form the subject of the claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] For a more complete understanding of the present disclosure, and the advantages thereof, reference is now made to the following descriptions to be taken in conjunction with the accompanying drawings describing specific embodiments of the disclosure, wherein:

[0018] FIGURE 1 shows an illustrative arrangement of the basic elements of a Faradaic capacitor;

[0019] FIGURE 2 shows an illustrative arrangement of the basic elements of an electric double layer capacitor;

[0020] FIGURE 3 shows an illustrative arrangement of the basic elements of a battery;

[0021] FIGURE 4 shows an illustrative electron micrograph of hydroxyapatite plates having diameters of 3 – 15 μm ;

[0022] FIGURE 5 shows an illustrative electron micrograph of hydroxyapatite nanorods having lengths of 100 – 200 nm;

[0023] FIGURE 6A shows an illustrative electron micrograph of as-received multi-wall carbon nanotubes; FIGURE 6B shows an illustrative electron micrograph of multi-wall carbon nanotubes exfoliated using hydroxyapatite nanorods;

[0024] FIGURE 7A shows an illustrative EDX spectrum of precipitated exfoliated, multi-wall carbon nanotubes; FIGURE 7B shows an illustrative EDX spectrum of precipitated, exfoliated multi-wall carbon nanotubes after acid washing;

[0025] FIGURE 8 shows an illustrative electron micrograph of exfoliated multi-wall carbon nanotubes after precipitation and washing;

[0026] FIGURE 9 shows an illustrative electron micrograph of exfoliated carbon nanotubes obtained from 3:1 $\text{H}_2\text{SO}_4\text{:HNO}_3$;

[0027] FIGURE 10 shows an illustrative electron micrograph of exfoliated double-wall carbon

nanotubes following acid exfoliation and treatment with sodium dodecyl sulfate; and

[0028] FIGURE 11 shows an illustrative electron micrograph of exfoliated carbon nanotubes decorated with copper oxide nanoparticles.

DETAILED DESCRIPTION

[0029] In the following description, certain details are set forth such as specific quantities, sizes, etc. so as to provide a thorough understanding of the present embodiments disclosed herein. However, it will be evident to those of ordinary skill in the art that the present disclosure may be practiced without such specific details. In many cases, details concerning such considerations and the like have been omitted inasmuch as such details are not necessary to obtain a complete understanding of the present disclosure and are within the skills of persons of ordinary skill in the relevant art.

[0030] Referring to the drawings in general, it will be understood that the illustrations are for the purpose of describing particular embodiments of the disclosure and are not intended to be limiting thereto. Drawings are not necessarily to scale.

[0031] While most of the terms used herein will be recognizable to those of ordinary skill in the art, it should be understood, however, that when not explicitly defined, terms should be interpreted as adopting a meaning presently accepted by those of ordinary skill in the art. In cases where the construction of a term would render it meaningless or essentially meaningless, the definition should be taken from Webster's Dictionary, 3rd Edition, 2009. Definitions and/or interpretations should not be incorporated from other patent applications, patents, or publications, related or not, unless specifically stated in this specification or if the incorporation is necessary for maintaining validity.

[0032] Various embodiments presented hereinbelow reference carbon nanotubes. In particular, in various embodiments, bundled carbon nanotubes can be debundled according to the methods described herein to produce exfoliated carbon nanotube solids. The carbon nanotubes being debundled can be made from any known means such as, for example, chemical vapor deposition, laser ablation, and high pressure carbon monoxide synthesis (HiPco). The bundled carbon

nanotubes can be present in a variety of forms including, for example, soot, powder, fibers, and bucky paper. Furthermore, the bundled carbon nanotubes may be of any length, diameter, or chirality. Carbon nanotubes may be metallic, semi-metallic, semi-conducting or non-metallic based on their chirality and number of walls. In various embodiments, the bundled and/or exfoliated carbon nanotubes may include, for example, single-wall carbon nanotubes (SWNTs), double-wall carbon nanotubes (DWNTs), multi-wall carbon nanotubes (MWNTs), shortened carbon nanotubes, oxidized carbon nanotubes, functionalized carbon nanotubes, and combinations thereof. One of ordinary skill in the art will recognize that many of the specific embodiments referenced hereinbelow utilizing a particular type of carbon nanotube may practiced equivalently within the spirit and scope of the disclosure utilizing other types of carbon nanotubes.

[0033] Functionalized carbon nanotubes of the present disclosure generally refer to the chemical modification of any of the carbon nanotube types described hereinabove. Such modifications can involve the nanotube ends, sidewalls, or both. Chemical modifications may include, but are not limited to covalent bonding, ionic bonding, chemisorption, intercalation, surfactant interactions, polymer wrapping, cutting, solvation, and combinations thereof. In some embodiments, the carbon nanotubes may be functionalized before being exfoliated. In other embodiments, the carbon nanotubes are functionalized after being exfoliated.

[0034] In some embodiments, the carbon nanotubes may be further associated or functionalized with an electroactive material. In some embodiments, an electroactive material may be transition metals or oxides of transition metals such as, for example, Ru, Ir, W, Mo, Mn, Ni and Co. In some embodiments, the electroactive material may be a conducting polymer such as, for example, polyaniline, polyvinylpyrrole or polyacetylene. In some embodiments, the electroactive material may be a nanoparticle or plurality of nanoparticles bound to the carbon nanotubes. For example, in some embodiments, an electroactive nanoparticle may include materials such as SnO_2 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$, silicon nanotubes, silicon nanoparticles and various combinations thereof. Carbon nanotubes associated or functionalized with an electroactive material may be particularly advantageous for applications involving electrical conductivity.

[0035] Any of the embodiments herein referencing carbon nanotubes may also be modified within the spirit and scope of the disclosure to substitute other tubular nanostructures, including, for example, inorganic or mineral nanotubes. Inorganic or mineral nanotubes include, for example, silicon nanotubes, boron nitride nanotubes and carbon nanotubes having heteroatom substitution in the nanotube structure. In various embodiments, the nanotubes may include elements such as, for example, carbon, silicon, boron and nitrogen. In further embodiments, the inorganic or mineral nanotubes may also include metallic and non-metallic elements. For example, in some embodiments, the inorganic or mineral nanotubes can be associated with metals, organic compounds, and inorganic compounds. Association may be on the interior or exterior of the inorganic or mineral nanotubes. Exterior association may be a physical association, such as, for example, van der Waals association. Exterior association of these materials may also include either ionic or covalent bonding to the nanotube exterior.

[0036] In various embodiments, the present disclosure describes compositions containing exfoliated carbon nanotubes. The exfoliated carbon nanotubes are not dispersed in a continuous matrix that maintains the carbon nanotubes in an exfoliated state. Illustrative continuous matrices include, for example, a solution or a polymer matrix that maintains the carbon nanotubes in at least a partially or substantially exfoliated state. In various embodiments, the exfoliated carbon nanotubes comprise a carbon nanotube mat. As such, the exfoliated carbon nanotubes of the present disclosure are distinguished over exfoliated carbon nanotubes presently known in the art, which may re-agglomerate once removed from solution.

[0037] The exfoliated carbon nanotubes of the present disclosure take advantage of physical properties offered by individual carbon nanotubes that are not apparent when the carbon nanotubes are aggregated into bundles. For example, in various embodiments, the exfoliated carbon nanotubes may be advantageously used in a wide range of applications including capacitors, batteries, photovoltaics, sensors, membranes, static dissipators, electromagnetic shields, video displays, pharmaceuticals and medical devices, polymer composites and gas storage vessels. In various embodiments, the exfoliated carbon nanotubes may also be used in fabrication and assembly techniques including, for example, ink-jet printing, spraying, coating, melt extruding, thermoforming, blow-molding and injection molding.

[0038] In various embodiments, the exfoliated carbon nanotubes may be single-wall carbon nanotubes, double-wall carbon nanotubes, multi-wall carbon nanotubes and various combinations thereof. In some embodiments, the carbon nanotubes are full-length carbon nanotubes. That is, in embodiments having full-length carbon nanotubes, the exfoliated carbon nanotubes are about the same length as the as-grown carbon nanotubes from which they are produced, and the carbon nanotube ends are generally closed in certain various embodiments. However, in other various embodiments, the carbon nanotubes are full-length carbon nanotubes that have open ends.

[0039] In some embodiments, the carbon nanotubes are substantially free of catalytic residues, non-nanotube carbon and various combinations thereof. In some embodiments, the carbon nanotubes are purified to remove catalytic residues and non-nanotube carbon. Such purification may take place either before or after the exfoliation of the carbon nanotubes takes place.

[0040] In some embodiments, the exfoliated carbon nanotubes are selectively precipitated by diameter. In various embodiments, exfoliated carbon nanotubes generally have a diameter of between about 0.7 nm and about 20 nm. Single-wall carbon nanotubes are generally about 0.7 nm to about 10 nm in diameter, whereas multi-wall nanotubes are generally greater than about 10 nm in diameter and up to about 100 nm in diameter in some embodiments. In some embodiments, the exfoliated carbon nanotubes have a diameter between about 1 nm and about 10 nm. In some embodiments, the exfoliated carbon nanotubes have a diameter between about 10 nm and about 100 nm.

[0041] In some embodiments, the exfoliated carbon nanotubes are selectively precipitated by length. The carbon nanotube length varies between about 500 nm and about 10 mm in some embodiments, between about 500 nm and 1 mm in some embodiments, between about 500 nm and 500 μ m in some embodiments, between about 500 nm and 1 μ m in some embodiments and various subranges thereof. In some embodiments, the exfoliated carbon nanotubes have an average length that is not substantially different than that of the bundled carbon nanotubes from which they are produced. That is, in some embodiments, the carbon nanotubes are full length carbon nanotubes that are not shortened during exfoliation. In some embodiments, the exfoliated carbon nanotubes are prepared from bundled carbon nanotubes, and the exfoliated carbon

nanotubes have a narrower distribution of lengths than do the bundled carbon nanotubes. That is, a subrange of exfoliated carbon nanotube lengths may be obtained from a population of bundled carbon nanotubes having a distribution of lengths.

[0042] The carbon nanotubes have a length to diameter ratio (aspect ratio) of least about 100 in some embodiments and at least about 1000 in other embodiments. In some embodiments, the exfoliated carbon nanotubes are prepared from bundled carbon nanotubes, and the exfoliated carbon nanotubes have a narrower distribution of diameters than do the bundled carbon nanotubes. That is, a subrange of exfoliated carbon nanotube diameters may be obtained from a population of bundled carbon nanotubes having a distribution of diameters.

[0043] In various embodiments, the exfoliated carbon nanotubes are further separated by chirality. For example, in the process of exfoliating bundled carbon nanotubes, exfoliated carbon nanotubes of a specific chirality or range of chiral forms may be produced. For example, in some embodiments, the exfoliated carbon nanotubes produced may be metallic, semi-metallic or semiconducting.

[0044] In some embodiments, the exfoliated carbon nanotubes are further functionalized. Functionalization may take place either before or after exfoliation. However, Applicants envision that functionalization after exfoliation may be advantageous to take advantage of the greater surface area available in the exfoliated carbon nanotubes compared to their bundled counterparts. In some embodiments, the exfoliated carbon nanotubes are functionalized to include an electroactive material bound to the carbon nanotubes, as set forth in more detail hereinabove.

[0045] Methods for exfoliating carbon nanotubes are also described herein. In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a first quantity of a nanocrystalline material, precipitating a first quantity of exfoliated carbon nanotubes from the solution and isolating the first quantity of exfoliated carbon nanotubes.

[0046] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing hydroxyapatite, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes.

[0047] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a nanorod material, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes.

[0048] In some embodiments of the methods, the carbon nanotubes may be further oriented in an alignment step after isolating the exfoliated carbon nanotubes. In some embodiments, the exfoliated carbon nanotubes may be shaped into a form such as, for example, a mat, film, fiber, cloth, non-woven fabric or felt.

[0049] An illustrative process for exfoliating carbon nanotubes follows. Carbon nanotubes can be effectively exfoliated using nanoplates of zirconium phosphate treated with a surfactant such as, for example, tetrabutylammonium hydroxide. The carbon nanotubes and the nanoplates are sonicated for short times to obtain full exfoliation of the carbon nanotubes in aqueous media. By controlling the ionic strength of the mixture after sonication, exfoliated carbon nanotubes can be obtained by simple separation techniques such as, for example, centrifugation. The carbon nanotubes after centrifuging and separating exist in a disordered but non-aggregated state and can easily be resuspended with other surfactant addition. Suitable surfactants for resuspension include, for example, both ionic and non-ionic surfactants, such as, for example, polyvinyl pyrrolidone, sodium dodecyl sulfate and PLURONICS. Cationic surfactants may be used for dispersion in non-polar media, such as chloroform and toluene. Application of an electric potential to the suspension may be used alternatively to or in combination with adjusting the ionic strength.

[0050] Although the above process may be used to cleanly separate single-wall carbon nanotubes, multi-wall carbon nanotubes and particularly oxidized multi-wall carbon nanotubes may not be separated as cleanly due to their broader range of ionic potentials. As a result, it is difficult to achieve separation of zirconium phosphate from the exfoliated carbon nanotubes when multi-wall carbon nanotubes are used. Furthermore, zirconium phosphate is particularly difficult to dissolve in acids (solubility = 0.12 mg/L in 6 M HCl), and it cannot typically be

removed by simple acid washing even after isolating the exfoliated carbon nanotubes. However, other various embodiments of the present disclosure are particularly applicable for exfoliating multi-wall carbon nanotubes.

[0051] In various embodiments, the methods for preparing exfoliated carbon nanotubes further include utilizing a solution that contains both a surfactant and a quantity of a nanocrystalline material. Surfactants are well known in the carbon nanotube art to aid in solubilization. Without being bound by theory or mechanism, Applicants believe that when a surfactant is used in preparing exfoliated carbon nanotubes, the surfactant may aid in the initial solubilization or suspension of the carbon nanotubes. Precipitation of exfoliated carbon nanotubes takes place thereafter. In various embodiments of the present disclosure, the surfactant may include, for example, sodium dodecyl sulfate, sodium dodecylbenzene sulfonate, or tetralkylammonium hydroxide (e.g., tetrabutylammonium hydroxide). In some embodiments, the surfactant may also modify the surface of the nanocrystalline material used for exfoliating the carbon nanotubes.

[0052] In general, exfoliated carbon nanotubes are prepared according to some embodiments of the present disclosure by precipitating exfoliated carbon nanotubes from a solution containing a nanocrystalline material. In some embodiments, the ionic strength of the solution is adjusted to induce precipitation of exfoliated carbon nanotubes. In some embodiments, the electrical potential of the solution is adjusted to induce precipitation of exfoliated carbon nanotubes. In some embodiments, the pH of the solution is adjusted to induce precipitation of exfoliated carbon nanotubes. In some embodiments, a combination of ionic strength, electrical potential and/or pH is adjusted to induce precipitation of exfoliated carbon nanotubes.

[0053] In some embodiments, the methods for exfoliating carbon nanotubes include adding a release species to the carbon nanotube suspension to adjust the ionic strength and precipitate exfoliated carbon nanotubes. In some embodiments, the ionic strength can be adjusted with an ionic species such as, for example, a solution of KCl. Although one of ordinary skill in the art will recognize the benefits of using an ionic species for adjustment of ionic strength, non-ionic species such as organic compounds may be used for ionic strength adjustment as well. Release species may be organic or inorganic compounds. In some embodiments, an electromagnetic field can be applied to the suspension of exfoliated carbon nanotubes in lieu of or in combination

with adjustment of the ionic strength with a release species to induce precipitation of the exfoliated carbon nanotubes.

[0054] After precipitation, exfoliated carbon nanotubes can be isolated by simple separation techniques such as, for example, centrifuging, filtering or settling. The separated, exfoliated carbon nanotubes exist in a disordered but non-aggregated state and can be easily redispersed in various media such as, for example, a liquid or polymer melt. In some embodiments, the redispersion may be aided by addition of a surfactant. Suitable surfactants include, but are not limited, to both ionic and non-ionic surfactants, sodium dodecyl sulfate, sodium dodecylbenzene sulfonate, and PLURONICS. Cationic surfactants are chiefly used for dispersion in non-polar media, such as, for example, chloroform and toluene. As noted above, other types of molecules such as, for example, cyclodextrins, polysaccharides, polypeptides, water soluble polymers, DNA, nucleic acids, polynucleotides, and polymers such as polyimides and polyvinyl pyrrolidone, can be used to redisperse the exfoliated carbon nanotubes in some embodiments.

[0055] In some embodiments, a second quantity of exfoliated carbon nanotubes may be precipitated from the suspension of carbon nanotubes. For example, in an embodiment, adding a second quantity of nanocrystalline material to the suspension results in precipitation of a second quantity of exfoliated carbon nanotubes. In some embodiments, the first quantity of carbon nanotubes and the second quantity of carbon nanotubes have different properties from one another such as, for example, different average lengths, diameters or chiralities. Repeated precipitation of carbon nanotube fractions may be repeated as many times as desired.

[0056] In some embodiments, the methods further include removing residual nanocrystalline material from the exfoliated carbon nanotubes. In some embodiments, the carbon nanotubes remain exfoliated after removing the nanocrystalline material. Hence, once the carbon nanotubes become fully exfoliated, they are no longer prone to becoming bundled. In some embodiments, the nanocrystalline material may be removed by washing the exfoliated carbon nanotubes. In some embodiments, the carbon nanotubes may be washed with an acid to remove the nanocrystalline material.

[0057] The redispersability of the carbon nanotubes after removal of the nanocrystalline material may be controlled by changing the surfactant concentration and the rate of addition of the release species. Hence, the redispersibility may be controlled by changing the rate of precipitation of exfoliated carbon nanotubes. In other words, in some embodiments the kinetic rate of carbon nanotube precipitation influences the rate of their redissolution following removal of the nanocrystalline material.

[0058] In general, the nanocrystalline materials of the present disclosure include any amorphous or crystalline material of nanometer-scale dimensions. In general, according to embodiments of the present disclosure, a nanocrystalline material is of nanometer-scale dimensions if it has at least one measured dimension that is less than about 1000 nm. In various embodiments of the present disclosure, carbon nanotubes are exfoliated from bundles of carbon nanotubes using a nanocrystalline material having a crystalline form such as, for example, nanorods, nanoplates, or nanowhiskers, to intersperse between individual carbon nanotubes. Nanorods include any inorganic or organic compound that may be induced to crystallize in a rod-like crystalline form. Nanowhiskers include any inorganic or organic compound that may be induced to crystallize in a whisker-like crystalline form. In various embodiments, the nanocrystalline material may include, for example, clays, graphite, inorganic crystalline materials, organic crystalline materials and various combinations thereof.

[0059] In some embodiments, the methods for preparing exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing hydroxyapatite, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes.

[0060] In various embodiments, the nanocrystalline material may be, for example, hydroxyapatite and hydroxyapatite derivatives. Hydroxyapatite derivatives include, for example, fluorapatite. In some embodiments, the hydroxyapatite has a crystalline form such as, for example, nanorods, nanoplates and nanowhiskers. In some embodiments, the methods further include removing the hydroxyapatite from the exfoliated carbon nanotubes. In some embodiments, removing can be accomplished, for example, through washing the exfoliated carbon nanotubes with an acid after their being isolated.

[0061] Various sizes of the nanocrystalline material may be used to exfoliate the carbon nanotubes. In some embodiments, the nanocrystalline material may be equal to or larger in size than the longest carbon nanotube present in the sample before exfoliation. In such embodiments, the exfoliated carbon nanotubes can be obtained in discrete fractions following addition of a release species such as, for example, KCl. In other embodiments, the nanocrystalline material has a size that is equal to or less than the longest carbon nanotube present in the sample before exfoliation. In this case, carbon nanotubes equal to or less than the size of the nanocrystalline material may be separated from the carbon nanotube suspension. In various embodiments, larger or smaller sizes of nanocrystalline material can be added to the carbon nanotube suspension to exfoliate carbon nanotube fractions having various carbon nanotube sizes.

[0062] In various embodiments, the exfoliated carbon nanotubes are further purified to remove impurities such as, for example, residual metal catalyst and non-nanotube carbon residue. With exfoliated carbon nanotubes, further purification is more easily conducted than like purifications conducted on bundled carbon nanotubes due to the comparatively greater surface area present in the exfoliated carbon nanotubes. Purification techniques include conventional techniques such as, for example, oxidation at elevated temperature (e.g., about 200°C to about 400°C) or acid extraction to remove metallic impurities. Illustrative acids that may be used to extract metallic impurities from the exfoliated carbon nanotubes include, for example, various concentrations of hydrochloric, hydrobromic, nitric, chlorosulfonic and phosphoric acids and various combinations thereof. In general, the acid and impurities are removed from the exfoliated carbon nanotubes by rinsing with water, organic solvents or combinations thereof. In some embodiments, supercritical fluids such as, for example, highly compressed CO₂ or hydrocarbons such as, for example, propane or butane, may also be employed to remove impurities from the exfoliated carbon nanotubes.

[0063] In various embodiments, the methods for producing exfoliated carbon nanotubes further include derivatization of the exfoliated carbon nanotubes with at least one functional group. Derivatization may occur either before or after exfoliation has occurred. Numerous methods to derivatize carbon nanotubes are known to those of ordinary skill in the art. For example, diazonium chemistry can be utilized to introduce alkyl or aryl groups, either of which may bear further functionalization, on to the carbon nanotubes. In additional embodiments, treating

nanotubes with lithium in liquid ammonia, followed by reaction with an alkyl halide may be used to alkylate carbon nanotubes. Reaction of fluorinated carbon nanotubes with ammonia or amines in the presence of a catalyst such as, for example, pyridine, may be used to functionalize the nanotubes through amine-bearing functionalities. Likewise, fluorinated carbon nanotubes may be functionalized with hydroxyl-containing moieties, which may be functionalized to bear an ether linkage OR, wherein R may be any combination of alkyl, aryl, acyl, and arylacyl groups. Furthermore, R may be further functionalized, for example, with halogens, thiols, amino groups and other common organic functionalities. In addition, the carbon nanotubes may be directly functionalized with thiols, alkyl substituted thiols, aryl substituted thiols, and halogens.

[0064] In some embodiments, the first quantity or second quantity of exfoliated carbon nanotubes are selectively precipitated by a physical property such as, for example, chirality, diameter or length. In various embodiments, carbon nanotubes are exfoliated using a nanocrystalline material in the form of nanoplates and then further separated by chirality, nanotube length, or nanotube diameter. In various embodiments, carbon nanotubes are exfoliated using a nanocrystalline material in the form of nanorods and then further separated by chirality, nanotube length, or nanotube diameter. In various embodiments, carbon nanotubes are exfoliated using a nanocrystalline material in the form of nanowhiskers and then further separated by chirality, nanotube length, or nanotube diameter. Regardless of how the exfoliated carbon nanotubes are prepared, separation by chirality, length or diameter may be more facile in some instances after the carbon nanotubes are exfoliated.

[0065] In some embodiments, a direct separation of carbon nanotubes by chirality, length or diameter may be accomplished by choice of the nanocrystalline material in combination with additional agents. For example, using a nanocrystalline material either alone or in combination with chiral surfactants and/or polymers may allow exfoliated carbon nanotubes to be separated based on length, diameter, chirality, type, and functionality such as, for example, oxidation state and/or defect structure.

[0066] In some embodiments, the suspension of carbon nanotubes further includes a chiral agent, resulting in selective precipitation of exfoliated carbon nanotubes by chirality. Chiral agents include, for example, surfactants, polymers and combinations thereof. Chiral agents include

molecules such as, for example, R,R-tartaric acid, which has been useful for separation of enantiomeric drugs in electrokinetic chromatography, and enantiomers of polylactic acid. Other molecules which are conventionally used in chiral separations also lie within the spirit and scope of the present disclosure. In some embodiments, the chiral agents may be used to separate exfoliated carbon nanotubes of a single chirality or a limited number of chiral configurations from a mixture of carbon nanotubes containing a range of carbon nanotube chiralities. In some embodiments, the chiral agent may be a surfactant that both helps disperse the carbon nanotubes and facilitates the chiral separation. The chiral agent may be associated with or chemically bound to the carbon nanotube surface. In some embodiments, carbon nanotubes separated by chirality also are separated by electronic type (i.e., metallic, semi-metallic and semiconducting).

[0067] By using polymers and/or surfactants having a defined chirality, separated populations of exfoliated metallic, semi-metallic, or semi-conducting carbon nanotubes can be obtained in some embodiments of the present disclosure. Without being bound by mechanism or theory, Applicants believe that polymers and/or surfactants of defined chirality preferentially wrap a carbon nanotube of a complementary chirality type. By selective carbon nanotube precipitation as described hereinabove, carbon nanotubes may be selectively separated by chirality. Selective carbon nanotube precipitation may occur either in the presence or absence of a nanocrystalline material. In some embodiments, separation techniques such as, for example, solvent/non-solvent addition, co-surfactant addition, and differential temperature gradients may be used to selectively precipitate a chiral population of carbon nanotubes.

[0068] In various embodiments, chiral polymers and/or surfactants may be mixtures of tactic molecules. By using tactic polymers with a low thermal degradation temperature, isolated, exfoliated carbon nanotubes can be easily recovered by thermal degradation of the polymer. Illustrative tactic polymers include, for example, atactic polystyrene, isotactic polystyrene, syndiotactic polystyrene, d and l polylactic acid, d and l polypropylene carbonate and the like. For example, polypropylene carbonate can be thermally degraded at less than about 300°C without damaging carbon nanotubes. In further embodiments, the tactic molecules may be a mixture dissolved in a hydrocarbon solvent such as, for example, toluene or decalin. In still further embodiments, the carbon nanotubes in the polymers can be oriented or aligned by various methods known to those of ordinary skill in the art.

[0069] The technique of separating carbon nanotubes by chirality by using a chiral polymer may be further extended to a chromatography column for continuous separation. For example, carbon nanotubes wrapped in a chiral polymer may be applied to a chromatography column and then be separated by chirality. Alternatively, a suspension of exfoliated carbon nanotubes lacking a chiral agent may be applied to a chromatography column having a chiral stationary phase. In these alternative embodiments, separation by chirality is based on a selective interaction of the chiral stationary phase with the various carbon nanotube chiralities.

[0070] In still further embodiments, exfoliated carbon nanotubes either with or without a wrapping of chiral polymers and/or surfactants may be separated by electronic type by applying an electric potential to a solution of exfoliated carbon nanotubes. For example, exfoliated metallic carbon nanotubes will migrate toward the potential for collection and separation.

[0071] In some embodiments of the present disclosure, alternative methods for producing exfoliated carbon nanotubes not utilizing a nanocrystalline material are disclosed. In some embodiments, the methods for producing exfoliated carbon nanotubes include preparing a solution of carbon nanotubes in an acid and filtering the solution through a filter to collect exfoliated carbon nanotubes on the filter. In some embodiments, the acid is sulfuric acid. In some embodiments, the acid is a mixture of nitric acid and sulfuric acid. In some embodiments, the acid is a superacid. In some embodiments, the superacid is chlorosulfonic acid.

[0072] In general, the acid solutions used in preparing dispersed carbon nanotubes have a concentration of carbon nanotubes that is below the percolation threshold of carbon nanotubes in the acid. Filtration of such acid solutions of exfoliated carbon nanotubes produces a mat of exfoliated carbon nanotubes on the filter in various embodiments of the present disclosure. Although some acids and superacids are known to dissolve and exfoliate bundles of carbon nanotubes in solution, particularly when a liquid crystalline phase is formed, Applicants believe that there has been no recognition in the art the exfoliation can be maintained upon removal of the acid solvent. The mat of exfoliated carbon nanotubes may be further modified on the filter in some embodiments of the present disclosure. For example, in some embodiments, the mat of exfoliated carbon nanotubes may be modified by functionalizing or treating with a surfactant to maintain the carbon nanotubes in an exfoliated state. The exfoliated carbon nanotubes may be

subsequently removed from the filter. In addition, the exfoliated carbon nanotubes may be further processed according to any of the methods described hereinabove.

[0073] The exfoliated carbon nanotubes prepared by the techniques described hereinabove are typically longer than are carbon nanotubes exfoliated using existing technology. For instance, as described previously, other separation techniques often result in carbon nanotube damage and shortened carbon nanotube lengths. In certain applications, particularly those involving electrical conduction or mechanical reinforcement, shorter carbon nanotubes may not provide adequate electrical conductivity or structural integrity. For example, by having at least a portion of longer carbon nanotubes present with electrical devices such as energy storage device, a higher degree of electrical connectivity at a fixed carbon nanotube volume fraction can be achieved. Furthermore, longer carbon nanotube lengths may increase the toughness of the polymer composites over those made with shorter carbon nanotubes.

[0074] The present disclosure also relates to improved energy storage devices containing exfoliated carbon nanotubes and particularly to capacitors, supercapacitors, ultracapacitors and batteries having components containing exfoliated carbon nanotubes. The improved energy storage devices include components such as, for example, current collectors, electrodes, insulators, electrolytes and separators, each capable of containing exfoliated carbon nanotubes. In some embodiments, the improved energy storage devices have at least one of at least two electrodes containing exfoliated carbon nanotubes. The improved energy storage devices also include a dielectric medium or electrolyte, each optionally including carbon nanotubes. The improved energy storage devices have a high energy density and power density.

[0075] FIGURE 1 shows an illustrative arrangement of the basic elements of a Faradaic capacitor. As shown in FIGURE 1, current collectors **1** and **5** contact with electrodes **2** and **4**, which are separated by dielectric **3**. In an embodiment of the present disclosure, at least one of the electrodes **2** and **4** contains exfoliated carbon nanotubes. In various embodiments, current collectors **1** and **5** can be metals such as, for example, copper and other highly conductive metals. In some embodiments, the current collectors can contain conductive exfoliated carbon nanotubes. In an embodiment, the carbon nanotubes may be full length exfoliated carbon nanotubes. In some embodiments, the carbon nanotubes may be separated metallic carbon

nanotubes. In various embodiments, at least one of electrodes **2** and **4** contains exfoliated carbon nanotubes.

[0076] FIGURE 2 shows an illustrative arrangement of the basic elements of an electric double layer capacitor. As shown in FIGURE 2, current collectors **11** and **17** contact electrodes **12** and **16**, and electrolytes **13** and **15** contact electrodes **12** and **16**. Non-conducting separator **14** separates electrolytes **13** and **15** and is permeable to ions flowing between the electrodes **12** and **16**. In some embodiments, current collectors **11** and **17** can be metals such as, for example, copper and like conductive metals. In some embodiments, current collectors **11** and **17** contain exfoliated carbon nanotubes. In some embodiments, the carbon nanotubes may be separated metallic carbon nanotubes. In various embodiments, electrolytes **13** and **15** can contain exfoliated carbon nanotubes, which may be exfoliated conductive carbon nanotubes in some embodiments. In various embodiments, at least one of electrodes **12** and **16** contains exfoliated carbon nanotubes. Electrolytes **13** and **15** may be fully intermixed with the electrodes **12** and **16**, or they may contact along a portion of the electrodes. For example electrolytes **13** and **15** may contact along a single side of electrodes **12** and **16** along a plane. In various embodiments, non-conducting separator **4** may contain non-conducting carbon nanotubes. In various embodiments, the non-conducting separator **4** may be made from porous polyethylene or fiberglass mats.

[0077] FIGURE 3 shows an illustrative arrangement of the basic elements of a battery. As shown in FIGURE 3, electrodes **21** and **23** contact electrolyte **22**. The electrolyte **22** conveys ions between electrodes **21** and **23**. In an embodiment, the ions are metal ions such as, for example, lithium ions. Hence, the present disclosure describes a lithium battery containing exfoliated carbon nanotubes. In some embodiments, at least one of the electrodes **21** and **23** contains exfoliated carbon nanotubes. In some embodiments, the electrolyte **22** contains exfoliated carbon nanotubes.

[0078] In various embodiments of the present disclosure, the energy storage device containing exfoliated carbon nanotubes is a battery containing at least two electrodes and an electrolyte in contact with the at least two electrodes. At least one of the electrodes contains exfoliated carbon nanotubes.

[0079] In some embodiments of the energy storage devices, the exfoliated carbon nanotubes are multi-wall carbon nanotubes. In some embodiments, the exfoliated carbon nanotubes are single-wall carbon nanotubes. In some embodiments, the at least one electrode containing exfoliated carbon nanotubes is the anode.

[0080] In various embodiments of the energy storage devices, the electrode may contain exfoliated carbon nanotubes dispersed in a polymer or viscous liquid. In various embodiments, the electrode may be laminated to another medium such as, for example, a dielectric or an electrolyte.

[0081] In various embodiments, the electrolyte of the energy storage devices can be a solid or a fluid. Electrolytes are generally chosen to minimize internal electrical resistance. Aqueous electrolytes such as potassium hydroxide or sulfuric acid are generally employed in conventional batteries and capacitors. Due to water's low electrochemical decomposition potential of 1.24 volts, the energy density is limited with these types of electrolytes. Organic electrolytes such as, for example, organic carbonates and tetraalkylammonium salts provide good solubility and reasonable conductivity. In general, organic electrolytes have lower conductivity than aqueous electrolytes, but they can operate at higher voltages, for example, up to about 5 volts. Other electrolytes can be of a polymer-gel type such as, for example, polyurethane–lithium perchlorate, polyvinyl alcohol-KOH-H₂O and the related systems. Organic electrolytes such as, for example tetraethylammonium tetrafluoroborate and tetrabutylammonium tetrafluoroborate, can simultaneously serve as an electrolyte and surfactant for dispersing and exfoliating carbon nanotubes in embodiments where carbon nanotubes are contained in the electrolyte. Electrolyte salts may also be used for dispersing the carbon nanotubes or maintaining exfoliated carbon nanotubes in an exfoliated state.

[0082] In some embodiments of the energy storage devices, the exfoliated carbon nanotubes are modified with an electroactive material. In some embodiments, the electroactive material is a transition metal or transition metal oxide. Electroactive transition metals include, for example, Ru, Ir, W, Mo, Mn, Ni, and Co. In some embodiments, the electroactive material may be a conducting polymers such as, for example, polyaniline, polyacetylene and polyvinylpyrrole. In some embodiments, the electroactive material is a nanomaterial bound to the exfoliated carbon

nanotubes. In some embodiments, the nanomaterial may be, for example, SnO_2 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$, silicon nanotubes, silicon nanoparticles and various combinations thereof.

[0083] In other various embodiments, the present disclosure describes layered structures containing exfoliated carbon nanotubes suitable for use in energy storage devices. For example, co-extrusion of liquids or melts containing exfoliated carbon nanotubes through multilayer dies or multilayer generators may be used in making the energy storage devices of the present disclosure. The resultant layered structures can be stacked and connected in series to give higher voltages in energy storage devices. In other embodiments, the components of the energy storage devices may be processed from a solution of exfoliated carbon nanotubes by solvent casting, spraying, paste spreading, compression stretching, or combinations thereof.

[0084] In some embodiments, the present disclosure also relates to an ion diffusion separator of electrical double-wall capacitors. In various embodiments, the separator contains non-metallic single-wall carbon nanotubes.

[0085] In some embodiments of the present disclosure, insulators of the energy storage devices contain non-metallic single-wall carbon nanotubes. In embodiments where the insulator contains carbon nanotubes, the dielectric constant of the insulator/carbon nanotube mixture may be greater than that of the insulator alone.

[0086] In various embodiments, exfoliated carbon nanotubes can be aligned in forming electrodes for use in the energy storage devices. In some embodiments, the alignment may occur through melt extrusion.

[0087] In some embodiments of the energy storage devices of the present disclosure, incorporation of exfoliated carbon nanotubes to electrodes, electrolytes or dielectrics of the energy storage devices provides enhanced strength and ruggedness to the device. These features allow further shaping of the device for functioning under demanding environments, such as high vibration or extreme thermal cycling environments.

[0088] In still additional embodiments of the present disclosure, polymer composites containing exfoliated carbon nanotubes and methods for making such polymer composites are described

herein. Polymer composites of the present disclosure are advantageous over those conventionally prepared in the art by having fully exfoliated carbon nanotubes dispersed in the polymer matrix. As such, the polymer composites of the present disclosure are advantageous in having all of the carbon nanotube surface area being available for load transfer when the composite is placed under stress. Further, the exfoliated state of the carbon nanotubes allows the carbon nanotubes to be easily mixed into the polymer matrix in a non-exfoliated state, as opposed to high shear mixing techniques which are used conventionally to disperse carbon nanotubes in polymer composites.

[0089] In some embodiments, methods for making polymer composites according to the present disclosure include providing exfoliated carbon nanotubes and mixing the exfoliated carbon nanotubes in a polymer material to form a polymer composite. The carbon nanotubes remain in an exfoliated state after being mixed in the polymer material.

[0090] In some embodiments of the methods for making polymer composites, the polymer material is an epoxy. In some embodiments, the methods further include curing the epoxy. In some embodiments, the polymer material is a monomer of a thermoplastic material, which is subsequently polymerized. In some embodiments, the polymer material is a polymer melt, which is hardened after mixing the exfoliated carbon nanotubes.

Experimental Examples

[0091] The following experimental examples are included to demonstrate particular aspects of the present disclosure. It should be appreciated by those of ordinary skill in the art that the methods described in the examples that follow merely represent illustrative embodiments of the disclosure. Those of ordinary skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments described and still obtain a like or similar result without departing from the spirit and scope of the present disclosure.

[0092] Example 1: Exfoliation of Carbon Nanotubes Using $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$ Nanoplates and Tetrabutylammonium Hydroxide Surfactant. A dispersed solution of carbon nanotubes was prepared from 10 mg of multi-wall carbon nanotubes placed in 2 mL of a solution of $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$ nanoplates and tetrabutylammonium hydroxide (5 wt% $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$; 1:0.8

ratio of $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$:tetrabutylammonium hydroxide). The solution was subsequently diluted to 30 mL and then sonicated for 2 hours. The solution was stable for at least 24 hours. An aliquot of 0.01 M KCl was added, resulting in precipitation of a quantity of exfoliated multi-wall carbon nanotubes. The precipitated fraction was removed by centrifugation. The quantity of isolated nanotubes was approximately 1/10 the mass of carbon nanotubes originally suspended. The filtrate was treated with another aliquot of 0.01 M KCl, resulting in a second precipitation of multi-wall carbon nanotubes. The precipitation/centrifugation process was repeated until substantially all nanotubes had been precipitated from the suspension.

[0093] Example 2: Exfoliation of Carbon Nanotubes Using $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$ Nanoplates of Varying Sizes (Prophetic Example). The experimental procedure described in Example 1 hereinabove will be repeated, except with a nanoplate size of about 1/10 the length of the longest carbon nanotube present in the sample. After removal of the first precipitation fraction following addition of 0.01 M KCl, a second quantity of nanoplates of a different (larger) size will be added. The second quantity of nanoplates will fractionate a second quantity of nanotubes following addition of 0.01 M KCl. The second precipitation fraction of nanotubes may have a different length distribution than did the first precipitation fraction. The precipitation/centrifugation process will be repeated with progressively larger nanoplates until substantially all carbon nanotubes have been precipitated from the suspension.

[0094] Example 3: Synthesis of Hydroxyapatite Nanoplates. Hydroxyapatite nanoplates of controlled sizes were synthesized by dissolving 10 g of hydroxyapatite (Sigma Aldridge reagent grade) in 400 mL of dilute nitric acid ($\text{pH} = 2$) at room temperature, followed by very slow dropwise addition of 48 mL of 1% v/v ammonium hydroxide. Crystals collected at $\text{pH} = 4$ and $\text{pH} = 5$ were found by microscopy to be plates having an aspect ratio about 7 to 8 and a diameter ranging between 3 – 15 μm . FIGURE 4 shows an illustrative electron micrograph of hydroxyapatite plates of 3 – 15 μm diameter. Increasing the addition rate of the 1% v/v ammonium hydroxide reduced the average plate size.

[0095] Example 4: Synthesis of Hydroxyapatite Nanorods. 2 g of hydroxyapatite was first dissolved in 40 mL of dilute nitric acid ($\text{pH} = 2$) containing a 3:1 ethanol:water ratio. The mixture was then quenched into 80 mL of 5 vol% ammonium hydroxide, also in a 3:1

ethanol:water ratio. The resultant pH was 8.5. A milky, jelly-like precipitate resulted. The resulting mixture containing the precipitate was then heated at between 70°C and 80°C on a magnetic stirrer hotplate for 24 hours. Thereafter, hydroxyapatite crystals were filtered, washed with deionized water and dried. Electron microscopy showed that hydroxyapatite nanorods having an aspect ratio of about 25 and lengths between 100 – 200 nm were formed. FIGURE 5 shows an illustrative electron micrograph of hydroxyapatite nanorods having lengths of 100 – 200 nm.

[0096] Example 5: Exfoliation of Carbon Nanotubes Using Hydroxyapatite. 0.5142 g hydroxyapatite nanorods were added to 50 mL water and 0.8280 g tetrabutylammonium hydroxide (Sigma Aldrich reagent grade; TBAH; 1:1 molar ratio of hydroxyapatite:TBAH). The resultant mixture was sonicated for one hour at 25°C and then diluted with deionized water to give a 0.2 wt% solution based on hydroxyapatite content.

[0097] Multi-wall carbon nanotubes were received as a powder that contained highly entangled nanotube bundles having a grain size of 1 – 10 µm in diameter. The lengths of the individual multi-wall carbon nanotubes were found to be in excess of 1 µm, and the diameters were found to be 10 – 20 nm. 1 g of the multi-wall carbon nanotubes was added to 50 mL of a mixture of concentrated sulfuric and nitric acid in a 3:1 volume ratio. The mixture was placed in a sonicator bath (Branson sonicator, model 250) and treated for two hours while sonicating at temperature of 25 – 35°C. The mixture was then filtered using a polyvinylidene fluoride microporous filter (5 µm pore size), followed by washing of the resultant solid with deionized water until the pH of the filtrate was 4.5. The multi-wall carbon nanotubes were then dried in vacuo for 2 hours at 80°C. The multi-wall carbon nanotubes were not substantially shortened by the acid treatment.

[0098] Samples were prepared by adding the dried multi-wall carbon nanotubes to the hydroxyapatite/TBAH solution prepared above to give carbon nanotube:hydroxyapatite weight ratios of 1:1, 1:2, 1:3, 1:4 and 1:5. The mixture was sonicated at room temperature for 2 hours and then left for 24 hours. At a weight ratio of 1:1, a portion of the multi-wall carbon nanotubes settled out as agglomerated particles. At a 1:2 weight ratio the solution had a few multi-wall carbon nanotube particles present after 24 hours. All higher weight ratios examined gave stable dispersions for at least 24 hrs. A control experiment at a weight ratio of 1:3 multi-wall carbon

nanotubes: TBAH with no hydroxyapatite present showed mostly aggregated carbon nanotubes settling after 24 hours. FIGURE 6A shows an illustrative electron micrograph of as-received multi-wall carbon nanotubes, and FIGURE 6B shows an illustrative electron micrograph of multi-wall carbon nanotubes exfoliated using hydroxyapatite nanorods.

[0099] The precipitated exfoliated multi-wall carbon nanotubes contained residual hydroxyapatite as evidenced by energy-dispersive X-ray (EDX) spectroscopy. FIGURE 7A shows an illustrative EDX spectrum of precipitated exfoliated multi-wall carbon nanotubes. As shown in the EDX spectrum, strong Ca and P signals indicated the presence of hydroxyapatite. The precipitated multi-wall carbon nanotubes were subsequently washed with 50 mL of 1 N nitric acid, followed by 250 mL of deionized water, which removed substantially all the hydroxyapatite as evidenced by EDX. FIGURE 7B shows an illustrative EDX spectrum of precipitated exfoliated multi-wall carbon nanotubes after acid washing. In contrast, the exfoliated multi-wall carbon nanotubes of Example 1 contained residual $\text{Zr}(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$, which could not be removed by washing with acids such as nitric, hydrochloric or sulfuric acids.

[0100] Unentangled multi-wall carbon nanotubes were obtained after exfoliation, precipitation and washing. FIGURE 8 shows an illustrative electron micrograph of the exfoliated multi-wall carbon nanotubes after precipitation and washing. Exfoliation of the multi-wall carbon nanotubes could be conducted equivalently using hydroxyapatite plates.

[0101] Example 6: Exfoliation of Carbon Nanotubes Using Concentrated Acid Solutions. 40 mg of multi-wall carbon nanotubes were added to 40 mL of a 3:1 sulfuric:nitric acid mixture and sonicated for 60 minutes at 25 °C. A drop of the mixture was placed on a PVDF filter and allowed to dry. FIGURE 9 shows an illustrative electron micrograph of exfoliated carbon nanotubes obtained from 3:1 $\text{H}_2\text{SO}_4:\text{HNO}_3$. As shown in FIGURE 9, exfoliation was maintained after removal of the acid by drying.

[0102] Example 7: Exfoliation of Carbon Nanotubes Using Concentrated Acid Solutions, Followed by Surfactant Addition. A 1% by weight double-wall carbon nanotube solution in 3:1 sulfuric:nitric acid was treated for 2 hours as described previously. After filtering the concentrated acid solution to immobilize the double wall carbon nanotubes, the immobilized carbon nanotubes were washed with deionized water until the washings were pH = 4.5. While

still wet, the PVDF filter paper and the double-wall carbon nanotubes were sonicated for 30 minutes with a 0.2% by weight sodium dodecyl sulfate (SDS) solution in deionized water such that the weight of double-wall carbon nanotubes to SDS was 1:3. The mixture was stable for at least 24 hours. A drop of the mixture was placed on a carbon tape and dried for examination by electron microscopy, which showed exfoliated carbon nanotubes. FIGURE 10 shows an illustrative electron micrograph of exfoliated double-wall carbon nanotubes following acid exfoliation and treatment with sodium dodecyl sulfate.

[0103] Example 8: Epoxy Composite Containing Exfoliated Carbon Nanotubes. 5 mg of acid-treated multi-wall carbon nanotubes were placed in 10 mL of tetraethylenetetramine (TETA), and various additions of sodium dodecylsulfate (SDS) were added such that the weight ratio of multi-wall carbon nanotubes to SDS was 5, 2.5, 1, and 0.33 to 1. The mixture was sonicated at 30°C for 30 minutes and allowed to stand. After 7 days the 1:1 and 1:0.33 ratio was seen to be stable toward precipitation.

[0104] 49 g of Bisphenol F epoxy was admixed with 0.242 g of acid-treated multi-wall carbon nanotubes and sonicated for 10 minutes at 60°C. The mixture was cooled to 25°C and then degassed for 10 minutes at 25 inches Hg. 7 g of TETA containing 0.5% wt treated multi-wall carbon nanotubes and 0.5% wt. SDS was sonicated and degassed separately as above. The two degassed mixtures were then carefully mixed and poured into a mold. The mold was cured for 2 hours at 100°C. Controls were prepared as above without carbon nanotubes (control 1) and with as-received multi-wall carbon nanotubes (control 2).

[0105] Table 1 shows the mechanical strength improvement in the epoxy composite containing exfoliated multi-wall carbon nanotubes. K_q is the maximum stress before failure on tensile testing of a notched specimen at 0.01/min initial strain rate and 1 mm razor notch. Relative fatigue lifetime improvement is the lifetime of the notched specimen counted as the number of cycles to failure at 1 Hz, at about 16.7 MPa maximum tensile stress with stress amplitude of 0.1 (stress minimum/stress maximum). As shown in Table 1, significant mechanical property enhancement was observed when exfoliated carbon nanotubes were used.

Table 1: Mechanical Properties of Carbon Nanotube Composites

Material	Relative K _q improvement	Relative fatigue lifetime improvement
Control 1	1	1
Control 2	1.2	1.1
Exfoliated Carbon Nanotubes	1.5	4.7

[0106] Example 8: Capacitor Containing Exfoliated Multi-Wall Carbon Nanotubes.

Control 1: 10 g of poly(ethylene oxide) (PEO; 1500 molecular weight) was melted, and 1 mL of 4 N potassium hydroxide added to make the electrolyte. 1 wt % of as-received multi-wall carbon nanotubes were added to the electrolyte mixture and sonicated for 15 minutes in a sonicator bath. Approximately 2.1 g of the mixture was poured into one part of a polystyrene petri dish 6 cm in diameter with a strip of copper adhered as the current collector. A piece of clean writing paper was then placed on the molten liquid electrolyte, and 2 g of the electrolyte was poured on to the paper, taking care not to weep at the edges. The other side of the petri dish with a copper strip adhered was then inserted to make a capacitor. After cooling to room temperature for 15 minutes the capacitance was measured using an HP 4282A capacitance meter. The measured capacitance was 0.0645 microfarads. Control 2: Control 2 was prepared as for control 1, except as-received graphene (Rice University) was substituted for the multi-wall carbon nanotubes. The measured capacitance was 0.176 microfarads. Exfoliated carbon nanotube capacitor: The capacitor was prepared as for Control 1, except exfoliated multi-wall carbon nanotubes were used in place of as-received multi-wall carbon nanotubes. The measured capacitance was 0.904 microfarads, a 14-fold improvement over control 1 and a 5.1-fold improvement over control 2.

[0107] Example 9: Exfoliated Carbon Nanotubes Decorated with Copper Nanoparticles.

102 mg of exfoliated multi-wall carbon nanotubes were added to 100 mg copper sulfate, 640 mg sodium EDTA, 15 mg of polyethylene glycol, 568 mg of sodium sulfate and 60 mL of deionized water. The mixture was sonicated for 10 minutes and then heated to 40°C. 3 mL of formaldehyde (37% solution) and 500 mg of sodium hydroxide were added to bring the pH to 12.2. The mixture was stirred for 30 minutes at 85°C and then filtered using a 5 micron PVDF filter and washed with 200 mL of deionized water. FIGURE 11 shows an illustrative electron

micrograph of exfoliated carbon nanotubes decorated with copper oxide nanoparticles obtained from the mixture.

[0108] From the foregoing description, one of ordinary skill in the art can easily ascertain the essential characteristics of this disclosure, and without departing from the spirit and scope thereof, can make various changes and modifications to adapt the disclosure to various usages and conditions. The embodiments described hereinabove are meant to be illustrative only and should not be taken as limiting of the scope of the disclosure, which is defined in the following claims.

CLAIMS

What is claimed is the following:

1. A composition comprising exfoliated carbon nanotubes,
wherein the exfoliated carbon nanotubes are not dispersed in a continuous matrix that maintains the carbon nanotubes in an exfoliated state.
2. The composition of claim 1, wherein the composition comprises a mat of exfoliated carbon nanotubes.
3. The composition of claims 1 or 2, wherein the carbon nanotubes are selected from the group consisting of single-wall carbon nanotubes, double-wall carbon nanotubes, multi-wall carbon nanotubes and combinations thereof.
4. The composition of any one of claims 1 – 3, wherein the carbon nanotubes are full-length carbon nanotubes.
5. The composition of any one of claims 1 – 4, wherein the carbon nanotubes are substantially free of impurities selected from the group consisting of catalytic residues, non-nanotube carbon and combinations thereof.
6. The composition of any one of claims 1 – 5, wherein the exfoliated carbon nanotubes are separated by chirality.
7. The composition of any one of claims 1 – 6, wherein the exfoliated carbon nanotubes are prepared from bundled carbon nanotubes;
wherein the exfoliated carbon nanotubes have a narrower distribution of lengths than do the bundled carbon nanotubes.
8. The composition of any one of claims 1 – 7, wherein the exfoliated carbon nanotubes are prepared from bundled carbon nanotubes;
wherein the exfoliated carbon nanotubes have a narrower distribution of diameters than do the bundled carbon nanotubes.

9. The composition of any one of claims 1 – 8, wherein the exfoliated carbon nanotubes are further functionalized.
10. The composition of any one of claims 1 – 9, wherein the exfoliated carbon nanotubes further comprise an electroactive material bound to the carbon nanotubes.
11. A method for preparing exfoliated carbon nanotubes, said method comprising:
 - a) suspending carbon nanotubes in a solution comprising a first quantity of a nanocrystalline material;
 - b) precipitating a first quantity of exfoliated carbon nanotubes from the solution;and
 - c) isolating the first quantity of exfoliated carbon nanotubes.
12. The method of claim 11, wherein the solution further comprises a surfactant.
13. The method of claims 11 or 12, wherein precipitating further comprises adjusting the ionic strength of the solution.
14. The method of any one of claims 11 – 13, wherein precipitating further comprises applying an electrical potential to the solution.
15. The method of any one of claims 11 – 14, wherein precipitating further comprises adjusting the pH of the solution.
16. The method of any one of claims 11 – 15, wherein the solution further comprises a chiral agent; and
wherein the first quantity of exfoliated carbon nanotubes is selectively precipitated by chirality.
17. The method of claim 16, wherein the chiral agent is selected from the group consisting of a surfactant, a polymer and combinations thereof.
18. The method of any one of claims 11 – 17, wherein the exfoliated carbon nanotubes are selectively precipitated by length.

19. The method of any one of claims 11 – 17, wherein the exfoliated carbon nanotubes are selectively precipitated by diameter.
20. The method of any one of claims 11 – 15, further comprising:
 - adding a second quantity of nanocrystalline material to the solution; and
 - precipitating a second quantity of exfoliated carbon nanotubes.
21. The method of claim 20, wherein the first quantity of exfoliated carbon nanotubes and the second quantity of exfoliated carbon nanotubes have different properties from one another.
22. The method of any one of claims 11 – 21, wherein the nanocrystalline material has a crystalline form selected from the group consisting of nanoplates, nanorods and nanowhiskers.
23. The method of any one of claims 11 – 22, wherein the nanocrystalline material is selected from the group consisting of clays, graphite, inorganic crystalline materials, organic crystalline materials, and combinations thereof.
24. The method of any one of claims 11 – 23, wherein the nanocrystalline material is selected from the group consisting of hydroxyapatite and hydroxyapatite derivatives.
25. A method for preparing exfoliated carbon nanotubes, said method comprising:
 - a) suspending carbon nanotubes in a solution comprising hydroxyapatite;
 - b) precipitating exfoliated carbon nanotubes from the solution; and
 - c) isolating the exfoliated carbon nanotubes.
26. The method of claim 25, wherein the hydroxyapatite has a crystalline form selected from the group consisting of nanorods, nanoplates and nanowhiskers.
27. The method of claims 25 or 26, further comprising:
 - removing residual hydroxyapatite from the exfoliated carbon nanotubes.
28. The method of claim 27, wherein removing comprises washing the exfoliated carbon nanotubes with an acid after their being isolated.

29. A method for preparing exfoliated carbon nanotubes, said method comprising:
 - a) suspending carbon nanotubes in a solution comprising a nanorod material;
 - b) precipitating exfoliated carbon nanotubes from the solution; and
 - c) isolating the exfoliated carbon nanotubes.
30. A method for preparing exfoliated carbon nanotubes, said method comprising:
 - a) preparing a solution of carbon nanotubes in an acid; and
 - b) filtering the solution through a filter to collect exfoliated carbon nanotubes on the filter.
31. The method of claim 30, wherein a concentration of carbon nanotubes in the acid is below a percolation threshold of the carbon nanotubes in the acid.
32. The method of claims 30 or 31, wherein the acid comprises a superacid.
33. The method of claim 32, wherein the superacid comprises chlorosulfonic acid.
34. The method of any one of claims 30 – 33, further comprising:
 - c) modifying the exfoliated carbon nanotubes on the filter.
35. The method of claim 34, wherein modifying comprises functionalizing the exfoliated carbon nanotubes.
36. The method of claim 34, wherein modifying comprises treating the exfoliated carbon nanotubes with a surfactant.
37. An energy storage device comprising:
 - a) at least two electrodes;
wherein at least one of the electrodes comprises the exfoliated carbon nanotubes of claim 1; and
 - b) an electrolyte in contact with the at least two electrodes.
38. The energy storage device of claim 37, wherein the exfoliated carbon nanotubes are modified with an electroactive material.

39. The energy storage device of claim 38, wherein the electroactive material comprises a transition metal.
40. The energy storage device of claim 38, wherein the electroactive material comprises a nanomaterial bound to the exfoliated carbon nanotubes.
41. The energy storage device of claim 40, wherein the nanomaterial is selected from the group consisting of SnO_2 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$, silicon nanotubes, silicon nanoparticles and combinations thereof.
42. The energy storage device of any one of claims 38 – 42, wherein the exfoliated carbon nanotubes comprise multi-wall carbon nanotubes.
43. The energy storage device of claim 37, wherein the at least one electrode comprising the exfoliated carbon nanotubes is the anode.
44. A method for making a polymer composite, said method comprising:
 - a) providing the exfoliated carbon nanotubes of claim 1; and
 - b) mixing the exfoliated carbon nanotubes in a polymer material to form a polymer composite;wherein exfoliated carbon nanotubes remain in an exfoliated state after being mixed in the polymer material.
45. The method of claim 44, wherein the polymer material comprises an epoxy.
46. The method of claim 45, further comprising:
 - curing the epoxy.

FIGURE 1

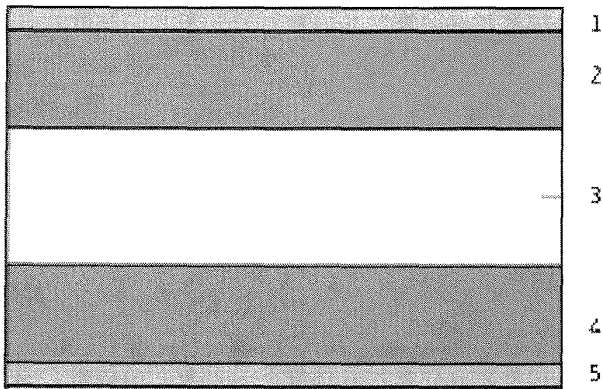


FIGURE 2

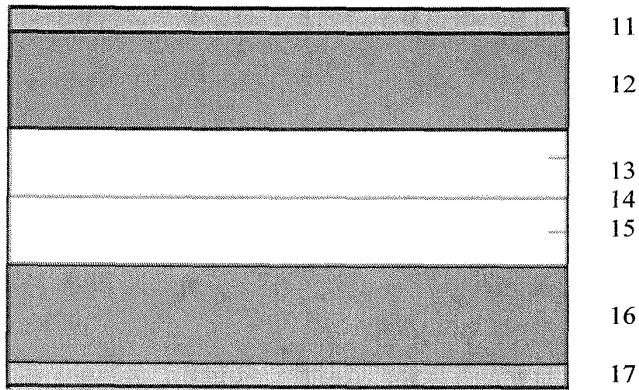


FIGURE 3

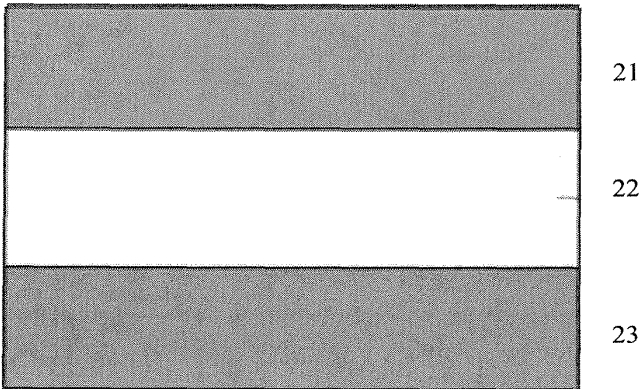


FIGURE 4

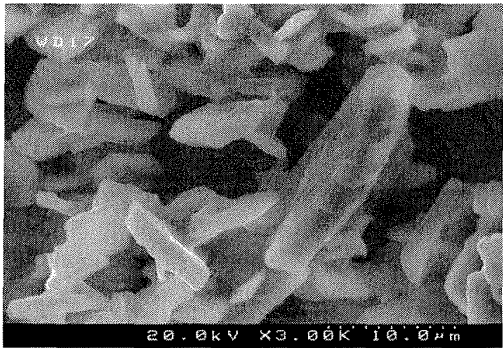
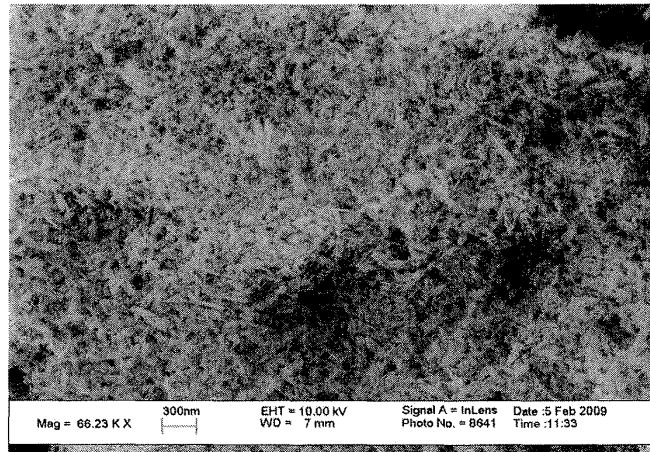


FIGURE 5



FIGURES 6A and 6B

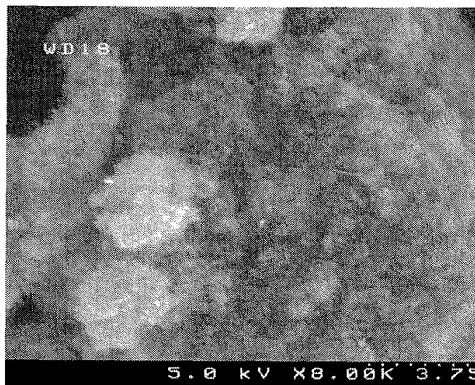


FIGURE 6A

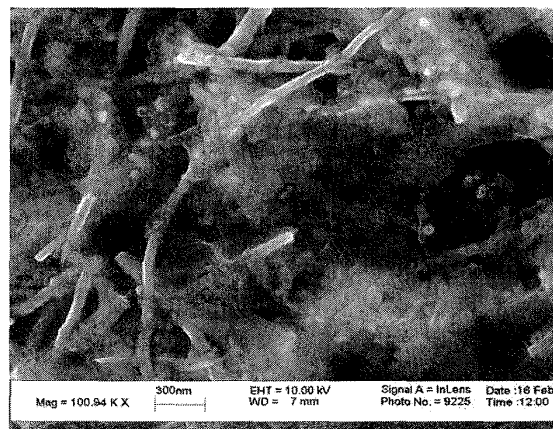


FIGURE 6B

FIGURES 7A and 7B

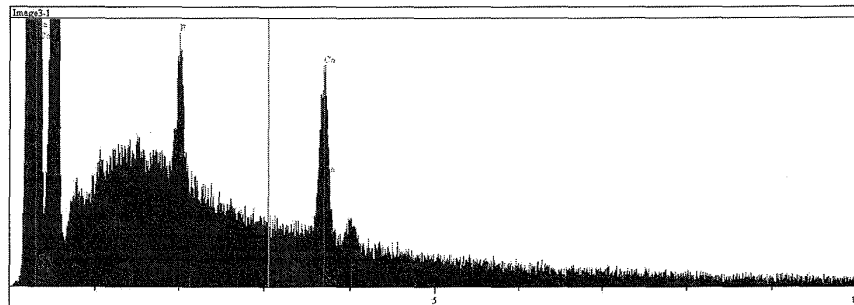


FIGURE 7A

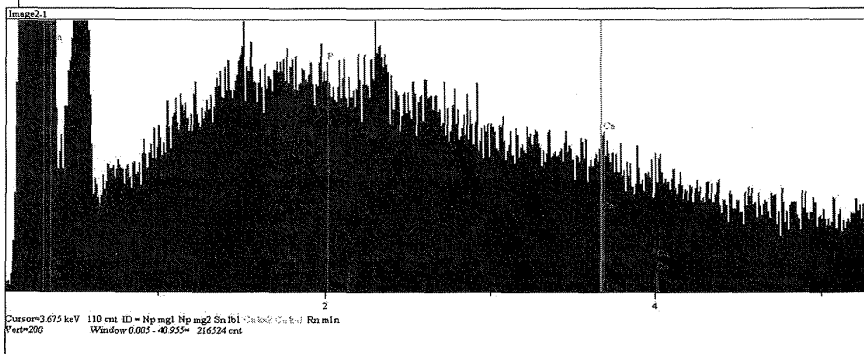


FIGURE 7B

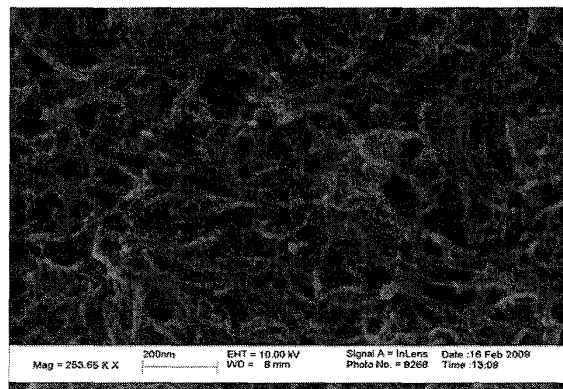


FIGURE 8

FIGURE 9

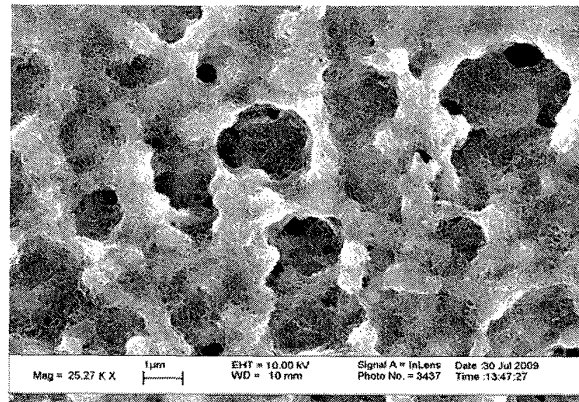


FIGURE 10

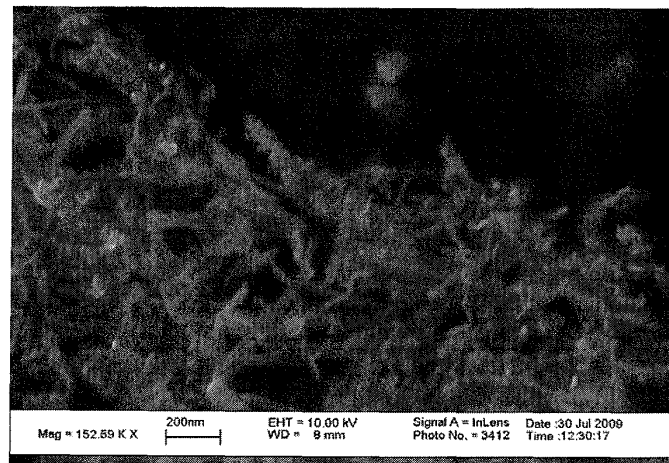
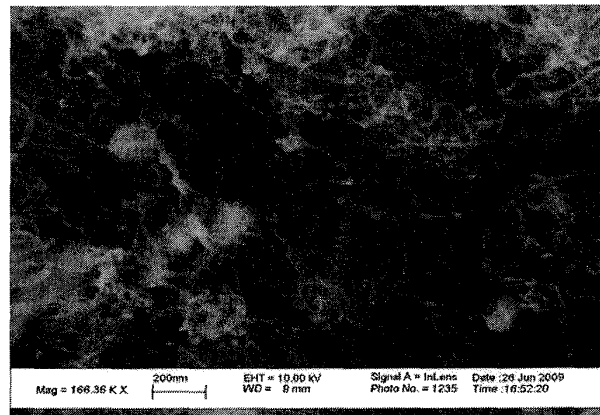


FIGURE 11



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 09/68781

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - B32B 27/02; C23C 4/04
USPC - 428/368; 427/535

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)

IPC: B32B 27/02; C23C 4/04
USPC: 428/368; 427/535

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

IPC: B32B 27/02; C23C 4/04 (text search only)
USPC: 428/368; 427/535; 977/742; 977/842; 977/844 (text search only)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

(USPT, PGPB, EPAB, JPAB); Google Patent, Google Scholar
Search terms on extra sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2005/0006623 A1 (Stanislaus et al.) 13 Jan 2005 (13.01.2005) (para [0015], [0017], [0019], [0025], [0040], [0045], [0064], [0067], [0071], [0072], [0073], [0074], [0105], [0106], [0117], [0120], [0144]).	1-3, 44-45
Y		11-13, 25-29, 37-43, 46
Y	US 2007/0280876 A1 (Tour et al.) 06 Dec 2007 (06.12.2007) (Abstract, para [0010], [0011], [0014], [0033], [0034], [0045], [0047], [0056]).	30-33
Y	US 2008/0220148 A1 (Clarkson et al.) 16 Aug 2007 (16.08.2007) (Abstract, para [0007], [0011], [0015], [0048], [0080]).	11-13, 25-29
Y	US 2006/0286456 A1 (Fu et al.) 21 Dec 2006 (21.12.2006) (Abstract, para [0007], [0008], [0009], [0017], [0022], [0028]).	37-43
Y	US 2007/0170071 A1 (Suh et al.) 26 Jul 2007 (26.07.2006) (para [0189], [0220]).	46

☐ Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

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"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

04 Sep 2010 (04.09.2010)

Date of mailing of the international search report

17 SEP 2010

Name and mailing address of the ISA/US

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Facsimile No. 571-273-3201

Authorized officer:

Lee W. Young

PCT Helpdesk: 571-272-4300
PCT OSP: 571-272-7774

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 09/68781

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☒ Claims Nos.: Claims 4-10, 14-24, 34-36
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

Form PCT/ISA/210 (continuation of first sheet (2)) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 09/68781

continued from box B

Search terms:

exfoliate, CNT, carbon nanotube, carbon nano-tube, mat, solid state, single walled, double walled, multi-walled, SWNT, MWNT, DWNT, ionic strength, precipitate, wash, epoxy, electrode, anode, transition, cured, curing, curable, crystalline, nanocrystalline, clay, graphite, solution, suspension, lithium titanate, oxide, $\text{Li}_4\text{Ti}_5\text{O}_{12}$, nanocrystals, surfactant, acid, superacid, filter, filtration, battery, energy storage, chlorosulfonic, chlorosulphonic, chlorosulfuric, hydroxyapatite, nanorod, nanowhisker, nanoplate, dispersant, pluornic

Form PCT/ISA/210 (extra sheet) (July 2009)

Electronic Acknowledgement Receipt	
EFS ID:	14004049
Application Number:	13529797
International Application Number:	
Confirmation Number:	1004
Title of Invention:	LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM
First Named Inventor/Applicant Name:	Clive P. Bosnyak
Customer Number:	25555
Filer:	Sara Kay Borrelli/Debra Pepper
Filer Authorized By:	Sara Kay Borrelli
Attorney Docket Number:	DENA-0008 (137124.00041)
Receipt Date:	17-OCT-2012
Filing Date:	21-JUN-2012
Time Stamp:	11:42:25
Application Type:	Utility under 35 USC 111(a)

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	Information Disclosure Statement (IDS) Form (SB08)	DENA0008IDS.pdf	611782 966f667fdb67e42ecf1e85b82648996c1634ca4d	no	4
Warnings:					
Information:					

2	Other Reference-Patent or Application Document	DENA0008ISR.pdf	725522	no	9
			1ac0c943bb0bf04eee702b310ab6f39e80b22910		
Warnings:					
Information:					
3	Foreign Reference	DENA0008WO117392.pdf	3729070	no	44
			c937ba9816415162936df4e505e5f238046eb4b2		
Warnings:					
Information:					
Total Files Size (in bytes):			5066374		
<p>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.</p> <p><u>New Applications Under 35 U.S.C. 111</u> 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.</p> <p><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.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> 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.</p>					



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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
13/529,797	06/21/2012	1726	970	DENA-0008 (137124.00041)	13	6

CONFIRMATION NO. 1004

UPDATED FILING RECEIPT



25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

Date Mailed: 08/13/2012

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**

Applicant(s)

Clive P. Bosnyak, Dripping Springs, TX;
Kurt W. Swogger, Austin, TX;

Assignment For Published Patent Application

Designed Nanotubes, LLC, Austin, TX

Power of Attorney: The patent practitioners associated with Customer Number 25555

Domestic Priority data as claimed by applicant

This appln claims benefit of 61/500,560 06/23/2011

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.)

If Required, Foreign Filing License Granted: 07/20/2012

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/529,797**

Projected Publication Date: 12/27/2012

Non-Publication Request: No

Early Publication Request: No

**** SMALL ENTITY ****

Title

LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM

Preliminary Class

429

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.

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Title 37, Code of Federal Regulations, 5.11 & 5.15

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page 2 of 3

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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875						Application or Docket Number 13/529,797			
APPLICATION AS FILED - PART I									
(Column 1)		(Column 2)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY			
FOR	NUMBER FILED	NUMBER EXTRA	RATE(\$)	FEE(\$)		RATE(\$)	FEE(\$)		
BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A	95		N/A			
SEARCH FEE (37 CFR 1.16(k), (l), or (m))	N/A	N/A	N/A	310		N/A			
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A	125		N/A			
TOTAL CLAIMS (37 CFR 1.16(i))	13	minus 20 = *	x 30 =	0.00	OR				
INDEPENDENT CLAIMS (37 CFR 1.16(h))	6	minus 3 = *	x 125 =	375					
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					
			TOTAL	905		TOTAL			
* If the difference in column 1 is less than zero, enter "0" in column 2.									
APPLICATION AS AMENDED - PART II									
(Column 1)		(Column 2)		(Column 3)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY	
AMENDMENT A	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)	
	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))					OR			
	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.									
AMENDMENT B	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)	
	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))					OR			
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))					OR			
			TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE			



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/529,797	06/21/2012	Clive P. Bosnyak	DENA-0008 (137124.00041)

CONFIRMATION NO. 1004

POA ACCEPTANCE LETTER



OC000000055861838

25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

Date Mailed: 08/13/2012

NOTICE OF ACCEPTANCE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 08/02/2012.

The Power of Attorney in this application is accepted. Correspondence in this application will be mailed to the above address as provided by 37 CFR 1.33.

/ydemisse/

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Clive P. Bosnyak, et al.
Serial No.: 13/529,797
Filed: June 21, 2012
Group Art Unit: 1726
Examiner: Unknown
Title: LITHIUM ION BATTERIES USING DISCRETE CARBON
NANOTUBES, METHODS FOR PRODUCTION THEREOF
AND PRODUCTS OBTAINED THEREFROM

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Dear Sir:

RESPONSE TO NOTICE TO FILE MISSING PARTS

In response to the Notice to File Missing Parts mailed July 24, 2012, enclosed please find the following documents:

1. One (1) Declaration (4 pages) executed by all inventors.

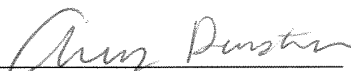
Also enclosed for filing are the following:

2. Power of Attorney; and
3. Statement under 37 C.F.R. 3.73(b).

Please charge the surcharge fee (\$65) to Jackson Walker L.L.P. Deposit Account No. 10-0096. Any additional fees associated with this filing should be charged to Deposit Account No. 10-0096. A prompt examination and allowance of the pending claims is earnestly solicited.

Respectfully submitted,

Date: 8/2/12

By: 
Amy Ulfers Dunstan
Reg. No. 61,069

Jackson Walker L.L.P.
901 Main Street, Suite 6000
Dallas, Texas 75202
Tel.: (214) 953-5982

Doc Code: Oath

Document Description: Oath or declaration filed

PTO/SB/01 (04-09)

Approved for use through 01/31/2014. OMB 0651-0032
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.

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63) ☐ Declaration Submitted With Initial Filing OR ☒ Declaration Submitted After Initial Filing (surcharge (37 CFR 1.16(f)) required)	Attorney Docket Number	DENA-0008 (137124.41)
	First Named Inventor	Clive P. Bosnyak
	COMPLETE IF KNOWN	
	Application Number	13/529,797
	Filing Date	June 21, 2012
	Art Unit	
Examiner Name		

I hereby declare that: (1) Each inventor's residence, mailing address, and citizenship are as stated below next to their name; and (2) I believe the inventor(s) named below to be the original and first inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention titled:

**IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES,
METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM**

(Title of the Invention)

the application of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) 06/21/2012 as United States Application Number or PCT International
Application Number 13/529,797 and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified application, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

Authorization To Permit Access To Application by Participating Offices

☐ If 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 World Intellectual Property Office (WIPO), and any other intellectual property offices in which a foreign application claiming priority to the above-identified patent application is filed access to the above-identified patent application. See 37 CFR 1.14(c) and (h). This box should not be checked if the applicant does not wish the EPO, JPO, KIPO, WIPO, or other intellectual property office in which a foreign application claiming priority to the above-identified patent application is filed to have access to the above-identified patent application.

In accordance with 37 CFR 1.14(h)(3), access will be provided to a copy of the above-identified patent application with respect to: 1) the above-identified patent application-as-filed; 2) any foreign application to which the above-identified patent application claims priority under 35 U.S.C. 119(a)-(d) if a copy of the foreign application that satisfies the certified copy requirement of 37 CFR 1.55 has been filed in the above-identified patent application; and 3) any U.S. application-as-filed from which benefit is sought in the above-identified patent application.

In accordance with 37 CFR 1.14(c), access may be provided to information concerning the date of filing the Authorization to Permit Access to Application by Participating Offices.

[Page 1 of 3]

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. 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 21 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.

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.

DECLARATION — Utility or Design Patent Application

Claim of Foreign Priority Benefits

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or (f), or 365(b) of any foreign application(s) for patent, inventor's or plant breeder's rights certificate(s), or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent, inventor's or plant breeder's rights certificate(s), or any PCT international application having a filing date before that of the application on which priority is claimed.

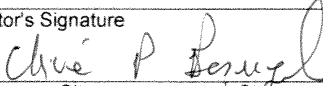
Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			☐	☐	☐
			☐	☐	☐
			☐	☐	☐
			☐	☐	☐

☐ Additional foreign application number(s) are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

[Page 2 of 3]

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.

DECLARATION — Utility or Design Patent Application

Direct all correspondence to: ☒		The address associated with Customer Number: 25555		OR ☐ Correspondence address below	
Name					
Address					
City		State		Zip	
Country		Telephone		Email	
<p align="center">WARNING:</p> <p>Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2038 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2038 submitted for payment purposes are not retained in the application file and therefore are not publicly available. Petitioner/applicant is advised that documents which form the record of a patent application (such as the PTO/SB/01) are placed into the Privacy Act system of records DEPARTMENT OF COMMERCE, COMMERCE-PAT-7, System name: <i>Patent Application Files</i>. Documents not retained in an application file (such as the PTO-2038) are placed into the Privacy Act system of COMMERCE/PAT-TM-10, System name: <i>Deposit Accounts and Electronic Funds Transfer Profiles</i>.</p> <p>I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.</p>					
NAME OF SOLE OR FIRST INVENTOR:			☐ A petition has been filed for this unsigned inventor		
Given Name (first and middle [if any])			Family Name or Surname		
Clive P.			Bosnyak		
Inventor's Signature 			Date 06-27-12		
Residence: City		State		Country	
Dripping Springs		Texas		U.S.A.	
Citizenship		U.S.A.			
Mailing Address					
12001 Settlers Trail					
City		State		Zip	
Dripping Springs		Texas		78620	
Country		U.S.A.			
☒ Additional inventors or a legal representative are being named on the <u>1</u> supplemental sheet(s) PTO/SB/02A or 02LR attached hereto					

[Page 3 of 3]

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.

DECLARATION	ADDITIONAL INVENTOR(S) Supplemental Sheet
Page <u>1</u> of <u>1</u>	

Name of Additional Joint Inventor, if any:		☐ A petition has been filed for this unsigned inventor	
Given Name (first and middle (if any))		Family Name or Surname	
Kurt W.		Swogger	
Inventor's Signature <i>Kurt W Swogger</i>		Date <i>6/27/12</i>	
Austin Residence: City	Texas State	U.S.A. Country	U.S.A. Citizenship
4211 Waters Edge Cove			
Mailing Address			
Austin City	Texas State	78731 Zip	U.S.A. Country
Name of Additional Joint Inventor, if any:		☐ A petition has been filed for this unsigned inventor	
Given Name (first and middle (if any))		Family Name or Surname	
Inventor's Signature		Date	
Residence: City	State	Country	Citizenship
Mailing Address			
City	State	Zip	Country
Name of Additional Joint Inventor, if any:		☐ A petition has been filed for this unsigned inventor	
Given Name (first and middle (if any))		Family Name or Surname	
Inventor's Signature		Date	
Residence: City	State	Country	Citizenship
Mailing Address			
City	State	Zip	Country

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. 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 21 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 (1-800-786-9199) and select option 2.

POWER OF ATTORNEY OR REVOCATION OF POWER OF ATTORNEY WITH A NEW POWER OF ATTORNEY AND CHANGE OF CORRESPONDENCE ADDRESS	Application Number	13/529,797
	Filing Date	June 21, 2012
	First Named Inventor	Clive P. Bosnyak
	Title	IMPROVED LITHIUM ION BATTERIES..
	Art Unit	
	Examiner Name	
	Attorney Docket Number	DENA-0008 (137124.41)

I hereby revoke all previous powers of attorney given in the above-identified application.

☐ A Power of Attorney is submitted herewith.

OR

☒ I hereby appoint Practitioner(s) associated with the following Customer Number as my/our attorney(s) or agent(s) to prosecute the application identified above, and to transact all business in the United States Patent and Trademark Office connected therewith:

25555

OR

☐ I hereby appoint Practitioner(s) named below as my/our attorney(s) or agent(s) to prosecute the application identified above, and to transact all business in the United States Patent and Trademark Office connected therewith:

Practitioner(s) Name	Registration Number

Please recognize or change the correspondence address for the above-identified application to:

☐ The address associated with the above-mentioned Customer Number.

OR

☒ The address associated with Customer Number:

25555

OR

☐ Firm or Individual Name

Address

City State Zip

Country

Telephone Email

I am the:

☐ Applicant/Inventor.

OR

☒ Assignee of record of the entire interest. See 37 CFR 3.71.
Statement under 37 CFR 3.73(b) (Form PTO/SB/96) submitted herewith or filed on _____

SIGNATURE of Applicant or Assignee of Record

Signature	x <i>Clive P. Bosnyak</i>	Date	x 06-27-12
Name	x Clive P. Bosnyak	Telephone	x (512) 394 0922
Title and Company	x CSO, Designed Nanotubes		

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required, see below.

☒ *Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.31, 1.32 and 1.33. 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 3 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.

STATEMENT UNDER 37 CFR 3.73(b)

Applicant/Patent Owner: Designed NanoTubes, LLC

Application No./Patent No.: 13/529,797

Filed/Issue Date: June 21, 2012

Titled: IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM

Designed NanoTubes, LLC

, a limited liability company

(Name of Assignee)

(Type of Assignee, e.g., corporation, partnership, university, government agency, etc.)

states that it is:

1. ☒ the assignee of the entire right, title, and interest in;
2. ☐ an assignee of less than the entire right, title, and interest in
(The extent (by percentage) of its ownership interest is _____ %); or
3. ☐ the assignee of an undivided interest in the entirety of (a complete assignment from one of the joint inventors was made)
the patent application/patent identified above, by virtue of either:

A. ☒ An assignment from the inventor(s) of the patent application/patent identified above. The assignment was recorded in the United States Patent and Trademark Office at Reel _____, Frame _____, or for which a copy therefore is attached.

OR

B. ☐ A chain of title from the inventor(s), of the patent application/patent identified above, to the current assignee as follows:

1. From: _____ To: _____

The document was recorded in the United States Patent and Trademark Office at
Reel _____, Frame _____, or for which a copy thereof is attached.

2. From: _____ To: _____

The document was recorded in the United States Patent and Trademark Office at
Reel _____, Frame _____, or for which a copy thereof is attached.

3. From: _____ To: _____

The document was recorded in the United States Patent and Trademark Office at
Reel _____, Frame _____, or for which a copy thereof is attached.

☐ Additional documents in the chain of title are listed on a supplemental sheet(s).

☒ As required by 37 CFR 3.73(b)(1)(i), the documentary evidence of the chain of title from the original owner to the assignee was, or concurrently is being, submitted for recordation pursuant to 37 CFR 3.11.

[NOTE: A separate copy (i.e., a true copy of the original assignment document(s)) must be submitted to Assignment Division in accordance with 37 CFR Part 3, to record the assignment in the records of the USPTO. See MPEP 302.08]

The undersigned (whose title is supplied below) is authorized to act on behalf of the assignee.

x Clive P. Bosnyak
Signature

x 06-27-12
Date

x Clive P. Bosnyak
Printed or Typed Name

x CSO
Title

This collection of information is required by 37 CFR 3.73(b). 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. 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.

ASSIGNMENT

Whereas, we, Clive P. Bosnyak and Kurt W. Swogger, have invented certain new and useful improvements in "LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM" for which a provisional application was filed with the U.S. Patent and Trademark Office on June 23, 2011, Application Number 61/500,560, and

Whereas, Designed NanoTubes, LLC, 4211 Waters Edge Cove, Austin, Texas 78731, (hereinafter referred to as "ASSIGNEE"), is desirous of acquiring the entire right, title and interest in and to the said invention, and to the said application and any Letters Patent that may issue thereon;

NOW, THEREFORE, for good and valuable consideration, the receipt of which is hereby acknowledged, I/we hereby sell, assign, and transfer unto said ASSIGNEE, its successors, assigns and legal representatives, the full and exclusive right, title and interest in and to said invention and in and to said application, including any non-provisional application(s) for this invention which claims priority to the filing date of the aforementioned provisional application, or any eventual utility application converted from the aforementioned provisional application, and all divisions, reissues, reexaminations, substitutions, continuations, continuations-in-part, and extensions thereof, and any other applications that have been, or may be, filed for the inventions in the United States and in any foreign countries; and I/We hereby authorize and request the Commissioner for Patents and Trademarks or any such foreign counterpart to issue all patents for said inventions, or patents resulting therefrom to said ASSIGNEE of the entire right, title and interest.

I/We also hereby sell and assign to said ASSIGNEE, its successors, assigns and legal representatives, the entire right, title and interest to the invention disclosed in said application throughout the world, including the right to file applications and obtain patents, utility models, industrial models, and designs for said invention in its own name throughout the world including all rights of priority, all rights to publish cautionary notices reserving ownership of said invention and all rights to register said invention in appropriate registries; and I/we further agree to execute any and all powers of attorney, applications, assignments, declarations, affidavits, and any other papers in connection therewith necessary to perfect such rights, title and interest in ASSIGNEE, its successors, assigns and legal representatives.

I/We hereby warrant and covenant that no assignment, grant, mortgage, license or other agreement affecting the rights and property herein conveyed has been or will be made to others.

I/We hereby further agree that I/we will communicate to said ASSIGNEE, or to its successors, assigns and legal representatives, any facts known to me/us respecting any improvements; and, at the expense of said ASSIGNEE, to testify in any legal proceedings, sign all lawful papers, execute all divisional, continuation, continuation-in-part, reissue, reexamination, and substitute applications, and make all lawful oaths, and generally do everything possible to vest title in said ASSIGNEE and to aid said ASSIGNEE, its successors, assigns and legal representatives to obtain and enforce proper protection for said invention and any improvements in the United States and all other countries.

This Assignment is made effective as of June 23, 2011.

IN WITNESS WHEREOF, I have hereunto set my hand and seal on the date below indicated.

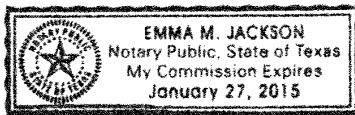
Kurt W Swogger
Kurt W. Swogger

STATE OF Texas

COUNTY OF Hays

§
§
§

On this 19th day of June, ^{as 2012 KWS JWS} 2011, before me personally came Kurt W. Swogger, to me known to be the individual described herein and who executed the foregoing instrument, and acknowledged execution of the same.



Emma M. Jackson
Notary Public

My Commission Expires:

Jan. 27, 2015

Seal

IN WITNESS WHEREOF, I have hereunto set my hand and seal on the date below indicated.

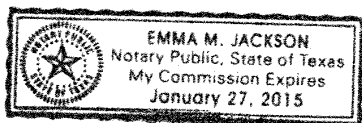
Clive P. Bosnyak
Clive P. Bosnyak

STATE OF Texas

COUNTY OF Hays

§
§
§

On this 19th day of June, ²⁰¹² ~~2011~~, before me personally came Clive P. Bosnyak, to me known to be the individual described herein and who executed the foregoing instrument, and acknowledged execution of the same.



Emma M. Jackson
Notary Public

My Commission Expires:

Jan. 27, 2015

Seal

Electronic Patent Application Fee Transmittal				
Application Number:		13529797		
Filing Date:		21-Jun-2012		
Title of Invention:		LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
First Named Inventor/Applicant Name:		Clive P. Bosnyak		
Filer:		Amy L. Ulfers/Debra Pepper		
Attorney Docket Number:		DENA-0008 (137124.00041)		
Filed as Small Entity				
Utility under 35 USC 111(a) Filing Fees				
Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Late filing fee for oath or declaration	2051	1	65	65
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Miscellaneous:				
Total in USD (\$)				65

Electronic Acknowledgement Receipt	
EFS ID:	13400548
Application Number:	13529797
International Application Number:	
Confirmation Number:	1004
Title of Invention:	LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM
First Named Inventor/Applicant Name:	Clive P. Bosnyak
Customer Number:	25555
Filer:	Amy L. Ulfers/Debra Pepper
Filer Authorized By:	Amy L. Ulfers
Attorney Docket Number:	DENA-0008 (137124.00041)
Receipt Date:	02-AUG-2012
Filing Date:	21-JUN-2012
Time Stamp:	11:06:05
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Deposit Account
Payment was successfully received in RAM	\$65
RAM confirmation Number	10108
Deposit Account	100096
Authorized User	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
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1	Applicant Response to Pre-Exam Formalities Notice	DENA0008RESPONSE.pdf	54447 c2a1c598f5bd6d5a0ffcd97006a4600507972a07	no	1
Warnings:					
Information:					
2	Oath or Declaration filed	DENA0008DECLARATION.pdf	548348 14612fa6b927c8fd8ffcd905aaff907d1636fde3f	no	4
Warnings:					
Information:					
3	Power of Attorney	DENA0008POA.pdf	176826 6b7935e44fa7497814fed537bb36950a064a2b5a	no	1
Warnings:					
Information:					
4	Assignee showing of ownership per 37 CFR 3.73(b).	DENA0008STMT.pdf	390249 74690254a62e155c61fe186f95547628ddffce7c	no	4
Warnings:					
Information:					
5	Fee Worksheet (SB06)	fee-info.pdf	30741 1a7ab1b736b543d7f31c8ae83ec07952019baa2	no	2
Warnings:					
Information:					
Total Files Size (in bytes):			1200611		
<p>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.</p> <p><u>New Applications Under 35 U.S.C. 111</u> 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.</p> <p><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.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> 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.</p>					

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875						Application or Docket Number 13/529,797			
APPLICATION AS FILED - PART I									
(Column 1)		(Column 2)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY			
FOR	NUMBER FILED	NUMBER EXTRA		RATE(\$)	FEE(\$)	RATE(\$)	FEE(\$)		
BASIC FEE <small>(37 CFR 1.16(a), (b), or (c))</small>	N/A	N/A		N/A	95	N/A	N/A		
SEARCH FEE <small>(37 CFR 1.16(k), (l), or (m))</small>	N/A	N/A		N/A	310	N/A	N/A		
EXAMINATION FEE <small>(37 CFR 1.16(o), (p), or (q))</small>	N/A	N/A		N/A	125	N/A	N/A		
TOTAL CLAIMS <small>(37 CFR 1.16(i))</small>	13	minus 20 =	*	x 30 =	0.00	OR			
INDEPENDENT CLAIMS <small>(37 CFR 1.16(h))</small>	6	minus 3 =	3	x 125 =	375	OR			
APPLICATION SIZE FEE <small>(37 CFR 1.16(s))</small>	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	OR			
MULTIPLE DEPENDENT CLAIM PRESENT <small>(37 CFR 1.16(j))</small>					0.00	OR			
* If the difference in column 1 is less than zero, enter "0" in column 2.				TOTAL	905	TOTAL			
APPLICATION AS AMENDED - PART II									
(Column 1)		(Column 2)		(Column 3)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY	
AMENDMENT A	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)	RATE(\$)	ADDITIONAL FEE(\$)		
	Total <small>(37 CFR 1.16(i))</small>	*	Minus **	=	x =	OR	x =		
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus ***	=	x =	OR	x =		
	Application Size Fee <small>(37 CFR 1.16(s))</small>					OR			
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>					OR			
TOTAL ADD'L FEE				TOTAL ADD'L FEE					
AMENDMENT B	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)	RATE(\$)	ADDITIONAL FEE(\$)		
	Total <small>(37 CFR 1.16(i))</small>	*	Minus **	=	x =	OR	x =		
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus ***	=	x =	OR	x =		
	Application Size Fee <small>(37 CFR 1.16(s))</small>					OR			
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>					OR			
TOTAL ADD'L FEE				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.									



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Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY. DOCKET NO.	TOT CLAIMS	IND CLAIMS
13/529,797	06/21/2012	1726	905	DENA-0008 (137124.00041)	13	6

CONFIRMATION NO. 1004

FILING RECEIPT



25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

Date Mailed: 07/24/2012

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**

Applicant(s)

Clive P. Bosnyak, Dripping Springs, TX;
Kurt W. Swogger, Austin, TX;

Assignment For Published Patent Application

Designed Nanotubes, LLC, Austin, TX

Power of Attorney: None

Domestic Priority data as claimed by applicant

This appln claims benefit of 61/500,560 06/23/2011

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.)

If Required, Foreign Filing License Granted: 07/20/2012

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/529,797**

Projected Publication Date: To Be Determined - pending completion of Missing Parts

Non-Publication Request: No

Early Publication Request: No

**** SMALL ENTITY ****

Title

LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM

Preliminary Class

429

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 (1-866-999-4158).

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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page 2 of 3

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This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/529,797	06/21/2012	Clive P. Bosnyak	DENA-0008 (137124.00041)

CONFIRMATION NO. 1004

FORMALITIES LETTER



OC000000055511300

25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

Date Mailed: 07/24/2012

NOTICE TO FILE MISSING PARTS OF NONPROVISIONAL APPLICATION

FILED UNDER 37 CFR 1.53(b)

Filing Date Granted

Items Required To Avoid Abandonment:

An application number and filing date have been accorded to this application. The item(s) indicated below, however, are missing. Applicant is given **TWO MONTHS** from the date of this Notice within which to file all required items below to avoid abandonment. Extensions of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR 1.136(a).

- The oath or declaration is missing.

A properly signed oath or declaration in compliance with 37 CFR 1.63, identifying the application by the above Application Number and Filing Date, is required.

Note: If a petition under 37 CFR 1.47 is being filed, an oath or declaration in compliance with 37 CFR 1.63 signed by all available joint inventors, or if no inventor is available by a party with sufficient proprietary interest, is required.

The applicant needs to satisfy supplemental fees problems indicated below.

The required item(s) identified below must be timely submitted to avoid abandonment:

- A surcharge (for late submission of filing fee, search fee, examination fee or oath or declaration) as set forth in 37 CFR 1.16(f) of **\$65** for a small entity in compliance with 37 CFR 1.27, must be submitted.

SUMMARY OF FEES DUE:

Total fee(s) required within **TWO MONTHS** from the date of this Notice is **\$65** for a small entity

- **\$65** Surcharge.

Replies should be mailed to:

Mail Stop Missing Parts
Commissioner for Patents
P.O. Box 1450
Alexandria VA 22313-1450

Registered users of EFS-Web may alternatively submit their reply to this notice via EFS-Web.
<https://portal.uspto.gov/authenticate/AuthenticateUserLocalEPF.html>

For more information about EFS-Web please call the USPTO Electronic Business Center at **1-866-217-9197** or visit our website at <http://www.uspto.gov/ebc>.

If you are not using EFS-Web to submit your reply, you must include a copy of this notice.

/nhassani/

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875						Application or Docket Number 13/529,797			
APPLICATION AS FILED - PART I									
(Column 1)		(Column 2)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY			
FOR	NUMBER FILED	NUMBER EXTRA	RATE(\$)	FEE(\$)		RATE(\$)	FEE(\$)		
BASIC FEE <small>(37 CFR 1.16(a), (b), or (c))</small>	N/A	N/A	N/A	95		N/A			
SEARCH FEE <small>(37 CFR 1.16(k), (l), or (m))</small>	N/A	N/A	N/A	310		N/A			
EXAMINATION FEE <small>(37 CFR 1.16(o), (p), or (q))</small>	N/A	N/A	N/A	125		N/A			
TOTAL CLAIMS <small>(37 CFR 1.16(i))</small>	13	minus 20 = *	x 30	= 0.00	OR				
INDEPENDENT CLAIMS <small>(37 CFR 1.16(h))</small>	6	minus 3 = *	x 125	= 375					
APPLICATION SIZE FEE <small>(37 CFR 1.16(s))</small>	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 <small>(37 CFR 1.16(j))</small>				0.00					
			TOTAL	905		TOTAL			
* If the difference in column 1 is less than zero, enter "0" in column 2.									
APPLICATION AS AMENDED - PART II									
(Column 1)		(Column 2)		(Column 3)		SMALL ENTITY		OR OTHER THAN SMALL ENTITY	
AMENDMENT A	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)	
	Total <small>(37 CFR 1.16(i))</small>	*	Minus **	=	x	=	OR	x	=
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus ***	=	x	=	OR	x	=
	Application Size Fee <small>(37 CFR 1.16(s))</small>						OR		
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>						OR		
				TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE		
AMENDMENT B	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)	
	Total <small>(37 CFR 1.16(i))</small>	*	Minus **	=	x	=	OR	x	=
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus ***	=	x	=	OR	x	=
	Application Size Fee <small>(37 CFR 1.16(s))</small>						OR		
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>						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.									



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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
13/529,797	06/21/2012	1726	905	DENA-0008 (137124.00041)	13	6

CONFIRMATION NO. 1004

FILING RECEIPT



OC000000055511299

25555
JACKSON WALKER LLP
901 MAIN STREET
SUITE 6000
DALLAS, TX 75202-3797

Date Mailed: 07/24/2012

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**

Applicant(s)

Clive P. Bosnyak, Dripping Springs, TX;
Kurt W. Swogger, Austin, TX;

Assignment For Published Patent Application

Designed Nanotubes, LLC, Austin, TX

Power of Attorney: None

Domestic Priority data as claimed by applicant

This appln claims benefit of 61/500,560 06/23/2011

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.)

If Required, Foreign Filing License Granted: 07/20/2012

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/529,797**

Projected Publication Date: To Be Determined - pending completion of Missing Parts

Non-Publication Request: No

Early Publication Request: No

**** SMALL ENTITY ****

Title

LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM

Preliminary Class

429

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

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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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page 2 of 3

the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

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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UTILITY PATENT APPLICATION TRANSMITTAL <small>(Only for new nonprovisional applications under 37 CFR 1.53(b))</small>	<i>Attorney Docket No.</i>	DENA-0008 (137124.00041)
	<i>First Inventor</i>	Clive P. Bosnyak
	<i>Title</i>	IMPROVED LITHIUM ION BATTERIES...
	<i>Express Mail Label No.</i>	Electronic Filing

APPLICATION ELEMENTS <small>See MPEP chapter 600 concerning utility patent application contents.</small>	ADDRESS TO: Commissioner for Patents P.O. Box 1450 Alexandria VA 22313-1450										
<p>1. ☐ Fee Transmittal Form (e.g., PTO/SB/17)</p> <p>2. ☒ Applicant claims small entity status. See 37 CFR 1.27.</p> <p>3. ☒ Specification [Total Pages <u>18</u>] Both the claims and abstract must start on a new page (For information on the preferred arrangement, see MPEP 608.01(a))</p> <p>4. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets <u>2</u>]</p> <p>5. Oath or Declaration [Total Sheets <u> </u>] a. ☐ Newly executed (original or copy) b. ☐ A copy from a prior application (37 CFR 1.63(d)) (for continuation/divisional with Box 18 completed) i. ☐ DELETION OF INVENTOR(S) Signed statement attached deleting inventor(s) name in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).</p> <p>6. ☒ Application Data Sheet. See 37 CFR 1.76</p> <p>7. ☐ CD-ROM or CD-R in duplicate, large table or Computer Program (Appendix) ☐ Landscape Table on CD</p> <p>8. Nucleotide and/or Amino Acid Sequence Submission (if applicable, items a. – c. are required) a. ☐ Computer Readable Form (CRF) b. ☐ Specification Sequence Listing on: i. ☐ CD-ROM or CD-R (2 copies); or ii. ☐ Paper c. ☐ Statements verifying identity of above copies</p>	ACCOMPANYING APPLICATION PARTS <p>9. ☐ Assignment Papers (cover sheet & document(s)) Name of Assignee _____</p> <p>10. ☐ 37 CFR 3.73(b) Statement (when there is an assignee) ☐ Power of Attorney</p> <p>11. ☐ English Translation Document (if applicable)</p> <p>12. ☐ Information Disclosure Statement (PTO/SB/08 or PTO-1449) ☐ Copies of citations attached</p> <p>13. ☐ Preliminary Amendment</p> <p>14. ☐ Return Receipt Postcard (MPEP 503) (Should be specifically itemized)</p> <p>15. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)</p> <p>16. ☐ Nonpublication Request under 35 U.S.C. 122(b)(2)(B)(i). Applicant must attach form PTO/SB/35 or equivalent.</p> <p>17. ☐ Other: _____</p>										
<p>18. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in the first sentence of the specification following the title, or in an Application Data Sheet under 37 CFR 1.76:</p> <p>☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No.: _____</p> <p>Prior application information: _____ Examiner: _____ Art Unit: _____</p>											
19. CORRESPONDENCE ADDRESS											
<p>☒ The address associated with Customer Number: 25555 OR ☐ Correspondence address below</p>											
<table border="1" style="width: 100%; border-collapse: collapse;"><tr><td colspan="2">Name</td></tr><tr><td colspan="2">Address</td></tr><tr><td>City</td><td>State</td></tr><tr><td>Country</td><td>Zip Code</td></tr><tr><td>Telephone</td><td>Email</td></tr></table>		Name		Address		City	State	Country	Zip Code	Telephone	Email
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<table border="1" style="width: 100%; border-collapse: collapse;"><tr><td>Signature</td><td>//Amy Ulfers Dunstan//</td><td>Date</td><td>2012-06-21</td></tr><tr><td>Name (Print/Type)</td><td>Amy Ulfers Dunstan</td><td>Registration No. (Attorney/Agent)</td><td>61069</td></tr></table>		Signature	//Amy Ulfers Dunstan//	Date	2012-06-21	Name (Print/Type)	Amy Ulfers Dunstan	Registration No. (Attorney/Agent)	61069		
Signature	//Amy Ulfers Dunstan//	Date	2012-06-21								
Name (Print/Type)	Amy Ulfers Dunstan	Registration No. (Attorney/Agent)	61069								

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	
		Application Number	
Title of Invention	IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
<p>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.</p> <p>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.</p>			

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☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Applicant Information:

Applicant 1					Remove	
Applicant Authority		☒ Inventor		☐ Legal Representative under 35 U.S.C. 117		☐ Party of Interest under 35 U.S.C. 118
Prefix	Given Name	Middle Name	Family Name		Suffix	
	Clive	P.	Bosnyak			
Residence Information (Select One)		☒ US Residency ☐ Non US Residency ☐ Active US Military Service				
City	Dripping Springs	State/Province	TX	Country of Residence i	US	
Citizenship under 37 CFR 1.41(b) i		US				
Mailing Address of Applicant:						
Address 1		12001 Settlers Trail				
Address 2						
City	Dripping Springs	State/Province	TX			
Postal Code	78620	Countryi	US			
Applicant 2					Remove	
Applicant Authority		☒ Inventor		☐ Legal Representative under 35 U.S.C. 117		☐ Party of Interest under 35 U.S.C. 118
Prefix	Given Name	Middle Name	Family Name		Suffix	
	Kurt	W.	Swogger			
Residence Information (Select One)		☒ US Residency ☐ Non US Residency ☐ Active US Military Service				
City	Austin	State/Province	TX	Country of Residence i	US	
Citizenship under 37 CFR 1.41(b) i		US				
Mailing Address of Applicant:						
Address 1		4211 Waters Edge Cove				
Address 2						
City	Austin	State/Province	TX			
Postal Code	78731	Countryi	US			
All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the Add button.						
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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	
		Application Number	
Title of Invention	IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
Customer Number	25555		
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Application Information:

Title of the Invention	IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
Attorney Docket Number		Small Entity Status Claimed	☒
Application Type	Nonprovisional		
Subject Matter	Utility		
Suggested Class (if any)		Sub Class (if any)	
Suggested Technology Center (if any)			
Total Number of Drawing Sheets (if any)	3	Suggested Figure for Publication (if any)	

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.	

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Prior Application Status	Pending	Remove	
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)
	non provisional of	61500560	2011-06-23
Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the Add button.			Add

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	
		Application Number	
Title of Invention	IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
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			Remove
Application Number	Country ⁱ	Parent Filing Date (YYYY-MM-DD)	Priority Claimed
			☒ Yes ☐ No
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Assignee 1				Remove
If the Assignee is an Organization check here. ☐				
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Address 1				
Address 2				
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Signature:

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Signature	//Amy Dunstan//		Date (YYYY-MM-DD)	2012-06-21
First Name	Amy	Last Name	Dunstan	Registration Number
				61069

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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)).
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**IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES,
METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED
THEREFROM**

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**IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES,
METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM**

[0001] This application claims priority to U.S. Provisional Patent Application Serial No. 61/500,560, entitled "LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM," filed on June 23, 2011, the entire content of which is hereby incorporated by reference.

FIELD OF THE INVENTION

[0002] The present invention relates generally to the field of improved technologies for lithium batteries. In some embodiments, the invention relates to discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

BACKGROUND

[0003] Lithium batteries are used extensively for portable electronic equipment and are increasingly being used to provide electrical back-up for wind and solar energy. The commercially used lithium ion (Li-ion) rechargeable batteries based on cobalt, nickel or manganese oxides for the cathode materials are known for their poor electrical conductivity and poor electrochemical stability which results in poor cycling (charge/discharge) ability. Olivine structures such as lithium iron phosphate, LiFePO_4 or lithium manganese phosphate, LiMnPO_4 , operate at higher charge and discharge rates, but are still limited by low electrical conductivity and the kinetics of Li-ion diffusion.

[0004] Attempts to overcome the above deficiencies have included synthesis of nano-sized crystals, termed nanocrystals, of lithium salts as well as mixtures of lithium salts and carbon nanotubes. Even though the mixtures of carbon nanotubes and nanocrystals of lithium salts show improvements in charge/discharge rates, the carbon nanotubes previously employed are not considered to be essentially discrete, i.e., individual uniformly dispersed tubes. Carbon nanotubes as conventionally made by gas phase reaction result in a tangled bundle of tubes with particle diameters ranging from 50 to 200 micrometers. Also, the lithium nanocrystals are not ionically or covalently attached to the surface of the discrete carbon nanotubes to provide for intimate electron

Page 2 of 16

transfer and enhanced mechanical strength of the active material. During charging and discharging of conventional lithium ion batteries, the nanocrystals expand and contract. Over a number of cycles, this leads to microcrack formation in the layer of active material and hence to higher internal resistance and decline of the battery performance. With crystals bound to and interconnected with discrete carbon nanotubes the microcrack formation due to severe mechanical vibration, bending, or expansion and contraction will be retarded.

[0005] Lithium salts have also been carbon coated with very thin coatings on the nanometer scale to enhance inter-particle electrical conductivity, but the carbon coating can slow the Lithium ion transport and may also react unfavorably with electrolytes over time. The carbon coating is considered to be amorphous in structure and is more likely to react with electrolyte than the crystalline carbon structure of the carbon nanotubes. Likewise, carbon particles have been added to the crystals of lithium salts to enhance inter-particle conductivity but these generally reduce the mechanical strength of dried pastes in the battery leading to cracking and reduced performance over time.

[0006] The present invention overcomes the difficulties of low electrical conduction, particularly due to expansion and contraction of the material during charging and discharging of the battery, improved lithium ion transport and mitigation of potentially damaging chemical side reactions by attaching nanosized crystals or nanosized layers of lithium ion active material to the surface of discrete, functionalized and well dispersed carbon nanotubes.

[0007] Likewise, for the lithium battery anode materials, active anode materials, such as carbon particles, tin oxide or silicon, can be attached to the discrete carbon nanotube surfaces to provide numerous benefits such as improved capacity, electron and ion conductivity and mechanical strength.

[0008] In addition to the discrete carbon nanotube network providing support and spatial stabilization of nanosized particles or layers for cathode or anode material of the lithium battery, other benefits include improved heat transfer media to avoid thermally-induced runaway, structural strength to the paste during manufacturing and high surface area per weight of lithium ion active material to provide good energy density. The uniform dispersion of discrete tubes will also provide a more uniform voltage gradient across the cathode or anode layer, therefore reducing

the probability of locally high electrically resistive regions that can cause accelerated decay of performance in that local region.

SUMMARY

[0009] One aspect of the invention is a composition useful for lithium ion batteries, comprising discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers. For a cathode, the lithium ion active material comprises a lithium metal salt which may further comprise iron, manganese, cobalt, copper, nickel, vanadium, titanium, or mixtures thereof. The composition can further comprise the lithium metal salt with an olivine crystal structure. For an anode, the lithium ion active material may comprise carbon, tin, silicon, copper, antimony, aluminum, germanium, titanium or mixtures thereof. The discrete carbon nanotubes of the composition preferably have an aspect ratio of 10 to 500 and oxidation levels from 1 to 15 weight percent of the carbon nanotube. The composition can further comprise graphene or oxygenated graphene. In one embodiment, the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

[0010] Yet another aspect of this invention is a process for the production of discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers, comprising the steps of:

- a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1 to 15 weight percent,
- b) mixing the carbon nanotubes with a high boiling point liquid, optionally with a non-ionic surfactant,
- c) optionally sonicating the carbon nanotube liquid mixture,
- d) adding reagents in correct balance to synthesize the desired lithium salt,
- e) reacting the mixture at the desired temperature to form the lithium salt while sonicating the mixture
- f) separating the solid salts from the liquid; and
- g) drying and annealing under an inert atmosphere the solid discrete carbon nanotubes with attached lithium salts at sufficient temperature to obtain the olivine crystal structure.

[0011] A further aspect of the invention is a process for the production of the discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers, comprising the steps of:

- a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1 to 15 weight percent,
- b) mixing the carbon nanotubes and desired lithium salt in a liquid with surfactant,
- c) sonicating the carbon nanotube/lithium salt liquid mixture,
- d) separating the solid salts from the liquid,
- e) drying and annealing under an inert atmosphere the solid discrete carbon nanotubes with attached lithium salts at sufficient temperature to obtain the olivine crystal structure.

[0012] An additional aspect of this invention is a composition useful for lithium ion batteries comprising discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

[0013] Yet another aspect of this invention useful for lithium ion batteries is a composition comprising oxidized carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers wherein the carbon nanotubes are uniformly dispersed without clustering on a dimension scale less than one micrometer.

[0014] A further aspect of this invention is a composition useful for lithium ion batteries comprising uniformly dispersible oxidized carbon nanotubes having lithium ion active materials attached to their surface, whether ionically or chemically.

BRIEF DESCRIPTION OF THE FIGURES

[0015] Figure 1 depicts lithium salt coated as a nanolayer on discrete carbon nanotubes.

[0016] Figure 2 shows LiMnO_4 with 5% oxidized carbon nanotubes of this invention.

[0017] Figure 3 shows titanium dioxide crystals attached to discrete carbon nanotubes.

DETAILED DESCRIPTION

[0018] The term “carbon nanotubes” as used herein refers to allotropes of carbon with a cylindrical nanostructure. The nanotubes may be single, double or multiwall.

[0019] The term “discrete carbon nanotubes” as used herein refers to carbon nanotubes capable of being separated to give individual tubes.

[0020] The term “graphene” as used herein refers to individual atomic thickness plates of carbon.

[0021] The term “oxidized graphene” as used herein refers to the products of oxidizing graphene plates. The oxidized species are generally at the edges of the graphene plates.

[0022] The term “oxidized carbon nanotubes” as used herein refers to the products of oxidizing the carbon nanotubes. Generally the oxidized species are carboxylic, ketone or hydroxyl functionalities.

[0023] The term “weight percent” or “percent weight” or “% by weight” as used herein is calculated by dividing the weight of a reagent by the total weight of a mixture to which it is added subsequent to the addition of the reagent. For example, adding 1 gram of a reagent A to 99 grams of a reagent B, thereby forming 100 grams of a mixture A+B would constitute adding 1 weight percent of the reagent A to the mixture.

[0024] During the process of making discrete carbon nanotubes (which can be single, double and multiwall configurations), the nanotubes are cut into segments and residual catalyst particles that are interior to the carbon nanotubes as received from the manufacturer are dissolved. This cutting of the tubes helps with exfoliation. The cutting of the tubes reduces the length of the tubes into carbon nanotube segments that are defined here as Molecular Rebar. Proper selection of the carbon nanotube feed stock related to catalyst particle type and distribution in the carbon nanotubes allows more control over the resulting individual tube lengths and overall tube length distribution. A preferred selection is where the internal catalyst sites are evenly spaced and where the catalyst is most efficient. The selection can be evaluated using electron microscopy and determination of the exfoliated tube distribution. Molecular Rebar gives advantageous mechanical

and transport properties when added to other materials compared to materials with no Molecular Rebar.

Types of migratable ions and ion active materials

[0025] The ion active material which can be attached to the discrete carbon nanotubes is defined here as meaning an ion can migrate into or out of the ion active material thereby exchanging electrons. The migrating ion is preferably lithium ion, and is preferably 50 percent by mole of the available migratable ions, more preferably 75 mole percent or higher, most preferably 95 mole percent or higher and can be as high as 100 mole percent of the total migratable ions. Other migratable ions can include magnesium, aluminium, iron, zinc, manganese, sodium, potassium, lead, mercury, silver, gold, nickel, cadmium and the like.

[0026] Examples of ion active materials are lithium iron phosphate, lithium manganese phosphate, lithium cobalt oxide, silica, manganese dioxide, cadmium, titanium dioxide and carbon.

[0027] The ion active materials can be the same or comprise different active materials. Further, ion species in these ion active materials can be the same, or they can be different. When using a mixture of migratable ions, it is preferred that the lithium ions comprise the majority of the ions of the mixture. Mixtures of migratable ions, when used, can be of 2 different ions, or can be three or more different ions in various proportions. In addition, the ion mixture can be varying valence (or oxidation) states of the same ion, such as Fe^{2+} or Fe^{3+} .

[0028] When mixing ion active materials for attachment to the carbon nanotubes, the mixture may be merely accidental, such as that resulting from either natural states of the ion active material compounds, or the mixture can be an intentional mixing of the different ion active materials, including those in differing chemical or physical state. In some cases comprising migratable ion mixture using lithium ion, the lithium ion may comprise the minority ionic component, of the mix, although this is not usually the preferred configuration. When more than one ion is used, then the ratio of Li ion to other total ions is preferably at least 2:1, or 3:1 or 10:1 and can be as high as 25:1 (mole ratios).

[0029] The ion active material attached to the discrete carbon nanotubes can also be useful for applications such as fuel cells, sensors, capacitors, photovoltaics and catalysts.

Experimental Examples

[0030] The following experimental examples are included to demonstrate particular aspects of the present disclosure. It should be appreciated by those of ordinary skill in the art that the methods described in the examples that follow merely represent illustrative embodiments of the disclosure. Those of ordinary skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments described and still obtain a like or similar result without departing from the spirit and scope of the present disclosure.

EXAMPLE 1

Discrete carbon nanotubes with attached copper oxide nanoparticles suitable for a lithium ion battery anode material

[0031] 102 mg of multi-wall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and aspect ratio about 60 are added to 100 mg copper sulfate, 640 mg sodium EDTA, 15 mg of polyethylene glycol, 568 mg of sodium sulfate and 60 mL of deionized water. The mixture is sonicated for 10 minutes and then heated to 40°C. 3 mL of formaldehyde (37% solution) and 500 mg of sodium hydroxide is added to bring the pH to 12.2. The mixture is stirred for 30 minutes at 85°C and then filtered using a 5 micron PVDF filter and washed with 200 mL of deionized water. The resultant coated multiwall tubes show a coppery hue color. An electron micrograph shows copper oxide particles of diameter about 50 to 150 nm interconnected by well-dispersed carbon nanotubes.

EXAMPLE 2

Discrete carbon nanotubes with lithium iron phosphate coated on the surface of the tubes, suitable for a lithium ion battery cathode material

[0032] 0.2 grams of multi-wall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and aspect ratio about 60 are added to 23.5 grams of diethylene glycol and sonicated for 15 minutes. The solution is grey. 1.16 grams of iron acetate dissolved in 4 ml of

deionized water is added and the mixture heated under nitrogen, stirring and sonication. After 1 hour at 140°C a solution of 0.65 grams of lithium acetate and 0.74 grams of ammonium phosphate in 4.2 ml of deionized water is added. The temperature is raised to about 185°C over 30 minutes, then held at 185°C for 4 hours. The mixture is cooled to 110°C and sonicated for 5 minutes followed by further cooling to ambient temperature while stirring. The solids are separated from the mixture by centrifugation followed by ethanol washing. The resultant powder is dried to constant weight *in vacuo*.

[0033] The resultant dried powder is determined to have a resistance of 2,000 ohm-cm compared to a commercially obtained sample determined as 19,000 ohm-cm.

EXAMPLE 3

[0034] Lithium iron phosphate obtained commercially is ground with multiwall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and of aspect ratio about 60, in a mortar and pestle at weight ratios of 5:1 and 1:1 lithium salt to carbon nanotube. Sodium dodecyl sulfate (SDS) is then added at a weight ratio of 0.7:1 compared to the carbon nanotube and deionized water added to resultant mixture to give a 0.5 weight percent solution of carbon nanotubes in the presence of lithium salt and surfactant. The mixture is sonicated for 1 hour, then the solids filtered using a polyvinylidene fluoride filter (0.2 micron), washed with deionized water and dried. A micrograph of the discrete carbon nanotubes coated with lithium iron phosphate is shown in Figure 1. The results of the resistance of the powders are given in Table 1, units of resistance are ohm-cm.

Table 1

Material	5:1 Weight ratio Lithium salt:carbon nanotube	1:1 Weight ratio Lithium salt:carbon nanotube
Dry mix	860	26
Coated	46.2	6.9
Improvement of coating	18.6 X	3.8X

EXAMPLE 4**Preparation of lithium manganese phosphate in the presence of five percent by weight oxidized carbon nanotubes**

[0035] 37.439g manganese II acetate is dissolved in 5.0ml of water and added to 589mL of diethylene glycol (DEG) already in the reaction vessel. The mixture is then heated to a target temperature of 140°C, under stirring and gentle nitrogen flow to distill off the water. 1.119g of oxidized carbon nanotubes (originally CNano, grade C9000, average diameter 13 nm) nanotubes having an oxidation of 4% and average aspect ratio of 60 are dispersed in 222.2 mL diethylene glycol (0.5% w/v) using sonication while stirring for 30 minutes. Additionally, 14.4434g lithium acetate and 16.4497g ammonium dihydrogen phosphate are dissolved in 90 mL of water. The dispersion of oxidized carbon nanotubes, lithium acetate and ammonium dihydrogen phosphate are added to the reaction medium while stirring, and with sonication. Heating is increased to maintain a target temperature of 180 degrees centigrade for 3 hours, while maintaining constant nitrogen flow. Following the end of the reaction, heating and sonication are turned off while stirring and nitrogen are maintained as the system cools to room temperature.

[0036] The product is a highly viscous, dark-grey colored fluid. The carbon nanotube coated with lithium manganese phosphate is centrifuged and the solids washed with anhydrous ethanol three times. An electron micrograph of the product is shown in Figure 2. In figure 2 the majority of tubes are shown to have coatings of lithium manganese phosphate about 20-40nm thick. The coatings are not smooth as seen in Example 1.

EXAMPLE 5**A composition useful as an anode active material in lithium batteries - *In situ* synthesis of TiO₂-oxidized carbon nanotube composition from titanium isopropoxide as a precursor**

[0037] 0.179g of carbon nanotubes (originally CNano, grade C-9000) oxidized to about 4 % by weight and aspect ratio about 60 is added to 27.3ml NMP and 2.7ml RO water and sonicated for 1 hour to obtain a fully exfoliated solution. A 19% v/v solution is prepared by adding 3.41ml of titanium isopropoxide (Sigma-Aldrich) to 18ml of isopropanol (Ultra Pure Solutions, Inc.) and

Page 12 of 16

is added dropwise with stirring to the exfoliated carbon nanotube dispersion while under sonication. The reaction vessel is then sealed and gently heated to slightly above room temperature for 1 hour. Sonication and a medium stirring speed are maintained over the entire duration of the reaction. The reaction products are obtained by filtering with a Buchner filter under vacuum, washing with ethanol to remove NMP and drying *in vacuo* to constant weight. An electron micrograph, Figure 3, shows crystals of titanium dioxide of scale 50-100nm bound to discrete well-dispersed carbon nanotubes.

CLAIMS

What is claimed is the following:

1. A composition useful for lithium ion batteries comprising:
discrete carbon nanotubes, the discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface.
2. The composition of claim 1, wherein the crystals or layers of lithium ion active material comprise a lithium metal salt and an element selected from the group consisting of: iron, manganese, cobalt, copper, nickel, vanadium, titanium, and mixtures thereof.
3. The composition of claim 2, wherein the lithium metal salt has an olivine crystal structure.
4. The composition of claim 1, wherein the crystals or layers of lithium ion active material comprise tin, silicon, copper, antimony, aluminum, germanium, titanium, or mixtures thereof.
5. The composition of claim 1, wherein the discrete carbon nanotubes have an aspect ratio of 10 to 500 and oxidation levels from 1% to 15% by weight of the carbon nanotube.
6. The composition of claim 1, further comprising graphene or oxygenated graphene.
7. The composition of claim 6, wherein the weight ratio of grapheme to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.
8. A process for the production of the discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, comprising the steps of:
 - a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% percent by weight;
 - b) mixing the carbon nanotubes with a high boiling point liquid;
 - c) adding reagents in correct balance to synthesize a lithium salt;
 - d) reacting the mixture at a temperature to form the lithium salt while sonicating the mixture;

Page 14 of 16

- e) separating the solid discrete carbon nanotube salts from the liquid; and
 - f) drying and annealing the solid discrete carbon nanotube salts under an inert atmosphere at sufficient temperature to obtain an olivine crystal structure.
9. A process for the production of the discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, comprising the steps of:
- a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level of 1% to 15% by weight;
 - b) mixing the carbon nanotubes and a lithium salt in a liquid with surfactant;
 - c) sonicating the liquid carbon nanotube/lithium salt mixture;
 - d) separating the solid salts from the liquid; and
 - e) drying and annealing the solid discrete carbon nanotubes with attached lithium salts under an inert atmosphere at sufficient temperature to obtain an olivine crystal structure.
10. A composition useful for lithium ion batteries, comprising discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.
11. A composition useful for lithium ion batteries, comprising oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface, wherein the carbon nanotubes are uniformly dispersed without clustering on a dimension scale less than 1 micrometer
12. A composition useful for lithium ion batteries, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.
13. The composition of claim 12, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

ABSTRACT

[0038] Compositions, and methods of obtaining them, useful for lithium ion batteries comprising discrete oxidized carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers. The composition can further comprise graphene or oxygenated graphene.

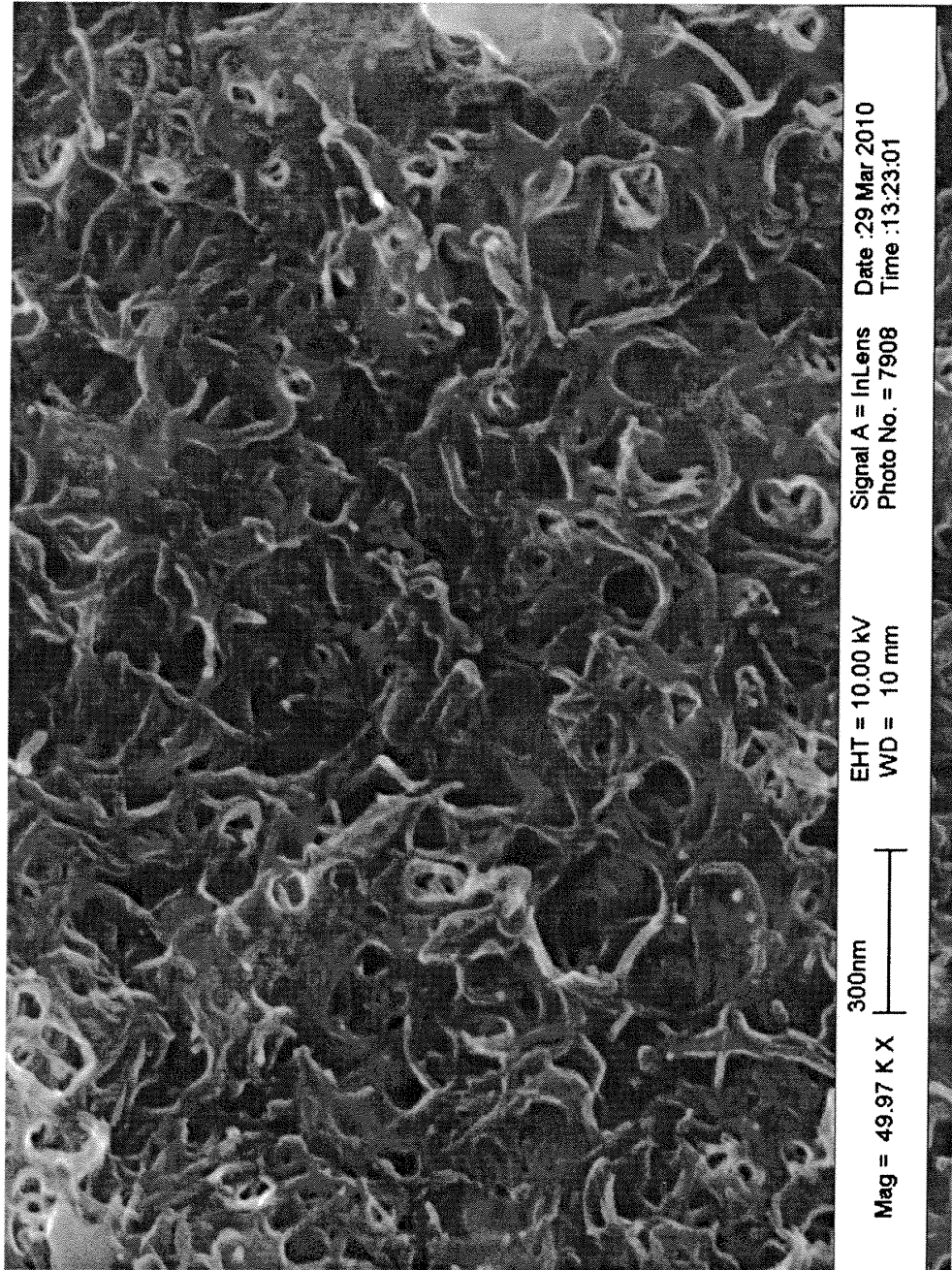


Figure 1

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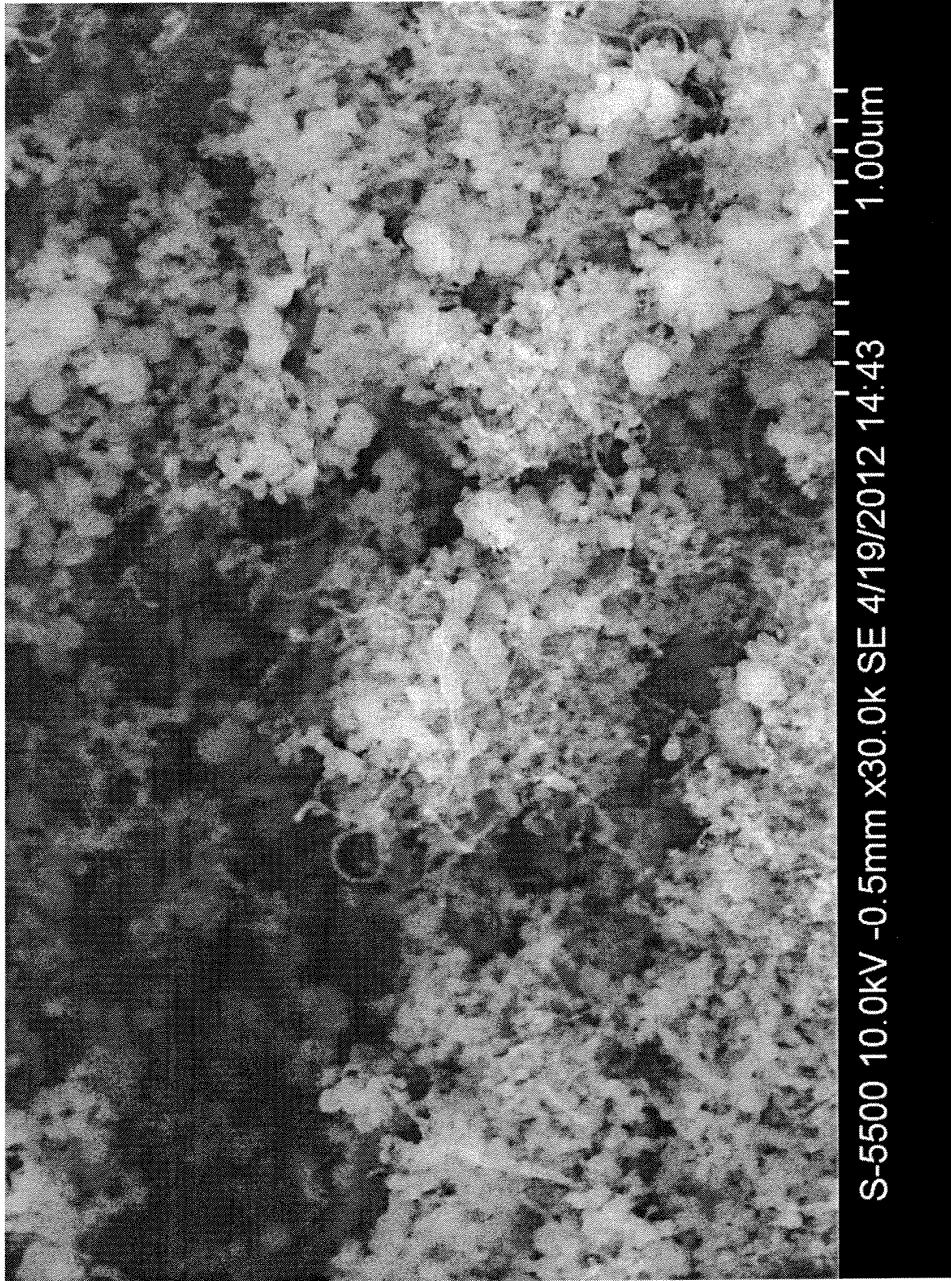


Figure 2

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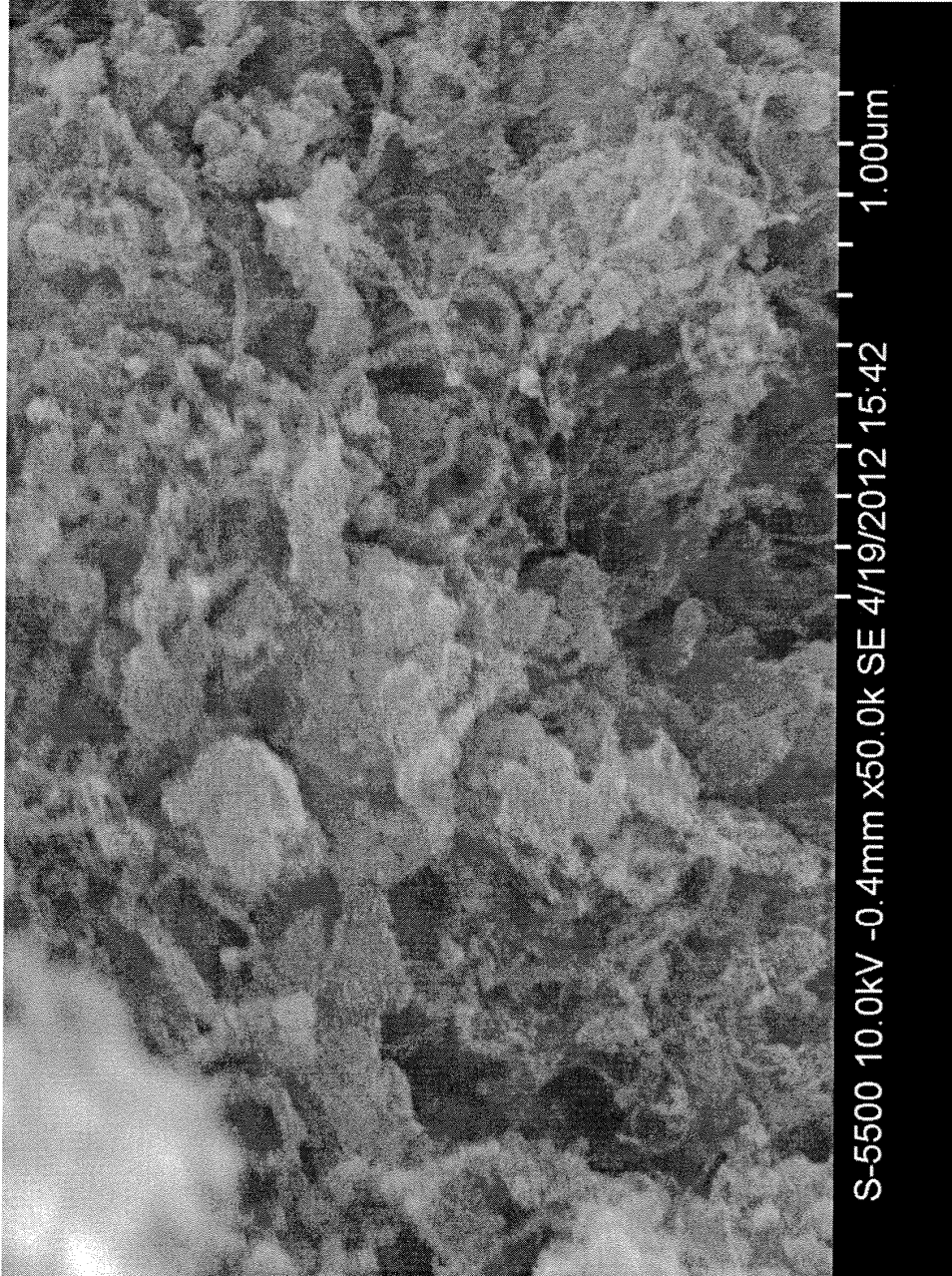


Figure 3

8237993v.1

Electronic Patent Application Fee Transmittal				
Application Number:				
Filing Date:				
Title of Invention:		IMPROVED LITHIUM BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM		
First Named Inventor/Applicant Name:		Clive P. Bosnyak		
Filer:		Amy L. Ulfers/Debra Pepper		
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Utility under 35 USC 111(a) Filing Fees				
Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:				
Utility filing Fee (Electronic filing)	4011	1	95	95
Utility Search Fee	2111	1	310	310
Utility Examination Fee	2311	1	125	125
Pages:				
Claims:				
Independent claims in excess of 3	2201	3	125	375
Miscellaneous-Filing:				
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 (\$)				905

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First Named Inventor/Applicant Name:	Clive P. Bosnyak
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Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
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1	Transmittal of New Application	DENA0008TRANSMITTAL.pdf	388250 f83a440807696fcb8919ab051fa6935d707e3480	no	2
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Information:					
2	Application Data Sheet	DENA0008ADS.pdf	1022835 c4a04497a60c157021b51b15caf645cd48e0c6	no	4
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3		DENA0008USAPPLICATION.pdf	3901317 868dfde8092b356913e7162c2d585160e8427319	yes	19
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	Claims		14	15	
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