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Pepper Cano, Emmanuel

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
20.03.2019

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

Communication to the parties concerning termination of opposition proceedings

No appeal against the Opposition Division's rejection of the opposition(s) dated 03.12.18 has reached the files within the time limit for appeal.

The opposition proceedings are accordingly terminated and the European patent is maintained unamended.

For the Opposition Division

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Pepper Cano, Emmanuel

Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date: 20.03.2019

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

Communication to the parties concerning termination of opposition proceedings

No appeal against the Opposition Division's rejection of the opposition(s) dated 03.12.18 has reached the files within the time limit for appeal.

The opposition proceedings are accordingly terminated and the European patent is maintained unamended.

For the Opposition Division

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Termination of opposition proceedings of patent no. 12731264.3 - 1108/ 2724403 with maintenance of the patent 15.03.19

I. **Findings**

1.1 The opposition proceedings are terminated for the following reason:

☐ The notice of opposition is deemed not to have been filed
There are no other admissible notices of opposition.
LOPR 3 coded and Form 2360 or 2361 despatched to the parties concerned.
OPPO(3) '3' = deemed not to have been filed.

☐ The notice of opposition is inadmissible
There are no other admissible notices of opposition.
LOPR 3 coded and Form 2362 despatched to the parties concerned.
OPPO(3) '2' = inadmissible.

☒ The notice/notices of opposition was/were rejected
REJO 03.12.18
REJO 2 coded and Form 2363 despatched to the parties concerned.

☐ The opposition proceedings were discontinued
OPPC
OPPC 2 coded and Form 2364 despatched to the parties concerned.

☐ The patent was maintained as amended
EPPU03
Form 2350 despatched with the new certificate.

1.2 ☒ The time limit pursuant to Article 108 and Rule 112(2) EPC has expired. No appeals or requests pursuant to Rule 112(2) EPC have been filed.

1.3 ☐ The decision under 1.1 was confirmed by the Board of Appeal.

2. Fees situation:

OPPO04	010	01005679	05.07.17	EUR	785,00

Checked for correct fee payment. Refunds made on . . . . . .

3. Any models still to hand were returned on . . . . . .

4. Disposal of paper file.

15.03.2019
. . . . . .
Date

Pepper Cano, Emmanuel
. . . . . .
Formalities officer

EPO Form 2357.2 02.15

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Art der Sendung /Nature de l'envoi / Type

☐ Brief / Lettre / Letter - Einschreiben / Recommandé / Registered

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03.12.18

RH 53 773 266 5DE

V.O.
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PAYS-BAS

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Anmeldung Nr./Application No./Demande r
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Bonnet, Claudine
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FRANCE

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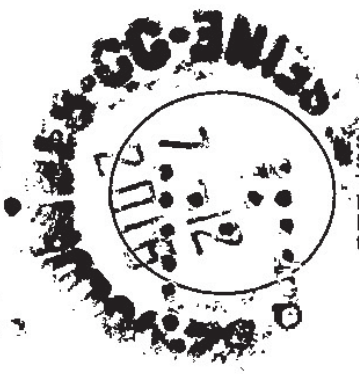
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Sonnenschmidt, Sandra

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
03.12.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

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420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

06 DEC. 2018

Date 03.12.2018

Reference	Application No./Patent No.
AL 0378 /CBL OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

EPA/EPO/OEB Formblatt/Form/Formulaire : 2330 & 2916O

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Sonnenschmidt, Sandra

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Date
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Molecular Rebar Design, LLC	

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420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date
03.12.2018

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Applicant/Proprietor	
Molecular Rebar Design, LLC	

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Application No.: 12 731 264.3

Patent No.: EP-B-2 724 403

Direct Decision:

( ) yes (X) no

Rejection of the opposition (Art. 101(2) EPC)

The Opposition Division has decided:

The opposition(s) filed against the European Patent No. EP-B-2 724 403 is/are rejected.

The Grounds for the decision (Form 2916) are enclosed.

21.11.18
Date

Chairman	1st Examiner	2nd Examiner	Legally qualified member
Götz, Heide	Bettio, Andrea	Kelly, Michael	

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Formalities Officer
Name: Sonnenschmidt-Rogge
Tel: +49 89 2399 - 4748
or call
+31 (0)70 340 45 00

Application No. / Patent No.	Ref.	Date
12 731 264.3 - 1108 / 2 724 403 /	P100028EP00	03.12.2018
Proprietor Molecular Rebar Design, LLC		

Decision rejecting the opposition (Art. 101(2) EPC)

The Opposition Division has decided:

The opposition(s) against the European patent EP-B- 2 724 403 is/are rejected.
The reasons for the decision are enclosed.

Possibility of appeal
This decision is open to appeal. Attention is drawn to the attached text of Articles 106 to 108 and Rules 97 to 98 EPC.

Registered letter with advice of delivery
EPO Form 2330 12.07TRI

Opposition Division:

Chairman:	Götz, Heide
2nd Examiner:	Kelly, Michael
1st Examiner:	Bettio, Andrea



Sonnenschmidt-Rogge
Formalities Officer
Tel. No.: +49 89 2399-4748

Enclosure(s): 10 page(s) reasons for the decision (Form 2916)
Wording of Articles 106 - 108 and Rules 97 - 98 EPC (Form 2019)
claims of the patent as granted

to EPO postal service: 28.11.18

Registered letter with advice of delivery
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I. Summary of Facts and Submissions

European patent 2 724 403 having the title "LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM" is based upon European patent application No. 12 731 264.3 filed on 21-06-2012. It claims priority of US 201161500560 filed on 23.06.2011.

The mention of the grant of the patent has been published in the European Patent Bulletin of 05.10.2016. Proprietor of the patent is Molecular Rebar Design, LLC, 13477 Fitzhugh Road Austin, TX 78736 US.

Notice of opposition has been filed by ARKEMA FRANCE, Département Propriété Industrielle 420, rue d'Estienne d'Orves 92700 Colombes FR on 05.07.2017.
The opponent (O) requested revocation of the patent in its entirety based on to Art. 101(2) EPC for lack of novelty and inventive step (Art. 100(a) EPC), lack of sufficiency of disclosure in the sense of Art 83 EPC (Art. 100(b) EPC) and extension of subject-matter (Art. 100(c) EPC). O additionally requested oral proceedings according to Art. 116 EPC.

The following evidence has been submitted within the opposition period:

A1: US61/500,560 priority document

A2: WO2011/117530 A9, filed on 22.03.2011 published on 29.09.2011

A3: WO95/07551, A1 published on 16.03.95

A4: WO2010/117392 A1, published on 14.10.2010

A5: US 2011/0104551 A1, published on 05.05.2011

A6: US2009/0185327A1, published on 23.07.2009

A7: WO2009/127901 A1, published on 22.10.2009

A8: US2011/0121240 A1, published on 26.05.2011

In a letter received on 19.12.2017, the proprietor requested the rejection of the opposition (Article 101(2) EPC) and maintenance of the patent as granted. Oral proceedings in the sense of Art. 116 EPC have been requested as auxiliary measure.

On 07.05.2018 the opposition division issued a summons to attend oral proceedings on 15.11.2018. In the attached communication the opposition division expressed the preliminary opinion that the patent as granted complied with the requirements of Art. 54 EPC, Art. 56 EPC, Art. 83 EPC and Art. 123(2) EPC.

On 06.09.2018 O submitted further arguments concerning the non-compliance with the requirements of Art. 56 EPC and Art. 83 EPC of the claims as granted and filed document

A9: WO94/25268 A1, published on 10.11.1994.

On 14.09.2018 P filed 2 sets of claims representing auxiliary requests I and II.

On 07.11.2018 O submitted that the opponent will not be represented at oral proceedings on 15.11.2018.

On 14.11.2018 oral proceedings were cancelled by the opposition division.

The decision is based on the following request:

Claims 1-12 as granted
The claims under consideration are attached to this decision. As to the other application documents, reference is made to the file.

II. Reasons for the Decision

The opposition is admissible because it meets all the requirements of Articles 99(1) and 100 EPC and Rules 3(1) and 76(1)(2) EPC.

Art. 100(a) EPC

Priority

The opponent (O) with the notice of opposition has submitted that claim 1 of the opposed patent did not benefit of the right to priority from application US 61/500,560 (A1) filed on 23.06.2011 because claim 5 of said US application disclosed carbon nanotubes (CNT) having an aspect ratio of 25 to 500 and not of 10 to 500 as in granted claim 1.

The opposition division considers that according to G1/15 claim 1 of the opposed patent enjoys the right to partial priority for the aspect ratio range of 25-500 because the subject matter of claim 1 of the opposed patent, as far as the aspect ratio 25-500 is considered, is clearly and unambiguously disclosed in claim 5 of A1.

With regard to opposed claims 2-5 the opposition division considers that the additional features disclosed in said claims are disclosed in claims 2-4 and 6 of A1.

As claims 2-6 of A1 are depending only on claim 1 and there is no interdependency of the claims from each other, the priority cannot be entirely acknowledged for the subject matter of opposed claims 2-5; the priority is partially valid only for iron and copper in opposed claim 2, and for copper in opposed claim 4 with regard to the range of aspect ratio of 25-500 because said three embodiments find a basis in examples 1, 2 and 3 of A1.

Opposed claims 6-9 do not find any basis in A1 and the priority right for the subject matter of said claims cannot be acknowledged.

Opposed claims 11 and 12 find a basis in claims 7 and 8 of A1 and the priority for said claims is partially valid for the range of aspect ratio of 25-500.

Art. 54 EPC

In the notice of opposition O has referred to the passages on page 8 line 7-9 and 24-27, page 20 line 24-page 21 line 21, page 22 lines 20-31 of A2 to conclude that claim 1 of the opposed patent was not novel over A2. O further submitted that opposed claims 2 and 4 were not novel in view of the teaching of page 20 line 24-page 21 line 21 of A2.

The same applied to opposed claim 7 in view of the teaching of example 2 of A2 and opposed claim 9 in view of the teaching on page 22 line 20-31 of A2.

The opposition division considers that A2 aims at providing ready-to-use master batches suitable for the immediate employ in the fabrication of electrodes in order to increase the electric conductivity (page 4, lines 26-31 of A2).

A2 teaches master batches in form of a solid agglomerate comprising nanofibers and/or carbon nanotube and/or carbon black, at least one solvent and at least one binding polymer (page 7, lines 5-15 of A2).

The teaching of the passage on page 22 line 20 to page 23 line 9 of A2 is that it is possible to distribute the carbon nanofibers and nanotube in a way that they form a mesh around the active material, said active material being selected among i) transition metal oxides having a spinel structure, ii) transition metal oxides having a lamellar structure, iii) polyanionic lithium oxides, iv) vanadium based oxides, v) graphites and vi) titanates (page 20 line 24-page 21 line 25 of A2).

A2 therefore does not teach discrete carbon nanotubes as required by the wording of opposed claim 1.

Another difference with the respect of the subject matter claimed in the opposed patent is that, despite the mention on page 8 line 24-27 of A2 that the nanotubes may be oxidized, A2 does not disclose any range of oxidation levels of the CNT.

The subject matter of the opposed claims is therefore novel in the sense of Art 54 EPC over A2.

Art. 56 EPC

In the notice of opposition O has argued that the technical effect deriving from the features of the aspect ratio and the oxidation degree had not been demonstrated in the opposed patent and therefore it could not be taken into account for the assessment of inventive step.

According to O the subject matter of opposed claim 1 was not inventive over A2.

O continued that as the claimed priority was not valid, A2 represented a prior art document in the sense of Art 54(2) EPC. Even if the novelty over A2 was acknowledged, the features of the aspect ratio and the oxidation degree were at least suggested by the general teaching of A2, which taught the advantages deriving from the association of CNT with particles of active material. O argued that P had not provided any comparative test demonstrating that the CNT of A2 did not show the claimed features and were not suitable to solve the technical problem.

O also submitted that opposed claim 1 lacked inventive step in view of the teaching of A3 which disclosed carbon fibrils corresponding to the CNT of the opposed patent.

Furthermore O submitted that opposed claims 1-12 were not inventive in view of the disclosure of A4; even if said document did not explicitly teach steps f) and e) of opposed claims 11 and 12 respectively, the use of inert atmosphere was obvious to the skilled person and the olivine crystal structure in the opposed had not been demonstrated.

For the same reasons used in the previous attacks, according to O even if A5 did not explicitly disclose the aspect ratio and the oxidation degree, opposed claims 1-10 lacked inventive step in view of A5.

Without specifying which of the opposed claims were concerned, O submitted that there was a lack of inventive step in view of A6, A7 and A8 because said documents taught CNT attached to active materials for lithium ion batteries.

With letter dated 06.09.2018 O has submitted that A4 was to be considered as the closest prior art and that the distinguishing features as identified by opposition division in the preliminary opinion had no technical effect; the objective technical problem was therefore the provision of an alternative composition suitable for Li-ion batteries.

Furthermore based on the teaching of paragraph 32 of A4, nothing would have prevented the skilled person from using oxidized CNT instead of the nanotubes disclosed in A4 and working in a range of oxidation 1-15% could result from the routine experimentation aimed at improving the composition.

The same reasoning could be applied to A3.

The opposition division observes that the problem to be solved by the invention underlying the opposed patent is how to overcome the difficulties of low electrical conduction, how to improve lithium ion transport and mitigate potentially damaging chemical side reactions in lithium ion batteries (paragraph 8 of the opposed patent).

A4 is the most promising starting point to reach the solution proposed by the opposed claims because it aims at improving the properties of carbon nanotubes in applications including energy storage devices (paragraph 7 of A4).

Furthermore among the cited documents A4 is the only one to deal with exfoliated carbon nanotubes and methods for preparing exfoliated carbon nanotubes (claims 1 and 11 of A4) by use of nanoplates of zirconium phosphate treated with a surfactant and sonication in aqueous media (paragraph 49 of A4). The obtained CNT after centrifugation and separation exist in a disordered **but not aggregate state** (paragraph 49 of A4).

For the above mentioned reasons the opposition division considers A4 as the closest prior art.

A4 teaches that in some embodiments the exfoliated CNT may be incorporated in electrodes of energy storage devices and in some embodiments electroactive nanomaterial selected from a list comprising $Li_4Ti_5O_{12}$ may be bound to the exfoliated carbon nanotubes of said active material (paragraph 82 A4).

Paragraph 42 of A4 discloses that the carbon nanotubes have an aspect ratio of least (*sic*) about 100 in some embodiments and at least about 1000 in other embodiments.

Example 5 (paragraph 97) of A4 deals with the preparation of exfoliated CNT and teaches that 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. This disclosure corresponds to a range of aspect ratio from 50 to 100 or higher.

The CNT of example 5 however were not modified with crystals or layers of lithium ion active material as required by opposed claim 1.

None of the examples of A4 refers to exfoliated CNT being modified with crystals or layers of lithium ion active material as required by opposed claim 1.

From said observations the opposition division concludes that the teachings of paragraph 42 (aspect ratio) and paragraph 82 (modification with lithium active material) relate to different embodiments of A4 and do not have to be necessarily combined.

The disclosure of paragraph 82 of A4 read in combination with paragraph 8 and claim 1 of A4 represents therefore the closest prior art.

The exfoliated CNT composition of said disclosure differs from the subject matter of opposed claim 1 because it does not disclose the following features:

i. the aspect ratio of 10 to 500

ii. the oxidation level from 1% to 15% by weight of the carbon nanotubes

Paragraph 6 of the published application underlying the opposed patent teaches that "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."

Paragraph 8 of said published application discloses that "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."

Paragraph 9 of said application teaches that "...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 nanotubes".

The opposition division considers that from said passages the skilled reader realizes that the discrete carbon nanotubes of the invention have a uniform dispersion because their aspect ratio is comprised between 10 to 500 and their oxidation level ranges from 1 to 15 weight percent; said "uniformity" confers the technical effect of preventing the accelerated decay of performance in local regions of the electrode.

As said technical effect is not disclosed in A4, no experimental evidence is required for concluding that the subject-matter of opposed claim 1 solves vis-à-vis the closest prior art A4 the objective technical problem of providing a CNT composition suitable for lithium ion batteries and capable of preventing accelerated decay of performance in local regions of the electrode (see T692/09 r. 2.4.3).

None of the documents on file mentions the problem of preventing accelerated decay of performance in local regions of the electrode.The skilled person has therefore no incentive to modify the CNT compositions of A4 so as to obtain a uniform composition having an aspect ratio of 10 to 500 and an oxidation level from 1% to 15% by weight of the carbon nanotubes.

The subject matter of opposed claim 1 is therefore inventive in the sense of Art. 56 EPC.

The processes of Opposed claims 11 and 12 contain the step (step a) in both claims) of selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level of 15 to 15% by weight.

Thus the subject matter of said process claims is also inventive in the sense of Art. 56 EPC for the same reasons outlined for claim 1.

Art 100(b) EPC

in the notice of opposition O has substantiated the lack of sufficiency disclosure using three line of argumentation:

1) the opposed patent did provide the skilled person with neither the indication concerning the method for determining the oxidation level of the CNT nor the teaching of how to perform the selection of the CNT with respect to the aspect ratio.

Furthermore the opposed patent did not disclose how to obtain the oxidized CNT especially with regard to the oxidation level disclosed in claim 1.

2) The patent did not provide any information concerning the selection of the size of the crystals or layers of active material that had to be attached to the surface of the CNT: the skilled person could only base himself on the teaching of the examples of the opposed patent but he had no guidance to obtain the crystals and the layers of active material having the required size.

3) In step b) of claim 11 the opposed patent disclosed a “high boiling point liquid” but the skilled person was not able to select such a liquid; furthermore a high number of experiments in order to correctly adjust the temperature and reagents in steps c) and d) was necessary. Furthermore the skilled person did not know at which temperature the annealing process in step f) of opposed claim 11 had to be performed.

Furthermore O has argued that the terms “liquid” and “surfactant” in step b) of claim 12 were vague and the description did not provide any indication allowing the skilled person to identify the liquids and the surfactants which were suitable to carry out said step.

In the letter of 06.09.2018 O has remarked that examples 1 and 5 of the patent did not fall under the subject matter of claim 1 and, referring to paragraph 22 of the opposed patent, submitted that the skilled person willing to carry out the invention did not know how to express the presence of the oxidized species mentioned in paragraph 22. There were many experimental techniques allowing to quantify the oxidation degree and the skilled person could not know whether he worked under the scope of protection of the opposed patent.

The opposition division observes that the opposed patent contains 5 different examples; as O has not provided any evidence or argumentation of the contrary, the opposition division considers that even if only examples examples 2, 3 and 4 fall within the scope of the claims, they all are reproducible.

In examples 1-5 of the opposed patent commercially available multi-wall carbon nanotubes (CNano, grade C9000) are employed. Said nanotubes are disclosed as being oxidized at about 8 percent and having an aspect ratio of about 60 in examples 1-3 whilst in examples 4 and 5 the same value of aspect ratio of about 60 and an oxidation level of 4% are disclosed.

Paragraphs 22 and 24 of the opposed patent teach the meaning of the term “oxidized carbon nanotubes” and the of definition “weight percent”.

With regard to point 1) of the opponent´s argumentation, the opposition division considers that the patent provides sufficient guidance to the skilled person to carry out the invention and the fact the two parameters of the aspect ratio and level of oxidation may not be clearly defined does not represent a ground for opposition in the sense of Art 100(b) EPC but rather a matter to be discussed under Art 84 EPC.

Furthermore the carbon oxidation is a well established chemical process which belong to the common general knowledge of every organic chemist. The fact that different analytical methods are available to the skilled person in order to determine the level of carbon oxidation does not mean that this parameter cannot be

unambiguously determined because the results obtained by use of different analytical methods on the same sample are compatible within the margins of experimental error of each analytical method.

With regard to point 2) the opposition division considers that the formation of crystals or layers of lithium ion active material attached to the surface of the CNT results from the processes described in the examples of the opposed patent and the skilled person has no need to select the size of the crystals or layers of lithium ion active material in order to carry out the invention.

With regard to point 3) the opposition division considers that the examples of the opposed patent provide sufficient guidance with regard to the liquid solvents, the annealing temperature and the surfactants to be used and no undue experimentation is necessary to the skilled person to adjust and adapt to each other said features.

Thus the subject matter of the claims of the patent as granted complies with the requirements of Art 83 EPC.

Art. 100(c) EPC

O has submitted that claims 5-10 of the opposed patent were dependent on the previous claims, which meant that the claims as granted disclosed combinations of features which were not originally disclosed in the application as filed; in the filed application said features were disclosed as separated and not in combination.

The opposition division considers that the teaching of paragraph 9 of the application as published represents a basis for claim 5 as granted. Said paragraph 9 teaches in fact 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 preferably the discrete carbon nanotubes of the composition have an aspect ratio of 10 to 500 and oxidation levels from 1 to 15 weight percent and wherein the composition can further comprises graphene or oxygenated graphene.

The additional features of granted claim 6 find a basis in the last sentence of paragraph 9, and those granted claims 7-10 in paragraphs 12, 13 and 14 respectively of the published application.

The patent as granted therefore complies with the requirements of Art 123(2) EPC.

III. Decision

The grounds of opposition do not prejudice maintenance of the patent as granted.
The opposition is rejected (Art. 101(2) EPC).

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Application No.: 12 731 264.3

Patent No.: EP-B- 2 724 403

A copy of the communication (communication, decision, minutes) was printed for and notified to each of the following representatives/parties:

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Article 106
Decisions subject to appeal

(1) An appeal shall lie from decisions of the Receiving Section, Examining Divisions, Opposition Divisions and the Legal Division. It shall have suspensive effect.

(2) A decision which does not terminate proceedings as regards one of the parties can only be appealed together with the final decision, unless the decision allows a separate appeal.

(3) The right to file an appeal against decisions relating to the apportionment or fixing of costs in opposition proceedings may be restricted in the Implementing Regulations.

Rule 97
Appeal against apportionment and fixing of costs

(1) The apportionment of costs of opposition proceedings cannot be the sole subject of an appeal.

(2) A decision fixing the amount of costs of opposition proceedings cannot be appealed unless the amount exceeds that of the fee for appeal.

Rule 98
Surrender or lapse of the patent

The decision of an Opposition Division may be appealed even if the European patent has been surrendered in all the designated Contracting States or has lapsed in all those States.

Article 107
Persons entitled to appeal and to be parties to appeal proceedings

Any party to proceedings adversely affected by a decision may appeal. Any other parties to the proceedings shall be parties to the appeal proceedings as of right.

Article 108
Time limit and form

Notice of appeal shall be filed, in accordance with the Implementing Regulations, at the European Patent Office within **two months** of notification of the decision. Notice of appeal shall not be deemed to have been filed until the fee for appeal has been paid. Within **four months** of notification of the decision, a statement setting out the grounds of appeal shall be filed in accordance with the Implementing Regulations.

Further information concerning the filing of an appeal

(a) Notice of appeal can be filed in accordance with Rule 1 and Rule 2(1) EPC, by delivery by hand, by post, or by technical means of communication. The filing has to comply with the details and conditions and, where appropriate, any special formal or technical requirements laid down by the President of the European Patent Office (R. 99(3) EPC).

(b) The addresses of the filing offices of the European Patent Office are as follows:

(i) European Patent Office
D-80298 Munich
Germany

Fax: +49 89 2399-4465

(ii) European Patent Office
Postbus 5818
NL-2280 HV Rijswijk (ZH)
The Netherlands

Fax: +31 70 340-3016

(iii) European Patent Office
D-10958 Berlin
Germany

Fax: +49 30 259 01-840

(c) The notice of appeal must contain the name and address of the appellant in accordance with the provisions of Rule 41(2)(c) EPC, an indication of the decision impugned, and a request defining the subject of the appeal. In the statement of grounds of appeal the appellant shall indicate the reasons for setting aside the decision impugned, or the extent to which it is to be amended, and the facts and evidence on which the appeal is based (R. 99(1) and (2) EPC). The notice of appeal and any subsequent submissions stating the grounds for appeal must be signed (R. 50(3) EPC).

(d) The fee for appeal is laid down in the Rules relating to Fees. The schedule of fees and expenses of the EPO or a reference to the current version is regularly published in the Official Journal of the European Patent Office under the heading "Guidance for the payment of fees, expenses and prices". Fee information is also published on the EPO website under www.epo.org/ fees.

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 TiO_2-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 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

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, 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.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, 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.

9. The composition of any one of claims 1-8, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

10. The composition of claim 9, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

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a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

12. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Patentansprüche

1. Zusammensetzung, verwendbar für Lithiumionenbatterien, umfassend:

diskrete Kohlenstoffnanoröhren, wobei die diskreten Kohlenstoffnanoröhren Kristalle oder Schichten von Lithiumionen-aktivem Material, befestigt auf ihrer Oberfläche aufweisen, wobei die diskreten Kohlenstoffnanoröhren ein Aspektverhältnis von 10 bis 500 und Oxidationsstufen von 1 Gew.-% bis 15 Gew.-% des Kohlenstoffnanorohrs aufweisen.

2. Zusammensetzung nach Anspruch 1, wobei die Kristalle oder Schichten von Lithiumionen-aktivem Material ein Lithiummetallsalz und ein Element, ausgewählt aus der Gruppe bestehend aus: Eisen, Mangan, Cobalt, Kupfer, Nickel, Vanadium, Titan und Gemischen davon, umfassen.

3. Zusammensetzung nach Anspruch 2, wobei das Lithiummetallsalz eine Olivin-Kristallstruktur hat.

4. Zusammensetzung nach Anspruch 1, wobei die Kristalle oder Schichten von Lithiumionen-aktivem Material Zinn, Silikon, Kupfer, Antimon, Aluminium, Germanium, Titan oder Gemische davon umfassen.

5. Zusammensetzung nach einem der Ansprüche 1-4, ferner umfassend Graphen oder oxygeniertes Graphen.

6. Zusammensetzung nach Anspruch 5, wobei das Gewichtsverhältnis von Graphen zum Kohlenstoffnanoröhren im Bereich von 0,1:99,9 bis 99,9:0,1 liegt.

7. Zusammensetzung nach einem der Ansprüche 1-6, wobei die Kohlenstoffnanoröhren in einem Gewichtsverhältnis von Kohlenstoffnanoröhren zu Lithiumsalzen von 0,1 % bis 10 % vorliegen.

8. Zusammensetzung nach einem der Ansprüche 1-7, umfassend oxidierte Kohlenstoffnanoröhren mit Kristallen oder Schichten von Lithiumionen-aktiven Materialien, befestigt auf ihrer Oberfläche, wobei die Kohlenstoffnanoröhren gleichförmig ohne Clusterbildung auf einer Dimensionsskala von weniger als 1 Mikrometer dispergiert sind.

9. Zusammensetzung nach einem der Ansprüche 1-8, umfassend gleichförmig dispergierbare oxidierte Kohlenstoffnanoröhren mit Kristallen oder Schichten von Lithiumionen-aktiven Materialien, befestigt auf ihrer Oberfläche.

10. Zusammensetzung nach Anspruch 9, wobei die Kristalle oder Schichten von Lithiumionen-aktiven Materialien chemisch auf der Oberfläche der dispergierbaren oxidierten Kohlenstoffnanoröhren befestigt sind.

11. Verfahren zur Herstellung diskreter Kohlenstoffnanoröhren mit Kristallen oder Schichten von Lithiumionen-aktiven Materialien, befestigt auf ihrer Oberfläche, wie in Anspruch 3 definiert, umfassend die Schritte von:

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GESCANL
20 NOV. 2018

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Received with thanks!
21 NOV 2018

By fax in advance on 09.11.2018
To: 0031 70 4166 799

Date
14.11.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403

Applicant/Proprietor
Molecular Rebar Design, LLC

BRIEF COMMUNICATION

Oral Proceedings on 15.11.18

Subject:
- ☐ Letter from the proprietor of the patent of
- ☒ Letter from the opponent 01 of 07.11.2018

Communication:
- ☒ The summons to attend oral proceedings / taking of evidence on 15.11.18 has been cancelled.
- ☒ The procedure will be continued in writing.
- ☐ The date fixed for oral proceedings is maintained.
- ☐ A new date will be set later.
- ☒ Please confirm receipt of fax with date and stamp.

Please take note.

For the Opposition Division

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Registered letter

Received at the EPO on Nov 21, 2018 10:56:07. Page 1 of 1
Duration: 11.21.2018 10:55:47 AM - 11.21.2018 10:56:07 AM. This page 1 of 1 was completed at 11.21.2018 10:56:07 AM

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By fax in advance on 09.11.2018
To. 0033 149 00 80 87

Date
14.11.2018

Reference		Application No./Patent No.
AL 0378	OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor		
Molecular Rebar Design, LLC		

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12 Nov. 2018

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EPO Form 2310A 12.07 (09/11/18)

Received at the EPO on Nov 14, 2018 11:42:02. Page 1 of 1
Duration: 11.14.2018 11:41:42 AM - 11.14.2018 11:42:02 AM. This page 1 of 1 was completed at 11.14.2018 11:42:02 AM

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AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

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By fax in advance on 09.11.2018
To: 0031 70 4166 799

Date
14.11.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Oral Proceedings on 15.11.18

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Communication:
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Date: 13.11.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☒ Oral Proceedings on 15.11.2018

Enclosure(s):
- ☐ Letter from the proprietor of the patent of
- ☒ Letter from the opponent 01 of 07.11.2018
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

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ARKEMA
INNOVATIVE CHEMISTRY

URGENT

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Colombes, le 7 novembre 2018

Objet : Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA France
Procédure orale du 15 novembre 2018

Mesdames, Messieurs,

Nous faisons suite à la convocation à la procédure orale prévue le 15 novembre 2018.

La soussignée vous informe que l'opposant 01, ARKEMA FRANCE, n'y sera pas représenté, et s'en tient aux écrits présentés dans le mémoire d'opposition ainsi que dans son courrier du 6 septembre 2018.

Ce courrier est envoyé également au mandataire du titulaire du brevet.

Veuillez agréer, Mesdames, Messieurs, l'expression de nos salutations distinguées

Claudine BONNEL
PG n° 8646

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European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact
Sonnenschmidt, Sandra

V.O.
P.O. Box 87930
2508 DH Den Haag
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By fax in advance on 09.11.2018.
To: 0031 70 4166 799

Date
14.11.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Oral Proceedings on 15.11.18

Subject:
- ☐ Letter from the proprietor of the patent of
- ☒ Letter from the opponent 01 of 07.11.2018

Communication:
- ☒ The summons to attend oral proceedings / taking of evidence on 15.11.18 has been cancelled.
- ☒ The procedure will be continued in writing.
- ☐ The date fixed for oral proceedings is maintained.
- ☐ A new date will be set later.
- ☒ Please confirm receipt of fax with date and stamp.

- 9 NOV 2018

Please take note.

For the Opposition Division

Europäisches Patentamt · European Patent Office · Office européen des brevets

Registered letter
EPO Form 2310A 12.07 (09/11/18)

Received at the EPO on Nov 09, 2018 16:28:36. Page 1 of 1
Duration: 11.09.2018 04:28:16 PM - 11.09.2018 04:28:36 PM. This page 1 of 1 was completed at 11.09.2018 04:28:36 PM

SENDEBERICHT

ZEIT : 09/11/2018 10:44
NAME : EPA
FAX : +49-89-2399-4465
TEL :
S-NR. : E71294F2J162015

DATUM/UHRZEIT	09/11 10:43
FAX-NR./NAME	00031704166799
Ü.-DAUER	00:00:25
SEITE(N)	01
ÜBERTR	OK
MODUS	STANDARD

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SENDEBERICHT

ZEIT : 09/11/2018 10:45
NAME : EPA
FAX : +49-89-2399-4465
TEL :
S-NR. : E71294F2J162015

DATUM/UHRZEIT	09/11 10:45
FAX-NR./NAME	00033149008087
Ü.-DAUER	00:00:25
SEITE(N)	01
ÜBERTR	OK
MODUS	STANDARD ECM

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By fax in advance on 09.11.2018
To. 0033 149 00 80 87

Date
14.11.2018

Reference		Application No./Patent No.
AL 0378	OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor		
Molecular Rebar Design, LLC		

BRIEF COMMUNICATION

Oral Proceedings on 15.11.18

Subject:
- ☐ Letter from the proprietor of the patent of
- ☒ Letter from the opponent 01 of 07.11.2018

Communication:
- ☒ The summons to attend oral proceedings / taking of evidence on 15.11.18 has been cancelled.
- ☒ The procedure will be continued in writing.
- ☐ The date fixed for oral proceedings is maintained.
- ☐ A new date will be set later.
- ☒ Please confirm receipt of fax with date and stamp.

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Accusé de réception

Par la présente, nous accusons réception des pièces suivantes produites postérieurement au dépôt:

Numéro de soumission	6884936	
Numéro de demande	EP12731264.3	
Date de réception	07 novembre 2018	
Office récepteur	Office européen des brevets, La Haye	
Votre référence	AL 0378	
Nom du demandeur	Tous les déposants mentionnés dans le dossier	
Documents envoyés	package-data.xml epf1038.pdf (1 p.)	ep-sfd-request.xml ORAL-1.pdf\AL 0378 - 2018-11-07 - E - Courrier PO.pdf (1 p.)
Effectué par	CN=Claudine Bonnel 7787	
Méthode de soumission	En ligne	
Date et heure de réception	07 novembre 2018, 14:28 (CET)	
Message condensé	05:D4:5D:D5:66:4E:AE:0F:A3:8F:14:20:A8:F3:B7:3B:06:83:7F:DB	

Correction par l'OEB d'erreurs figurant dans les ordres de débit transmis via eOLF
Les erreurs figurant dans les ordres de débit transmis via eOLF et causées par une modification des informations saisies dans le formulaire 1038E ou une utilisation persistante de logiciels dépassés (tous les formulaires) peuvent être corrigées automatiquement par l'OEB, sans que la date de paiement soit modifiée (cf. décision T 152/82, JO OEB 1984, 301 et point 6.3 s. RCC, supplément au JO OEB 10/2007).

/Office européen des brevets/

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Lettre d'accompagnement relative à des pièces produites ultérieurement

Mandataire:

Mme Claudine BONNEL
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La (Les) pièce(s) désignées ci-après est (sont) produite(s) postérieurement au dépôt, pour la demande suivante :

N° de la demande	12731264.3
Référence du demandeur ou du mandataire	AL 0378

	Description des pièces	Nom du dossier original	Nom du dossier attribué
1	Lettre concernant une procédure orale	AL 0378 - 2018-11-07 - E - Courrier PO.pdf	ORAL-1.pdf

	Paiement	
1	Méthode de paiement	Non spécifié

Signatures

Lieu:	**Colombes**
Date:	**07 novembre 2018**
Signé par:	**Claudine Bonnel 7787**
Nom du mandataire:	**Claudine BONNEL**
Fonction:	**(Mandataire)**

ARKEMA
INNOVATIVE CHEMISTRY

URGENT

ARKEMA France
Direction Recherche Développement
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Colombes, le 7 novembre 2018

Objet : Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA France
Procédure orale du 15 novembre 2018

Mesdames, Messieurs,

Nous faisons suite à la convocation à la procédure orale prévue le 15 novembre 2018.

La soussignée vous informe que l'opposant 01, ARKEMA FRANCE, n'y sera pas représenté, et s'en tient aux écrits présentés dans le mémoire d'opposition ainsi que dans son courrier du 6 septembre 2018.

Ce courrier est envoyé également au mandataire du titulaire du brevet.

Veuillez agréer, Mesdames, Messieurs, l'expression de nos salutations distinguées

Claudine BONNEL
PG n° 8646

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European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
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Département Propriété Industrielle
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92705 Colombes Cedex
FRANCE

Date: 20.09.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☒ Oral Proceedings on 15.11.2018

Enclosure(s):
- ☒ Letter from the proprietor of the patent of 14.09.2018
- ☐ Letter from the opponent of
- ☒ Copy (copies) claims, aux. requests 1-3, clean and amended

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

☒ **Registered letter**

Patents & Trademarks

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European Patent Office
D-80298 Munich
Germany

Your ref. 12731264.3/ 1108
Our ref. HPM/P100028EP00

The Hague,
14 September 2018

Re: European Patent No. 2724403
In the name of Molecular Rebar Design, LLC

With reference to the Summons dated May 7, 2018 and in preparation for the Oral Proceedings scheduled for November 15, 2018, I inform you that I intend to attend these oral proceedings accompanied by Mr Stephen P. Krupp, Patentee's Chief Patent Counsel, and by Dr Clive P. Bosnyak, one of the inventors mentioned on the Patent.

Dr Bosnyak obtained his BSc with Honors in Chemistry with Minors in Mathematics and Physics in 1977 and his Ph.D. in Chemistry from the University of Birmingham, England in 1980. He was adjunct professor at the University of Illinois at Chicago for 8 years, leading research and teaching of polymers as materials in engineering.

From 1981-2006, he held various research positions within The Dow Chemical Company in the field of polymers bridging business needs to new technologies and products. This included the development of new materials and structures for building and construction, novel polyolefins, environmentally friendly blow-moldable styrenic block copolymers, new engineering thermoplastic polyesters, blends, nanocomposites, smart materials, thermosets, composites, films and foams. Special emphasis has been

on the design of polymeric materials, both chemical and physical, for specific process and structure-property relationships. Dr Bosnyak also became a credited trainer for new business opportunity identification and analysis in Marketing & New Business Development. On his retirement from Dow, he was Lead Scientist.

As of 2006, he is co-owner of and Chief Scientific Officer in Molecular Rebar Design, LLC.

He is recognized internationally as an inventor and innovator in the development of new polymeric materials and technologies. He is also a consultant for selected international companies and specializes in innovative technologies and business development. He is a founding member of several start-up companies, including Creative Polymers, LLC, Microlayer Energy, LLC and Planned Innovation. He has coauthored 64 papers and has over 70 patents and patent applications. Dr Bosnyak has presented a seminar by invitation at the US Patent Office on the subject of new advances in Carbon Nanotubes.

Dr Bosnyak will attend the Oral Proceedings as technical expert.

He may, under my responsibility and supervision, provide background information on the technical field of the invention and provide answers to technical questions which the Opposition Division may have.

Mr Krupp received his Bachelor of Science in Chemical Engineering from Washington University in St. Louis, MO, and his Juris Doctorate, *Cum Laude*, from Thomas M. Cooley Law School in Lansing, MI. He is licensed to practice before the USPTO and the CAFC, and is admitted to practice by the State Bars of Missouri and Texas.

Mr Krupp began his career at The Dow Chemical Company in 1981, as a Research Engineer. He held various R&D and TS&D positions in Dow, including co-inventorship on 8 U.S. Patents. He transferred into Dow's Legal Department in 1989, and later graduated from law school. He retired from Dow in 2010 after 29 years combined R&D and legal experience, has his own law practice and has been with Patentee since 2010. As Chief Patent Counsel, he is responsible for filing, prosecuting, coordinating, and executing all matters relating to MRD's global intellectual property protection.

Patentee notes that the preliminary opinion of the Opposition Division appears to be a positive one from our perspective.

However, with an eye on the Rules of Procedure before the Boards of Appeal, which become stricter and stricter, Patentee has decided to file a number of Auxiliary Claim Requests. These Auxiliary Claim Requests are enclosed in a form showing revisions and in retyped form.

Patentee wishes to maintain its right to change the order of its Auxiliary Claim Requests, to combine one or more of these Auxiliary Claim Requests and/or to file further or other Auxiliary Claim Requests.

Auxiliary Claim Request I is based on a combination of claims 1 and 8 as granted. Claim 8 is deleted with the remaining claims being renumbered.

Auxiliary Claim Request II is based on a combination of claims 1 and 7 as granted. Claim 7 as granted is deleted with the remaining claims being renumbered.

Both Auxiliary Request I and II introduce additional limitation in respect of lithium components in claim 1 and bring the claims even further away from the prior art cited by Opponent.

Auxiliary Claim Request III is based on a combination of claims 1 and 9 as granted. I additionally refer to §§ [0014] and [0004] of the application as published and §§ [0016] and [0004] of the Patent as granted. Claim 9 is deleted and the remaining claims are adapted to new claim 1 and deleted claim 9. Where some prior art references on file refer to carbon nanotubes in agglomerated or aggregated state or in the form of assemblages, this Auxiliary Request even brings the claims further away from this prior art.

Finally, I note that with my letter of May 22, 2018, I requested interpretation from French into English in case Opponent Arkema would use the French language during the Oral Proceedings.

The professional representative,



H.A.M. Marsman

Encl.: Auxiliary Claim Request I-III (with revisions and clean version)

Auxiliary Claim Request I

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersed without clustering on a dimension scale less than 1 micrometer.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

9. The composition of claim 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request II

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, 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, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, 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.

8. The composition of any one of claims 1-7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

9. The composition of claim 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request III

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersible.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, 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.

9. The composition of claim 1, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request I

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersed without clustering on a dimension scale less than 1 micrometer.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

~~8. The composition of any one of claims 1-7, 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.~~

~~9.~~8. The composition of any one of claims 1-~~8~~7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

~~10.~~9. The composition of claim ~~9~~ 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request II

1. 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, 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, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

~~7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.~~

~~8.~~7. The composition of any one of claims 1-~~7~~6, 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.

~~9.~~8. The composition of any one of claims 1-~~8~~7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

~~10.~~9. The composition of claim ~~9~~ 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request III

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersible.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, 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.

~~9. The composition of any one of claims 1-8, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.~~

~~10.~~9. The composition of claim ~~9~~ 1, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

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Acknowledgement of receipt

We hereby acknowledge receipt of the following subsequently filed document(s):

Submission number	6713870
Application number	EP12731264.3
Date of receipt	14 September 2018
Receiving Office	European Patent Office, The Hague
Your reference	P100028EP00
Applicant	All applicants as on file

Documents submitted	
package-data.xml	ep-sfd-request.xml
epf1038.pdf (2 p.)	ORAL-1.pdf\P100028EP00 Response written submissions.pdf (4 p.)
CLMS-1.pdf\P100028EP00 Auxiliary Claim Request I (clean version).pdf (3 p.)	CLMS-HWA-1.pdf\P100028EP00 Auxiliary Claim Request I (with revisions).pdf (3 p.)
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CLMS-3.pdf\P100028EP00 Auxiliary Claim Request III (clean version).pdf (3 p.)	CLMS-HWA-3.pdf\P100028EP00 Auxiliary Claim Request III (with revisions).pdf (3 p.)

Submitted by	CN=Huub Maas 34471
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Message Digest	44:FE:26:23:19:76:B0:5C:58:07:66:72:4A:B2:C9:4A:5D:F7:A6:05

Correction by the EPO of errors in debit instructions filed by eOLF

Errors in debit instructions filed by eOLF that are caused by the editing of Form 1038E entries or the continued use of outdated software (all forms) may be corrected automatically by the EPO, leaving the payment date unchanged (see decision T 152/82, OJ EPO 1984, 301 and point 6.3 ff ADA, Supplement to OJ EPO 10/2007).

/European Patent Office/

Auxiliary Claim Request I

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersed without clustering on a dimension scale less than 1 micrometer.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

~~8. The composition of any one of claims 1-7, 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.~~

~~9.~~8. The composition of any one of claims 1-~~8~~7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

~~10.~~9. The composition of claim ~~9~~ 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request II

1. 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, 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, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

~~7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.~~

~~8.~~7. The composition of any one of claims 1-~~7~~6, 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.

~~9.~~8. The composition of any one of claims 1-~~8~~7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

~~10.~~9. The composition of claim ~~9~~ 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request III

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersible.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, 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.

~~9. The composition of any one of claims 1-8, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.~~

~~10.~~9. The composition of claim ~~9~~ 1, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

~~11.~~10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:

a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
f) 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.

~~12.~~11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request III

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersible.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, 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.

9. The composition of claim 1, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request I

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, 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, and wherein the oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surfaces are uniformly dispersed without clustering on a dimension scale less than 1 micrometer.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

8. The composition of any one of claims 1-7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

9. The composition of claim 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

Auxiliary Claim Request II

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, 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, wherein the carbon nanotubes are in a weight ratio of carbon nanotubes to lithium salts of 0.1% to 10%.

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 any one of claims 1-4, further comprising graphene or oxygenated graphene.

6. The composition of claim 5, wherein the weight ratio of graphene to carbon nanotube is in the range of 0.1:99.9 to 99.9:0.1.

7. The composition of any one of claims 1-6, 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.

8. The composition of any one of claims 1-7, comprising uniformly dispersible oxidized carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface.

9. The composition of claim 8, wherein the crystals or layers of lithium ion active materials are chemically attached to the surface of the dispersible oxidized carbon nanotubes.

10. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, comprising the steps of:
    a) selecting carbon nanotubes of aspect ratio 10 to 500 and oxidation level 1% to 15% 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;
    e) separating the solid discrete carbon nanotubes with attached lithium salts from the liquid; and
    f) 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.

11. A process for the production of discrete carbon nanotubes having crystals or layers of lithium ion active materials attached to their surface as defined in claim 3, 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 discrete carbon nanotubes with attached lithium 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.

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Letter accompanying subsequently filed items

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The document(s) listed below is (are) subsequently filed documents pertaining to the following application:

Application number	12731264.3
Applicant's or representative's reference	P100028EP00

	Description of document	Original file name	Assigned file name
1	Letter dealing with Oral proceedings	P100028EP00 Response written submissions.pdf	ORAL-1.pdf
2	Amended claims (clean copy)	P100028EP00 Auxiliary Claim Request I (clean version).pdf	CLMS-1.pdf
3	Amended claims with annotations	P100028EP00 Auxiliary Claim Request I (with revisions).pdf	CLMS-HWA-1.pdf
4	Amended claims (clean copy)	P100028EP00 Auxiliary Claim Request II (clean version).pdf	CLMS-2.pdf
5	Amended claims with annotations	P100028EP00 Auxiliary Claim Request II (with revisions).pdf	CLMS-HWA-2.pdf
6	Amended claims (clean copy)	P100028EP00 Auxiliary Claim Request III (clean version).pdf	CLMS-3.pdf
7	Amended claims with annotations	P100028EP00 Auxiliary Claim Request III (with revisions).pdf	CLMS-HWA-3.pdf

	Payment	
1	Method of payment	Not specified

Signatures

Place:	**Utrecht**
Date:	**14 September 2018**
Signed by:	**/H.P.A. Maas/**
Association:	**V.O.**
Representative name:	**H.P.A. Maas**
Capacity:	**(Representative)**

P100028EP00

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Patents & Trademarks

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Your ref. 12731264.3/ 1108
Our ref. HPM/P100028EP00

The Hague,
14 September 2018

Re: European Patent No. 2724403
In the name of Molecular Rebar Design, LLC

With reference to the Summons dated May 7, 2018 and in preparation for the Oral Proceedings scheduled for November 15, 2018, I inform you that I intend to attend these oral proceedings accompanied by Mr Stephen P. Krupp, Patentee's Chief Patent Counsel, and by Dr Clive P. Bosnyak, one of the inventors mentioned on the Patent.

Dr Bosnyak obtained his BSc with Honors in Chemistry with Minors in Mathematics and Physics in 1977 and his Ph.D. in Chemistry from the University of Birmingham, England in 1980. He was adjunct professor at the University of Illinois at Chicago for 8 years, leading research and teaching of polymers as materials in engineering.

From 1981-2006, he held various research positions within The Dow Chemical Company in the field of polymers bridging business needs to new technologies and products. This included the development of new materials and structures for building and construction, novel polyolefins, environmentally friendly blow-moldable styrenic block copolymers, new engineering thermoplastic polyesters, blends, nanocomposites, smart materials, thermosets, composites, films and foams. Special emphasis has been

on the design of polymeric materials, both chemical and physical, for specific process and structure-property relationships. Dr Bosnyak also became a credited trainer for new business opportunity identification and analysis in Marketing & New Business Development. On his retirement from Dow, he was Lead Scientist.

As of 2006, he is co-owner of and Chief Scientific Officer in Molecular Rebar Design, LLC.

He is recognized internationally as an inventor and innovator in the development of new polymeric materials and technologies. He is also a consultant for selected international companies and specializes in innovative technologies and business development. He is a founding member of several start-up companies, including Creative Polymers, LLC, Microlayer Energy, LLC and Planned Innovation. He has coauthored 64 papers and has over 70 patents and patent applications. Dr Bosnyak has presented a seminar by invitation at the US Patent Office on the subject of new advances in Carbon Nanotubes.

Dr Bosnyak will attend the Oral Proceedings as technical expert.

He may, under my responsibility and supervision, provide background information on the technical field of the invention and provide answers to technical questions which the Opposition Division may have.

Mr Krupp received his Bachelor of Science in Chemical Engineering from Washington University in St. Louis, MO, and his Juris Doctorate, *Cum Laude*, from Thomas M. Cooley Law School in Lansing, MI. He is licensed to practice before the USPTO and the CAFC, and is admitted to practice by the State Bars of Missouri and Texas.

Mr Krupp began his career at The Dow Chemical Company in 1981, as a Research Engineer. He held various R&D and TS&D positions in Dow, including co-inventorship on 8 U.S. Patents. He transferred into Dow's Legal Department in 1989, and later graduated from law school. He retired from Dow in 2010 after 29 years combined R&D and legal experience, has his own law practice and has been with Patentee since 2010. As Chief Patent Counsel, he is responsible for filing, prosecuting, coordinating, and executing all matters relating to MRD's global intellectual property protection.

Patentee notes that the preliminary opinion of the Opposition Division appears to be a positive one from our perspective.

However, with an eye on the Rules of Procedure before the Boards of Appeal, which become stricter and stricter, Patentee has decided to file a number of Auxiliary Claim Requests. These Auxiliary Claim Requests are enclosed in a form showing revisions and in retyped form.

Patentee wishes to maintain its right to change the order of its Auxiliary Claim Requests, to combine one or more of these Auxiliary Claim Requests and/or to file further or other Auxiliary Claim Requests.

Auxiliary Claim Request I is based on a combination of claims 1 and 8 as granted. Claim 8 is deleted with the remaining claims being renumbered.

Auxiliary Claim Request II is based on a combination of claims 1 and 7 as granted. Claim 7 as granted is deleted with the remaining claims being renumbered.

Both Auxiliary Request I and II introduce additional limitation in respect of lithium components in claim 1 and bring the claims even further away from the prior art cited by Opponent.

Patents & Trademarks

Auxiliary Claim Request III is based on a combination of claims 1 and 9 as granted. I additionally refer to §§ [0014] and [0004] of the application as published and §§ [0016] and [0004] of the Patent as granted. Claim 9 is deleted and the remaining claims are adapted to new claim 1 and deleted claim 9. Where some prior art references on file refer to carbon nanotubes in agglomerated or aggregated state or in the form of assemblages, this Auxiliary Request even brings the claims further away from this prior art.

Finally, I note that with my letter of May 22, 2018, I requested interpretation from French into English in case Opponent Arkema would use the French language during the Oral Proceedings.

The professional representative,



H.A.M. Marsman

Encl.: Auxiliary Claim Request I-III (with revisions and clean version)

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Date 12-09-2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☑ Oral P. 15.11.18

Enclosure(s):
- ☐ Letter from the proprietor of the patent of
- ☑ Letter from the opponent of 6.9.18
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

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Lettre d'accompagnement relative à des pièces produites ultérieurement

Mandataire:

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La (Les) pièce(s) désignées ci-après est (sont) produite(s) postérieurement au dépôt, pour la demande suivante :

N° de la demande	12731264.3
Référence du demandeur ou du mandataire	AL 0378CBL

	Description des pièces	Nom du dossier original	Nom du dossier attribué
1	Lettre concernant une procédure orale	AL 0374 CBL.pdf	ORAL-1.pdf
2	Document brevet présenté par un tiers	A9-wo9425268.pdf	TDOCP-1.pdf

	Paiement	
1	Méthode de paiement	Non spécifié

Signatures

Lieu:	**Colombes**
Date:	**06 septembre 2018**
Signé par:	**Claudine Bonnel 7787**
Nom du mandataire:	**Claudine BONNEL**
Fonction:	**(Mandataire)**

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INNOVATIVE CHEMISTRY

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Colombes, le 6 septembre 2018

Objet : Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA FRANCE

Madame, Monsieur,

Nous faisons suite à la notification datée du 7 mai 2018 concernant la citation à une procédure orale le 15 novembre 2018.

Nous présentons ci-dessous nos commentaires sur l'opinion préliminaire de la Division d'Opposition, ainsi que le document A9 (WO 94/25268).

Au vu des arguments présentés ci-après et de ceux présentés dans le mémoire d'opposition, l'opposant considère que le brevet opposé doit être révoqué dans sa totalité.

Nous vous prions d'agréer, Madame, Monsieur, nos salutations distinguées.

Veuillez agréer, Madame, Monsieur, nos salutations distinguées.

Claudine BONNEL
PG n° 8646

1) Insuffisance de description

L'opposant estime, comme mentionné dans le mémoire d'opposition, que l'objet de la revendication 1 n'est pas suffisamment décrit pour qu'un homme du métier puisse l'exécuter.

En effet, le brevet, comme la demande dont il est issu, ne contient aucune information sur la méthode de mesure utilisée pour déterminer le niveau d'oxydation des NTC. Ce paramètre est pourtant présenté comme une caractéristique essentielle de l'invention.
Selon la Division d'opposition, cela relèverait plutôt de l'Article 84 CBE, et l'Article 83 CBE serait satisfait.

La Division d'opposition se base sur les 5 exemples du brevet et à leur reproductibilité.
Cependant, il est fait remarqué que l'exemple 1 (illustrant des NTC oxydés - taux d'oxydation 8%- avec des particules d'oxyde de cuivre attachées à leur surface) et l'exemple 5 (illustrant des NTC oxydés - taux d'oxydation 4% - avec des particules d'oxyde de titane attachées à leur surface) ne sont pas des exemples selon l'invention revendiquée dans la revendication 1.
Seuls les exemples 2, 3 et 4 illustrent des NTC oxydés (à un taux de 4% ou 8%) comportant à leur surface des particules comportant du lithium.

Le paragraphe [0022] du brevet contesté donne une définition pour les NTC oxydés, à savoir des produits d'oxydation des NTC, qui sont « généralement » de type carboxylique, cétone ou hydroxyle.

L'incertitude pour l'homme du métier qui veut reproduire l'invention est donc de savoir comment exprimer la présence de ces espèces oxydées, sous la forme d'un taux d'oxydation.

Il existe de nombreuses façons pour quantifier des espèces oxydées, par exemple une teneur en oxygène, (qui fait appel aussi à plusieurs techniques possibles, XPS, ESCA, analyse élementaire), une acidité (quand il s'agit d'espèces carboxyliques), un indice de cétone ou un indice d'hydroxyle, ou une thermogravimétrie (décomposition sous une certaine température).

L'homme du métier n'est pas en mesure de savoir s'il travaille ou non dans la portée du brevet contesté, compte tenu de l'absence de méthode de détermination du taux d'oxydation.

L'opposant est d'avis que l'objet des revendications du brevet opposé ne satisfait pas aux exigences de l'Article 83 CBE et ne partage pas l'opinion de la Division d'opposition.

2) Activité inventive

a) Afin de compléter l'argumentation présentée dans le mémoire d'opposition vis-à-vis de A3, nous fournissons comme document A9, la demande WO 94/25268 qui revendique la priorité de la demande US 08/057,328 déposée le 05.05.1993 mentionnée dans A3 (page 6, l.32-36).

b) L'opposant partage l'avis de la Division d'opposition selon lequel A4 peut être considéré comme un document de la technique le plus proche de l'invention.

Comme l'a indiqué la Division d'opposition dans son opinion préliminaire, à juste titre, aucun effet technique dérivé des différences entre l'invention du brevet contesté et A4 n'a été démontré ; le problème technique objectif est ainsi la fourniture d'une composition alternative utilisable pour les batteries Li-ion.

Cependant, la Division d'opposition estimerait qu'une activité inventive pourrait résulter du fait que l'homme du métier aurait du faire plusieurs sélections dans A4 pour parvenir à son invention.

L'opposant ne partage pas cet avis, pour les raisons suivantes :

- Dans A4 [0032], il est clairement stipulé que « 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 ».

  L'utilisation de NTC oxydés n'est pas une selection motivée, mais équivalente à l'utilisation d'autres types de NTC. De plus, rien dans le brevet contesté ne permet de prouver que les NTC oxydés sont avantageux par rapport à des NTC « non oxydés ».

  L'utilisation de NTC ayant un rapport d'aspect d'au moins 100 n'est pas une selection motivée, mais équivalente à l'utilisation de NTC ayant un autre rapport d'aspect.

- Même si A4 ne divulgue pas spécifiquement un niveau d'oxydation lorsqu'il s'agit de NTC oxydés, la détermination de la gamme 1-15% telle que revendiquée dans le brevet contesté peut résulter d'expérimentations classiques de routine visant l'optimisation de la composition.
  La sélection ne peut impliquer une activité inventive que si elle entraine un effet particulier ; sans effet démontré, la sélection peut résulter de simples expérimentations que l'homme du métier peut exécuter sans exercer d'activité inventive.

c) Le même raisonnement relatif à A3 peut s'appliquer : la détermination d'un taux d'oxydation optimal pour les NTC discrets (non agrégés) oxydés divulgués dans A3 peut être réalisée par de simples expérimentation de routine.
En conséquence, l'invention revendiquée n'implique pas d'activité inventive.

Pour toutes ces raisons, la composition revendiquée dans la revendication 1 ne satisfait pas l'Article 56 CBE.

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(54) Title: THREE DIMENSIONAL MACROSCOPIC ASSEMBLAGES OF RANDOMLY ORIENTED CARBON FIBRILS AND COMPOSITES CONTAINING SAME

(57) Abstract

It has now been found that highly advantageous three-dimensional, macroscopic assemblages of randomly oriented carbon fibrils can be prepared which have relatively uniform physical properties along one, preferably two and most desirably three-dimensional axis of the three-dimensional assemblage. Preferred compositions prepared according to the methods of the invention have uniform physical properties along at least one dimensional axis and have relatively isotropic physical properties in at least one plane of the assemblage and most desirably are isotropic throughout the entire three-dimensional structure.

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THREE DIMENSIONAL MACROSCOPIC ASSEMBLAGES OF RANDOMLY ORIENTED CARBON FIBRILS AND COMPOSITES CONTAINING SAME

Field of the Invention

The invention relates generally to assemblages of carbon fibrils. More specifically, the invention relates to three dimensional, macroscopic, assemblages of randomly oriented carbon fibrils having a bulk density of from 0.001 to 0.50 gm/cc and to methods for preparing such assemblages. Even more specifically, the invention relates to such assemblages for use as catalyst supports, electrodes, chromatographic media, etc. and to composite structures comprising the assemblage and a second material contained within the assemblage.

Background of the Invention

Carbon fibrils are vermicular carbon deposits having diameters less than 500 nanometers. They exist in a variety of forms, and have been prepared through the catalytic decomposition of various carbon-containing gases at metal surfaces.

Tennent, U.S. 4,663,230, describes carbon fibrils that are free of a continuous thermal carbon overcoat and have multiple graphitic outer layers that are substantially parallel to the fibril axis. As such they may be characterized as having their c-axes, the axes which are perpendicular to the tangents of the curved layers of graphite, substantially perpendicular to their cylindrical axes. They generally have diameters no greater than 0.1 micron and length to diameter ratios of at least 5. Desirably they are substantially free of a continuous thermal carbon overcoat, i.e., pyrolytically deposited carbon resulting from thermal cracking of the gas feed used to prepare them.

Tubular fibrils having graphitic layers that are substantially parallel to the microfiber axis and diameters between 3.5 and 75 nanometers, are described in Tennent et al., U.S.S.N. 871,676 filed June 6, 1986

("Novel Carbon Fibrils, Method for Producing Same and Compositions Containing Same"), Tenant et al., U.S.S.N. 871,675 filed June 6, 1986 ("Novel Carbon Fibrils, Method for Producing Same and Encapsulated Catalyst"), Snyder et al., U.S.S.N. 149,573 filed January 28, 1988 ("Carbon Fibrils"), Mandeville et al., U.S.S.N. 285,817 filed December 16, 1988 ("Fibrils"), and McCarthy et al ., U.S.S.N. 351,967 filed May 15, 1989 ("Surface Treatment of Carbon Microfibers"), all of which are assigned to the same assignee as the present application and are hereby incorporated by reference.

Fibrils are useful in a variety of applications. For example, they can be used as reinforcements in fiber-reinforced composite structures or hybrid composite structures (i.e. composites containing reinforcements such as continuous fibers in addition to fibrils). The composites may further contain fillers such as a carbon black and silica, alone or in combination with each other. Examples of reinforceable matrix materials include inorganic and organic polymers, ceramics (e.g., lead or copper). When the matrix is an organic polymer, it may be a thermoset resin such as epoxy, bismaleimide, polyamide, or polyester resin; a thermoplastic resin; or a reaction injection molded resin.

Objects of the Invention

It is an object of the invention to provide a composition of matter which comprises carbon fibrils and more specifically an assemblage of randomly oriented carbon fibrils which has a low bulk density and which can be used as a substrate or medium for various industrial and scientific purposes.

It is another object of the invention to provide a composition of matter comprising a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils having a low bulk density to which can be added one or more functional

second materials in the nature of active catalysts, electroactive species, etc. so as to form composites having novel industrial properties.

It is yet another object of the invention to provide three-dimensional, macroscopic assemblages of a multiplicity of randomly oriented carbon fibrils which have isotropic physical properties so that such compositions can be used reliably and interchangeably for multiple industrial purposes.

It is a further object of the invention to provide processes for the preparation of such three-dimensional microscopic assemblages of carbon fibrils which are efficient and convenient to use in the preparation of low-density compositions.

It is a still further object of the invention to provide improved catalyst supports, filter media, chromato- graphic media, EMI shielding and other compositions of industrial value based on three-dimensional assemblages of carbon fibrils.

SUMMARY OF THE INVENTION

Definitions

The term "assemblage" refers to any configuration of a mass of individual fibrils and embraces intertwined as well as discrete fibril embodiments.

The term "macroscopic" means that the assemblages may be of any suitable size to achieve an industrial or scientific purpose.

The term "physical property" means an inherent, measurable property of the assemblage, e.g. resistivity.

The term "isotropic" means that all measurements of a physical property within a plane or volume of the assemblage, independent of the direction of the measurement, are of a constant value. It is understood that measurements of such non-solid compositions must be taken on a representative sample of the assemblage so that the average value of the void

spaces is taken into account.

The term "relatively" means that ninety-five percent of the values of the physical property when measured along an axis of, or within a plane of or within a volume of the assemblage, as the case may be, will be within plus or minus fifty percent of a mean value.

The term "substantially" means that ninety-five percent of the values of the physical property when measured along an axis of, or within a plane of or within a volume of the assemblage, as the case may be, will be within plus or minus ten percent of a mean value.

The terms" relatively isotropic" and "substantially isotropic" correspond to the ranges of variability in the values of a physical property set forth above.

The Invention

The invention is broadly in a composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc.

The assemblages described above can be used to great advantage as three-dimensional matrixes for a number of industrial purposes. For example, the assemblages can be used as filter media, as catalyst supports, as electroactive materials for use, e.g. in electrodes in fuel cells and batteries, and as chromatography media. It has been found that the assemblages are useful in the formation of composites which comprise the assemblage together with either a particulate solid, an electroactive component or a catalytically active metal or metal-containing compound,

as well as in composites with polymers.

It has now been found that highly advantageous three-dimensional, macroscopic assemblages of randomly oriented carbon fibrils can be prepared which have relatively uniform physical properties along one, preferably two and most desirably three-dimensional axis of the three-dimensional assemblage. Preferred compositions prepared according to the methods of the invention have uniform physical properties along at least one dimensional axis and have relatively isotropic physical properties in at least one plane of the assemblage and most desirably are isotropic throughout the entire three-dimensional structure.

These advantageous compositions can be prepared by dispersing fibrils in aqueous or organic solid media and then filtering the fibrils. Low density compositions are advantageously prepared by forming a gel or paste of carbon fibrils in a fluid, e.g. an organic solvent such as propane and then heating that gel or paste to above the critical temperature of the medium, removing supercritical fluid and finally removing a low-density porous mat or plug from the vessel in which the process has been carried out.

DETAILED DESCRIPTION

In the Drawings

Fig. 1 is a photomicrograph of a three dimensional assemblage of randomly oriented carbon fibrils prepared by the method of EXAMPLE 1.

Production of Carbon Fibrils

Fibrils are prepared by contacting a carbon-containing gas with a metal catalyst in a reactor for an appropriate period of time, at a suitable pressure, and at a temperature sufficient to produce fibrils with the above-described morphology. Reaction temperatures are generally 400-850°C, more preferably 600-750°C. Fibrils are advantageously prepared continuously by bringing the reactor to the reaction temperature, adding metal

catalyst particles, and then continuously contacting the catalyst with a carbon-containing gas.

Examples of suitable feed gases, catalysts and reaction conditions are given in the several patent applications referenced above as well as in Moy et al., U.S. Patent Applications Ser. Nos. 887,307 and 887,314 filed May 22, 1992 which are hereby incorporated by reference.

Fibrils may be prepared such that at least a portion of the fibrils are in the form of aggregates. As used herein, an aggregate is defined as two or more entangled fibrils. Fibril aggregates typically have macroscopic morphologies, as determined by scanning electron microscopy, in which they are randomly entangled with each other to form entangled balls of fibrils resembling a bird's nest ("BN"); or as aggregates consisting of bundles of straight to slightly bent or kinked carbon fibrils having substantially the same relative orientation, and having the appearance of combed yarn ("CY") e.g., the longitudinal axis of each fibril, despite individual bends or kinks, extends in the same direction as that of the surrounding fibrils in the bundles; or, as aggregates consisting of straight to slightly bent or kinked fibrils which are loosely entangled with each other to form an "open net" ("ON") structure. In open net structures the degree of fibril entanglement is greater than observed in the combed yarn aggregates (in which the individual fibrils have substantially the same relative orientation) but less than that of bird's nest.

In addition to fibrils such as are described in Tennent, U.S. Patent No. 4,663,230, fibrils may be prepared having different macromorphologies, such as the so-called fishbone ("FB") morphology described in published European Patent Application No. 198,558 to J.W. Geus (published October 22, 1986). Fibrils of the so-called fishbone morphology may be characterized as having

their c-axes (as defined above) at some angle less than perpendicular to the cylindrical axes of the fibrils. The invention relates to such fishbone fibrils as well as to those described in Tennent, U.S. Patent No. 4,663,230.

Carbon Fibrils

The carbon fibrils preferably comprise a combination of discrete fibrils and fibril aggregates. However, the fibrils may all be in the form of aggregates. The aggregates, when present, are generally of the bird's nest, combed yarn or open net morphologies. The more "entangled" the aggregates are, the more processing will be required to achieve a suitable composition. This means that the selection of combed yarn or open net aggregates is most preferable for the majority of applications. However, bird's nest aggregates will generally suffice.

The Assemblages

Broadly, the invention is in a composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc. Preferably the assemblage has relatively or substantially uniform physical properties along at least one dimensional axis and desirably have relatively or substantially uniform physical properties in one or more planes within the assemblage, i.e. they have isotropic physical properties in that plane. In other embodiments, the entire assemblage is relatively or substantially isotropic with respect to one or more of its physical properties.

The physical properties which can be easily measured and by which uniformity or isotrophy are determined include resistivity and optical density.

Composites Containing the Assemblages

Broadly, the fibril assemblages may be used for any purpose for which porous media are known to be useful. These include filtration, electrodes, catalyst supports, chromatography media, etc. In addition, the assemblages are a convenient bulk form of carbon fibrils and may thus be used for any known applications including especially EMI shielding, polymer composites, active electrodes, etc.

For some applications like EMI shielding, filtration and current collection, unmodified fibril assemblages can be used. For other applications, the fibril assemblages are a component of a more complex material, i.e. they are part of a composite. Examples of such composites are polymer molding compounds, chromatography media, electrodes for fuel cells and batteries, fibril supported catalyst and ceramic composites, including bioceramics like artificial bone.

In some of these composites, like molding compound and artificial bone, it is desirable that the non-fibril components fill - or substantially fill - the porosity of the fibril assemblage. For others, like electrodes, catalysts, and chromatography media, their usefulness depends on the composite retaining at least some of the porosity of the fibril assemblage.

Methods of Preparing Fibril Assemblages

While fibrils of any morphology may be used to prepare the assemblages of the invention by using the methods of the invention, it is preferred to use fibrils having a parallel type morphology such as CC, DD or CY. Methods for the preparation of fibrils having these morphologies are described in Moy et al., U.S. Patent Application Ser. Nos. 887,307 and 887,314 filed May 22, 1992.

Mats with a thickness between 0.02 and 0.50 millimeters have a density of typically 0.20 g/cc corresponding to a pore volume fraction of 0.90. Their electrical resistivity in the plane of the mat is typically 0.02 ohm/cm; resistivity perpendicular to the mat is typically 1.0 ohm/cm.

Solid ingredients can be incorporated within the fibril mat by mixing them with the fibril dispersion prior to mat formation. The content of other solids in the dry mat may be made as high as fifty parts solids per part of fibrils.

Fibrils from the synthesis reactor are dispersed at high shear in a high-shear mixer, e.g. a Waring Blender. The dispersion may contain broadly from 0.01 to 10% fibrils in water, ethanol, mineral spirits, etc.. This procedure adequately opens fibril bundles, i.e. tightly wound bundles, of fibrils and disperses fibrils to form self-supporting mats after filtration and drying. The application of high shear mixing may take up to several hours. Mats prepared by this method are not free of aggregates.

If the high shear procedure is followed by ultrasonication, dispersion is improved. Dilution to 0.1% or less aids ultrasonication. Thus, 200 cc of 0.1% fibrils may be sonified by a Bronson Sonifier Probe (450 watt power supply) for 5 minutes or more to further improve the dispersion.

To achieve the highest degrees of dispersion, i.e. a dispersion which is free or virtually free of fibril aggregates, sonication must take place either at very low concentration in a compatible liquid, e.g. at 0.001% to 0.01% concentration in ethanol or at higher concentration e.g. 0.1% in water to which a surfactant, e.g. Triton X-100 has been added in a concentration of about 0.5%. The mat which is subsequently formed may be rinsed free or substantially free of surfactant by sequential additions of water followed by vacuum

filtration.

Particulate solids such as MnO_2 (for batteries) and Al_2O_3 (for high temperature gaskets) may be added to the fibril dispersion prior to mat formation at up to 50 parts added solids per part of fibrils.

Reinforcing webs and scrims may be incorporated on or in the mats during formation. Examples are polypropylene mesh and expanded nickel screen.

Methods of Improving the Stability of Assemblages

In order to increase the stability of the fibril assemblages, it is possible to deposit polymer at the intersections of the assemblage. This may be infiltrating the assemblage with a dilute solution of polymer cement and allowing the solvent to evaporate. Capillary forces will concentrate the polymer at fibril intersections. It is understood that in order to substantially improve the stiffness and integrity of the assemblage, only a small fraction of the fibril intersections need be cemented.

EXAMPLES

The invention is further described in the following examples.

EXAMPLE I

Preparation of a Porous Fibril Mat

A dilute dispersion of fibrils is used to prepare porous mats or sheets. A suspension of fibrils is prepared containing 0.5% fibrils in water using a Waring Blender. After subsequent dilution to 0.1%, the fibrils are further dispersed with a probe type sonifier. The dispersion is then vacuum filtered to form a mat, which is then oven dried.

The mat has a thickness of about 0.20 mm and a density of about 0.20 gm/cc corresponding to a pore volume of 0.90. The electrical resistivity in the plane of the mat is about 0.02 ohm/cm. The resistivity in the direction perpendicular to the mat is about 1.0 ohm/cm.

EXAMPLE II

Preparation of a Porous Fibril Mat

A suspension of fibrils is prepared containing 0.5% fibrils in ethanol using a Waring Blendor. After subsequent dilution to 0.1%, the fibrils are further dispersed with a probe type sonifier. The ethanol is then allowed to evaporate and a mat is formed. The mat has the same physical properties and characteristics as the mat prepared in EXAMPLE I.

EXAMPLE III

Preparation of a Low-Density Porous Fibril Plug

Supercritical fluid removal from a well dispersed-fibril paste is used to prepare low density shapes. 50 cc of a 0.5% dispersion in n-pentane is charged to a pressure vessel of slightly larger capacity which is equipped with a needle valve to enable slow release of pressure. After the vessel is heated above the critical temperature of pentane (Tc = 196.6°), the needle valve is cracked open slightly to bleed the supercritical pentane over a period of about an hour.

The resultant solid plug of Fibrils, which has the shape of the vessel interior, has a density of 0.005 g/cc, corresponding to a pore volume fraction of 0.997%. The resistivity is isotropic and about 20 ohm/cm.

EXAMPLE IV

Preparation of EMI Shielding

A fibril paper is prepared according to the procedures of EXAMPLE I. Table I below sets forth the attenuation achieved at several paper thickness.

TABLE I

FIBRIL PAPER

EMI SHIELDING

ATTENUATION 30 MHz TO 1 GHz

THICKNESS, INCHES (MM)	WEIGHT	ATTENUATION
0.002 (0.5)	12 G/M^2	27 Db
0.005 (.125)	30	37Db
0.017 (.425)	120	48Db

EXAMPLE V

A fibril mat prepared by the method of EXAMPLE I is used as an electrode in an electrochemiluminescence cell such as is described in PCT U.S. 85/02153 (WO 86/02734) and U.S. Patents Nos. 5,147,806 and 5,068,088. When the voltage is pulsed in the presence of ruthenium trisbipyridyl, electrochemiluminescence is observed.

WHAT IS CLAIMED IS:

1. A composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of a randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc.

2. A composite material comprising:

(a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc; and

(b) a second component comprising (i) particulate solids, or (ii) an electroactive material; or (iii) a catalytically active metal or metal-containing compound, in an amount up to fifty parts per part of fibrils.

3. A composite material as recited in claim 2 wherein said electroactive component is lead or a lead compound or manganese or a manganese compound.

4. A composite material as recited in claim 2 wherein said particulate material is an aluminum oxide, silicon dioxide or silicon carbide.

5. A composite material as recited in claim 2 wherein said particulate material is a catalyst.

6. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively uniform physical properties along at least one dimensional axis thereof and a bulk density of from 0.001 to 0.50 gm/cc.

7. A three-dimensional assemblage as recited in claim 6 having substantially uniform physical properties along said one dimensional axis.

8. A composite material comprising (a) an assemblage of fibrils as recited in claim 6, and (b) a second material at least partially contained within said assemblage.

9. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties in at least one plane thereof and a bulk density of from 0.001 to 0.50 gm/cc.

10. A three-dimensional assemblage as recited in claim 9 having substantially isotropic physical properties in said plane.

11. A composite material comprising (a) an assemblage of fibrils as recited in claim 9 and (b) a second material at least partially contained within said assemblage.

12. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties and a bulk density of from 0.001 to 0.50 gm/cc.

13. A three-dimensional assemblage as recited in claim 12 having substantially isotropic physical properties.

14. A composite material comprising (a) an assemblage of fibrils as recited in claim 12, and (b) a second material at least partially contained within said assemblage.

15. A method of preparing a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, comprising the steps of:

(a) dispersing a multiplicity of said fibrils in a medium; and

(b) separating said assemblage from said medium.

16. A method as recited in claim 15 for the formation of a porous mat or sheet of said carbon fibrils, said mat or sheet having a bulk density of from 0.05 to 0.50 gm/cc comprising the steps of:

(a) vigorously dispersing a multiplicity of said fibrils in water or an organic solvent and thereby forming a dispersion, said dispersion containing less than 10.0 percent by weight of fibrils; and

(b) filtering the dispersion to form said porous mat or sheet.

17. A method as recited in claim 15 for the formation of a porous mat or sheet of said carbon fibrils, said mat or sheet having a bulk density of from 0.05 to 0.50 gm/cc comprising the steps of:

(a) vigorously dispersing a multiplicity of said fibrils in water or an organic solvent and thereby forming a dispersion, said dispersion containing less than 10.0 percent by weight of fibrils; and

(b) permitting the water or organic solvent to evaporate from said dispersion thereby leaving said porous mat or sheet.

18. A method as recited in claim 15 for the formation of a low-density porous plug of said carbon fibrils, said plug having a bulk density of from 0.001 to 0.05 gm/cc comprising the steps of:

(a) forming a gel or paste comprising a multiplicity of carbon fibrils in a fluid, said gel or paste comprising less than 10.0 percent by weight of fibrils;

(b) heating said gel or paste in a pressure vessel to a temperature above the critical temperature of said fluid;

(c) removing supercritical fluid from said pressure vessel; and

(d) removing said porous plug from said pressure vessel.

19. A method of preparing a composite of (a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, and (b) a second material, comprising the steps of:

(a) dispersing a multiplicity of said fibrils and a second material in a medium; and

(b) separating said composite from said medium.

20. A three-dimensional, macroscopic assemblage of randomly oriented carbon fibrils prepared by a method of claim 15.

21. A composite prepared by a method of claim 19.

22. A composite material comprising:

(a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc; and

(b) a second component comprising (i) particulate solids, or (ii) an electroactive material; or (iii) a catalytically active metal or metal-containing compound, in an amount up to fifty parts per part of fibrils.

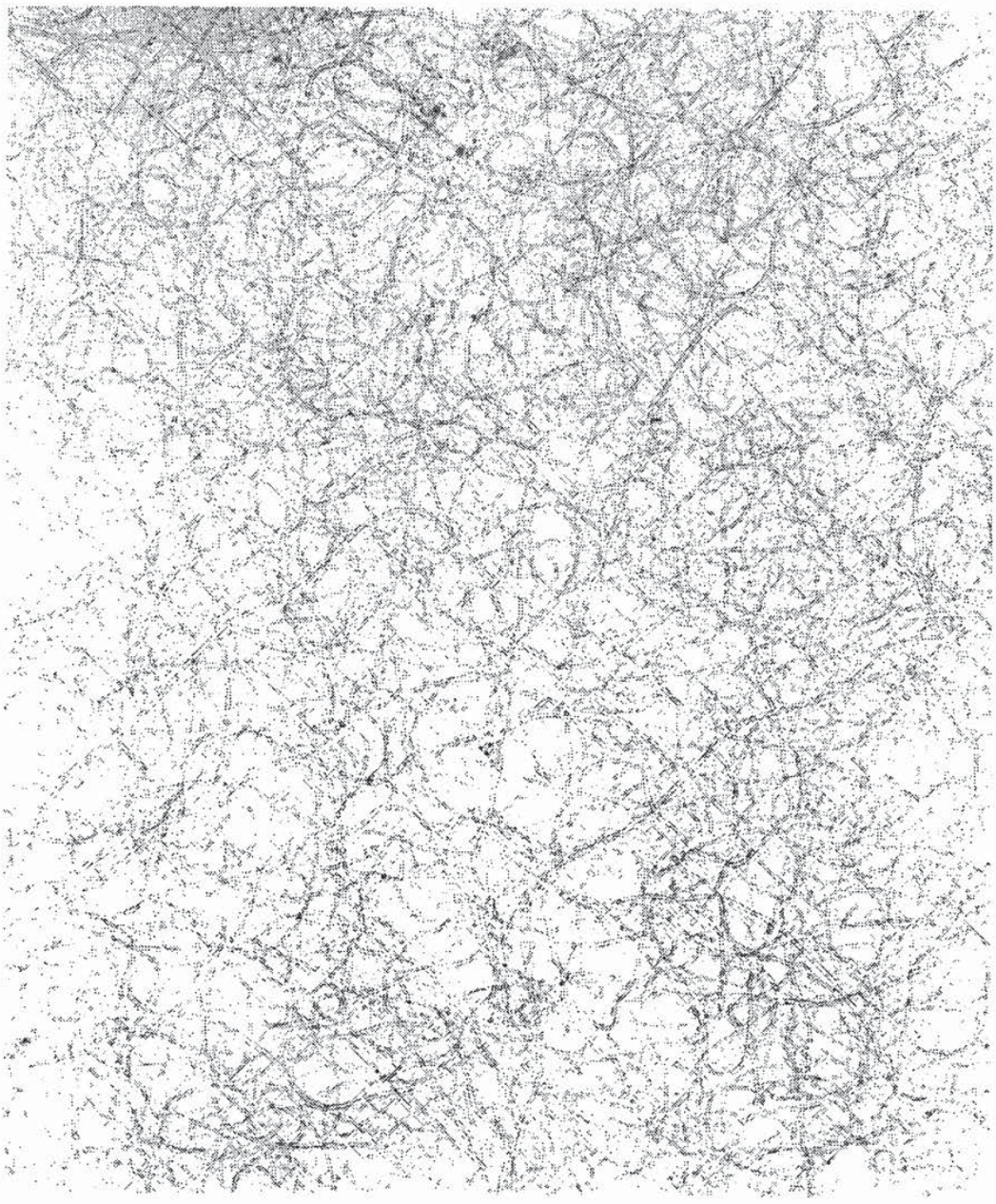
23. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively uniform physical properties along at least one dimensional axis thereof and a bulk density of from 0.001 to 0.50 gm/cc.

24. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties and a bulk density of from 0.001 to 0.50 gm/cc.

25. A method of preparing a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, comprising the steps of:

(a) dispersing a multiplicity of said fibrils in a medium; and

(b) separating said assemblage from said medium.



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/04879

A. CLASSIFICATION OF SUBJECT MATTER

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US CL :428/367
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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 428/359, 360, 367, 372, 408; 210/500.1, 500.25, 500.43, 503, 505, 510.11; 423/447.1, 447.2

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

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,663,230 (TENNENT) 05 May 1987, see Abstract.	1-25
Y	WO, A, WO 86/03455 (HYPERION CATALYSIS INTERNATIONAL, INC.) 19 June 1986, see Abstract.	1-25
Y	WO, A, WO 89/07163 (HYPERION CATALYSIS INTERNATIONAL, INC.) 10 August 1989, see Abstract.	1-25
Y	WO, A, WO 90/07023 (HYPERION CATALYSIS INTERNATIONAL, INC.) 28 June 1990, see Abstract.	1-25
Y	WO, A, WO 91/05089 (HYPERION CATALYSIS INTERNATIONAL, INC.) 18 April 1991, see Abstract.	1-25

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO, A, WO 91/01219 (HYPERION CATALYSIS INTERNATIONAL, INC.) 07 February 1991, see Abstract.	1-25

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(54) Title: THREE DIMENSIONAL MACROSCOPIC ASSEMBLAGES OF RANDOMLY ORIENTED CARBON FIBRILS AND COMPOSITES CONTAINING SAME

(57) Abstract

It has now been found that highly advantageous three-dimensional, macroscopic assemblages of randomly oriented carbon fibrils can be prepared which have relatively uniform physical properties along one, preferably two and most desirably three-dimensional axis of the three-dimensional assemblage. Preferred compositions prepared according to the methods of the invention have uniform physical properties along at least one dimensional axis and have relatively isotropic physical properties in at least one plane of the assemblage and most desirably are isotropic throughout the entire three-dimensional structure.

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THREE DIMENSIONAL MACROSCOPIC ASSEMBLAGES OF RANDOMLY ORIENTED CARBON FIBRILS AND COMPOSITES CONTAINING SAME

Field of the Invention

The invention relates generally to assemblages of carbon fibrils. More specifically, the invention relates to three dimensional, macroscopic, assemblages of randomly oriented carbon fibrils having a bulk density of from 0.001 to 0.50 gm/cc and to methods for preparing such assemblages. Even more specifically, the invention relates to such assemblages for use as catalyst supports, electrodes, chromatographic media, etc. and to composite structures comprising the assemblage and a second material contained within the assemblage.

Background of the Invention

Carbon fibrils are vermicular carbon deposits having diameters less than 500 nanometers. They exist in a variety of forms, and have been prepared through the catalytic decomposition of various carbon-containing gases at metal surfaces.

Tennent, U.S. 4,663,230, describes carbon fibrils that are free of a continuous thermal carbon overcoat and have multiple graphitic outer layers that are substantially parallel to the fibril axis. As such they may be characterized as having their c-axes, the axes which are perpendicular to the tangents of the curved layers of graphite, substantially perpendicular to their cylindrical axes. They generally have diameters no greater than 0.1 micron and length to diameter ratios of at least 5. Desirably they are substantially free of a continuous thermal carbon overcoat, i.e., pyrolytically deposited carbon resulting from thermal cracking of the gas feed used to prepare them.

Tubular fibrils having graphitic layers that are substantially parallel to the microfiber axis and diameters between 3.5 and 75 nanometers, are described in Tennent et al., U.S.S.N. 871,676 filed June 6, 1986

("Novel Carbon Fibrils, Method for Producing Same and Compositions Containing Same"), Tenant et al., U.S.S.N. 871,675 filed June 6, 1986 ("Novel Carbon Fibrils, Method for Producing Same and Encapsulated Catalyst"), Snyder et al., U.S.S.N. 149,573 filed January 28, 1988 ("Carbon Fibrils"), Mandeville et al., U.S.S.N. 285,817 filed December 16, 1988 ("Fibrils"), and McCarthy et al ., U.S.S.N. 351,967 filed May 15, 1989 ("Surface Treatment of Carbon Microfibers"), all of which are assigned to the same assignee as the present application and are hereby incorporated by reference.

Fibrils are useful in a variety of applications. For example, they can be used as reinforcements in fiber-reinforced composite structures or hybrid composite structures (i.e. composites containing reinforcements such as continuous fibers in addition to fibrils). The composites may further contain fillers such as a carbon black and silica, alone or in combination with each other. Examples of reinforceable matrix materials include inorganic and organic polymers, ceramics (e.g., lead or copper). When the matrix is an organic polymer, it may be a thermoset resin such as epoxy, bismaleimide, polyamide, or polyester resin; a thermoplastic resin; or a reaction injection molded resin.

Objects of the Invention

It is an object of the invention to provide a composition of matter which comprises carbon fibrils and more specifically an assemblage of randomly oriented carbon fibrils which has a low bulk density and which can be used as a substrate or medium for various industrial and scientific purposes.

It is another object of the invention to provide a composition of matter comprising a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils having a low bulk density to which can be added one or more functional

second materials in the nature of active catalysts, electroactive species, etc. so as to form composites having novel industrial properties.

It is yet another object of the invention to provide three-dimensional, macroscopic assemblages of a multiplicity of randomly oriented carbon fibrils which have isotropic physical properties so that such compositions can be used reliably and interchangeably for multiple industrial purposes.

It is a further object of the invention to provide processes for the preparation of such three-dimensional microscopic assemblages of carbon fibrils which are efficient and convenient to use in the preparation of low-density compositions.

It is a still further object of the invention to provide improved catalyst supports, filter media, chromato- graphic media, EMI shielding and other compositions of industrial value based on three-dimensional assemblages of carbon fibrils.

SUMMARY OF THE INVENTION

Definitions

The term "assemblage" refers to any configuration of a mass of individual fibrils and embraces intertwined as well as discrete fibril embodiments.

The term "macroscopic" means that the assemblages may be of any suitable size to achieve an industrial or scientific purpose.

The term "physical property" means an inherent, measurable property of the assemblage, e.g. resistivity.

The term "isotropic" means that all measurements of a physical property within a plane or volume of the assemblage, independent of the direction of the measurement, are of a constant value. It is understood that measurements of such non-solid compositions must be taken on a representative sample of the assemblage so that the average value of the void

spaces is taken into account.

The term "relatively" means that ninety-five percent of the values of the physical property when measured along an axis of, or within a plane of or within a volume of the assemblage, as the case may be, will be within plus or minus fifty percent of a mean value.

The term "substantially" means that ninety-five percent of the values of the physical property when measured along an axis of, or within a plane of or within a volume of the assemblage, as the case may be, will be within plus or minus ten percent of a mean value.

The terms" relatively isotropic" and "substantially isotropic" correspond to the ranges of variability in the values of a physical property set forth above.

The Invention

The invention is broadly in a composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc.

The assemblages described above can be used to great advantage as three-dimensional matrixes for a number of industrial purposes. For example, the assemblages can be used as filter media, as catalyst supports, as electroactive materials for use, e.g. in electrodes in fuel cells and batteries, and as chromatography media. It has been found that the assemblages are useful in the formation of composites which comprise the assemblage together with either a particulate solid, an electroactive component or a catalytically active metal or metal-containing compound,

as well as in composites with polymers.

It has now been found that highly advantageous three-dimensional, macroscopic assemblages of randomly oriented carbon fibrils can be prepared which have relatively uniform physical properties along one, preferably two and most desirably three-dimensional axis of the three-dimensional assemblage. Preferred compositions prepared according to the methods of the invention have uniform physical properties along at least one dimensional axis and have relatively isotropic physical properties in at least one plane of the assemblage and most desirably are isotropic throughout the entire three-dimensional structure.

These advantageous compositions can be prepared by dispersing fibrils in aqueous or organic solid media and then filtering the fibrils. Low density compositions are advantageously prepared by forming a gel or paste of carbon fibrils in a fluid, e.g. an organic solvent such as propane and then heating that gel or paste to above the critical temperature of the medium, removing supercritical fluid and finally removing a low-density porous mat or plug from the vessel in which the process has been carried out.

DETAILED DESCRIPTION

In the Drawings

Fig. 1 is a photomicrograph of a three dimensional assemblage of randomly oriented carbon fibrils prepared by the method of EXAMPLE 1.

Production of Carbon Fibrils

Fibrils are prepared by contacting a carbon-containing gas with a metal catalyst in a reactor for an appropriate period of time, at a suitable pressure, and at a temperature sufficient to produce fibrils with the above-described morphology. Reaction temperatures are generally 400-850°C, more preferably 600-750°C. Fibrils are advantageously prepared continuously by bringing the reactor to the reaction temperature, adding metal

catalyst particles, and then continuously contacting the catalyst with a carbon-containing gas.

Examples of suitable feed gases, catalysts and reaction conditions are given in the several patent applications referenced above as well as in Moy et al., U.S. Patent Applications Ser. Nos. 887,307 and 887,314 filed May 22, 1992 which are hereby incorporated by reference.

Fibrils may be prepared such that at least a portion of the fibrils are in the form of aggregates. As used herein, an aggregate is defined as two or more entangled fibrils. Fibril aggregates typically have macroscopic morphologies, as determined by scanning electron microscopy, in which they are randomly entangled with each other to form entangled balls of fibrils resembling a bird's nest ("BN"); or as aggregates consisting of bundles of straight to slightly bent or kinked carbon fibrils having substantially the same relative orientation, and having the appearance of combed yarn ("CY") e.g., the longitudinal axis of each fibril, despite individual bends or kinks, extends in the same direction as that of the surrounding fibrils in the bundles; or, as aggregates consisting of straight to slightly bent or kinked fibrils which are loosely entangled with each other to form an "open net" ("ON") structure. In open net structures the degree of fibril entanglement is greater than observed in the combed yarn aggregates (in which the individual fibrils have substantially the same relative orientation) but less than that of bird's nest.

In addition to fibrils such as are described in Tennent, U.S. Patent No. 4,663,230, fibrils may be prepared having different macromorphologies, such as the so-called fishbone ("FB") morphology described in published European Patent Application No. 198,558 to J.W. Geus (published October 22, 1986). Fibrils of the so-called fishbone morphology may be characterized as having

their c-axes (as defined above) at some angle less than perpendicular to the cylindrical axes of the fibrils. The invention relates to such fishbone fibrils as well as to those described in Tennent, U.S. Patent No. 4,663,230.

Carbon Fibrils

The carbon fibrils preferably comprise a combination of discrete fibrils and fibril aggregates. However, the fibrils may all be in the form of aggregates. The aggregates, when present, are generally of the bird's nest, combed yarn or open net morphologies. The more "entangled" the aggregates are, the more processing will be required to achieve a suitable composition. This means that the selection of combed yarn or open net aggregates is most preferable for the majority of applications. However, bird's nest aggregates will generally suffice.

The Assemblages

Broadly, the invention is in a composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc. Preferably the assemblage has relatively or substantially uniform physical properties along at least one dimensional axis and desirably have relatively or substantially uniform physical properties in one or more planes within the assemblage, i.e. they have isotropic physical properties in that plane. In other embodiments, the entire assemblage is relatively or substantially isotropic with respect to one or more of its physical properties.

The physical properties which can be easily measured and by which uniformity or isotrophy are determined include resistivity and optical density.

Composites Containing the Assemblages

Broadly, the fibril assemblages may be used for any purpose for which porous media are known to be useful. These include filtration, electrodes, catalyst supports, chromatography media, etc. In addition, the assemblages are a convenient bulk form of carbon fibrils and may thus be used for any known applications including especially EMI shielding, polymer composites, active electrodes, etc.

For some applications like EMI shielding, filtration and current collection, unmodified fibril assemblages can be used. For other applications, the fibril assemblages are a component of a more complex material, i.e. they are part of a composite. Examples of such composites are polymer molding compounds, chromatography media, electrodes for fuel cells and batteries, fibril supported catalyst and ceramic composites, including bioceramics like artificial bone.

In some of these composites, like molding compound and artificial bone, it is desirable that the non-fibril components fill - or substantially fill - the porosity of the fibril assemblage. For others, like electrodes, catalysts, and chromatography media, their usefulness depends on the composite retaining at least some of the porosity of the fibril assemblage.

Methods of Preparing Fibril Assemblages

While fibrils of any morphology may be used to prepare the assemblages of the invention by using the methods of the invention, it is preferred to use fibrils having a parallel type morphology such as CC, DD or CY. Methods for the preparation of fibrils having these morphologies are described in Moy et al., U.S. Patent Application Ser. Nos. 887,307 and 887,314 filed May 22, 1992.

Mats with a thickness between 0.02 and 0.50 millimeters have a density of typically 0.20 g/cc corresponding to a pore volume fraction of 0.90. Their electrical resistivity in the plane of the mat is typically 0.02 ohm/cm; resistivity perpendicular to the mat is typically 1.0 ohm/cm.

Solid ingredients can be incorporated within the fibril mat by mixing them with the fibril dispersion prior to mat formation. The content of other solids in the dry mat may be made as high as fifty parts solids per part of fibrils.

Fibrils from the synthesis reactor are dispersed at high shear in a high-shear mixer, e.g. a Waring Blender. The dispersion may contain broadly from 0.01 to 10% fibrils in water, ethanol, mineral spirits, etc.. This procedure adequately opens fibril bundles, i.e. tightly wound bundles, of fibrils and disperses fibrils to form self-supporting mats after filtration and drying. The application of high shear mixing may take up to several hours. Mats prepared by this method are not free of aggregates.

If the high shear procedure is followed by ultrasonication, dispersion is improved. Dilution to 0.1% or less aids ultrasonication. Thus, 200 cc of 0.1% fibrils may be sonified by a Bronson Sonifier Probe (450 watt power supply) for 5 minutes or more to further improve the dispersion.

To achieve the highest degrees of dispersion, i.e. a dispersion which is free or virtually free of fibril aggregates, sonication must take place either at very low concentration in a compatible liquid, e.g. at 0.001% to 0.01% concentration in ethanol or at higher concentration e.g. 0.1% in water to which a surfactant, e.g. Triton X-100 has been added in a concentration of about 0.5%. The mat which is subsequently formed may be rinsed free or substantially free of surfactant by sequential additions of water followed by vacuum

filtration.

Particulate solids such as MnO_2 (for batteries) and Al_2O_3 (for high temperature gaskets) may be added to the fibril dispersion prior to mat formation at up to 50 parts added solids per part of fibrils.

Reinforcing webs and scrims may be incorporated on or in the mats during formation. Examples are polypropylene mesh and expanded nickel screen.

Methods of Improving the Stability of Assemblages

In order to increase the stability of the fibril assemblages, it is possible to deposit polymer at the intersections of the assemblage. This may be infiltrating the assemblage with a dilute solution of polymer cement and allowing the solvent to evaporate. Capillary forces will concentrate the polymer at fibril intersections. It is understood that in order to substantially improve the stiffness and integrity of the assemblage, only a small fraction of the fibril intersections need be cemented.

EXAMPLES

The invention is further described in the following examples.

EXAMPLE I

Preparation of a Porous Fibril Mat

A dilute dispersion of fibrils is used to prepare porous mats or sheets. A suspension of fibrils is prepared containing 0.5% fibrils in water using a Waring Blender. After subsequent dilution to 0.1%, the fibrils are further dispersed with a probe type sonifier. The dispersion is then vacuum filtered to form a mat, which is then oven dried.

The mat has a thickness of about 0.20 mm and a density of about 0.20 gm/cc corresponding to a pore volume of 0.90. The electrical resistivity in the plane of the mat is about 0.02 ohm/cm. The resistivity in the direction perpendicular to the mat is about 1.0 ohm/cm.

EXAMPLE II

Preparation of a Porous Fibril Mat

A suspension of fibrils is prepared containing 0.5% fibrils in ethanol using a Waring Blendor. After subsequent dilution to 0.1%, the fibrils are further dispersed with a probe type sonifier. The ethanol is then allowed to evaporate and a mat is formed. The mat has the same physical properties and characteristics as the mat prepared in EXAMPLE I.

EXAMPLE III

Preparation of a Low-Density Porous Fibril Plug

Supercritical fluid removal from a well dispersed-fibril paste is used to prepare low density shapes. 50 cc of a 0.5% dispersion in n-pentane is charged to a pressure vessel of slightly larger capacity which is equipped with a needle valve to enable slow release of pressure. After the vessel is heated above the critical temperature of pentane (Tc = 196.6°), the needle valve is cracked open slightly to bleed the supercritical pentane over a period of about an hour.

The resultant solid plug of Fibrils, which has the shape of the vessel interior, has a density of 0.005 g/cc, corresponding to a pore volume fraction of 0.997%. The resistivity is isotropic and about 20 ohm/cm.

EXAMPLE IV

Preparation of EMI Shielding

A fibril paper is prepared according to the procedures of EXAMPLE I. Table I below sets forth the attenuation achieved at several paper thickness.

TABLE I

FIBRIL PAPER

EMI SHIELDING

ATTENUATION 30 MHz TO 1 GHz

THICKNESS, INCHES (MM)	WEIGHT	ATTENUATION
0.002 (0.5)	12 G/M^2	27 Db
0.005 (.125)	30	37Db
0.017 (.425)	120	48Db

EXAMPLE V

A fibril mat prepared by the method of EXAMPLE I is used as an electrode in an electrochemiluminescence cell such as is described in PCT U.S. 85/02153 (WO 86/02734) and U.S. Patents Nos. 5,147,806 and 5,068,088. When the voltage is pulsed in the presence of ruthenium trisbipyridyl, electrochemiluminescence is observed.

WHAT IS CLAIMED IS:

1. A composition of matter consisting essentially of a three-dimensional, macroscopic assemblage of a multiplicity of a randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc.

2. A composite material comprising:

(a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc; and

(b) a second component comprising (i) particulate solids, or (ii) an electroactive material; or (iii) a catalytically active metal or metal-containing compound, in an amount up to fifty parts per part of fibrils.

3. A composite material as recited in claim 2 wherein said electroactive component is lead or a lead compound or manganese or a manganese compound.

4. A composite material as recited in claim 2 wherein said particulate material is an aluminum oxide, silicon dioxide or silicon carbide.

5. A composite material as recited in claim 2 wherein said particulate material is a catalyst.

6. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively uniform physical properties along at least one dimensional axis thereof and a bulk density of from 0.001 to 0.50 gm/cc.

7. A three-dimensional assemblage as recited in claim 6 having substantially uniform physical properties along said one dimensional axis.

8. A composite material comprising (a) an assemblage of fibrils as recited in claim 6, and (b) a second material at least partially contained within said assemblage.

9. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties in at least one plane thereof and a bulk density of from 0.001 to 0.50 gm/cc.

10. A three-dimensional assemblage as recited in claim 9 having substantially isotropic physical properties in said plane.

11. A composite material comprising (a) an assemblage of fibrils as recited in claim 9 and (b) a second material at least partially contained within said assemblage.

12. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties and a bulk density of from 0.001 to 0.50 gm/cc.

13. A three-dimensional assemblage as recited in claim 12 having substantially isotropic physical properties.

14. A composite material comprising (a) an assemblage of fibrils as recited in claim 12, and (b) a second material at least partially contained within said assemblage.

15. A method of preparing a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, comprising the steps of:

(a) dispersing a multiplicity of said fibrils in a medium; and

(b) separating said assemblage from said medium.

16. A method as recited in claim 15 for the formation of a porous mat or sheet of said carbon fibrils, said mat or sheet having a bulk density of from 0.05 to 0.50 gm/cc comprising the steps of:

(a) vigorously dispersing a multiplicity of said fibrils in water or an organic solvent and thereby forming a dispersion, said dispersion containing less than 10.0 percent by weight of fibrils; and

(b) filtering the dispersion to form said porous mat or sheet.

17. A method as recited in claim 15 for the formation of a porous mat or sheet of said carbon fibrils, said mat or sheet having a bulk density of from 0.05 to 0.50 gm/cc comprising the steps of:

(a) vigorously dispersing a multiplicity of said fibrils in water or an organic solvent and thereby forming a dispersion, said dispersion containing less than 10.0 percent by weight of fibrils; and

(b) permitting the water or organic solvent to evaporate from said dispersion thereby leaving said porous mat or sheet.

18. A method as recited in claim 15 for the formation of a low-density porous plug of said carbon fibrils, said plug having a bulk density of from 0.001 to 0.05 gm/cc comprising the steps of:

(a) forming a gel or paste comprising a multiplicity of carbon fibrils in a fluid, said gel or paste comprising less than 10.0 percent by weight of fibrils;

(b) heating said gel or paste in a pressure vessel to a temperature above the critical temperature of said fluid;

(c) removing supercritical fluid from said pressure vessel; and

(d) removing said porous plug from said pressure vessel.

19. A method of preparing a composite of (a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, and (b) a second material, comprising the steps of:

(a) dispersing a multiplicity of said fibrils and a second material in a medium; and

(b) separating said composite from said medium.

20. A three-dimensional, macroscopic assemblage of randomly oriented carbon fibrils prepared by a method of claim 15.

21. A composite prepared by a method of claim 19.

22. A composite material comprising:

(a) a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc; and

(b) a second component comprising (i) particulate solids, or (ii) an electroactive material; or (iii) a catalytically active metal or metal-containing compound, in an amount up to fifty parts per part of fibrils.

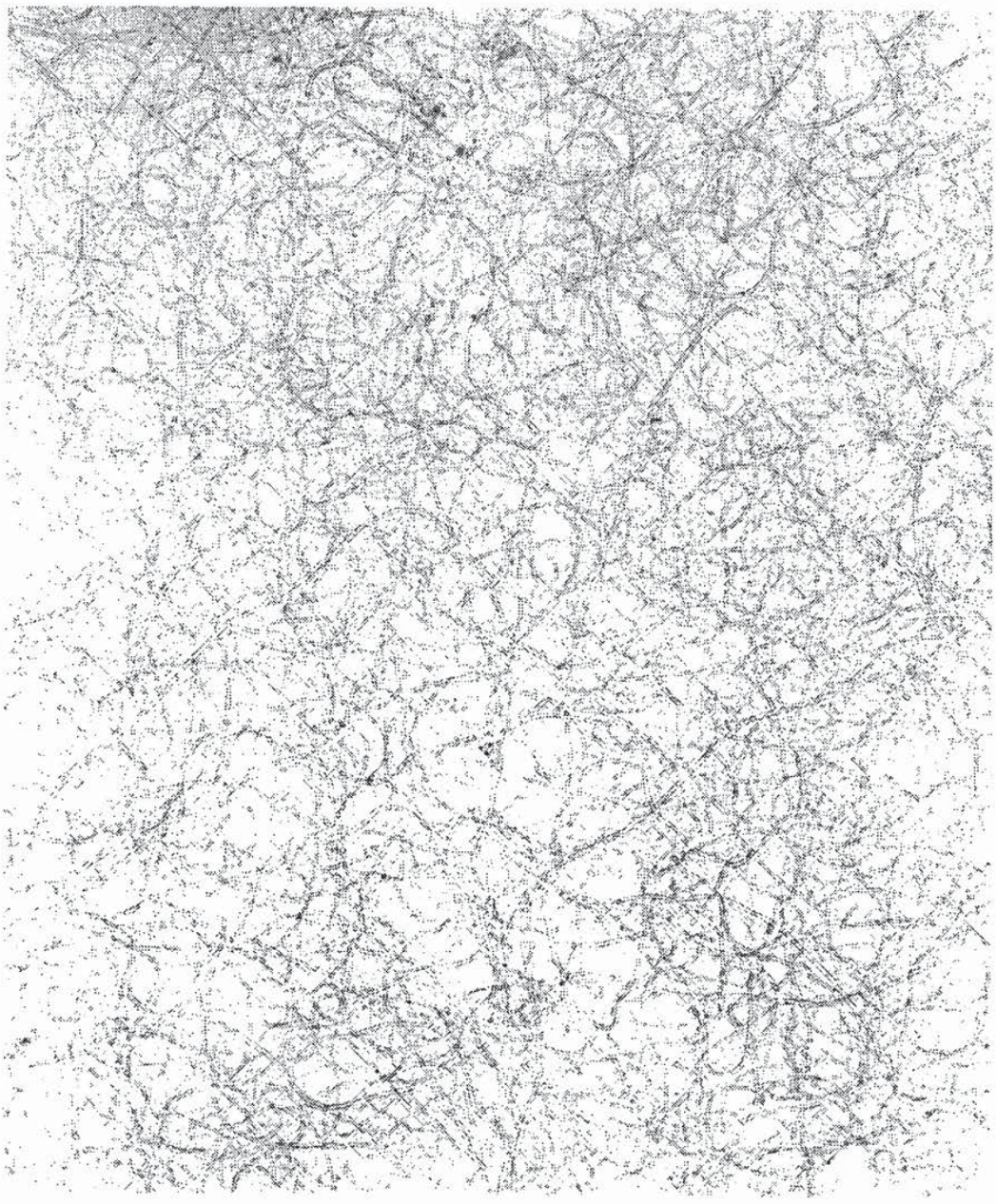
23. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively uniform physical properties along at least one dimensional axis thereof and a bulk density of from 0.001 to 0.50 gm/cc.

24. A three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having relatively isotropic physical properties and a bulk density of from 0.001 to 0.50 gm/cc.

25. A method of preparing a three-dimensional, macroscopic assemblage of a multiplicity of randomly oriented carbon fibrils, said fibrils being substantially cylindrical with a substantially constant diameter, having c-axes less than substantially perpendicular to their cylindrical axis, being substantially free of pyrolytically deposited carbon and having a diameter between about 3.5 and 70 nanometers, said assemblage having a bulk density of from 0.001 to 0.50 gm/cc, comprising the steps of:

(a) dispersing a multiplicity of said fibrils in a medium; and

(b) separating said assemblage from said medium.



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/04879

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :B32B 9/00; D02G 3/00
US CL :428/367
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)

U.S. : 428/359, 360, 367, 372, 408; 210/500.1, 500.25, 500.43, 503, 505, 510.11; 423/447.1, 447.2

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

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,663,230 (TENNENT) 05 May 1987, see Abstract.	1-25
Y	WO, A, WO 86/03455 (HYPERION CATALYSIS INTERNATIONAL, INC.) 19 June 1986, see Abstract.	1-25
Y	WO, A, WO 89/07163 (HYPERION CATALYSIS INTERNATIONAL, INC.) 10 August 1989, see Abstract.	1-25
Y	WO, A, WO 90/07023 (HYPERION CATALYSIS INTERNATIONAL, INC.) 28 June 1990, see Abstract.	1-25
Y	WO, A, WO 91/05089 (HYPERION CATALYSIS INTERNATIONAL, INC.) 18 April 1991, see Abstract.	1-25

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be part of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
22 JULY 1994	13 SEP 1994
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer PATRICK RYAN Telephone No. (703) 308-2351

Form PCT/ISA/210 (second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/04879

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO, A, WO 91/01219 (HYPERION CATALYSIS INTERNATIONAL, INC.) 07 February 1991, see Abstract.	1-25

Form PCT/ISA/210 (continuation of second sheet)(July 1992)*

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Département Propriété Industrielle

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Colombes, le 6 septembre 2018

Objet : Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA FRANCE

Madame, Monsieur,

Nous faisons suite à la notification datée du 7 mai 2018 concernant la citation à une procédure orale le 15 novembre 2018.

Nous présentons ci-dessous nos commentaires sur l'opinion préliminaire de la Division d'Opposition, ainsi que le document A9 (WO 94/25268).

Au vu des arguments présentés ci-après et de ceux présentés dans le mémoire d'opposition, l'opposant considère que le brevet opposé doit être révoqué dans sa totalité.

Nous vous prions d'agréer, Madame, Monsieur, nos salutations distinguées.

Veuillez agréer, Madame, Monsieur, nos salutations distinguées.

Claudine BONNEL
PG n° 8646

1) Insuffisance de description

L'opposant estime, comme mentionné dans le mémoire d'opposition, que l'objet de la revendication 1 n'est pas suffisamment décrit pour qu'un homme du métier puisse l'exécuter.

En effet, le brevet, comme la demande dont il est issu, ne contient aucune information sur la méthode de mesure utilisée pour déterminer le niveau d'oxydation des NTC. Ce paramètre est pourtant présenté comme une caractéristique essentielle de l'invention.
Selon la Division d'opposition, cela relèverait plutôt de l'Article 84 CBE, et l'Article 83 CBE serait satisfait.

La Division d'opposition se base sur les 5 exemples du brevet et à leur reproductibilité.
Cependant, il est fait remarqué que l'exemple 1 (illustrant des NTC oxydés - taux d'oxydation 8%- avec des particules d'oxyde de cuivre attachées à leur surface) et l'exemple 5 (illustrant des NTC oxydés - taux d'oxydation 4% - avec des particules d'oxyde de titane attachées à leur surface) ne sont pas des exemples selon l'invention revendiquée dans la revendication 1.
Seuls les exemples 2, 3 et 4 illustrent des NTC oxydés (à un taux de 4% ou 8%) comportant à leur surface des particules comportant du lithium.

Le paragraphe [0022] du brevet contesté donne une définition pour les NTC oxydés, à savoir des produits d'oxydation des NTC, qui sont « généralement » de type carboxylique, cétone ou hydroxyle.

L'incertitude pour l'homme du métier qui veut reproduire l'invention est donc de savoir comment exprimer la présence de ces espèces oxydées, sous la forme d'un taux d'oxydation.

Il existe de nombreuses façons pour quantifier des espèces oxydées, par exemple une teneur en oxygène, (qui fait appel aussi à plusieurs techniques possibles, XPS, ESCA, analyse élementaire), une acidité (quand il s'agit d'espèces carboxyliques), un indice de cétone ou un indice d'hydroxyle, ou une thermogravimétrie (décomposition sous une certaine température).

L'homme du métier n'est pas en mesure de savoir s'il travaille ou non dans la portée du brevet contesté, compte tenu de l'absence de méthode de détermination du taux d'oxydation.

L'opposant est d'avis que l'objet des revendications du brevet opposé ne satisfait pas aux exigences de l'Article 83 CBE et ne partage pas l'opinion de la Division d'opposition.

2) Activité inventive

a) Afin de compléter l'argumentation présentée dans le mémoire d'opposition vis-à-vis de A3, nous fournissons comme document A9, la demande WO 94/25268 qui revendique la priorité de la demande US 08/057,328 déposée le 05.05.1993 mentionnée dans A3 (page 6, l.32-36).

b) L'opposant partage l'avis de la Division d'opposition selon lequel A4 peut être considéré comme un document de la technique le plus proche de l'invention.

Comme l'a indiqué la Division d'opposition dans son opinion préliminaire, à juste titre, aucun effet technique dérivé des différences entre l'invention du brevet contesté et A4 n'a été démontré ; le problème technique objectif est ainsi la fourniture d'une composition alternative utilisable pour les batteries Li-ion.

Cependant, la Division d'opposition estimerait qu'une activité inventive pourrait résulter du fait que l'homme du métier aurait du faire plusieurs sélections dans A4 pour parvenir à son invention.

L'opposant ne partage pas cet avis, pour les raisons suivantes :

- Dans A4 [0032], il est clairement stipulé que « 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 ».

  L'utilisation de NTC oxydés n'est pas une selection motivée, mais équivalente à l'utilisation d'autres types de NTC. De plus, rien dans le brevet contesté ne permet de prouver que les NTC oxydés sont avantageux par rapport à des NTC « non oxydés ».

  L'utilisation de NTC ayant un rapport d'aspect d'au moins 100 n'est pas une selection motivée, mais équivalente à l'utilisation de NTC ayant un autre rapport d'aspect.

- Même si A4 ne divulgue pas spécifiquement un niveau d'oxydation lorsqu'il s'agit de NTC oxydés, la détermination de la gamme 1-15% telle que revendiquée dans le brevet contesté peut résulter d'expérimentations classiques de routine visant l'optimisation de la composition.
  La sélection ne peut impliquer une activité inventive que si elle entraine un effet particulier ; sans effet démontré, la sélection peut résulter de simples expérimentations que l'homme du métier peut exécuter sans exercer d'activité inventive.

c) Le même raisonnement relatif à A3 peut s'appliquer : la détermination d'un taux d'oxydation optimal pour les NTC discrets (non agrégés) oxydés divulgués dans A3 peut être réalisée par de simples expérimentation de routine.
En conséquence, l'invention revendiquée n'implique pas d'activité inventive.

Pour toutes ces raisons, la composition revendiquée dans la revendication 1 ne satisfait pas l'Article 56 CBE.

****

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V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date: 08.08.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☒ Oral Proceedings on 15.11.2018

Enclosure(s):
- ☐ Letter from the proprietor of the patent of
- ☒ Letter from the opponent 01 of 02.08.2018
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

☒ **Registered letter**

Europäisches Patentamt
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Lettre d'accompagnement relative à des pièces produites ultérieurement

Mandataire:

Mme Claudine BONNEL
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La (Les) pièce(s) désignées ci-après est (sont) produite(s) postérieurement au dépôt, pour la demande suivante :

N° de la demande	12731264.3
Référence du demandeur ou du mandataire	AL 0378 CBL

	Description des pièces	Nom du dossier original	Nom du dossier attribué
1	Lettre concernant une procédure orale	AL 0378.pdf	ORAL-1.pdf

	Paiement	
1	Méthode de paiement	Non spécifié

Signatures

Lieu:	**Colombes**
Date:	**02 août 2018**
Signé par:	**Claudine Bonnel 7787**
Nom du mandataire:	**Claudine BONNEL**
Fonction:	**(Mandataire)**

ARKEMA
INNOVATIVE CHEMISTRY

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Colombes, le 3 août 2018

Objet :Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA FRANCE

Madame, Monsieur,

Nous faisons suite à la notification datée du 7 mai 2018 concernant la citation à une procédure orale le 15 novembre 2018.

Nous vous informons que l'opposant sera représenté par la soussignée qui s'exprimera en langue française et demande une traduction simultanée pour toute autre langue que le français.

Veuillez agréer, Madame, Monsieur, nos salutations distinguées.

Claudine BONNEL
PG n° 8646

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Accusé de réception

Par la présente, nous accusons réception des pièces suivantes produites postérieurement au dépôt:

Numéro de soumission	6582111
Numéro de demande	EP12731264.3
Date de réception	02 août 2018
Office récepteur	Office européen des brevets, La Haye
Votre référence	AL 0378 CBL
Nom du demandeur	Tous les déposants mentionnés dans le dossier
Documents envoyés	package-data.xml epf1038.pdf (1 p.) ep-sfd-request.xml ORAL-1.pdf\AL 0378.pdf (1 p.)
Effectué par	CN=Claudine Bonnel 7787
Méthode de soumission	En ligne
Date et heure de réception	02 août 2018, 16:13 (CEST)
Message condensé	1B:2A:92:FC:A2:D4:BC:38:94:F8:CC:C5:F1:FD:86:3B:58:F8:42:F0

Correction par l'OEB d'erreurs figurant dans les ordres de débit transmis via eOLF
Les erreurs figurant dans les ordres de débit transmis via eOLF et causées par une modification des informations saisies dans le formulaire 1038E ou une utilisation persistante de logiciels dépassés (tous les formulaires) peuvent être corrigées automatiquement par l'OEB, sans que la date de paiement soit modifiée (cf. décision T 152/82, JO OEB 1984, 301 et point 6.3 s. RCC, supplément au JO OEB 10/2007).

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Lettre d'accompagnement relative à des pièces produites ultérieurement

Mandataire:

Mme Claudine BONNEL
ARKEMA FRANCE
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 COLOMBES CEDEX
France

Téléphone: 33 (0) 1 49 00 80 26
Télécopie : 33 (0) 1 49 00 80 87
Courriel : claudine.bonnel@arkema.com

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2280 HV Rijswijk
Netherlands
Tel. +31(0)70 340-2040 | Fax -3016

10958 Berlin
Germany
Tel. +49(0)30 25901-0 | Fax -840

La (Les) pièce(s) désignées ci-après est (sont) produite(s) postérieurement au dépôt, pour la demande suivante :

N° de la demande	12731264.3
Référence du demandeur ou du mandataire	AL 0378 CBL

	Description des pièces	Nom du dossier original	Nom du dossier attribué
1	Lettre concernant une procédure orale	AL 0378.pdf	ORAL-1.pdf

	Paiement	
1	Méthode de paiement	Non spécifié

Signatures

Lieu:	**Colombes**
Date:	**02 août 2018**
Signé par:	**Claudine Bonnel 7787**
Nom du mandataire:	**Claudine BONNEL**
Fonction:	**(Mandataire)**

ARKEMA
INNOVATIVE CHEMISTRY

ARKEMA France
Direction Recherche Développement
Département Propriété Industrielle

OFFICE EUROPEEN DES BREVETS
Direction Générale 2
D-80469 MÜNCHEN
ALLEMAGNE

N/Réf. : AL 0378 EP CBL/eo
☎ : (33 1) 49 00 80 26
Télécopie : (33 1) 49 00 80 87
E-mail : claudine.bonnel@arkema.com

Colombes, le 3 août 2018

Objet :Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA FRANCE

Madame, Monsieur,

Nous faisons suite à la notification datée du 7 mai 2018 concernant la citation à une procédure orale le 15 novembre 2018.

Nous vous informons que l'opposant sera représenté par la soussignée qui s'exprimera en langue française et demande une traduction simultanée pour toute autre langue que le français.

Veuillez agréer, Madame, Monsieur, nos salutations distinguées.

Claudine BONNEL
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ARKEMA France
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420, rue d'Estienne d'Orves
92705 Colombes Cedex
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Date: 25.05.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☒ Oral Proceedings on 15.11.2018

Enclosure(s):
- ☒ Letter from the proprietor of the patent of 22.05.2018
- ☐ Letter from the opponent of
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

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Germany

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Carnegieplein 5
P.O. Box 87930
2508 DH The Hague
The Netherlands

Telephone +31 70 416 67 11
Telefax +31 70 416 67 99

H.Marsman@vo.eu
www.vo.eu

Your ref. 12731264.3/1108
Our ref. MAS/P100028EP00

The Hague,
May 22, 2018

Re: European Patent No. 2724403
Molecular Rebar Design, LLC

Thank you for your cooperation in finding a new date for Oral Proceedings.

During the Oral Proceedings scheduled for November 15, 2018 I will use the English language.

Assuming that Opponent Arkema will use the French language, interpretation is requested from French into English.

The professional representative,



H.A.M. Marsman

EK

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Acknowledgement of receipt

We hereby acknowledge receipt of the following subsequently filed document(s):

Submission number	6344862	
Application number	EP12731264.3	
Date of receipt	22 May 2018	
Receiving Office	European Patent Office, The Hague	
Your reference	P100028EP00	
Applicant	All applicants as on file	
Documents submitted	package-data.xml epf1038.pdf (1 p.)	ep-sfd-request.xml ORAL-1.pdf\P100028EP00 Letter to EPO.pdf (1 p.)
Submitted by	CN=Hermanus Marsman 34472	
Method of submission	Online	
Date and time receipt generated	22 May 2018, 13:16 (CEST)	
Message Digest	70:57:F0:26:B1:6E:B4:C2:D4:DD:66:FA:65:31:BA:C1:8F:96:1B:3B	

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Anmeldung Nr./Application No./Demande n°
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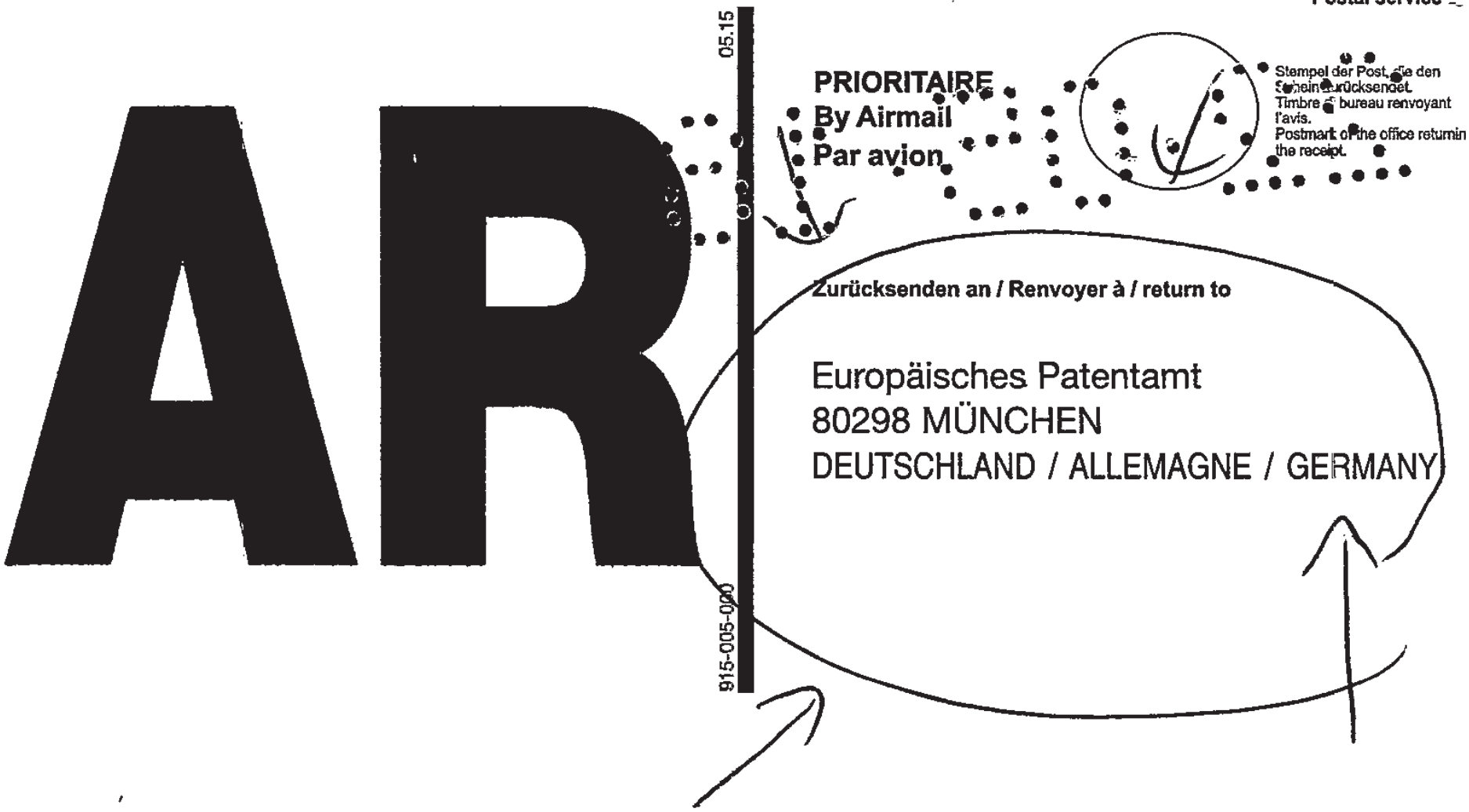
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Bonnel, Claudine
ARKEMA France
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92705 Colombes Cedex
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Letter accompanying subsequently filed items

Representative:
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10958 Berlin
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Tel. +49(0)30 25901-0 | Fax -840

The document(s) listed below is (are) subsequently filed documents pertaining to the following application:

Application number	12731264.3
Applicant's or representative's reference	P100028EP00

	Description of document	Original file name	Assigned file name
1	Letter dealing with Oral proceedings	P100028EP00 Letter to EPO.pdf	ORAL-1.pdf

	Payment	
1	Method of payment	Not specified

Signatures

Place:	**Utrecht**
Date:	**22 May 2018**
Signed by:	**/H.A.M. Marsman/**
Association:	**V.O.**
Representative name:	**H.A.M. Marsman**
Capacity:	**(Representative)**

Patents & Trademarks

European Patent Office
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Telefax +31 70 416 67 99

H.Marsman@vo.eu
www.vo.eu

Your ref. 12731264.3/1108
Our ref. MAS/P100028EP00

The Hague,
May 22, 2018

Re: European Patent No. 2724403
Molecular Rebar Design, LLC

Thank you for your cooperation in finding a new date for Oral Proceedings.

During the Oral Proceedings scheduled for November 15, 2018 I will use the English language.

Assuming that Opponent Arkema will use the French language, interpretation is requested from French into English.

The professional representative,



H.A.M. Marsman

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Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date: 07.05.2018

Reference		Application No./Patent No.
AL 0378	OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor		
Molecular Rebar Design, LLC		

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Received at the EPO on May 15, 2018 07:14:21. Page 1 of 1
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Reference	Application No./Patent No.
P100026EP00	12731264.3 - 1108 / 2724403

Applicant/Proprietor: Molecular Rebar Design, LLC

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Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
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92705 Colombes Cedex
FRANCE

Date: 07.05.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

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2508 DH Den Haag
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Date
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Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

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(Umschlag / envelope / enveloppe ISO C4 / DL / C6/C5 / C6)

Europäisches Patentamt
80298 MÜNCHEN
DEUTSCHLAND

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EPA/EPO/OEB Form 2936 08.10

SS51511

Europäisches Patentamt
European Patent Office
Office européen des brevets

Wichtige Hinweise zur mündlichen Verhandlung

Das Europäische Patentamt verfügt über keine eigenen Dolmetscher. Diese müssen im Bedarfsfall von außerhalb, teilweise sogar aus anderen Ländern, beigezogen werden, was mit einem hohen Aufwand an Kosten und organisatorischen Vorbereitungen verbunden ist. Muss ein Verhandlungstermin kurzfristig abberaumt werden, können Kosten für bestellte Dolmetscher nicht mehr vermieden werden.

Es wird daher gebeten, eine Simultanübersetzung nur bei wirklichem Bedarf in Anspruch zu nehmen. Es wäre wünschenswert, wenn sich die Beteiligten (zweckmäßigerweise gleichzeitig mit der Terminabstimmung) auf die Benutzung einer Amtssprache einigen könnten. Bei Verständigungsschwierigkeiten sind die Mitglieder der Einspruchsabteilung bereit zu helfen.

Die von den Verfahrensbeteiligten bevorzugte (abgestimmte) Verhandlungssprache und ggf. eine notwendige Simultanübersetzung sind dem Amt möglichst vor der in Regel 4(1) EPÜ angegebenen Frist mitzuteilen.

☐ Verfahrenssprache ist **Deutsch**

Von der/dem/den Einsprechenden wurde

☐ Englisch

☐ Französisch benutzt.

Es wird um eilige Mitteilung - möglichst per Telefax an den zuständigen Formalprüfer - gebeten,

möglichst bis

Datum 14.09.2018

1. welche Sprache(n) Sie in der mündlichen Verhandlung verwenden (**Sprechen**)
2. aus welcher Sprache Sie eine Simultanübersetzung benötigen (**Hören**).

Important information concerning oral proceedings

The European Patent Office has no interpreters of its own. When interpreters are needed they have to be brought in from outside, sometimes even from other countries, which is costly and involves considerable organisation. If oral proceedings have to be cancelled at short notice, the cost of interpreters already engaged still has to be borne.

Please therefore make use of simultaneous interpreting facilities only where strictly necessary. If possible the parties should agree on an official language for the proceedings, preferably at the time when they arrange a date. The members of the Opposition Division will be willing to help should any communication problems arise.

The EPO should be told if possible before the period mentioned in Rule 4 (1) EPC which language the parties prefer (agree on) and whether simultaneous interpreting facilities are required.

☒ Language of the proceedings is **English**

The language used by the opponent/s was

☐ German

☒ French.

Please inform us urgently - where possible by fax addressed to the formalities officer concerned -

if possible by

Date 14.09.2018

1. which language(s) you intend to use during the oral proceedings (**Speaking**)
2. from which language you need simultaneous interpretation (**Listening**).

Très important Procédure orale

L'Office européen des brevets ne dispose pas de son propre service d'interprètes. Aussi faut-il appel le cas échéant à des interprètes de l'extérieur, qui viennent même parfois de l'étranger, ce qui occasionne de frais élevés et demande un grand travail d'organisation. Si la date d'une procédure orale doit être annulée au dernier moment, il n'est plus possible d'éviter les frais d'interprètes.

Les parties à une procédure sont donc priées de ne demander une traduction simultanée qu'en cas de réel besoin. Il serait souhaitable qu'elles puissent se mettre d'accord en même temps qu'elles conviennent de la date sur l'utilisation d'une langue officielle comme langue des débats. Si les parties éprouvent des difficultés de compréhension lors des débats, les membres de la division d'opposition sont disposés à leur prêter leur assistance.

L'Office doit être avisé si possible avant le début du délai mentionné dans la règle 4(1) CBE de la langue préférée par les parties pour le déroulement des débat (et sur laquelle elles se sont préalablement mises d'accord) et de la nécessité éventuelle d'une traduction simultanée.

☐ La langue de la procédure est le **français**

La langue utilisée par l'opposant/les opposants était

☐ l'allemand

☐ l'anglais.

Prière d'indiquer d'urgence à l'agent des formalités compétent si possible par téléfax

si possible jusqu'au

Date 14.09.2018

1. quelle(s) langue(s) vous utiliserez au cours de la procédure orale (**pour parler**)
2. à partir de quelle langue vous aurez besoin d'une traduction simultanée (**pour écouter**).

EPA/EPO/OEB Form 2043 02.09

12731264.3

Sollten Sie Ihren Antrag auf mündliche Verhandlung zurückziehen oder zum anberaumten Verhandlungstermin nicht erscheinen wollen bzw. aus wichtigem Grund daran gehindert sein, werden Sie gebeten,

- **unverzüglich das Amt - möglichst per Telefax - davon zu benachrichtigen, wobei das Schriftstück mit einem deutlichen Vermerk "Dringend, mündliche Verhandlung am ..." oder sinngemäß gekennzeichnet sein sollte;**
- **in dringenden Fällen** (weniger als 1 Monat vor dem Verhandlungstermin) zusätzlich **auch dem/die anderen Verfahrensbeteiligten** bzw. ihre(n) Vertreter auf schnellstem Weg **direkt zu unterrichten.**

In jedem solchen Fall obliegt der Einspruchsabteilung die Entscheidung, ob die Verhandlung durchgeführt oder abberaumt wird. Es wird jedoch darauf hingewiesen, dass einem Verfahrensbeteiligten, der eine nicht rechtzeitige oder unterbliebene Benachrichtigung zu verantworten hat, die dadurch den anderen Beteiligten verursachten Kosten auferlegt werden können (Art. 104 EPÜ).

Hinweis auf Regel 4 EPÜ

Regel 4
Sprache im mündlichen Verfahren

(1) Jeder an einem mündlichen Verfahren vor dem Europäischen Patentamt Beteiligte kann sich anstelle der Verfahrenssprache einer anderen Amtssprache des Europäischen Patentamts bedienen, sofern er dies dem Europäischen Patentamt spätestens einen Monat vor dem angesetzten Termin mitgeteilt hat oder selbst für die Übersetzung in die Verfahrenssprache sorgt. Jeder Beteiligte kann sich einer Amtssprache eines Vertragsstaats bedienen, sofern er selbst für die Übersetzung in die Verfahrenssprache sorgt. Von diesen Vorschriften kann das Europäische Patentamt Ausnahmen zulassen.

Should you decide to withdraw your request for oral proceedings or not wish to attend on the date set, or if for some special reason you are unable to do so, you are requested

- **to notify the EPO immediately, where possible by fax, marking the document clearly with the words "Urgent, oral proceedings on ..." or similar;**
- **in urgent cases** (less than one month before the date set for the proceedings), additionally to **notify the other party/parties** and/or their representative(s) **direct as rapidly as possible.**

In all such cases the Opposition Division will decide whether the proceedings are to go ahead or be cancelled. You should however note that costs incurred by the other parties may be charged to a party who either fails to notify them or does not do so in good time (Article 104 EPC).

Attention is drawn to Rule 4 EPC

Rule 4
Language in oral proceedings

(1) Any party to oral proceedings before the European Patent Office may use an official language of the European Patent Office other than the language of the proceedings, if such party gives notice to the European Patent Office at least one month before the date of such oral proceedings or provides for interpretation into the language of the proceedings. Any party may use an official language of a Contracting State, if he provides for interpretation into the language of the proceedings. The European Patent Office may permit derogations from these provisions.

Si vous retirez votre requête tendant à recourir à la procédure orale ou si vous ne souhaitez pas vous présenter à la date fixée pour la procédure orale ou ne pouvez vous y présenter pour une raison sérieuse, veuillez

- **en faire avis sans retard à l'Office, si possible par téléfax, en partant sur votre communication clairement la mention "Urgent, procédure orale le ..." ou une indication similaire;**
- **dans les cas urgents** (moins d'un mois avant la date fixée pour la procédure orale) **en faire avis également directement** pas la voie la plus rapide à **l'autre/aux autres partie(s)** ou bien à son/leurs mandataire(s).

Il appartient alors à la division d'opposition de décider si la procédure orale aura lieu ou non. Il est néanmoins souligné que les frais causés aux autres parties par une partie qui est responsable de l'omission d'un tel avis ou de ce que cet avis n'a pas été fait en temps utile peuvent être mis à la charge de cette partie (art. 104 CBE).

Rappel de la Règle 4 CBE

Règle 4
Langues admissibles lors de la procédure orale

(1) Toute partie à une procédure orale devant l'Office européen des brevets peut utiliser une langue officielle de l'Office européen des brevets autre que la langue de la procédure, à condition soit d'en aviser l'Office européen des brevets un mois au moins avant la date de la procédure orale, soit d'assurer l'interprétation dans la langue de la procédure. Toute partie peut utiliser une langue officielle de l'un des Etats contractants à condition d'assurer l'interprétation dans la langue de la procédure. L'Office européen des brevets peut autoriser des dérogations aux présentes dispositions.

(2) Die Bediensteten des Europäischen Patentamts können sich im mündlichen Verfahren anstelle der Verfahrenssprache einer anderen Amtssprache des Europäischen Patentamts bedienen.

(2) In the course of oral proceedings, employees of the European Patent Office may use an official language of the European Patent Office other than the language of the proceedings.

(2) Au cours de la procédure orale, les agents de l'Office européen des brevets peuvent utiliser une langue officielle de l'Office européen des brevets autre que la langue de la procédure.

(3) In der Beweisaufnahme können sich die zu vernehmenden Beteiligten, Zeugen oder Sachverständigen, die sich in einer Amtssprache des Europäischen Patentamts oder eines Vertragsstaats nicht hinlänglich ausdrücken können, einer anderen Sprache bedienen. Erfolgt die Beweisaufnahme auf Antrag eines Beteiligten, so werden die Beteiligten, Zeugen oder Sachverständigen mit Erklärungen, die sie in einer anderen Sprache als in einer Amtssprache des Europäischen Patentamts abgeben, nur gehört, sofern dieser Beteiligte selbst für die Übersetzung in die Verfahrenssprache sorgt. Das Europäische Patentamt kann jedoch die Übersetzung in eine seiner anderen Amtssprachen zulassen.

(3) Where evidence is taken, any party, witness or expert to be heard who is unable to express himself adequately in an official language of the European Patent Office or of a Contracting State may use another language. Where evidence is taken upon request of a party, parties, witnesses or experts expressing themselves in a language other than an official language of the European Patent Office shall be heard only if that party provides for interpretation into the language of the proceedings. The European Patent Office may, however, permit interpretation into one of its other official languages.

(3) Lors de l'instruction, les parties, témoins ou experts appelés à être entendus, qui ne possèdent pas une maîtrise suffisante d'une langue officielle de l'Office européen des brevets ou d'un Etat contractant, peuvent utiliser une autre langue. Si la mesure d'instruction est ordonnée sur requête d'une partie, les parties, témoins ou experts qui s'expriment dans une langue autre qu'une langue officielle de l'Office européen des brevets ne sont entendus que si cette partie assure l'interprétation dans la langue de la procédure. L'Office européen des brevets peut toutefois autoriser l'interprétation dans l'une de ses autres langues officielles.

(4) Mit Einverständnis aller Beteiligten und des Europäischen Patentamts kann jede Sprache verwendet werden.

(4) If the parties and the European Patent Office agree, any language may be used.

(4) Sous réserve de l'accord des parties et de l'Office européen des brevets, toute langue peut être utilisée.

(5) Das Europäische Patentamt übernimmt, soweit erforderlich, auf seine Kosten die Übersetzung in die Verfahrenssprache und gegebenenfalls in seine anderen Amtssprachen, sofern ein Beteiligter nicht selbst für die Übersetzung zu sorgen hat.

(5) The European Patent Office shall, if necessary, provide at its own expense interpretation into the language of the proceedings, or, where appropriate, into its other official languages, unless such interpretation is the responsibility of one of the parties.

(5) L'Office européen des brevets assure à ses frais, en tant que de besoin, l'interprétation dans la langue de la procédure, ou, le cas échéant, dans ses autres langues officielles, à moins que cette interprétation ne doive être assurée par l'une des parties.

(6) Erklärungen von Bediensteten des Europäischen Patentamts, Beteiligten, Zeugen und Sachverständigen, die in einer Amtssprache des Europäischen Patentamts abgegeben werden, werden in dieser Sprache in die Niederschrift aufgenommen. Erklärungen in einer anderen Sprache werden in der Amtssprache aufgenommen, in die sie übersetzt worden sind. Änderungen einer europäischen Patentanmeldung oder eines europäischen Patents werden in der Verfahrenssprache in die Niederschrift aufgenommen.

(6) Statements by employees of the European Patent Office, parties, witnesses or experts, made in an official language of the European Patent Office, shall be entered in the minutes in that language. Statements made in any other language shall be entered in the official language into which they are translated. Amendments to a European patent application or European patent shall be entered in the minutes in the language of the proceedings.

(6) Les interventions des agents de l'Office européen des brevets, des parties, témoins et experts faites dans une langue officielle de l'Office européen des brevets sont consignées au procès-verbal dans cette langue. Les interventions faites dans une autre langue sont consignées dans la langue officielle dans laquelle elles sont traduites. Les modifications apportées à une demande de brevet européen ou à un brevet européen sont consignées au procès verbal dans la langue de la procédure.

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
07.05.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

The earlier summons dated 11.04.2018 are superseded herewith.

Summons to attend oral proceedings pursuant to Rule 115(1) EPC

You are hereby summoned to attend oral proceedings arranged in connection with the above-mentioned European patent.

The matters to be discussed are set out in the communication accompanying this summons (EPO Form 2906). *

The oral proceedings, which will be public, will take place before the opposition division

on 15.11.18 at 09.00 hrs in Room 1653 at the EPO,
Grasserstr. 2, PschorrHöfe, D-80335 München

No changes to the date of the oral proceedings can be made, except on serious grounds (see OJ EPO 1/2009, 68). If you do not appear as summoned, the oral proceedings may continue without you (R. 115(2) EPC).

Your attention is drawn to Rule 4 EPC, regarding the language of the oral proceedings, and to the Special edition No. 3 OJ EPO 2007, 128, concerning the filing of authorisations for company employees and lawyers acting as representatives before the EPO.

The final date for making written submissions and/or amendments (R. 116 EPC) is 14.09.18.

You are requested to report in good time beforehand to the porter in the EPO foyer. Room 1657 is available as waiting room.

Parking is available in the underground car park, accessible only via the entrance "Grasserstrasse 2/6". On presentation of the summons to oral proceedings at the porters' lodge in the main foyer "Bayerstrasse", the parking ticket will be revoked.

1st Examiner:	2nd Examiner:	Chairman:
Bettio A	Kelly M	Götz H

For the Opposition Division

Annexes:
Confirmation of receipt (Form 2936)
Rule 4 EPC (EPC Form 2043)
Communication (EPO Form 2906)
* enclosed in earlier summons

Registered letter with advice of delivery

to EPO postal service: 26.04.18

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date: 07.05.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

The earlier summons dated 11.04.2018 are superseded herewith.

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1st Examiner:	2nd Examiner:	Chairman:
Bettio A	Kelly M	Götz H

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

Annexes:
Confirmation of receipt (Form 2936)
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to EPO postal service: 26.04.18

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
03.05.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Oral Proceedings on 05.11.18

Subject:
- [x] Letter from the proprietor of the patent of 20.04.2018
- [ ] Letter from the opponent of

Communication:
- [x] The summons to attend oral proceedings / taking of evidence on 05.11.18 has been cancelled.
- [ ] The procedure will be continued in writing.
- [ ] The date fixed for oral proceedings is maintained.
- [x] A new date will be set later.
- [ ]

Please take note.

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

Registered letter
EPO Form 2310A 12.07 (26/04/18)

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date: 03.05.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Oral Proceedings on 05.11.18

Subject:
- [x] Letter from the proprietor of the patent of 20.04.2018
- [ ] Letter from the opponent of

Communication:
- [x] The summons to attend oral proceedings / taking of evidence on 05.11.18 has been cancelled.
- [ ] The procedure will be continued in writing.
- [ ] The date fixed for oral proceedings is maintained.
- [x] A new date will be set later.
- [ ]

Please take note.

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

Registered letter
EPO Form 2310A 12.07 (26/04/18)

Europäisches Patentamt
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Bonnel, Claudine
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420, rue d'Estienne d'Orves
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FRANCE

Date: 26.04.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☒ Oral Proceedings on 05.11.2018

Enclosure(s):
- ☒ Letter from the proprietor of the patent of 20.04.2018
- ☐ Letter from the opponent of
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

☒ **Registered letter**

Patents & Trademarks

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H.Marsman@vo.eu
www.vo.eu

Your ref. 12731264.3/1108
Our ref. MAS/P100028EP00

The Hague,
April 20, 2018

Re: European Patent No. 2724403
Molecular Rebar Design, LLC

I refer to the Summons dated April 11, 2018 to attend Oral Proceedings on **November 5, 2018**.

I request that this Hearing be rescheduled. It is not needed that the Hearing is postponed; it may also be planned earlier.

This request is because I am a tutor for the CEIPI in Strasbourg on the training for the EQE. From November 5-9, I am tutor for the Pre-Exam Seminar. (In the week of November 19-22, I have to tutor for the A and B-Seminar.)

To assist you on finding an alternative date I note that from October 1 – December 15 currently I only have inconvenience dates (except November 5-9 and 19-22) on October 18 and December 4-6, because of complicated Oral Proceedings before the Board of Appeal.

V.O.
Patents & Trademarks

Patentee is a relatively small company and so far I personally have dealt with their patent applications outside the U.S.A., it would be quite a burden to involve one of my colleagues.

If needed I can send you further evidence that indeed I have to tutor for the CEIPI.

A copy of this request is sent to Opponent's representative, as well.

Your assistance on this matter would be highly appreciated.

The professional representative,



H.A.M. Marsman

Cc. Opponent's representative

EK

Europäisches Patentamt
European Patent Office
Office européen des brevets

Acknowledgement of receipt

We hereby acknowledge receipt of the following subsequently filed document(s):

Submission number	6254824	
Application number	EP12731264.3	
Date of receipt	20 April 2018	
Receiving Office	European Patent Office, The Hague	
Your reference	P100028EP00	
Applicant	All applicants as on file	
Documents submitted	package-data.xml epf1038.pdf (1 p.)	ep-sfd-request.xml ORAL-1.pdf\P100028EP00 Letter to EPO.pdf (2 p.)
Submitted by	CN=Hermanus Marsman 34472	
Method of submission	Online	
Date and time receipt generated	20 April 2018, 16:41 (CEST)	
Message Digest	1D:D4:B1:C2:A7:10:D4:4F:A4:05:F2:44:0B:C6:CA:EB:7D:15:AA:2A	

Correction by the EPO of errors in debit instructions filed by eOLF
Errors in debit instructions filed by eOLF that are caused by the editing of Form 1038E entries or the continued use of outdated software (all forms) may be corrected automatically by the EPO, leaving the payment date unchanged (see decision T 152/82, OJ EPO 1984, 301 and point 6.3 ff ADA, Supplement to OJ EPO 10/2007).

/European Patent Office/

Europäisches Patentamt
European Patent Office
Office européen des brevets

Letter accompanying subsequently filed items

Representative:
V.O.
106850
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Tel. +31(0)70 340-2040 | Fax -3016

10958 Berlin
Germany
Tel. +49(0)30 25901-0 | Fax -840

The document(s) listed below is (are) subsequently filed documents pertaining to the following application:

Application number	12731264.3
Applicant's or representative's reference	P100028EP00

	Description of document	Original file name	Assigned file name
1	Letter dealing with Oral proceedings	P100028EP00 Letter to EPO.pdf	ORAL-1.pdf

	Payment	
1	Method of payment	Not specified

Signatures

Place:	**Utrecht**
Date:	**20 April 2018**
Signed by:	**/H.A.M. Marsman/**
Association:	**V.O.**
Representative name:	**H.A.M. Marsman**
Capacity:	**(Representative)**

Patents & Trademarks

European Patent Office
D-80298 Munich
Germany

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H.Marsman@vo.eu
www.vo.eu

Your ref. 12731264.3/1108
Our ref. MAS/P100028EP00

The Hague,
April 20, 2018

Re: European Patent No. 2724403
Molecular Rebar Design, LLC

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V.O.
Patents & Trademarks

Patentee is a relatively small company and so far I personally have dealt with their patent applications outside the U.S.A., it would be quite a burden to involve one of my colleagues.

If needed I can send you further evidence that indeed I have to tutor for the CEIPI.

A copy of this request is sent to Opponent's representative, as well.

Your assistance on this matter would be highly appreciated.

The professional representative,



H.A.M. Marsman

Cc. Opponent's representative

EK

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V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
11.04.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

EPA/EPO/OEB Formblatt/Form/Formulaire : 2310 & 2906O

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Date
11.04.2018

Reference		Application No./Patent No.
AL 0378	OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor		
Molecular Rebar Design, LLC		

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Claudine BONNEL

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SS51511

Introduction

In the notice of opposition of 05.07.2017 the opponent (O) has requested to revoke the patent in its entirety according to Art. 101(2) EPC for lack of novelty and inventive step (Art. 100(a) EPC), lack of sufficiency of disclosure in the sense of Art 83 EPC (Art. 100(b) EPC) and extension of subject-matter (Art. 100(c) EPC). O additionally requested oral proceedings according to Art. 116 EPC.

The following documents have been cited by O:

A1: US61/500,560 priority document

A2: WO2011/117530 A9, filed on 22.03.2011 published on 29.09.2011

A3: WO95/07551, A1 published on 16.03.95

A4: WO2010/117392 A1, published on 14.10.2010

A5: US 2011/0104551 A1, published on 05.05.2011

A6: US2009/0185327A1, published on 23.07.2009

A7: WO2009/127901 A1, published on 22.10.2009

A8: US2011/0121240 A1, published on 26.05.2011

With letter of 19.12.2017 the Proprietor (P) has requested that the opposition be rejected and the patent be maintained as granted. Oral proceedings in the sense of Art. 116 EPC have been requested as auxiliary measure.

Preliminary opinion of the opposition division

The opposition filed in due time, in proper form and supported by reasoned statements seems admissible in that it complies with the requirements of Art. 99(1), 100 EPC and Rule 76 EPC.

Taking into account the submissions made by the parties so far, the opposition division comes to the following preliminary opinion. This opinion is not binding as it is based on the facts and arguments presented until the issuance of this communication and cannot take into account further points which might be made at a later stage of the proceedings.

Art. 100(a) EPC

Priority

The opponent (O) has submitted that claim 1 of the opposed patent did not benefit of the right to priority from application US 61/500,560 (A1) filed on 23.06.2011 because claim 5 of said US application disclosed carbon nanotubes (CNT) having an aspect ratio of 25 to 500 and not of 10 to 500 as in granted claim 1.

The proprietor (P) has replied that in line with G1/15, the priority was at least (partially) valid for the aspect ratio range of 25-500.

The opposition division is of the preliminary opinion that according to G1/15 claim 1 of the opposed patent enjoys the right to partial priority for the aspect ratio range of 25-500 because the subject matter of claim 1 of the opposed patent, as far as the aspect ratio 25-500 is considered, is clearly and unambiguously disclosed in claim 5 of A1.

With regard to opposed claims 2-5 the opposition division observes that the additional features disclosed in said claims are disclosed in claims 2-4 and 6 of A1.

As claims 2-6 of A1 are depending only on claim 1 and there is no interdependency of the claims from each other, the priority cannot be entirely acknowledged for the subject matter of opposed claims 2-5; the priority is partially valid only for iron and copper in opposed claim 2, and for copper in opposed claim 4 with regard to the range of aspect ratio of 25-500 because said three embodiments find a basis in examples 1, 2 and 3 of A1.

Opposed claims 6-9 do not find any basis in A1 and the priority right for the subject matter of said claims cannot be acknowledged.

Opposed claims 11 and 12 find a basis in claims 7 and 8 of A1 and the priority for said claims is partially valid for the range of aspect ratio of 25-500.

Novelty

O has referred to the passages on page 8 line 7-9 and 24-27, page 20 line 24-page 21 line 21, page 22 lines 20-31, of A2 to conclude that claim 1 of the opposed patent was not novel over A2. O further submitted that opposed claims 2 and 4 were not novel in view of the teaching of page 20 line 24-page 21 line 21 of A2.

The same applied to opposed claim 7 in view of the teaching of example 2 of A2 and opposed claim 9 in view of the teaching on page 22 line 20-31 of A2.

P has reacted to these arguments by submitting that A2 did not disclose discrete carbon nanotubes but rather a master batch in agglomerated form of carbon nanofibers and /or carbon nanotubes and/or carbon black. Furthermore A2 did not disclose the degree of oxidation of opposed claim 1.

The preliminary opinion of the opposition division is that A2 aims at providing ready-to-use master batches suitable for the immediate employ in the fabrication of electrodes in order to increase the electric conductivity (page 4, lines 26-31 of A2).

A2 teaches master batches in form of a solid agglomerate comprising nanofibers and/or carbon nanotube and/or carbon black, at least one solvent and at least one binding polymer (page 7, lines 5-15 of A2).

The teaching of the passage on page 22 line 20 to page 23 line 9 of A2 is that it is possible to distribute the carbon nanofibers and nanotube in a way that they form a mesh around the active material, said active material being selected among i) transition metal oxides having a spinel structure, ii) transition metal oxides having a lamellar structure, iii) polyanionic lithium oxides, iv) vanadium based oxides, v) graphites and vi) titanates (page 20 line 24-page 21 line 25 of A2).

A2 therefore does not teach discrete carbon nanotubes as required by the wording of opposed claim 1.

Another difference with the respect of the subject matter claimed in the opposed patent is that, despite the mention on page 8 line 24-27 of A2 that the nanotubes may be oxidized, A2 does not disclose any range of oxidation levels of the CNT.

The subject matter of the opposed claims is therefore novel over A2.

Inventive step

O has argued that the technical effect deriving from the features of the aspect ratio and the oxidation degree had not been demonstrated in the opposed patent and therefore it could not be taken into account for the assessment of inventive step.

According to O the subject matter of opposed claim 1 was not inventive over A2.

As the claimed priority was not valid, A2 represented a prior art document in the sense of Art 54(2) EPC. Even if the novelty over A2 was acknowledged, the features of the aspect ratio and the oxidation degree were at least suggested by the general teaching of A2, which taught the advantages deriving from the association of CNT with particles of active material. O argued that P had not provided any comparative test demonstrating that the CNT of A2 did not show the claimed features and were not suitable to solve the technical problem.

O also submitted that opposed claim 1 lacked inventive step in view of the teaching of A3 which disclosed carbon fibrils corresponding to the CNT of the opposed patent.

Furthermore O submitted that opposed claims 1-12 were not inventive in view of the disclosure of A4; even if said document did not explicitly teach steps f) and e) of opposed claims 11 and 12 respectively, the use of inert atmosphere was obvious to the skilled person and the olivine crystal structure in the opposed had not been demonstrated.

For the same reasons used in the previous attacks, according to O even if A5 did not explicitly disclose the aspect ratio and the oxidation degree, opposed claims 1-10 lacked inventive step in view of A5.

Without specifying which of the opposed claims were concerned, O submitted that there was a lack of inventive step in view of A6, A7 and A8 because said documents taught CNT attached to active materials for lithium ion batteries.

P has reacted to the above mentioned attacks on inventive step by submitting that A2 focused on agglomeration of nanofibers, nanotubes or carbon black and not on discrete nanotubes.

A3 was mainly related to aggregates of carbon fibrils and the only reference to non-aggregated fibrils appeared on page 6 lines 32-36 of A3 which referred to a (unpublished?) US application 08/057,328 filed on 05.05.1993.

With regard to A4, P submitted that, as the aspect ratio and oxidation degree mentioned in opposed claim 1 were missing in A4, said document could never bring the skilled person to the claimed invention.

P further argued that A5 was concerned with aligned CNT which had a different structure from the discrete carbon nanotubes of the opposed patent.

Again the aspect ratio and oxidation degree mentioned in opposed claim 1 were missing in A5.

With regard to A6 P submitted that said document had nothing to do with the claimed invention because it dealt with a porous structure of nanotubes and it did not disclose discrete CNT.

Finally P submitted that A7 dealt with all kinds of carbon used together with $LiMPO_4$ and A8 disclosed a process wherein an electroactive material was partially coated with carbon.

Neither A7 nor A8 could bring the skilled person closer to the claimed subject matter.

The opposition division observes that the problem to be solved by the invention underlying the opposed patent is how to overcome the difficulties of low electrical conduction, how to improve lithium ion transport and mitigate potentially damaging chemical side reactions in lithium ion batteries (paragraph 8 of the present application).

A4 appears to the opposition division as the most promising starting point to reach the solution proposed by the opposed claims because it aims at improving the properties of carbon nanotubes in applications including energy storage devices (paragraph 7 of A4).

Furthermore among the cited documents A4 is the only one to deal with exfoliated carbon nanotubes and methods for preparing exfoliated carbon nanotubes (claims 1 and 11 of A4) by use of nanoplates of zirconium phosphate treated with a surfactant and sonication in aqueous media (paragraph 49 of A4). The obtained CNT after centrifugation and separation exist in a disordered **but not aggregate state** (paragraph 49 of A4).

Among the other documents cited by O only A3 discloses non-aggregated fibrils a on page 6 lines 32-36 which refers back to US application 08/057,328 filed on 05.05.1993. Said disclosure is considered as not enabling by the opposition division because the publication number of the US application is not provided.

For the above mentioned reasons the opposition division considers A4 as the closest prior art.

A4 also teaches that the exfoliated CNT may be incorporated in electrodes of energy storage devices and in some embodiments electroactive nanomaterial selected from a list comprising $Li_4Ti_5O_{12}$ may be bound to the exfoliated carbon nanotubes of said active material (paragraphs 82-83 of A4).

Said composition differs from the subject matter of claim 1 for the following features:

i. the aspect ratio of 10 to 500 is not disclosed

ii. the oxidation level from 1% to 15% by weight of the carbon nanotube is not disclosed

No technical effect can be derived from said differences.

The resulting objective technical problem may be formulated as the provision of alternative compositions useful for lithium ion batteries.

Paragraph 42 of A4 teaches that in some embodiments the aspect ratio of carbon nanotubes is at least about 100 and in some other embodiments it is at least about 1000.

Paragraph 32 of A4 discloses that the exfoliated carbon nanotubes may include a type of CNT selected from a list comprising oxidized carbon nanotubes.

The degree of oxidation is not disclosed.

The preliminary opinion of the opposition division is that the skilled person would have no incentive to combine the exfoliated CNT bound to $Li_4Ti_5O_{12}$ nanomaterial of paragraphs 82-83 with the feature of an aspect ratio of at least about 100 and with the feature of oxidized carbon nanotubes because two selections are involved is said process; and even applying said two selections, the skilled person would not necessarily obtain and oxidation level from 1% to 15% as disclosed in opposed claim 1.

The subject matter of opposed claim 1 is therefore inventive; the subject matter of dependent claims 2-10 is necessarily also inventive.

The subject matter of opposed claims 11 and 12 being said claims directed to a process for preparing compositions according to claim 3 is also inventive.

Art 100(b) EPC

O has substantiated the lack of sufficiency disclosure using three line of argumentation:

1) the opposed patent did provide the skilled person neither with any indication concerning the method for determining the oxidation level of the CNT nor concerning how to perform the selection of the CNT with respect to the aspect ratio.

Furthermore the opposed patent did not disclose how to obtain the oxidized CNT especially with regard to the oxidation level disclosed in claim 1.

2) The patent did not provide any information concerning the selection of the size of the crystals or layers of active material that had to be attached to the surface of the CNT: the skilled person could only base himself on the teaching of the examples of the opposed patent but he had no guidance to obtain the crystals and the layers of active material having the required size.

3) In step b) of claim 11 the opposed patent disclosed a “high boiling point liquid” but the skilled person was not able to select such a liquid; furthermore a high number of experiments in order to correctly adjust the temperature and reagents in steps c) and d) was necessary. Furthermore the skilled person did not know at which temperature the annealing process had to be performed in step f) of opposed claim 11.

Finally O has argued that the terms “liquid” and “surfactant” in step b) of claim 12 were vague and the description did not provide any indication allowing the skilled person to identify the liquids and the surfactants which were suitable to carry out said step.

P has replied that O´s arguments essentially focused on the definitions of the parameters in the claims and were rather clarity objections. According to P neither did O contest the reproducibility of the examples of the opposed patent nor did he put forward any evidence the claimed CNT could not be prepared by the skilled person. With regard to point 1) of O´s argumentation P has referred to paragraphs 22 and 24 of the opposed patent. With regard to point 2) P has submitted that O appeared to accept the fact that the skilled person could rely on the examples of the opposed patent and this was enough for sufficiency of disclosure. Concerning point 3) P replied that the raised objection regarded rather clarity than sufficiency of disclosure.

The opposition division observes that the opposed patent contains 5 different examples; as O has not provided any evidence or argumentation of the contrary, the opposition division takes the preliminary view that each of said 5 examples fall within the scope of the claims and that they are reproducible.

In examples 1-5 of the opposed patent commercially available multi-wall carbon nanotubes (CNano, grade C9000) are employed. Said nanotubes are disclosed as being oxidized at about 8 percent and having an aspect ratio of about 60 in examples 1-3 whilst in examples 4 and 5 the same value of aspect ratio of about 60 and an oxidation level of 4% are disclosed.

Paragraphs 22 and 24 of the opposed patent teach the meaning of the term "oxidized carbon nanotubes" and the of definition "weight percent"

With regard to point 1) of the opponent´s argumentation, the opposition division considers that the patent provides sufficient guidance to the skilled person to carry out the invention and the fact the two parameters of the aspect ratio and level of oxidation may not be clearly defined does not represent a ground for opposition in the sense of Art 100(b) EPC but rather a matter to be discussed under Art 84 EPC.

With regard to point 2) the opposition division considers that the formation of crystal or layers of lithium ion active material attached to the surface of the CNT i results from performing the processes described in the examples of the opposed patent and the skilled person has no need to select the size of the crystals or layers of lithium ion active material in order to carry out the invention.

With regard to point 3) the opposition division considers that the examples of the opposed patent provide sufficient guidance with regard to the liquid solvents, the annealing temperature and the surfactants to be used and no undue experimentation is necessary to the skilled person to adjust and adapt to each other said features.

The preliminary opinion of the opposition division is therefore that the patent in suit is sufficiently disclosed in the sense of Art 83 EPC.

Art. 100(c) EPC

O has submitted that claims 5-10 of the opposed patent were dependent on the previous claims, which meant that the claims as granted disclosed combinations of features which were not originally disclosed in the application as filed; in the filed application said features were disclosed as separated and not in combination.

P has replied that granted claim 5 found a basis in original claim 6 and the penultimate sentence of paragraph 9 of the published application; granted claim 6 found a basis in original claim 7 and in the last sentence of paragraph 9 of the published application; granted claims 7 and 8 found a basis in original claims 10 and

11 and in paragraphs 12 and 13 of the published application; granted claims 9 and 10 found a basis in originally filed claims 12 and 13 and in paragraph 14 of the published application.

The opposition division takes the preliminary view that the teaching of paragraph 9 of the application as published represents a basis for claim 5 as granted. Said paragraph 9 teaches in fact 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 preferably the discrete carbon nanotubes of the composition have an aspect ratio of 10 to 500 and oxidation levels from 1 to 15 weight percent and wherein the composition can further comprises graphene or oxygenated graphene.

The additional features of granted claim 6 find a basis in the last sentence of paragraph 9, and those granted claims 7-10 in paragraphs 12, 13 and 14 respectively of the published application.

The patent as granted therefore complies with the requirements of Art 123(2) EPC.

Further remarks

Following the requests of all parties, oral proceedings are arranged. The matter to be discussed during the oral proceedings has been laid down in the sections above. It is pointed out that an amended patent must meet all requirements of the EPC (see Art. 101(3)(a) EPC). Hence, as regards the first or any further auxiliary request further topics may arise in the conduct of the oral proceedings.

As a matter of precaution, the parties´ attention is drawn to Rule 116(1) EPC, 2nd to 4th sentence.

Any request for oral submission by an accompanying person should be made at least two months before the date of the oral proceedings and the request should state the name and the qualification of the accompanying person, and should specify the subject-matter of the proposed oral submissions – in accordance with the decision of the Enlarged Board of Appeal G04/95 – so that all parties are able to prepare

themselves in relation to the proposed oral submissions. A late filed request in the absence of exceptional circumstances will be refused unless each of the parties agrees to the making of the oral submissions requested.

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Es wird daher gebeten, eine Simultanübersetzung nur bei wirklichem Bedarf in Anspruch zu nehmen. Es wäre wünschenswert, wenn sich die Beteiligten (zweckmäßigerweise gleichzeitig mit der Terminabstimmung) auf die Benutzung einer Amtssprache einigen könnten. Bei Verständigungsschwierigkeiten sind die Mitglieder der Einspruchsabteilung bereit zu helfen.	Please therefore make use of simultaneous interpreting facilities only where strictly necessary. If possible the parties should agree on an official language for the proceedings, preferably at the time when they arrange a date. The members of the Opposition Division will be willing to help should any communication problems arise.	Les parties à une procédure sont donc priées de ne demander une traduction simultanée qu'en cas de réel besoin. Il serait souhaitable qu'elles puissent se mettre d'accord en même temps qu'elles conviennent de la date sur l'utilisation d'une langue officielle comme langue des débats. Si les parties éprouvent des difficultés de compréhension lors des débats, les membres de la division d'opposition sont disposés à leur prêter leur assistance.
Die von den Verfahrensbeteiligten bevorzugte (abgestimmte) Verhandlungssprache und ggf. eine notwendige Simultanübersetzung sind dem Amt möglichst vor der in Regel 4(1) EPÜ angegebenen Frist mitzuteilen.	The EPO should be told if possible before the period mentioned in Rule 4 (1) EPC which language the parties prefer (agree on) and whether simultaneous interpreting facilities are required.	L'Office doit être avisé si possible avant le début du délai mentionné dans la règle 4(1) CBE de la langue préférée par les parties pour le déroulement des débat (et sur laquelle elles se sont préalablement mises d'accord) et de la nécessité éventuelle d'une traduction simultanée.
☐ Verfahrenssprache ist **Deutsch**	☒ Language of the proceedings is **English**	☐ La langue de la procédure est le **français**
Von der/dem/den Einsprechenden wurde	The language used by the opponent/s was	La langue utilisée par l'opposant/les opposants était
☐ Englisch	☐ German	☐ l'allemand
☐ Französisch benutzt.	☒ French.	☐ l'anglais.
Es wird um eilige Mitteilung - möglichst per Telefax an den zuständigen Formalprüfer - gebeten,	**Please inform us urgently - where possible by fax addressed to the formalities officer concerned -**	**Prière d'indiquer d'urgence à l'agent des formalités compétent si possible par téléfax**
möglichst bis Datum 05.09.2018	if possible by Date 05.09.2018	si possible jusqu'au Date 05.09.2018
1. welche Sprache(n) Sie in der mündlichen Verhandlung verwenden (**Sprechen**)	1. which language(s) you intend to use during the oral proceedings (**Speaking**)	1. quelle(s) langue(s) vous utiliserez au cours de la procédure orale (**pour parler**)
2. aus welcher Sprache Sie eine Simultanübersetzung benötigen (**Hören**).	2. from which language you need simultaneous interpretation (**Listening**).	2. à partir de quelle langue vous aurez besoin d'une traduction simultanée (**pour écouter**).

EPA/EPO/OEB Form 2043 02.09

12731264.3

Sollten Sie Ihren Antrag auf mündliche Verhandlung zurückziehen oder zum anberaumten Verhandlungstermin nicht erscheinen wollen bzw. aus wichtigem Grund daran gehindert sein, werden Sie gebeten,

- **unverzüglich das Amt - möglichst per Telefax - davon zu benachrichtigen, wobei das Schriftstück mit einem deutlichen Vermerk "Dringend, mündliche Verhandlung am ..." oder sinngemäß gekennzeichnet sein sollte;**
- **in dringenden Fällen** (weniger als 1 Monat vor dem Verhandlungstermin) zusätzlich **auch dem/die anderen Verfahrensbeteiligten** bzw. ihre(n) Vertreter auf schnellstem Weg **direkt zu unterrichten.**

In jedem solchen Fall obliegt der Einspruchsabteilung die Entscheidung, ob die Verhandlung durchgeführt oder abberaumt wird. Es wird jedoch darauf hingewiesen, dass einem Verfahrensbeteiligten, der eine nicht rechtzeitige oder unterbliebene Benachrichtigung zu verantworten hat, die dadurch den anderen Beteiligten verursachten Kosten auferlegt werden können (Art. 104 EPÜ).

Hinweis auf Regel 4 EPÜ

Regel 4
Sprache im mündlichen Verfahren

(1) Jeder an einem mündlichen Verfahren vor dem Europäischen Patentamt Beteiligte kann sich anstelle der Verfahrenssprache einer anderen Amtssprache des Europäischen Patentamts bedienen, sofern er dies dem Europäischen Patentamt spätestens einen Monat vor dem angesetzten Termin mitgeteilt hat oder selbst für die Übersetzung in die Verfahrenssprache sorgt. Jeder Beteiligte kann sich einer Amtssprache eines Vertragsstaats bedienen, sofern er selbst für die Übersetzung in die Verfahrenssprache sorgt. Von diesen Vorschriften kann das Europäische Patentamt Ausnahmen zulassen.

Should you decide to withdraw your request for oral proceedings or not wish to attend on the date set, or if for some special reason you are unable to do so, you are requested

- **to notify the EPO immediately, where possible by fax, marking the document clearly with the words "Urgent, oral proceedings on ..." or similar;**
- **in urgent cases** (less than one month before the date set for the proceedings), additionally to **notify the other party/parties** and/or their representative(s) **direct as rapidly as possible.**

In all such cases the Opposition Division will decide whether the proceedings are to go ahead or be cancelled. You should however note that costs incurred by the other parties may be charged to a party who either fails to notify them or does not do so in good time (Article 104 EPC).

Attention is drawn to Rule 4 EPC

Rule 4
Language in oral proceedings

(1) Any party to oral proceedings before the European Patent Office may use an official language of the European Patent Office other than the language of the proceedings, if such party gives notice to the European Patent Office at least one month before the date of such oral proceedings or provides for interpretation into the language of the proceedings. Any party may use an official language of a Contracting State, if he provides for interpretation into the language of the proceedings. The European Patent Office may permit derogations from these provisions.

Si vous retirez votre requête tendant à recourir à la procédure orale ou si vous ne souhaitez pas vous présenter à la date fixée pour la procédure orale ou ne pouvez vous y présenter pour une raison sérieuse, veuillez

- **en faire avis sans retard à l'Office, si possible par téléfax, en partant sur votre communication clairement la mention "Urgent, procédure orale le ..." ou une indication similaire;**
- **dans les cas urgents** (moins d'un mois avant la date fixée pour la procédure orale) **en faire avis également directement** pas la voie la plus rapide à **l'autre/aux autres partie(s)** ou bien à son/leurs mandataire(s).

Il appartient alors à la division d'opposition de décider si la procédure orale aura lieu ou non. Il est néanmoins souligné que les frais causés aux autres parties par une partie qui est responsable de l'omission d'un tel avis ou de ce que cet avis n'a pas été fait en temps utile peuvent être mis à la charge de cette partie (art. 104 CBE).

Rappel de la Règle 4 CBE

Règle 4
Langues admissibles lors de la procédure orale

(1) Toute partie à une procédure orale devant l'Office européen des brevets peut utiliser une langue officielle de l'Office européen des brevets autre que la langue de la procédure, à condition soit d'en aviser l'Office européen des brevets un mois au moins avant la date de la procédure orale, soit d'assurer l'interprétation dans la langue de la procédure. Toute partie peut utiliser une langue officielle de l'un des Etats contractants à condition d'assurer l'interprétation dans la langue de la procédure. L'Office européen des brevets peut autoriser des dérogations aux présentes dispositions.

(2) Die Bediensteten des Europäischen Patentamts können sich im mündlichen Verfahren anstelle der Verfahrenssprache einer anderen Amtssprache des Europäischen Patentamts bedienen.

(2) In the course of oral proceedings, employees of the European Patent Office may use an official language of the European Patent Office other than the language of the proceedings.

(2) Au cours de la procédure orale, les agents de l'Office européen des brevets peuvent utiliser une langue officielle de l'Office européen des brevets autre que la langue de la procédure.

(3) In der Beweisaufnahme können sich die zu vernehmenden Beteiligten, Zeugen oder Sachverständigen, die sich in einer Amtssprache des Europäischen Patentamts oder eines Vertragsstaats nicht hinlänglich ausdrücken können, einer anderen Sprache bedienen. Erfolgt die Beweisaufnahme auf Antrag eines Beteiligten, so werden die Beteiligten, Zeugen oder Sachverständigen mit Erklärungen, die sie in einer anderen Sprache als in einer Amtssprache des Europäischen Patentamts abgeben, nur gehört, sofern dieser Beteiligte selbst für die Übersetzung in die Verfahrenssprache sorgt. Das Europäische Patentamt kann jedoch die Übersetzung in eine seiner anderen Amtssprachen zulassen.

(3) Where evidence is taken, any party, witness or expert to be heard who is unable to express himself adequately in an official language of the European Patent Office or of a Contracting State may use another language. Where evidence is taken upon request of a party, parties, witnesses or experts expressing themselves in a language other than an official language of the European Patent Office shall be heard only if that party provides for interpretation into the language of the proceedings. The European Patent Office may, however, permit interpretation into one of its other official languages.

(3) Lors de l'instruction, les parties, témoins ou experts appelés à être entendus, qui ne possèdent pas une maîtrise suffisante d'une langue officielle de l'Office européen des brevets ou d'un Etat contractant, peuvent utiliser une autre langue. Si la mesure d'instruction est ordonnée sur requête d'une partie, les parties, témoins ou experts qui s'expriment dans une langue autre qu'une langue officielle de l'Office européen des brevets ne sont entendus que si cette partie assure l'interprétation dans la langue de la procédure. L'Office européen des brevets peut toutefois autoriser l'interprétation dans l'une de ses autres langues officielles.

(4) Mit Einverständnis aller Beteiligten und des Europäischen Patentamts kann jede Sprache verwendet werden.

(4) If the parties and the European Patent Office agree, any language may be used.

(4) Sous réserve de l'accord des parties et de l'Office européen des brevets, toute langue peut être utilisée.

(5) Das Europäische Patentamt übernimmt, soweit erforderlich, auf seine Kosten die Übersetzung in die Verfahrenssprache und gegebenenfalls in seine anderen Amtssprachen, sofern ein Beteiligter nicht selbst für die Übersetzung zu sorgen hat.

(5) The European Patent Office shall, if necessary, provide at its own expense interpretation into the language of the proceedings, or, where appropriate, into its other official languages, unless such interpretation is the responsibility of one of the parties.

(5) L'Office européen des brevets assure à ses frais, en tant que de besoin, l'interprétation dans la langue de la procédure, ou, le cas échéant, dans ses autres langues officielles, à moins que cette interprétation ne doive être assurée par l'une des parties.

(6) Erklärungen von Bediensteten des Europäischen Patentamts, Beteiligten, Zeugen und Sachverständigen, die in einer Amtssprache des Europäischen Patentamts abgegeben werden, werden in dieser Sprache in die Niederschrift aufgenommen. Erklärungen in einer anderen Sprache werden in der Amtssprache aufgenommen, in die sie übersetzt worden sind. Änderungen einer europäischen Patentanmeldung oder eines europäischen Patents werden in der Verfahrenssprache in die Niederschrift aufgenommen.

(6) Statements by employees of the European Patent Office, parties, witnesses or experts, made in an official language of the European Patent Office, shall be entered in the minutes in that language. Statements made in any other language shall be entered in the official language into which they are translated. Amendments to a European patent application or European patent shall be entered in the minutes in the language of the proceedings.

(6) Les interventions des agents de l'Office européen des brevets, des parties, témoins et experts faites dans une langue officielle de l'Office européen des brevets sont consignées au procès-verbal dans cette langue. Les interventions faites dans une autre langue sont consignées dans la langue officielle dans laquelle elles sont traduites. Les modifications apportées à une demande de brevet européen ou à un brevet européen sont consignées au procès verbal dans la langue de la procédure.

Europäisches Patentamt
European Patent Office
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European Patent Office
80298 MUNICH
GERMANY
Tel: +49 89 2399 0
Fax: +49 89 2399 4465

Application No.: 12 731 264.3

Patent No.: EP-B-2 724 403

Preparation for oral proceedings - Instructions to Support Service

Oral proceedings are to be held in connection with the above patent application

1. The matters to be discussed are set out in the annex (Form 2906)

2. Dispatch the summons using Form 2008/2310 and Form 2906 for the parties to attend on:

Day 05.11.2018 Time 09:00

Archibus

2453

Conference Room booked

ORAL 01, 02, 03 and 05 coded

Date Initials

SS 06. 04. 18

2.1 If no room is available, notify the division on Form 2088

2.2 Parties' submissions in preparation for the oral proceedings, if any, should be made no later than

2 month(s)

before the date of the oral proceedings
(transfer to Form 2008.1 /2310.1)

2.3 Encode ORAL(04) coded

SS 06. 04. 18

Date Initials

SS 06. 04. 18

2.4 Dispatch Form 2008.7 /2310.7 to division

Date Initials

3. ☐ Arrange for the following special equipment to be provided in the conference room:

Date Initials

4. Return the dossier to primary examiner with Form 2041 (15 days before the oral proceedings)

Date Initials

23.3.'18
Date

Götz, Heide
Chairman

Kelly, Michael
2nd examiner

Bettio, Andrea
1st examiner

Legal member

Enclosure(s):

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date: 11.04.2018

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1108 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

Summons to attend oral proceedings pursuant to Rule 115(1) EPC

You are hereby summoned to attend oral proceedings arranged in connection with the above-mentioned European patent.

The matters to be discussed are set out in the communication accompanying this summons (EPO Form 2906).

The oral proceedings, which will be public, will take place before the opposition division

on 05.11.18 at 09.00 hrs in Room 2453
at the EPO, Bayerstr. 34, PschorrHöfe, D-80335 München

No changes to the date of the oral proceedings can be made, except on serious grounds (see OJ EPO 1/2009, 68). If you do not appear as summoned, the oral proceedings may continue without you (R. 115(2) EPC).

Your attention is drawn to Rule 4 EPC, regarding the language of the oral proceedings, and to the Special edition No. 3 OJ EPO 2007, 128, concerning the filing of authorisations for company employees and lawyers acting as representatives before the EPO.

The final date for making written submissions and/or amendments (R. 116 EPC) is 05.09.18.

You are requested to report in good time beforehand to the porter in the EPO foyer. Room 3473 and 3474 are available as waiting rooms.

Parking is available in the underground car park, accessible only via the entrance "Grasserstrasse 2/6". On presentation of the summons to oral proceedings at one of the porters' lodges in the PschorrHöfe, the parking ticket will be revoked.

1st Examiner:	2nd Examiner:	Chairman:
Bettio A	Kelly M	Götz H

For the Opposition Division

Annexes:
Confirmation of receipt (Form 2936)
Rule 4 EPC (EPC Form 2043)
Communication (EPO Form 2906)

Europäisches Patentamt • European Patent Office • Office européen des brevets

Registered letter with advice of delivery

to EPO postal service: 06.04.18

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date
11.04.2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1108 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

Summons to attend oral proceedings pursuant to Rule 115(1) EPC

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1st Examiner:	2nd Examiner:	Chairman:
Bettio A	Kelly M	Götz H

For the Opposition Division

Annexes:
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Rule 4 EPC (EPC Form 2043)
Communication (EPO Form 2906)

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Registered letter with advice of delivery
to EPO postal service: 06.04.18

Europäisches Patentamt
European Patent Office
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European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Bonnel, Claudine
ARKEMA France
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date: 03-01-2018

Reference	Application No./Patent No.
AL 0378 OPPO 01	12731264.3 - 1360 / 2724403
Applicant/Proprietor: Molecular Rebar Design, LLC	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☐

Enclosure(s):
- ☑ Letter from the proprietor of the patent of 19.12.17
- ☐ Letter from the opponent of
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are available via the European Patent Register at **www.epo.org/register.** Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

☐ **Registered letter**

Europäisches Patentamt
European Patent Office
Office européen des brevets

Acknowledgement of receipt

We hereby acknowledge receipt of the following subsequently filed document(s):

Submission number	5892466	
Application number	EP12731264.3	
Date of receipt	19 December 2017	
Receiving Office	European Patent Office, The Hague	
Your reference	P100028EP00	
Applicant	All applicants as on file	
Documents submitted	package-data.xml epf1038.pdf (1 p.)	ep-sfd-request.xml ORAL-1.pdf\P100028EP00 Response to Opposition.pdf (9 p.)
Submitted by	CN=Hermanus Marsman 34472	
Method of submission	Online	
Date and time receipt generated	19 December 2017, 16:15 (CET)	
Message Digest	A2:39:70:92:0E:75:FC:F1:40:45:FD:26:78:63:34:69:16:64:AA:11	

Correction by the EPO of errors in debit instructions filed by eOLF
Errors in debit instructions filed by eOLF that are caused by the editing of Form 1038E entries or the continued use of outdated software (all forms) may be corrected automatically by the EPO, leaving the payment date unchanged (see decision T 152/82, OJ EPO 1984, 301 and point 6.3 ff ADA, Supplement to OJ EPO 10/2007).

/European Patent Office/

Europäisches Patentamt
European Patent Office
Office européen des brevets

Letter accompanying subsequently filed items

Representative:
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Tel. +49(0)30 25901-0 | Fax -840

The document(s) listed below is (are) subsequently filed documents pertaining to the following application:

Application number	12731264.3
Applicant's or representative's reference	P100028EP00

	Description of document	Original file name	Assigned file name
1	Letter dealing with Oral proceedings	P100028EP00 Response to Opposition.pdf	ORAL-1.pdf

	Payment	
1	Method of payment	Not specified

Annotations

deposit-account(V.O.; 19.12.2017)
1. Remark(Annotate)
I herewith authorize the European Patent Office to deduct all necessary fees that are involved in this specific action from my deposit-account No. 2809.0002

Signatures

Place:	Den Bosch
Date:	19 December 2017
Signed by:	/H.A.M. Marsman/
Association:	V.O.
Representative name:	H.A.M. Marsman
Capacity:	(Representative)

V.O.
Patents & Trademarks

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Your ref. 12731264.3/1360
Our ref. MAS/P100028EP00

The Hague,
December 19, 2017

Re: European Patent No. 2724403
Molecular Rebar Design, LLC

In response to your Registered Letter of August 10, 2017 and with reference to the Communication of a Notice of Opposition dated July 13, 2017, I request that the Opposition be rejected and the Patent identified herein-above (herein-after: "the Patent") be maintained in the form as granted. If you believe that this request cannot be met, Oral Proceedings are requested under Art 116 EPC.

In support of my request, the following is observed.

I will use the document numbering as suggested by Opponent Arkema France.

Opponent has raised objections on the grounds of Art 100(a)-(c) EPC. None of these convince.

I will start discussing the enablement objections raised under Art 100(b) EPC. These start at the second half of page 4 of the Opposition Brief.

I note that the arguments raised on this ground essentially focus on the definitions of parameters in the claims. This means that Opponent raises clarity objections, rather than sufficiency of disclosure objections. Lack of clarity, however, is not a ground for opposition.

The working examples of the Patent describe discrete carbon nanotubes with attached crystals or layers of lithium ion active material. The carbon nanotubes described have aspect ratios and oxidation levels in the ranges present in the claims.

Opponent does not in any way contest these data nor the repeatability of these examples. Certainly, the Opponent does not put forward any evidence that the carbon nanotubes used in accordance with the present invention could not be prepared by the skilled person. This means that the invention is described in at least one enabling way, so that the requirements of Art 100(b) j^0 Art 83 EPC have been met.

What Opponent does argue (see under **1)** at page 4 of its Opposition Brief), is that the description of the Patent would not contain any indication on a definition of the aspect ratio, yet seems to accept that this concept is well known to the skilled person by referring to the ratio of the length over the diameter of the discrete carbon nanotubes (see the last paragraph of page 4 of the Opposition Brief). It then only observes (and that is also known to the skilled person) that the lengths and diameters could vary over "larges mesures".

It is noted that the Patent specification elaborates on this issue in § [0024] of the Patent as published.

More importantly, however, no evidence is given by Opponent on that this issue on the aspect ratio would make it not possible for a skilled person to work the invention.

Since Opponent's considerations may, at best for Opponent, have an effect on the endpoints of the range, this undoubtedly is a matter of clarity. Nowhere does Opponent provide evidence that hence the invention would not be enabled.

Also under item **1)** of the ground of Art 100(b) EPC, Opponent complains about the fact that the description would not provide any method to determine the amount of oxidation of the carbon nanotubes. Opponent does however accept that it understands from the description (see the first paragraph at page 5 of the Opposition Brief and § [0022] of the Patent as granted) what is meant with the type of oxidation. However, determining the presence and amounts of such groups is entirely within the routine of the skilled person.

Under item **2)** at page 5 of the Opposition Brief, Opponent states that "il subsiste donc un doute sur la taille des cristaux ou des couches de matériau actif Li-ion qui doivent être attachés à la surface des NTC pour résoudre le problème".

Apart from the fact that I fail to see the problem with which the skilled person would be confronted, I note that nowhere Opponent provides any data on that a problem would exist and that this problem would lead to non-enablement.

Rather on the contrary, it appears that Opponent accepts that the skilled person can rely on the examples given in the description. This is – unless evidence to the contrary is provided – more than enough to guarantee a sufficient description of the invention.

As a last argument under the ground of insufficient disclosure, Opponent considers a number of components and/or steps in the processes of the invention vaguely defined and would require "un nombre d'expérimentation". Again, these are only clarity issues, and are not issues of an insufficient description of the invention.

Hence, the invention is described in the Patent in a manner that is sufficiently clear and complete for the skilled person to work it.

At the top of page 6, Opponent argues that the subject-matter of dependent claims 5-10 would be (at least partially) based on combinations for which there would not be support in the application as originally filed.

This observation is incorrect.

Claim 5 as granted is not only based on claim 6 as originally filed, which dependent on claim 1 as filed, but also on the penultimate sentence is § [0009] of the application as published. Said sentence makes clear that the composition [of the invention] can further comprise graphene or oxygenated graphene. In said generally worded § [0009] all limitations of the claims 1-4 as granted are present.

Claim 6 as granted is based on original claim 7 and the last sentence of § [0009] of the application as published.

Neither do claims 7 and 8 as granted extend beyond the scope of the application as filed, because these not only have explicit support in original claims 10 and 11, respectively, but also in the generally worded §§ [0012] and [0013] of the application as published.

Claims 9 and 10 as granted find general basis in originally filed claims 12 and 13, respectively, and in the generally applicable paragraph [0014] of the application as published.

Hence, it is clear that Art 123(2) EPC has not been violated.

At page 4 of its Opposition Brief, Opponent indicates that it considers the priority claim invalid. While referring to priority document A1, it argues that claim 1 as granted cannot benefit from the priority of A1, because the aspect ratio in claim 1 as granted is in the range of 10-500, while the priority document A1 only sketches a range of 25-500.

This observation is not correct. At best for Opponent, the position can be taken that for the aspect ratio range of 10 to less than 25, the priority would not be valid. In line with G 1/15, the priority is at least (partially) valid for the range of 25-500.

Because the position taken by Opponent on the priority is incorrect, Opponent's A2 is merely a document citable under Art 54(3) EPC, for as far as it is entitled to its priority dates. A2 cannot be used to contest inventive step.

According to Opponent, A2 would anticipate claims 1, 2, 4, 7 and 9 as granted. This is incorrect.

Opponent at best creates a teaching by mosaicking unlinked passages from A2.

That is, Opponent seems to suggest that A2 at page 22, in lines 20-31, would teach discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface.

This is not taught in said passage.

In said passage, it is noted that with the process of A2, it is possible to distribute the carbon nanofibers and nanotubes so that they form a mesh around the particles of active material.

This passage does not teach discrete carbon nanotubes, nor attachment of crystals or layers of lithium ion active material.

Rather on the contrary, the invention of A2 does not talk about discrete carbon nanotubes. Its invention is to create a master batch in agglomerated form of carbon nanofibers and/or carbon nanotubes and/or carbon black, a solvent and a polymer binder.

Moreover, in order to come to crystals of lithium ion active material (nothing being said of attachment of these crystals to the carbon nanotubes), a selection is to be made from a group of active materials, which group does not consist of only lithium crystals. See in this light page 20, line 24 – page 21, line 21 of A2.

A further selection is made in the first full paragraph of page 8 of A2, from which Opponent picks the preferred aspect ratio of higher than 10 and, more preferred, higher than 100. However, this paragraph does not in any way relate to discrete carbon nanotubes.

Yet a further, and entirely unlinked selection is made based on the passage from lines 24-27 of page 8, teaching that "these nanotubes" (likely the raw carbon nanotubes of the previous paragraph, which are certainly not discrete carbon nanotubes) may be purified and/or treated (for example oxidized) and/or milled and/or functionalized. Even if the oxidation option would be picked, still the degree of oxidation is missing.
A2 does not directly and unambiguously teach the subject-matter of any of claims 1, 2, 4, 7 and 9 as granted. If the words of these claims are already there in A2, any direct links or pointers are missing.

As said, at best (for Opponent) A2 is citable for novelty and not for inventive step. Yet, if A2 would be used in an inventive step attack, it teaches away from the presently

claimed invention, because it is entirely focussing on agglomeration of nanofibers, nanotubes or carbon black.

With this observation, Opponent's first inventive step attack is already flawed.

As to the other inventive step objections, Opponent launches attacks starting from 6 other references, without indicating which document would be the closest prior art and without attempt to bring a commonly used problem-solution approach.

These 6 other attacks are essentially of the nature: "there are differences with the claimed subject-matter, but since we (Opponent Arkema) do not see unexpected effects for these differences, there is no inventive step".

A3 is directed to lithium batteries having an anode formed from a carbon fibril material comprised of "an aggregate of fibrils or non-aggregated mass of fibrils having an average particle size of 0.1 to 100 microns in which fine, filiform carbon fibrils of 3.5 to 75 nm in diameter are intertwined with each other, and fibrils being intercalated with lithium" (see *e.g.* the abstract). Particular reference is made to non-aggregated fibrils disclosed not in A3, but in a (unpublished?) U.S. Patent Application Serial No. 08/057,328, filed May 5, 1993 of which it is "now believed" that these "may be used in the electrodes of the invention". See page 6, lines 32-36 of A3.

Otherwise, A3 relates to aggregates and/or A3 uses "a mass" of fibrils and hence in fact teaches away from the subject-matter of the claims of the Patent opposed.

The starting fibrils may be oxidized (page 11, lines 6-20) without details on the degree of oxidation and among other treatments.

In the working examples, carbon fibril material is used with a polyethylene binder.

Only on the basis of hindsight a skilled person could pick and choose particular parts from A3 in order to arrive at something that would come close to what is claimed in the Patent.

A4 and A5 were already cited in the procedure up to grant as D1 and D2.

A4 comes from the present inventors. In A4, it is described that nanocrystalline material may be used to exfoliate the carbon nanotubes (§ [0061]) and that nanomaterial may be bound to the exfoliated carbon nanotubes, for which $Li_4Ti_5O_{12}$ is given as example. However, since A4 is not in any way pointing to the subjective problem underlying the invention in the Patent (see for example §§ [0008]-[0010] and [0002] of the description of the Patent) and Opponent itself already admits that the aspect ratio and oxidation degree mentioned in granted claim 1 are missing in A4, A4 can never bring the skilled person to the claimed invention.

A5 is a reference describing aligned nanotubular base materials comprising aligned carbon nanotubes. This alignment structure differs from the structure of having discrete carbon nanotubes, and also for this case Opponent itself already admits that the aspect ratio and oxidation degree mentioned in granted claim 1 are missing in A5, so that also A5 can never bring the skilled person to the claimed invention.

Reference A6 teaches a mixed oxide coating on a highly porous carbon structure. According to claim 6, the porous carbon structure may be an "unaligned carbon nanofiber structure". What is important in A6 is the highly porous support. This support is not one single discrete carbon nanotube which Opponent wishes to deduce from claim 6, but a structure of nanotubes so that pores are formed in that structure. Apart from many unlinked and unguided selections to be made in A6, this has nothing to do with the invention claimed in the Patent.

A7 does not bring a skilled person closer to the invention as claimed in the Patent, either. According to A7, all kinds of carbon can be used together with $LiMPO_4$ and these materials can be milled together with the aim of breaking covalent bonds in the carbon material. One of the carbon materials mentioned are carbon nanotubes. However, nothing in A7 points to any further aspects of the invention.

The same applies to A8. A8 is directed to a process wherein an electroactive material, which could be selected from for example lithium compounds, is partially coated by carbon. Apart from other differences, this concept seems 180^0 different than what the invention encompasses.

In view of the above, I conclude that the invention defined by the claims is described in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art, that the claimed subject-matter does not in any way extend beyond the scope of the application as originally filed, is novel and involves an inventive step.

The professional representative,



H.A.M. Marsman

EK

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
10-08-2017

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1360 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

Communication of notices of opposition (R. 79(1) EPC)

Notice of opposition has been filed within the opposition period by:

01.
ARKEMA FRANCE
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92700 Colombes
FRANCE

The notice of opposition indicated above has already been communicated to you.

You are requested to file your observations within a period of **four months** from notification of this communication.

You may also file amendments, where appropriate, to the description, claims and drawings within the period specified. One set of these documents is to be filed.

If you introduced documents which have not yet been mentioned during the proceedings, your attention is drawn to Rule 83 EPC.

Enclosures:

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

Registered letter

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Bonnel, Claudine
ARKEMA FRANCE
Département Propriété Industrielle
420, rue d'Estienne d'Orves
92705 Colombes Cedex
FRANCE

Date
10-08-2017

Reference		Application No./Patent No.
AL 0378	OPPO 01	12731264.3 - 1360 / 2724403
Applicant/Proprietor		
Molecular Rebar Design, LLC		

Communication of further notices of opposition pursuant to Rule 79(2) EPC

No further opposition has been filed.

If oral proceedings are to take place, parties are advised to check the electronic file via the European Patent Register at **www.epo.org/register** in advance of the hearing to ensure that they are in possession of all relevant documents. (OJ EPO 2009, 434)

A copy of the communication pursuant to Rule 79(1) EPC sent to the proprietor of the patent is enclosed for your information.

The observations of the patent proprietor on the notice of opposition will be communicated to the opponent without delay. A time limit for reply will be fixed if the Opposition Division considers this expedient.

If no reply to a communication is received within the time limit set, the proceedings will be continued. Your attention is drawn to Article 114(2) EPC.

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

Enclosures: copy of the communication pursuant to Rule 79(1) EPC (Form 2317A)

Europäisches Patentamt
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European Patent Office
80298 MUNICH
GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

V.O.
P.O. Box 87930
2508 DH Den Haag
PAYS-BAS

Date
13-07-2017

Reference	Application No./Patent No.
P100028EP00	12731264.3 - 1360 / 2724403
Applicant/Proprietor	
Molecular Rebar Design, LLC	

Communication of a notice of opposition

You are hereby informed of a notice of opposition to the European patent specified above (see attached copy in the enclosure). The documents specified as patent documents in the notice of opposition are available for inspection via the European Patent Register at **www.epo.org/register**. Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (see OJ EPO 2009, 434).

If oral proceedings are to take place, parties are advised to check the electronic file via European Patent Register in advance of the hearing to ensure they are in possession of all relevant documents.

An invitation to file observations and to file amendments, where appropriate, to the description, claims and drawings (R. 79(1) EPC) will be issued separately.

The period within which such observations may be filed will not be fixed until the following conditions are met:

(a) the opposition period has expired;

(b) the notice of opposition has been examined for certain formal requirements (R. 77 EPC).

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets •

Enclosure: Notice of opposition O1: ARKEMA FRANCE

Europäisches Patentamt
European Patent Office
Office européen des brevets

Accusé de réception

Par la présente, nous accusons réception de l'opposition :

Numéro de soumission	5424167
Numéro de demande	EP12731264.3
Numéro de brevet	EP2724703
Date de réception	05 juillet 2017
Votre référence	AL 0378
Nom d'opposant(e)	ARKEMA FRANCE
Titre	BATTERIES LITHIUM QUI UTILISENT DES NANOTUBES DE CARBONE DISCRETS, PROCEDES DE PRODUCTION DE CES DERNIERES ET PRODUITS AINSI OBTENUS.

Documents envoyés		
	package-data.xml	ep-opposition-data.xml
	ep-oppo.pdf (5 p.)	OPPO.pdf\AL 0378.pdf (9 p.)
	Published-Evidence-1.pdf\A1-US201161500560P.pdf (14 p.)	Published-Evidence-2.pdf\A2-wo11117530a9.pdf (55 p.)
	Published-Evidence-3.pdf\A3-wo9507551.pdf (21 p.)	Published-Evidence-4.pdf\A4-wo10117392.pdf (44 p.)
	Published-Evidence-5.pdf\A5-us2011104551.pdf (12 p.)	Published-Evidence-6.pdf\A6-us2009185327.pdf (8 p.)
	Published-Evidence-7.pdf\A7-wo0912 7901.pdf (34 p.)	Published-Evidence-8.pdf\A8-us2011121240.pdf (9 p.)

Effectué par	CN=Claudine Bonnel 7787
Méthode de soumission	En ligne

Date et heure de réception	05 juillet 2017, 16:59 (CEST)
Message condensé	F6:28:30:31:2F:47:BA:D7:A9:19:18:87:C9:9D:2F:AC:7C:A3:AB:D0

Correction par l'OEB d'erreurs figurant dans les ordres de débit transmis via eOLF
Les erreurs figurant dans les ordres de débit transmis via eOLF et causées par une modification des informations saisies dans le formulaire 1038E ou une utilisation persistante de logiciels dépassés (tous les formulaires) peuvent être corrigées automatiquement par l'OEB, sans que la date de paiement soit modifiée (cf. décision T 152/82, JO OEB 1984, 301 et point 6.3 s. RCC, supplément au JO OEB 10/2007).

/Office européen des brevets/

Europäisches Patentamt
European Patent Office
Office européen des brevets

Opposition à un brevet européen

I. Brevet attaqué

Numéro du brevet	EP2724703
Numéro de la demande	EP12731264.3
Date de la mention de la délivrance au Bulletin européen des brevets (art. 97(3) et 99(1) CBE)	05 octobre 2016
Titre de l'invention	BATTERIES LITHIUM QUI UTILISENT DES NANOTUBES DE CARBONE DISCRETS, PROCEDES DE PRODUCTION DE CES DERNIERES ET PRODUITS AINSI OBTENUS.

II. Titulaire du brevet

cité en premier lieu dans le fascicule du brevet	Molecular Rebar Design, LLC
Référence de l'opposant ou du mandataire	AL 0378

III. Opposant

Nom :	ARKEMA FRANCE
Département	Département Propriété Industrielle
Adresse :	420 rue d'Estienne d'Orves 92700 COLOMBES France
Etat du domicile ou du siège :	France
Téléphone/Télécopie :	+33 (0)1 49 00 80 80 / +33 (0)1 49 00 80 87
Opposition conjointe (cf. feuille additionnelle)	☐

IV. Pouvoir

1. Mandataire

	BONNEL, Mme Claudine
Société :	ARKEMA FRANCE
Service :	Département Propriété Industrielle
Adresse professionnelle :	420, rue d'Estienne d'Orves 92705 COLOMBES CEDEX

	France	
Téléphone/Télécopie :	33 (0) 1 49 00 80 68	33 (0) 1 49 00 80 87
Courriel :	claudine.bonnel@arkema.com	

Multiple representatives (see additional sheet) ☐

Pouvoir général NB° (art. 133(3) CBE) :

est/sont joint(s) ☐

est/sont enregistré(s) sous le n° ☒ 8646

V. L'opposition est formée contre

le brevet dans son ensemble ☒

les revendications n°

VI. Motifs d'opposition

L'opposition est fondée sur les motifs mentionnés ci-après :

(a) l'objet du brevet européen attaqué n'est pas brevetable (art. 100a) CBE) pour les motifs suivants:

- défaut de nouveauté (art. 52(1) et 54 CBE) ☒
- défaut d'activité inventive (art. 52(1) et 56 CBE) ☒
- autres motifs excluant la brevetabilité, à savoir articles ☐

(b) le brevet européen attaqué n'expose pas l'invention de façon suffisamment claire et complète pour qu'un homme du métier puisse l'exécuter (art. 100b) CBE ; cf. art. 83 CBE). ☒

(c) l'objet du brevet européen attaqué s'étend au-delà du contenu de la demande/demande antérieure telle qu'elle a été déposée (art.100c) CBE ; cf. art. 123(2) CBE). ☒

VII. Exposé des faits

L'exposé des faits (règle 76(2)c) CBE invoqués à l'appui de l'opposition figure dans le document joint ☒

VIII. Autres requêtes

IX. **Preuves produites**

A1	Document de brevet	USUS 61/500,560 (A) , 29.06.2012 fichier électronique originel : A1-US201161500560P.pdf annexé comme : Published-Evidence-1.pdf
A2	Document de brevet	WO2011/117530 (A9) , 29.09.2011 fichier électronique originel : A2-wo11117530a9.pdf annexé comme : Published-Evidence-2.pdf
A3	Document de brevet	WO95/07551 (A1) , 16.03.1995 fichier électronique originel : A3-wo9507551.pdf annexé comme : Published-Evidence-3.pdf
A4	Document de brevet	WO2010/117392 (A1) , 14.10.2010 fichier électronique originel : A4-wo10117392.pdf annexé comme : Published-Evidence-4.pdf
A5	Document de brevet	US2011/0104551 (A1) , 05.05.2011 fichier électronique originel : A5-us2011104551.pdf annexé comme : Published-Evidence-5.pdf
A6	Document de brevet	US2009/0185327 (A1) , 23.07.2009 fichier électronique originel : A6-us2009185327.pdf annexé comme : Published-Evidence-6.pdf
A7	Document de brevet	WO2009/127901 (A1) , 22.10.2009 fichier électronique originel : A7-wo09127901.pdf annexé comme : Published-Evidence-7.pdf
A8	Document de brevet	US2011/0121240 (A1) , 26.05.2011 fichier électronique originel : A8-us2011121240.pdf annexé comme : Published-Evidence-8.pdf

X. **Paiement**

Modalité de paiement	Débit du compte de dépôt

L'Office européen des brevets est autorisé à prélever du compte courant ci-après les taxes et frais indiqués.

Devise :	EUR
Numéro du compte courant :	28040093
Titulaire du compte :	

Remboursements

Les remboursements éventuels doivent être effectués sur le compte courant ci-contre ouvert auprès de l'OEB :	28040093
Titulaire du compte :	

Taxes	Coeff. appliqué	Barème des taxes	Montant à payer
010 Taxe d'opposition	1	785.00	785.00
Total :		EUR	785.00

	A Formulaires	Détails :	Fichier électronique :
A-1	Formulaire d'opposition		ep-oppo.pdf

	B Document(s) annexé(s)	Nom du fichier originel :	Fichier électronique :
B-1	1. Faits et arguments	AL 0378.pdf	OPPO.pdf

	C Fichiers de preuve joints	Nom du fichier originel :	Fichier électronique :
C-1	1. Document de brevet	A1-US201161500560P.pdf	Published-Evidence-1.pdf
C-2	2. Document de brevet	A2-wo11117530a9.pdf	Published-Evidence-2.pdf
C-3	3. Document de brevet	A3-wo9507551.pdf	Published-Evidence-3.pdf
C-4	4. Document de brevet	A4-wo10117392.pdf	Published-Evidence-4.pdf
C-5	5. Document de brevet	A5-us2011104551.pdf	Published-Evidence-5.pdf
C-6	6. Document de brevet	A6-us2009185327.pdf	Published-Evidence-6.pdf
C-7	7. Document de brevet	A7-wo09127901.pdf	Published-Evidence-7.pdf

C-8	8. Document de brevet	A8-us2011121240.pdf	Published-Evidence-8.pdf

Signature de l'opposant ou du mandataire

Lieu :	**Colombes**
Date :	**05 juillet 2017**
Signé par :	**Claudine Bonnel 7787**
Nom du mandataire:	**Claudine BONNEL**
Fonction :	**(Mandataire)**

ARKEMA FRANCE
Direction Recherche et Développement
Département Propriété Industrielle

Colombes, le 5 juillet 2017

OFFICE EUROPEEN DES BREVETS
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Objet : Opposition au Brevet EP 2 724 403 B1 au nom de Molecular Rebar Design, LLC
Opposant : ARKEMA FRANCE

Messieurs,

Par la présente, la Société ARKEMA FRANCE forme opposition au brevet européen EP 2 724 403 B1 délivré le 5 octobre 2016.

L'opposant requiert la révocation totale du brevet. L'opposition est fondée sur les motifs suivants : Article 100 (a), (b) et (c) de la CBE.

Nous vous prions de trouver ci-joint :

- le formulaire d'opposition
- le bordereau de taxes donnant instruction de payer la taxe d'opposition de notre compte courant n°28040093
- L'exposé des faits et motifs, précisant la mesure dans laquelle ledit brevet européen est contesté.
- Une copie des documents de l'état de la technique A1 à A8.

Si la Division d'opposition envisageait de ne pas révoquer ledit brevet, l'opposant requiert la tenue d'une procédure orale au sens de l'article 116(1) CBE.

Nous vous prions d'agréer nos salutations distinguées.

Claudine BONNEL
Mandataire Européen

OPPOSITION AU BREVET EUROPEEN EP 2 724 403 B1

EXPOSE DES FAITS ET MOTIFS

L'opposant met en cause la validité du brevet délivré EP 2 724 403 B1, ci-après nommé brevet opposé, pour toutes les revendications sur la base des articles 100a CBE (défaut de nouveauté et/ou activité inventive), 100b CBE (non-conformité à l'article 83), et 100c CBE (extension de l'objet du brevet) et sollicite par la présente sa révocation dans sa totalité par la Division d'Opposition en vertu de l'Article 101(2) de la CBE.

L'opposant requiert une procédure orale selon l'Art 116 CBE au cas où la Division d'Opposition ne souhaiterait pas rendre une décision selon l'article 101(2) CBE.

DOCUMENTS A CONSIDERER

A1 : US 61/500,560, document de priorité
A2 : WO 2011/117530 A9, déposé le 22.03.2011 et publié le 29.09.2011
A3 : WO 95/07551, publié le 16.03.95
A4 : WO 2010/117392 A1 publié le 14.10.2010, cité dans la procédure
A5 : US 2011/0104551 A1, publié le 5.05.2011, cité dans la procédure
A6 : US 2009/0185327, publié le 23.07.2009
A7 : WO 2009/127901, publié le 22.10.2009
A8 : US 2011/0121240, publié le 26.05.2011

Dans le mémoire, les abréviations "rev", "p.", "col.", "l." signifient respectivement revendication, page, colonne et ligne.
NTC signifie nanotube de carbone.

RAPPEL DE LA PROCEDURE DE DELIVRANCE DU BREVET EP 2 724 403 B1, ET DES REVENDICATIONS DU BREVET

La demande de brevet EP 2 724 403 a été déposée le 21.06.2012, et elle est issue de la demande PCT/US2012/043534, publiée sous le numéro WO 2012/177865. Cette demande de brevet revendique la priorité de la demande USP 61/500,560 du 23.06.2011.

Au cours de la procédure PCT, le titulaire a déposé le 23.04.2013 un nouveau jeu de 12 revendications sur lequel s'est basé un examen préliminaire international.

L'entrée en phase européenne a été réalisée sur la base du jeu de revendications déposé le 23.04.2013. Le déposant a modifié ses revendications en réponse à la notification selon l'Art. 94(3) CBE du 19.01.2015, pour répondre à des objections d'unité d'invention et de clarté.
Le déposant a modifié ses revendications en réponse à la notification selon l'Art. 94(3) CBE du 29.09.2015, pour répondre à des objections de clarté.
La communication selon la Règle 71(3) CBE a été émise le 29.04.2016, puis la mention de délivrance du brevet a été publiée le 5.10.2016.

Le brevet opposé concerne :

i) une composition utile pour des batteries lithium-ion, comprenant des NTC discrets, ces NTC discrets comportant des cristaux ou des couches de matériau actif lithium-ion attachés à leur surface, les NTC discrets ayant un rapport d'aspect de 10 à 500 et des niveaux d'oxydation de 1% à 15% en poids des NTC

(revendications 1 à 10)

ii) un procédé pour la production de NTC discrets comportant des cristaux ou des couches de matériau actif lithium-ion attachés à leur surface, tels que définis dans la revendication 3.

(revendication 11)

iii) un procédé pour la production de NTC discrets comportant des cristaux ou des couches de matériau actif lithium-ion attachés à leur surface, tels que définis dans la revendication 3.

(revendication 12)

PROBLEME A RESOUDRE TEL QUE PRESENTE DANS LE BREVET OPPOSE

Selon la description du brevet opposé ([0008]), le problème que cherche à résoudre l'invention est de surmonter les difficultés de faible conduction électrique dans les batteries lithium-ion, en particulier en raison de l'expansion et de la contraction du matériau lors de la charge et de la décharge de la batterie, l'amélioration du transport de l'ion lithium et l'atténuation des réactions chimiques secondaires potentiellement nuisibles.
La solution proposée par l'invention revendiquée est d'attacher des cristaux nanométriques ou des couches nanométriques de matériau actif Li-ion à la surface de NTC discrets, fonctionnalisés et bien dispersés ([0008]).

L'invention fournit ainsi une composition utile pour des batteries lithium-ion, comprenant des NTC discrets, ces NTC discrets comportant des cristaux ou des couches de matériau actif lithium-ion attachés à leur surface, les NTC discrets ayant un rapport d'aspect de 10 à 500 et des niveaux d'oxydation de 1% à 15% en poids des NTC.

L'invention fournit également 2 procédés pour préparer une telle composition dans laquelle les cristaux ou les couches de matériau actif lithium-ion comprennent un sel métallique de lithium et un élément choisi dans le groupe constitué par: le fer, le manganèse, le cobalt, le cuivre, le nickel, le vanadium, le titane et leurs mélanges, le sel métallique de lithium présentant une structure cristalline d'olivine.

REVENDICATION DE PRIORITE NON VALABLE

La demande US 201161500560P (jointe en annexe sous la référence A1) décrit à la revendication 5, que les NTC discrets ont un rapport d'aspect de 25 à 500 et des niveaux d'oxydation de 1% à 15% en poids des NTC, alors que la revendication 1 du brevet opposé décrit que les NTC ont un rapport d'aspect de 10 à 500 et des niveaux d'oxydation de 1% à 15% en poids des NTC.
La caractéristique sur le rapport d'aspect ayant été introduite dans la revendication 1 au cours de la procédure, il s'ensuit qu'elle est essentielle pour la définition de l'invention.

La revendication 1 du brevet opposé ne peut donc pas bénéficier de la priorité de la demande US 201161500560P déposée le 23.06.2011 qui ne comporte pas cette caractéristique.

ARTICLE 100b CBE - INSUFFISANCE DE DESCRIPTION (ART. 83 CBE)

L'invention revendiquée n'est pas décrite de façon suffisamment claire et complète pour qu'un homme du métier puisse l'exécuter.

1) Les NTC discrets présents dans la composition revendiquée sont caractérisés par la combinaison des deux caractéristiques suivantes :

i) un rapport d'aspect de 10 à 500, et
ii) un niveau d'oxydation de 1% à 15% en poids des NTC

Or, la description ne donne aucune indication sur la définition de ces caractéristiques, et ne fournit aucune méthode de détermination du niveau d'oxydation des NTC. La description n'est que sommaire sur la caractérisation des NTC utilisés dans l'invention, alors que ces caractéristiques sont des caractéristiques essentielles pour différencier l'invention de l'état de la technique.
Même si la définition du rapport d'aspect peut être connue en soi (rapport longueur/diamètre), la longueur et le diamètre des NTC peuvent varier dans de larges mesures. Aucune indication n'est fournie à l'homme du métier pour choisir les NTC ayant le rapport d'aspect requis.

Dans le paragraphe [0022], seule est mentionnée une définition de NTC oxydés, correspondant à des produits d'oxydation de NTC, les espèces oxydées étant généralement de type carboxylique, cétone ou hydroxyle.
Aucune indication n'est fournie pour obtenir de tels NTC oxydés, et plus particulièrement des NTC ayant un niveau d'oxydation de 1% à 15% en poids. En l'absence de définition du niveau d'oxydation, et en l'absence de méthode de détermination de ce niveau d'oxydation, l'homme du métier ne sait pas comment sélectionner les NTC adaptés à la composition revendiquée.

2) Selon l'invention revendiquée, les NTC discrets comportent des cristaux ou des couches de matériau actif lithium-ion attachés à leur surface. Dans la description ([0008] et [0015]), le problème à la base de l'invention est résolu en attachant des cristaux nanométriques (« nanosized ») ou des couches nanométriques de matériau actif Li-ion à la surface de NTC discrets, fonctionnalisés et bien dispersés.
Il subsiste donc un doute sur la taille des cristaux ou des couches de matériau actif Li-ion qui doivent être attachés à la surface des NTC pour résoudre le problème.
De plus, pour « attacher » les cristaux ou les couches de matériau actif sur les NTC, l'homme du métier ne peut se baser que sur les exemples fournis dans la description, mais ne dispose d'aucune information pour obtenir les cristaux ou les couches de matériau actif ayant une taille adaptée.

3) La description ne fournit aucune définition sur certaines caractéristiques essentielles des procédés revendiqués dans les rev. 11 et 12 :

- *Procédé de la rev. 11*
  L'étape b) met en œuvre un liquide de point d'ébullition élevé : l'homme du métier ne sait pas comment choisir un tel liquide, et la mesure du terme « élevé ».
  L'équilibre correct des réactifs et la température de réaction des étapes c) et d) sont des termes vagues qui nécessitent pour l'homme du métier de réaliser un nombre d'expérimentation important pour obtenir le sel de lithium.
  L'homme du métier ne sait pas non plus dans quel domaine de température le recuit des NTC doit être effectué à l'étape f) pour obtenir la structure cristalline d'olivine, à moins d'effectuer un nombre important d'expérimentation et de caractérisation.
- *Procédé de la rev . 12*
  Les termes « liquide » et « agent tensio-actif » dans l'étape b) sont vagues, et la description ne fournit aucune indication permettent à l'homme du métier de savoir quels sont les liquides ou les agents tensio-actifs utilisables pour réaliser cette étape du procédé de l'invention.

Il faut donc conclure que l'exposé de l'invention dans le brevet ne contient pas tout l'enseignement requis pour que l'homme du métier puisse exécuter l'invention.

ARTICLE 100c CBE – EXTENSION DE L'OBJET (ART. 123(2) CBE)

Les revendications 5 à 10 du brevet délivré sont dépendantes des revendications précédentes, ce qui conduit à des compositions revendiquées présentant des combinaisons de caractéristiques qui ne sont pas décrites explicitement dans la demande telle que déposée.
Dans la demande telle que déposée, ces caractéristiques ne sont décrites que de façon séparée, et non en combinaison, l'objet du brevet opposé s'étend donc au-delà du contenu de la demande telle qu'elle a été déposée.

ARTICLE 100a CBE – DEFAUT DE NOUVEAUTE (Art.54)

1) **Défaut de nouveauté de la revendication 1 au vu de A2**

A2 divulgue une composition comprenant des NTC discrets comportant des particules de matériau actif lithium-ion attachés à leur surface (A2, p.22 l.20-31, p.20 l.24 à p.21 l.21).
Les NTC ont un rapport longueur/diamètre supérieur à 10, et de préférence supérieur à 100 (A2, p.8 l.7-9), donc compris dans la gamme 10-500 revendiquée.
Les NTC peuvent être des NTC oxydés (A2, p.8 l.24-27), donc ayant un degré d'oxydation compris dans la gamme 1-15% revendiquée.

La revendication 1 du brevet opposé n'est donc pas nouvelle au vu de A2.

2) **Défaut de nouveauté des revendications dépendantes 2, 4, 7 et 9 au vu de A2**

Les caractéristiques additionnelles des revendications 2 et 4 sont décrites dans A2 (p.20 l.24 – p.21 l.21).
L'exemple 2 de A2 divulgue une composition comprenant 2% de NTC et 93% de matériau actif Li-ion qui rentre dans la gamme 0.1% à 10% de la revendication 7.
Il est clair selon l'enseignement de A2 (p.22 l.20-31), que les NTC sont dispersés de façon homogène, ce qui détruit la nouveauté de la revendication 9 du brevet opposé.

ARTICLE 100a CBE – DEFAUT D'ACTIVITE INVENTIVE (Art.56)

Remarque préliminaire

L'effet technique des caractéristiques de rapport d'aspect ou de degré d'oxydation pour les NTC n'a pas été démontré dans le brevet opposé, et ne peut donc pas en être tenu compte dans la formulation du problème technique objectif.

1) **Défaut d'activité inventive au vu de A2**

Etant donné que la priorité du brevet opposé n'est pas valable, la date effective à considérer est la date de dépôt de la demande PCT dont est issue la demande EP, c'est-à-dire le 21.06.2012, le document A2 est donc opposable au titre de l'article 54(2).

Si la nouveauté de la rev.1 au vu de A2 était malgré tout reconnue, la revendication 1 n'est pas inventive au vu de A2.
En effet les caractéristiques des NTC, en particulier leur rapport d'aspect et leur degré d'oxydation (sous réserve que ces caractéristiques soient reconnues comme bien définies dans l'exposé de l'invention), même si elles ne sont pas considérées comme explicites dans A2, elles sont au moins suggérées dans l'enseignement général de A2.
De plus, A2 (p. 22 l.20 – p.21 l.8) décrit les avantages apportés par l'association des NTC avec des particules de matériau actif, notamment au niveau des étapes de charge-décharge et de maintien mécanique.

La rev. 1 du brevet opposé résulte donc de A2 sans impliquer d'activité inventive.

Par ailleurs, le titulaire n'a pas montré que ces caractéristiques d'aspect et de degré d'oxydation sont essentielles dans la résolution du problème posé. Aucun exemple comparatif n'a été fourni permettant de justifier que des NTC ne présentant pas les caractéristiques requises ne sont pas adaptés pour résoudre le problème posé.

2) **Défaut d'activité inventive au vu de A3**

A3 a trait au domaine des batteries au lithium avec des électrodes contenant des fibrilles de carbone. Ces fibrilles de carbone correspondent à des NTC au sens du brevet opposé.
Dans A3, les NTC discrets (non aggrégés) (A3, p.6 l.32-36) peuvent avoir un rapport d'aspect supérieur à 5, de préférence supérieur à 100 (A3, p.9 l.20-24), et peuvent être oxydés (A3, p.11 l.6-20). Ces NTC sont intercalés avec du lithium (A3, p.7 l.1-3), ce qui suggère que des particules de matériau actif de lithium sont attachées à leur surface.

Comme déjà mentionné ci-dessus, l'effet technique des caractéristiques d'aspect de forme ou de degré d'oxydation pour les NTC n'a pas été démontré dans le brevet opposé, et ne peut donc pas en être tenu compte dans la formulation du problème technique objectif.

La rev. 1 du brevet opposé résulte de l'enseignement de A3 sans impliquer d'activité inventive.

3) **Défaut d'activité inventive au vu de A4**

A4 décrit une composition comprenant des NTC discrets ayant des cristaux ou couches de matériau actif de li-ion attachés à leur surface (p.15, [0061], p.21 [0082], [0083]).
Le rapport d'aspect et le degré d'oxydation des NTC ne sont pas explicitement décrits dans A4.

Pour les mêmes raisons que celles mentionnées ci-dessus, la rev. 1 du brevet opposé résulte de l'enseignement de A4 sans impliquer d'activité inventive.

Les revendications dépendantes 2 à 10 du brevet opposé ne comportent de caractéristiques justifiant une activité inventive au vu de l'enseignement de A4.

A4 décrit un procédé pour produire des NTC avec des cristaux ou des couches de matériau actif à leur surface, comprenant les étapes telles que décrites dans les rev. 11 et 12 du brevet opposé (A4, [0049]-[0052], [0082], [0083], exemple 9).
Les procédés des rev. 1 et 12 du brevet opposé se différencient de A4 par l'étape de recuit des NTC sous atmosphère inerte pour obtenir une structure d'olivine. Sous réserve de la description suffisante de cette étape, il peut être considéré qu'une étape de recuit est à la portée de l'homme du métier qui n'exerce ainsi pas d'activité inventive, la structure cristalline d'olivine n'ayant par ailleurs pas été démontrée dans le brevet opposé.

Les revendications 11 et 12 sont donc dépourvues d'activité inventive au vu de A4.

4) **Défaut d'activité inventive au vu de A5**

A5 décrit une composition comprenant des NTC discrets ayant des cristaux ou couches de matériau actif de li-ion attachés à leur surface ([0007] à [0009],[00039], [00042]).
Le rapport d'aspect et le degré d'oxydation des NTC ne sont pas explicitement décrits dans A5.
Pour les mêmes raisons que celles mentionnées ci-dessus, la rev. 1 du brevet opposé résulte de l'enseignement de A5 sans impliquer d'activité inventive.

Les revendications dépendantes 2 à 10 du brevet opposé ne comportent de caractéristiques justifiant une activité inventive au vu de l'enseignement de A5.

5) **Défaut d'activité inventive au vu de A6**

A6 concerne un composite d'électrode comprenant une structure poreuse de carbone revêtue d'un oxyde mixte métallique, utilisable dans des batteries. La structure carbonée peut être des NTC (A6, rev. 1 et 6).

Il est donc connu de A6 d'attacher des matériaux actifs d'électrode à des NTC pour améliorer le fonctionnement de batteries.
Partant de l'enseignement de A6, l'homme du métier aurait pu adapter la nature du matériau actif à celui d'une batterie li-ion sans exercer d d'activité inventive.

6) **Défaut d'activité inventive au vu de A7**

A7 décrit des nano composites à base de phosphate de lithium et de NTC et/ou graphène (A7, p. 3 l.13-16), utilisables comme matériaux actifs de cathode pour des batteries au lithium.

Il est donc connu de A7 de disposer de composition de NTC liés à des cristaux ou couches de matériau actif de li-ion.

Partant de l'enseignement de A7, l'homme du métier aurait pu parvenir à l'invention revendiquée sans exercer d d'activité inventive.

7) **Défaut d'activité inventive au vu de A8**

A8 concerne un matériau actif d'électrode revêtu de carbone à partir d'un précurseur de carbone qui est choisi parmi les NTC et/ou du graphène pouvant être oxygénés ou hydratés (A8, rev. 1, 3, 7). Le matériau actif est un oxyde mixte de lithium pouvant être de structure olivine (A8, rev.13).

Il est donc connu de A8 de disposer de composition de NTC liés à des cristaux ou couches de matériau actif de li-ion, en particulier de structure cristalline d'olivine dans le domaine des batteries li-ion.

Partant de l'enseignement de A8, l'homme du métier aurait pu parvenir à l'invention revendiquée sans exercer d d'activité inventive.

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(54) **COMPOSITE ELECTRODE COMPRISING A CARBON STRUCTURE COATED WITH A THIN FILM OF MIXED METAL OXIDES FOR ELECTROCHEMICAL ENERGY STORAGE**

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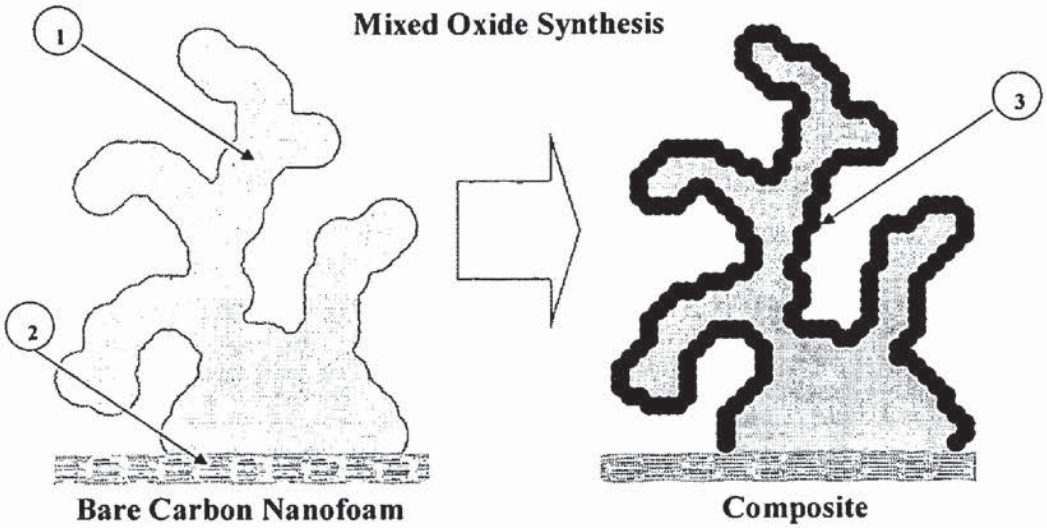
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H01G 9/042	(2006.01)

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(57) **ABSTRACT**

A composite electrode is created by forming a thin conformal coating of mixed metal oxides on a highly porous carbon structure. The highly porous carbon structure performs a role in the synthesis of the mixed oxide coating and in providing a three-dimensional, electronically conductive substrate supporting the thin coating of mixed metal oxides. The metal oxide mixture shall include two or more metal oxides. The composite electrode, a process for producing said composite electrode, an electrochemical capacitor and an electrochemical secondary (rechargeable) battery using said composite electrode are disclosed.



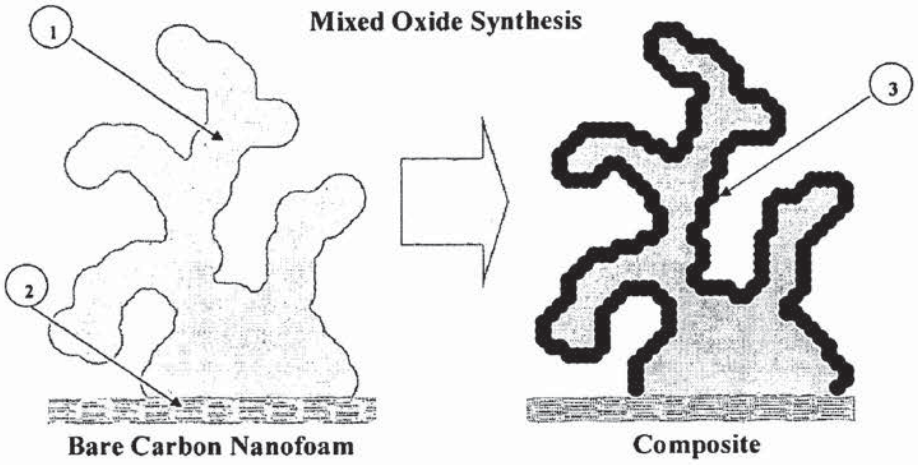


FIG. 1

Nickel Ratio	1	1	1	0
Manganese Ratio	4	2	1	1
Capacity (F/g in 1M LiCl)	131.4	107.58	94.78	99.27

FIG. 2

Temp (C)	Initial Mass	Final Mass	Oxide Mass	Mass Increase%	Oxide % of Final Mass
amb (~20)	0.0253	0.0355	0.0102	40.32	28.73
40	0.0248	0.0335	0.0087	35.08	25.97
60	0.026	0.0364	0.0104	40.00	28.57
80	0.0265	0.036	0.0095	35.85	26.39

FIG. 3

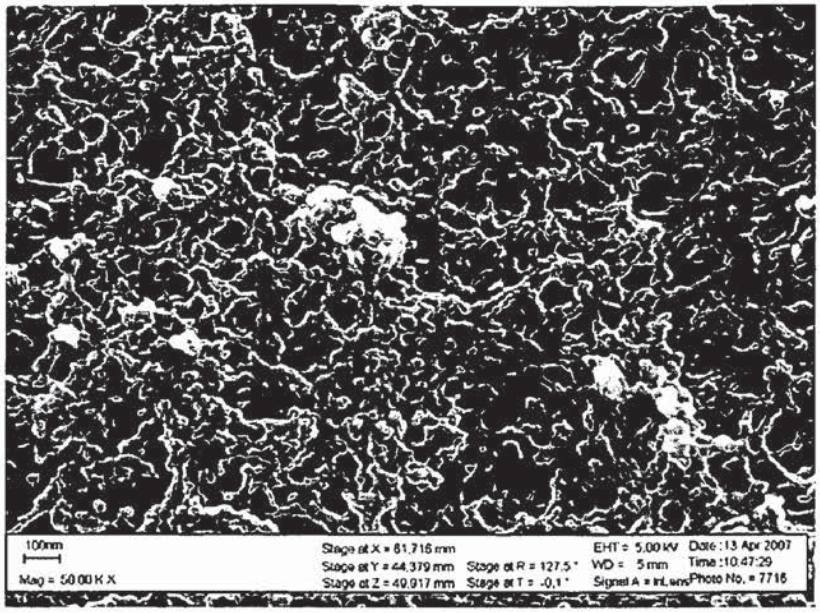

100nm
Mag = 50.00 K X
Stage at X = 81.716 mm
Stage at Y = 44.379 mm
Stage at Z = 49.017 mm
Stage at R = 127.5 °
Stage at T = -0.1 °
EHT = 5.00 kV
WD = 5 mm
Date :13 Apr 2007
Time :10:47:29
Photo No. = 7716

FIG. 4

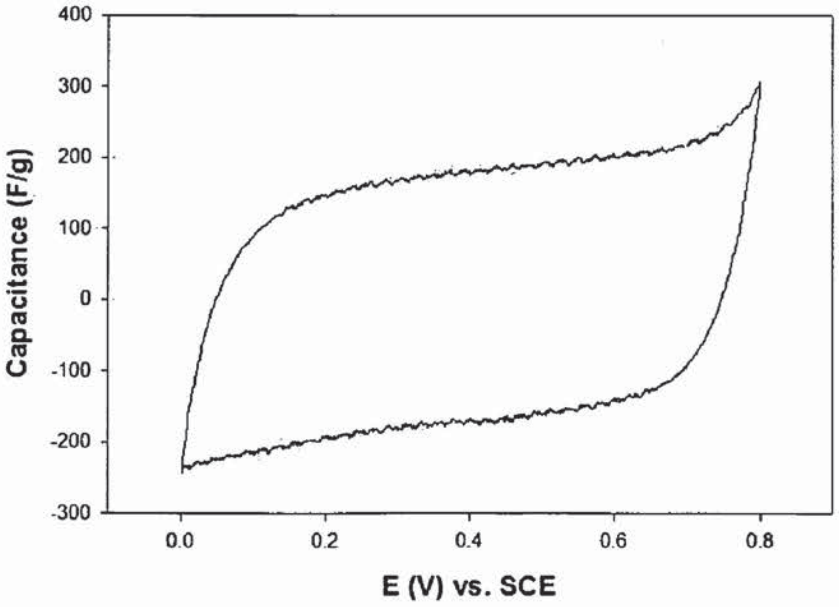

Capacitance (F/g)
400
300
200
100
0
-100
-200
-300
0.0
0.2
0.4
0.6
0.8
E (V) vs. SCE

FIG. 5



FIG. 6