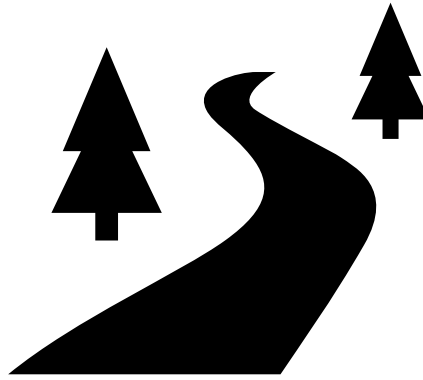




**Patent Owner Demonstrative Exhibits in
IPR2024-01006, IPR2024-01011, IPR2024-01013**

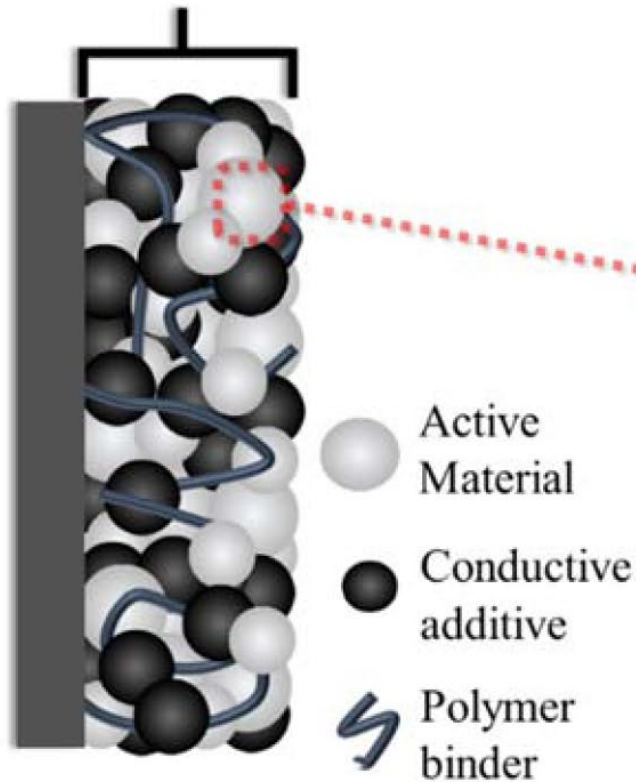
Consolidated Hearing
Patent Trial and Appeal Board
September 16, 2025



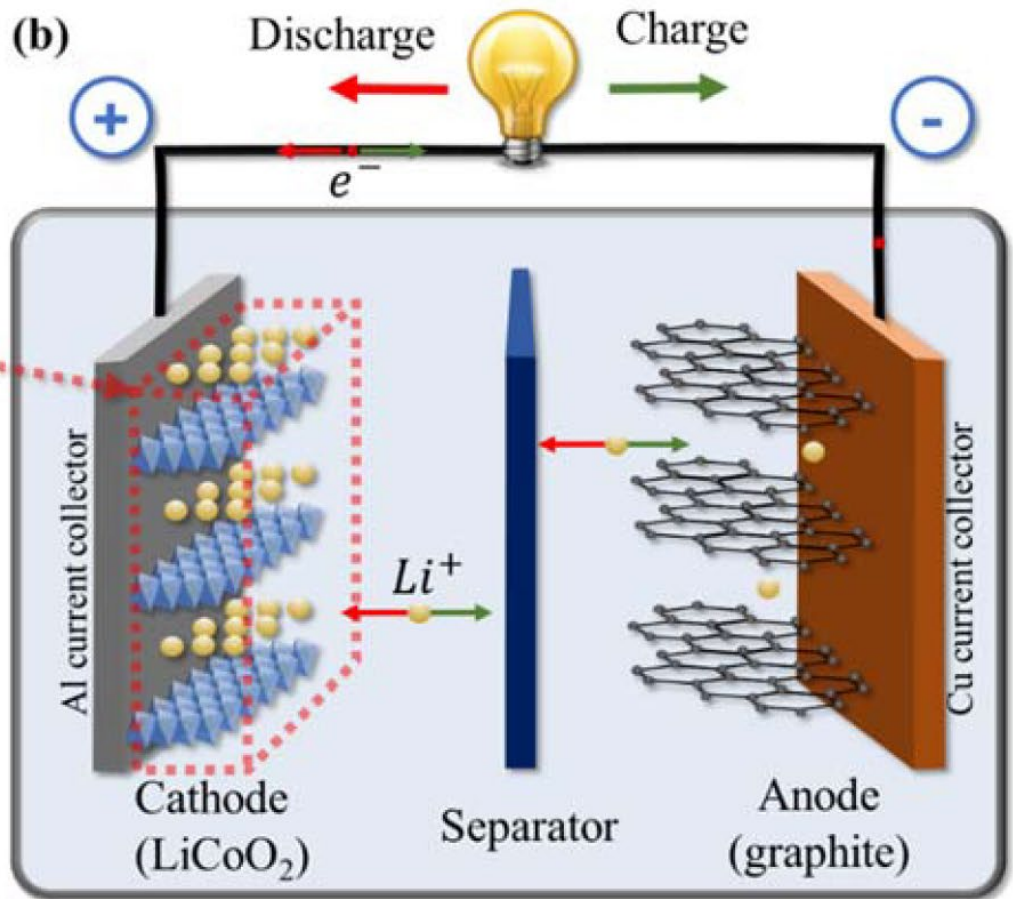
Overview of Technology

Electrodes Generally

(a) Composite electrode



(b)



MRD-2037, 2

Efforts to Improve Electrical Conductivity: Carbon Black

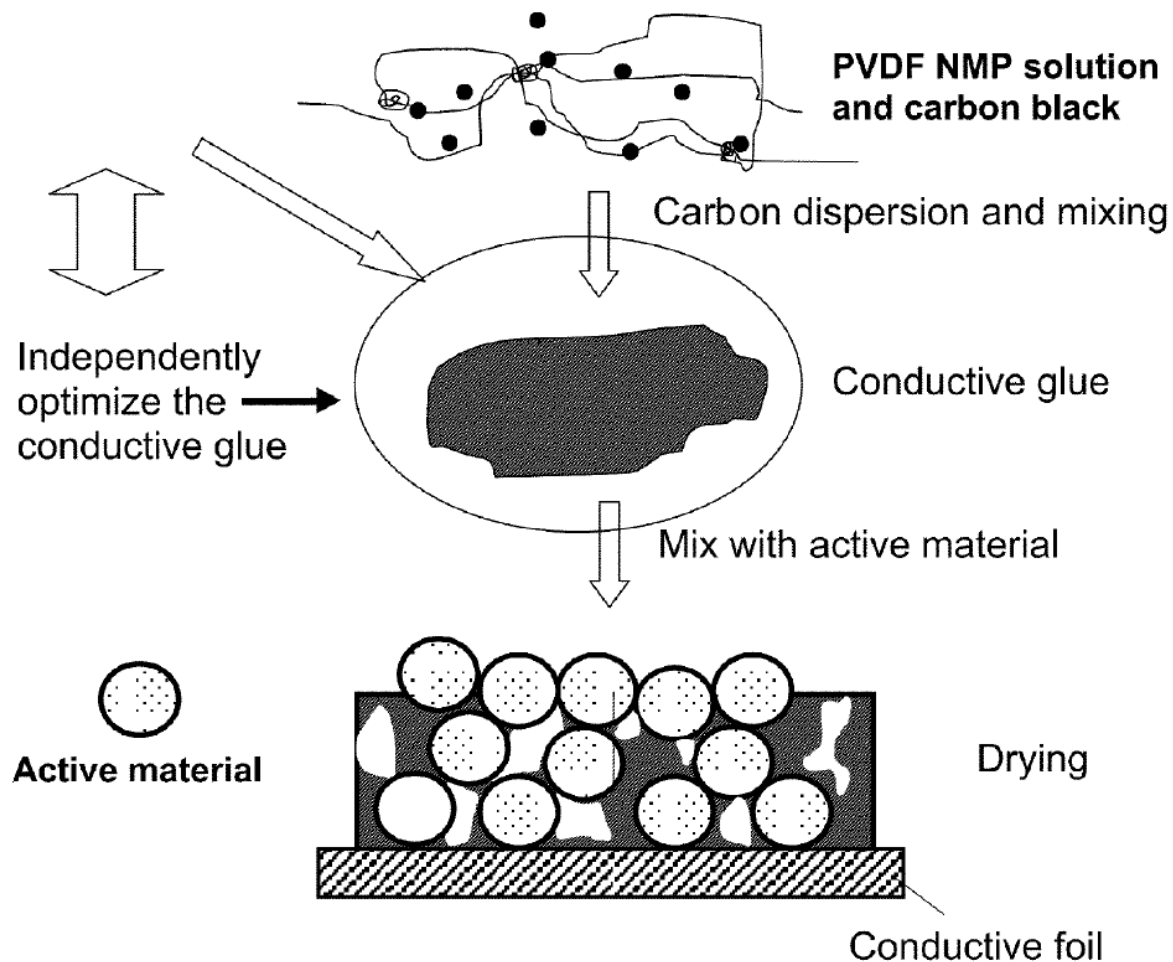
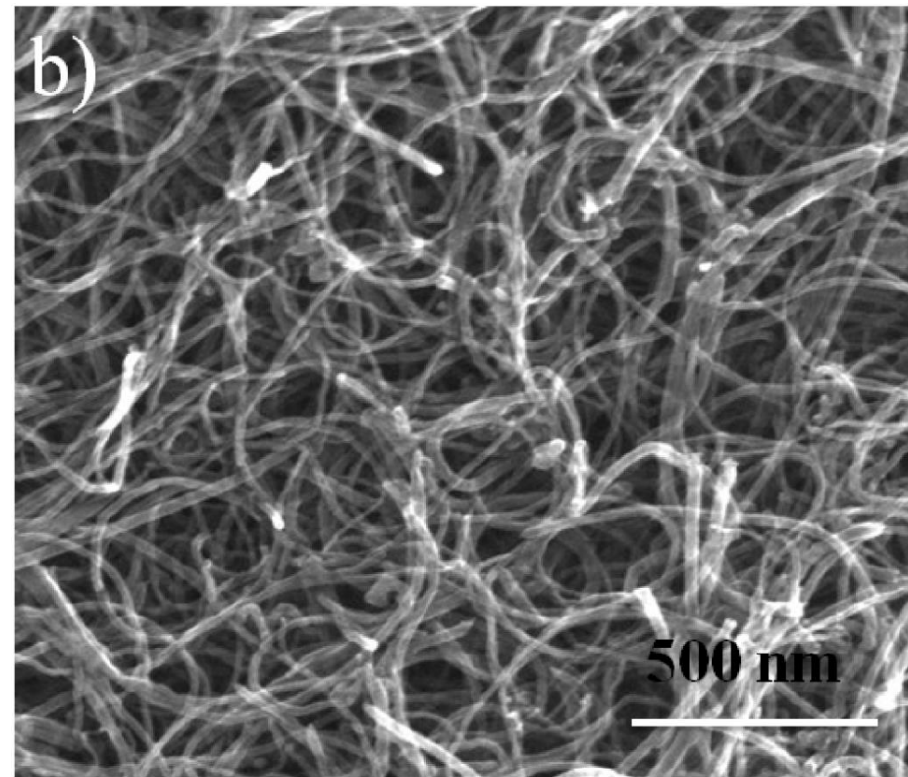
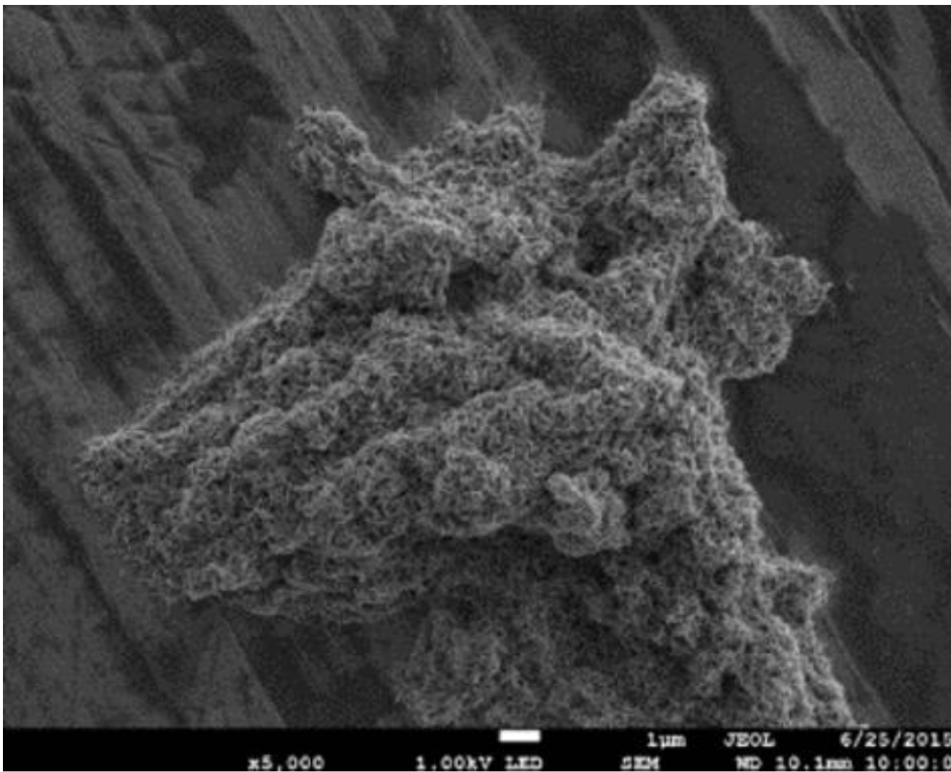


FIG. 6

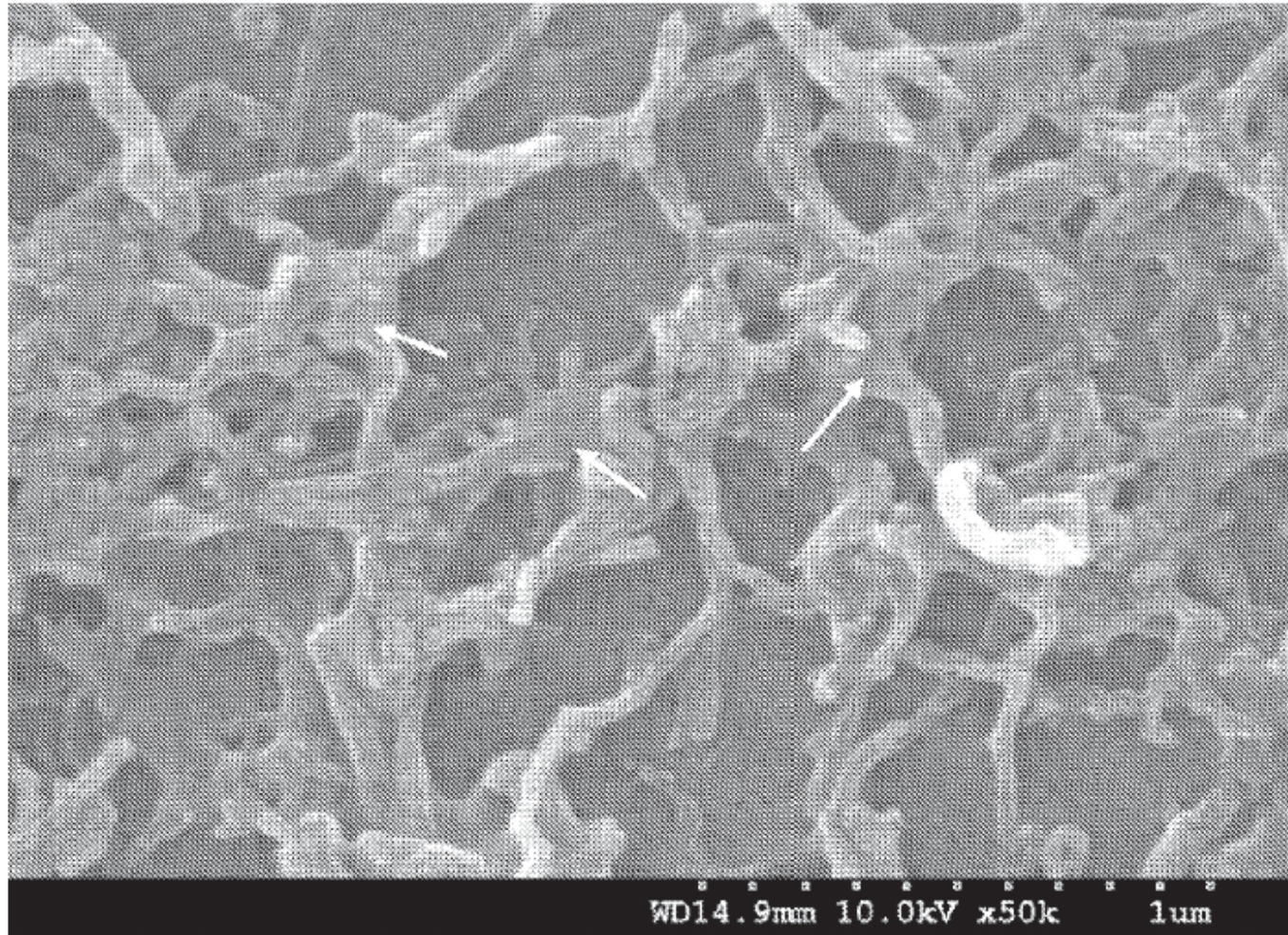
MRD-2008

Efforts to Improve Electrical Conductivity: Carbon Nanotubes



Prior Efforts to Improve Conductivity: Better Dispersed “Percolated” CNT Networks

Gan, Fig. 5



MRD 2019/LGES-1021

The Inventors' Discovery

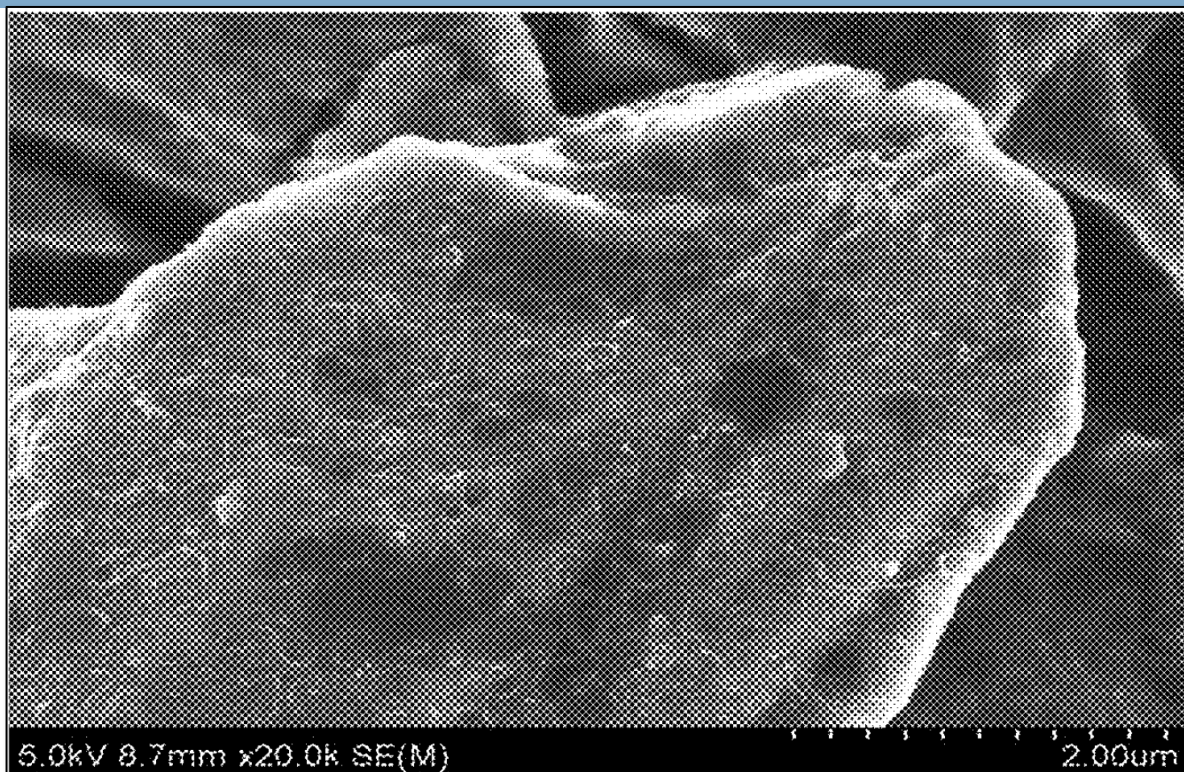
BACKGROUND

Attempts to overcome the above deficiencies have included synthesis of nano-sized crystals, termed nanocrystals, of lithium salts as well as mixtures of lithium salts and carbon nanotubes. Even though the mixtures of carbon nanotubes and nanocrystals of lithium salts show improvements in charge/discharge rates, the carbon nanotubes previously employed are not considered to be essentially discrete, i.e., individual uniformly dispersed tubes. Carbon nanotubes as conventionally made by gas phase reaction result in a tangled bundle of tubes with particle diameters ranging from 50 to 200 micrometers. Also, the lithium nanocrystals are not ionically or covalently attached to the surface of the discrete carbon nanotubes to provide for intimate electron transfer and enhanced mechanical strength of the active material. During charging and discharging of conventional lithium ion batteries, the nanocrystals expand and contract. Over a number of cycles, this leads to microcrack formation in the layer of active material and hence to higher internal resistance and decline of the battery performance. With crystals bound to and interconnected with discrete carbon nanotubes the microcrack formation due to severe mechanical vibration, bending, or expansion and contraction will be retarded.

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.

'483 Patent, 1:36-2:12

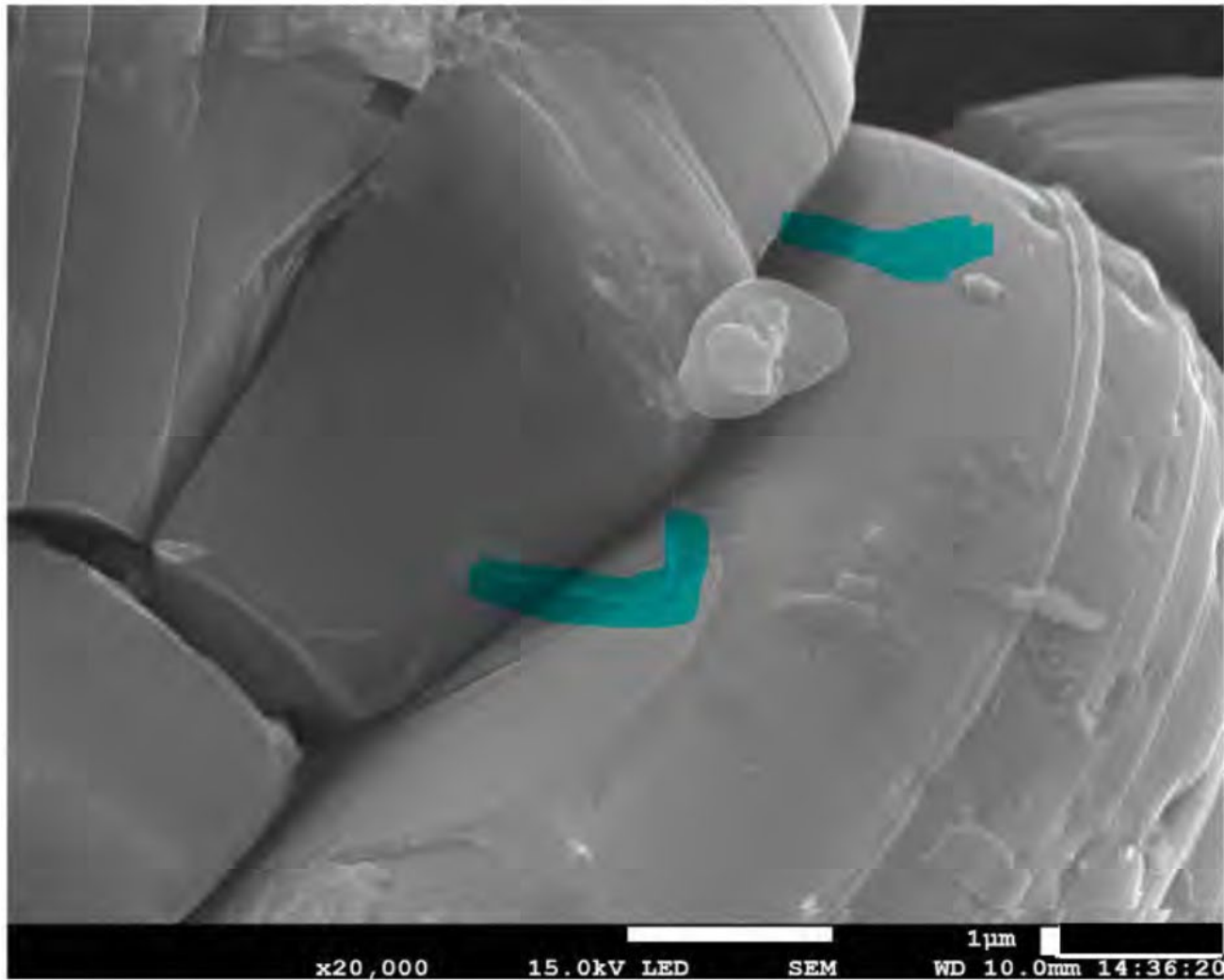
LGES' 2018 Patent Application



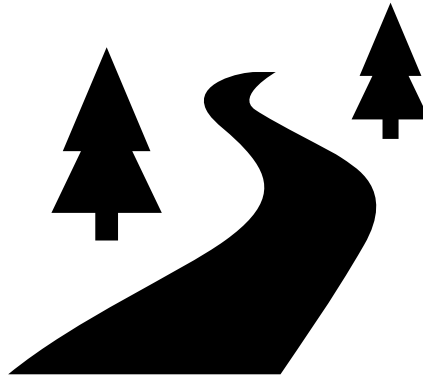
As a result, even when the silicon-based active material undergoes volumetric swelling during charge/discharge cycles to cause cracking of particles, the fibrous phase of carbon nanotubes is positioned on the surface of silicon-based particles so that it may still cover the whole of the cracked or shrunk silicon-based particles and crosslink the particles.

MRD-2028 ¶[0042]

LGES-1050: LGES' Products



LGES-1050, 32 ('483 & '909)/LGES-1033, 32 ('924)



Claim Construction

“discrete carbon nanotubes”

Patent Owner	Petitioner
carbon nanotubes that are <u>separate</u> and distinct from other nanotube fibers— <u>not attached</u> to other nanotubes	Petition: “ <u>fully isolated</u> ” carbon nanotubes Reply: “carbon nanotubes <i>capable of</i> being separated to give individual tubes.”

’483 POR, 13; ’483 Sur-reply, 7

’483 Pet., 36; MRD-2024, 11:4-8; ’483 Reply, 3

Described Problem/Solution

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

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.

'483 Patent, 1:36-2:12

The Disclosed Process

During the process of making discrete carbon nanotubes (which can be single, double and multiwall configurations), the nanotubes are cut into segments and residual catalyst particles that are interior to the carbon nanotubes as received from the manufacturer are dissolved. This cutting of the tubes helps with exfoliation. The cutting of the tubes reduces

Example 3

'483 Patent, 4:6-24

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

'483 Patent, 5:53-6:3

The Common Meaning of “discrete”

WEBSTER'S NEW WORLD COLLEGE DICTIONARY

Fourth Edition

dis·crete (di skrēt') *adj.* [ME *discret*: see DISCREET] 1 **separate and distinct; not attached to others; unrelated** 2 made up of distinct parts; discontinuous 3 designating or of an electronic circuit having separate transistors, resistors, etc. —**dis·crete'ly** *adv.* —**dis·crete'ness** *n.*

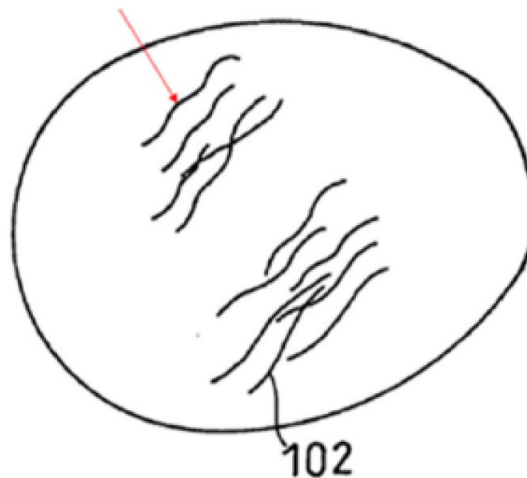
MRD-2007, 8

Dr. Zhu's Original Position

“And looking again to Ohata’s illustrative example, provided below, it can be seen that **most** of the nanotubes are depicted as being discrete, non-agglomerated, and exfoliated. Indeed, a POSITA would have understood a carbon nanotube that is, as shown below, **fully isolated** from each other to be discrete, non-agglomerated, and exfoliated.”

“discrete, non-agglomerated, and exfoliated
carbon nanotubes”

LGES-1003 ('483) ¶83



Excerpt from Ohata, Fig. 1(b) (annotated)

Dr. Zhu's Original Position

Q And so you're identifying that nanotube as discrete because it's fully isolated from the other nanotubes?

A Yeah. I say it is discrete, non-agglomerated and exfoliated carbon nanotubes.

MRD-2024, 11:4-8

Q And so you testified that a discrete nanotube is a non-bundled -- non-bundled -- or I'm sorry.

Your testimony was that discrete carbon nanotubes are non-bundled nanotubes that are separate from each other, correct?

MR. IN: Objection|. Form.

A Yeah. I think more accurately, the -- what I said in the declaration is discrete, non-agglomerated and exfoliated nanotubes are nanotubes that are uniformly distributed in the medium.

MRD-2024, 13:13-22

Dr. Zhu Was Not Taken “Out of Context”

7 What is the definition of a discrete
8 carbon nanotube in your own words?

9 MR. IN: Objection to form.

10 A Well, I think we don't really need to
11 have a definition for this discrete because this is a common

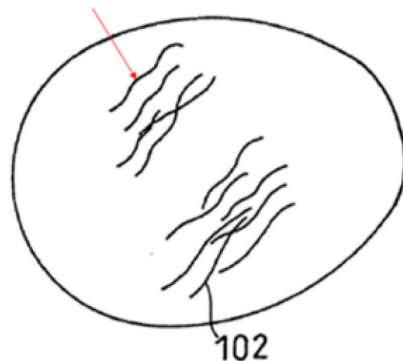
MRD-2024, 9:3-15

Dr. Zhu's Supplemental Testimony Should Be Given No Weight

To be sure, a fully isolated carbon nanotube that is not touching any other nanotube is likely “discrete.” However, this is a description, not a definition. Just as a carbon nanotube is undeniably a nanotube without all nanotubes needing to be carbon nanotubes, a fully isolated nanotube can be discrete without all discrete nanotubes needing to be fully isolated.

“discrete, non-agglomerated, and exfoliated
carbon nanotubes”

LGES-1056 ¶7



Excerpt from Ohata, Fig. 1(b) (annotated)

Petitioner's New "Capable of" Construction is Implausible

carbon nanotubes. Even though the mixtures of carbon nanotubes and nanocrystals of lithium salts show improvements in charge/discharge rates, the carbon nanotubes previously employed are not considered to be essentially discrete, i.e., individual uniformly dispersed tubes. Carbon

'483 Patent, 1:36-43

During the process of making discrete carbon nanotubes (which can be single, double and multiwall configurations), the nanotubes are cut into segments and residual catalyst particles that are interior to the carbon nanotubes as received from the manufacturer are dissolved. This cutting of the

Id., 4:6-9

Example 3

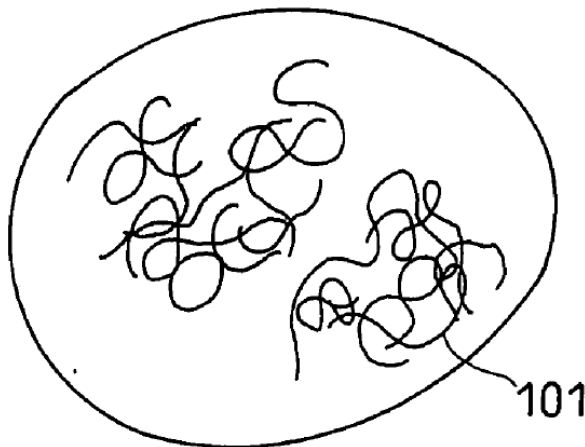
Lithium iron phosphate obtained commercially is ground with multiwall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and of aspect ratio about 60, in a mortar and pestle at weight ratios of 5:1 and 1:1 lithium salt to carbon nanotube. Sodium dodecyl sulfate (SDS) is then added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

Id., 5:54-6:3

Dr. Zhu's Testimony Undermines the "Capability" Construction

OHATA, FIG. 1(a)

(CNTs "tangled and twisted with one another")



Q. Right. But does Figure 1A depict discrete carbon nanotubes?

A. It's not a discrete carbon nanotube. Figure 1A is not a discrete carbon nanotube.

MRD-2043, 12:7-13

Q. (BY MR. ZINDA) Right. But if you apply the -- if you apply the appropriate process, isn't it true that any carbon nanotube is capable of being separated from another carbon nanotube?

MR. IN: Objection, form.

A. The carbon nanotube -- or the entangled carbon nanotube in Figure 1A, if it was under right procedure, there is always a potential to be separated.

MRD-2043, 13:13-20

Petitioner's "Capable" Construction is Inconsistent with its Position in the '282 Proceedings

Q. From the perspective of a person of ordinary skill in the art, what is a discrete carbon nanotube fiber?

MR. RIFFE: Objection, legal conclusion.

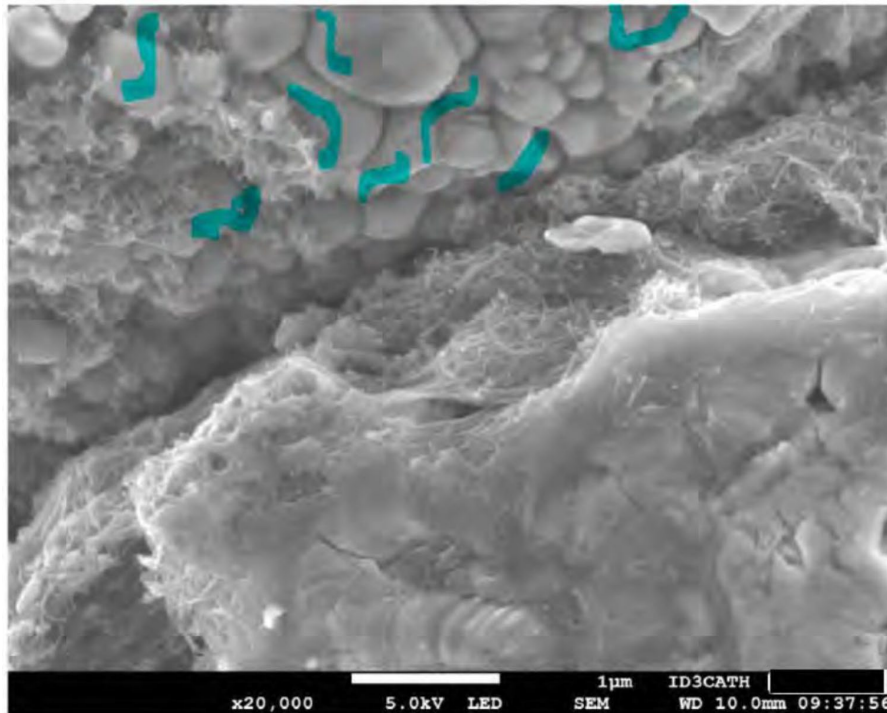
THE WITNESS: Well, I talked about this in my Declaration and should probably look at that, but I remember, you know, what I said, and basically individual nanotubes that are not associated with each other along the majority of their length, or something to that effect.

Q. Does a discrete carbon nanotube fiber have to be separate from another carbon nanotube?

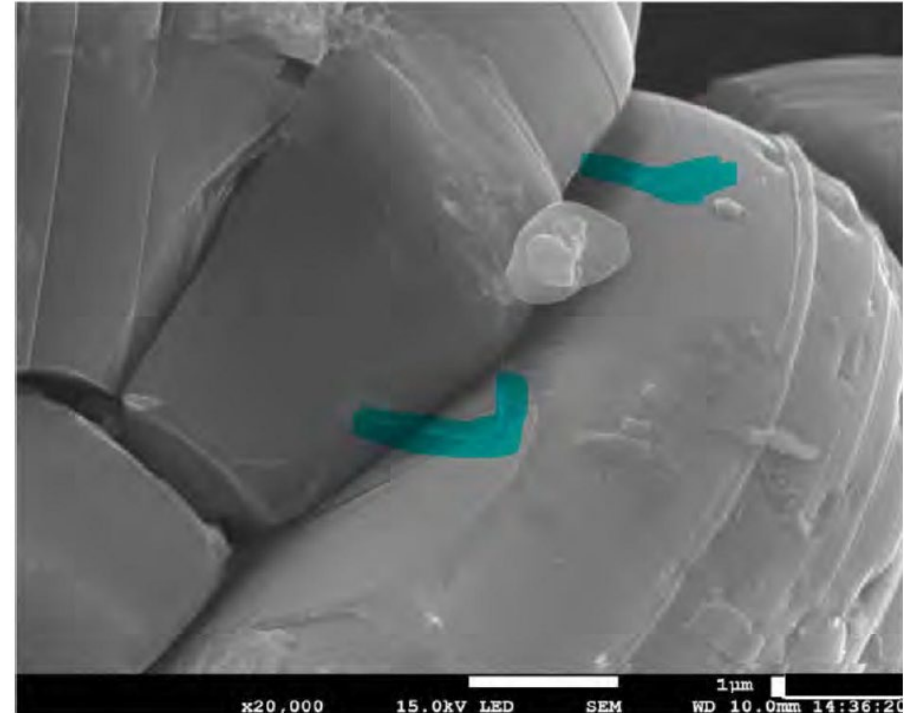
MR. RIFFE: Objection, form.

THE WITNESS: It has -- well, it should not be associated with other carbon nanotubes along a substantial portion of its length.

Petitioner's Reliance on the Infringement Contentions is Misplaced

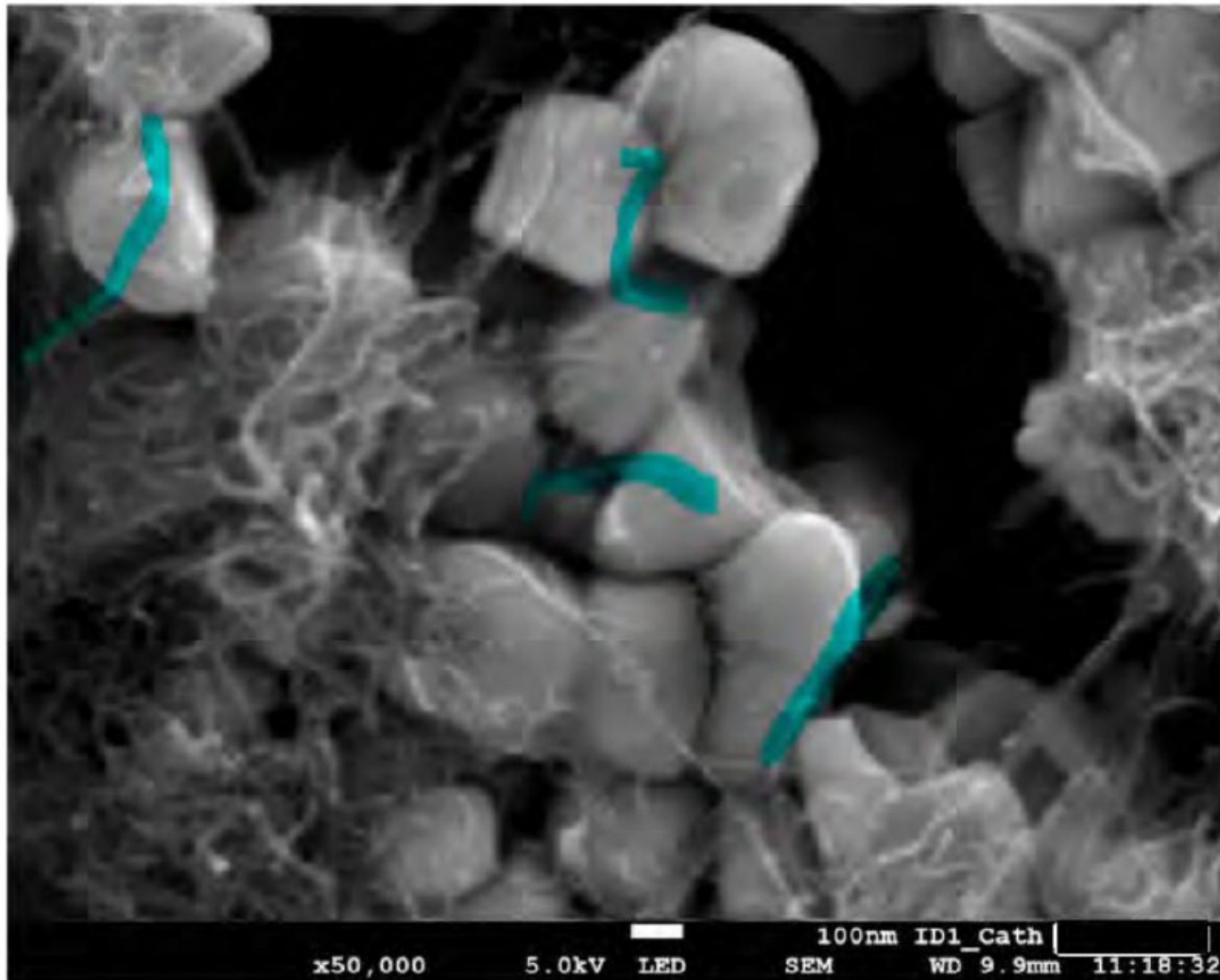


LGES-1050 ('483), 31



LGES-1050 ('483), 32

Petitioner's Reliance on the Infringement Contentions is Misplaced



LGES-1051 ('483), 36

Petitioner's Reliance on the Infringement Contentions is Misplaced

THE WITNESS: Yes. This drawing is two-dimensional. There's a third dimension that's not shown, you know. If you just look at a two-dimensional view, it looks like that they are in physical contact. But if one looks at the third dimension as well, they might not be.

LGES-1055, 127:21-128:1

POR, 42-43. However, in a two-dimensional projection of a three-dimensional sample, such as the microscope image of Sun's Fig. 6b, nanotubes that appear to cross in 2-D may, in fact, be present on entirely different planes and thus not in contact at all.

Paper 23, 19 in IPR2024-01005



Trials and appeals

Globus Medical, Inc. v. Spinelogik, Inc., IPR2025-00226, Paper 9 at 11 (PTAB July 14, 2025) (“[W]e decline to adopt Petitioner’s conclusory assumptions regarding what it purports to be Patent Owner’s applied construction” based on infringement contentions)

The Inventors Knew How to Claim “Capability”

8. A composition useful for lithium ion batteries, comprising oxidized discrete, non-agglomerated, and exfoliated carbon nanotubes having a surface, and mixtures of ion active materials comprising migratable ions said materials comprising crystals or layers of lithium ion active material, wherein a ratio of lithium ion to other total ions in the ion active materials is at least 2:1 to 25:1, wherein the discrete carbon nanotubes have crystals or layers of lithium ion active materials attached to their surface, and wherein the carbon nanotubes are uniformly dispersed without clustering on a dimension scale of less than about 1 micrometer and wherein the discrete carbon nanotubes comprise oxidized species selected from the group consisting of carboxylic, ketone, or hydroxyl functionalities, and 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.

9. A composition useful for lithium ion batteries, comprising uniformly dispersible oxidized, non-agglomerated carbon nanotubes having a surface, and mixtures of ion active materials comprising migratable ions said materials comprising crystals or layers of lithium ion active material, wherein a ratio of lithium ion to other total ions in the ion active materials is at least 2:1 to 25:1, and wherein the discrete carbon nanotubes have crystals or layers of lithium ion active materials ionically attached to their surface and wherein the discrete carbon nanotubes comprise oxidized species selected from the group consisting of carboxylic, ketone, or hydroxyl functionalities, and 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.

'483 Patent, Cls. 8, 9

Petitioner Attempts to Improperly Equate Claim Terms

'483 Patent, Claim 1

1. A composition useful for lithium ion batteries comprising: discrete, non-agglomerated, and exfoliated carbon nanotubes having a surface, and mixtures of ion active materials comprising migratable ions said materials comprising crystals or layers of lithium ion active material.

'483 Patent, Cls. 8, 9



“[T]he use of two terms in a claim requires that they connote different meanings.” *Applied Med. Res. v. U.S. Surgical Corp.*, 448 F.3d 1324, 1333 n.3 (Fed. Cir. 2006).

“Agglomerate,” “Exfoliate” and “Discrete” Have Different Meanings

ag·glom·er·ate (ə glām'ər āt'; for *adj.* & *n.*, -ər it) *vt.*, *vi.* -at'ed, -at'ing [*< L agglomeratus*, pp. of *agglomerare < ad-*, to + *glomerare*, to form into a ball *< IE base *glem-*, var. of **gel-*, form a ball, sphere, hence akin to *CLIMB*, *CLENCH*] to gather into a cluster, mass, or ball —*adj.* gathered into a mass or ball; clustered —*n.* 1 a jumbled heap, mass, etc. 2 *Geol.* a mass of fragments of volcanic rock fused by heat —**ag·glom'era'tive** (-āt'iv, -ə tiv) *adj.*

ex·fo·li·ate (eks fō'lē āt') *vt.*, *vi.* -at'ed, -at'ing [*< LL exfoliatus*, pp. of *exfoliare*, to strip of leaves *< L ex-*, out + *folium*, a leaf: see *FOIL*²] to cast or come off in flakes, scales, or layers, as skin, bark, rock, etc. —**ex·fo'lia'tion** *n.* —**ex·fo'lia'tive** *adj.*

dis·crete (di skrēt') *adj.* [*ME discret*: see *DISCREET*] 1 separate and distinct; not attached to others; unrelated 2 made up of distinct parts; discontinuous 3 designating or of an electronic circuit having separate transistors, resistors, etc. —**dis·crete'ly** *adv.* —**dis·crete'ness** *n.*

MRD-2007, 5, 8, 11

Ohata-Kavan Grounds

'483 Petition

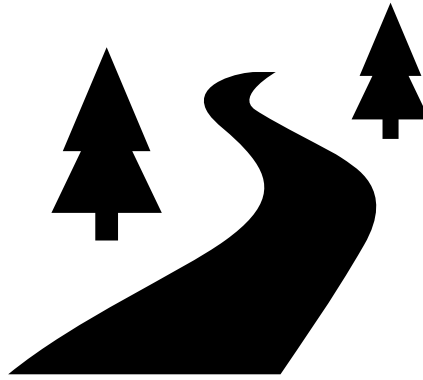
Ground	Claims	Basis for Rejection
1A/1B	1-4, 7-18	§103: Ohata and Kavan in view of Gong (1A) or Liu (1B)
2	1, 2, 7-18	§103: Ohata, Kavan, and Ichinose

'924 Petition

Ground	Claims	Basis for Rejection
1	1, 4, 5	§103: Ohata and Kavan
2A	1, 4, 5	§103: Cheng and Gan
2B	2, 3	§103: Cheng, Gan, and Zhao

'909 Petition

Ground	Claims	Basis for Rejection
1	1-2, 5-8	103: Ohata-Kavan
2A	1-2, 5-8	103: Lee-Wepasnick
2B	3-4	103: Lee-Wepasnick-Zhamu



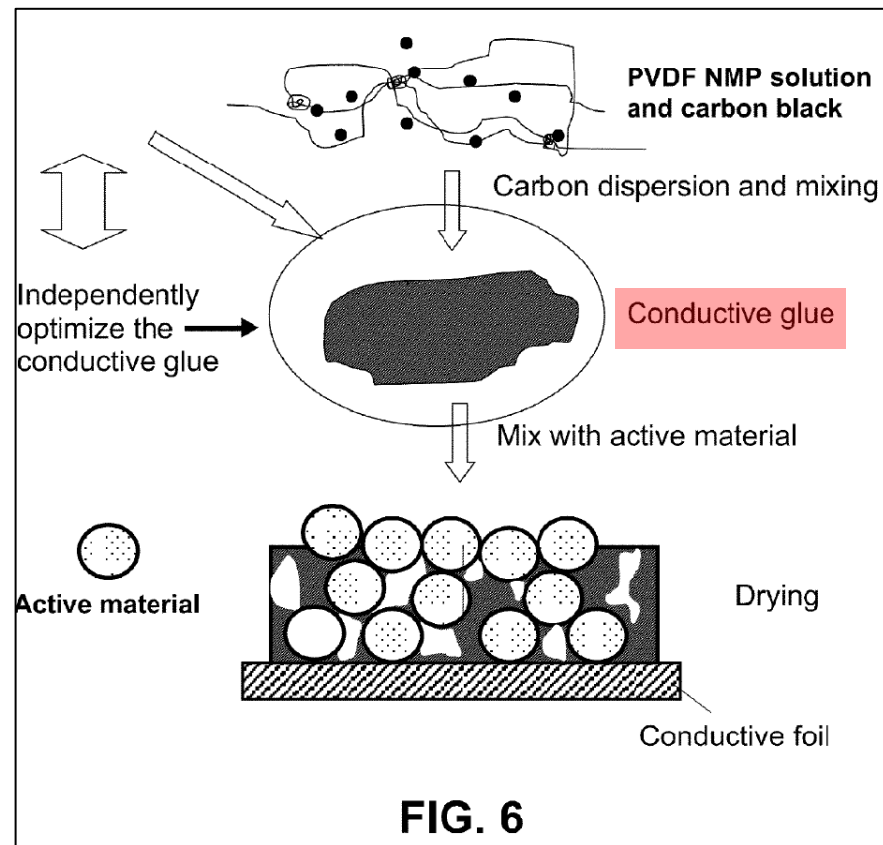
Overview of Ohata (LGES-1004)

Ohata: The Problem

battery capacity will be decreased. Accordingly, in order to reduce the amount of conductive material to be used, it is proposed to use a masterbatch process, whereby a conductive material is uniformly predispersed in a binder, in the production of a positive electrode (see Japanese Laid-Open Patent No. Hei 10-255844). As a conductive material, carbon black such as furnace black or acetylene black, and powdered graphite are used.

[0003] The primary particles of carbon black or powdered graphite are linked into a chain to form a conductive path. Such conductive path is likely to be damaged by shearing force. Shearing force is applied to the conductive material during the masterbatch process. For this reason, the use of the masterbatch process to uniformly disperse the conductive material in a binder does not yield an electrode superior in conductivity.

Ohata ¶¶[0002]-[0003]



MRD-2008

Ohata: The Superior “Masterbatch”

[0004] In view of this, it is an object of the present invention to **provide an electrode superior in conductivity with a small amount of conductive material**, yet with high capacity.

[0005] The present invention relates to a method for producing an electrode for a battery comprising the steps of: (a) **producing a masterbatch comprising at least carbon nanotubes and a resin**; (b) blending an electrode material mixture containing at least the masterbatch and an electrode active material with a dispersion medium to prepare an electrode material mixture paste; (c) applying the electrode

[0016] As shown in **FIG. 1(a)**, carbon nanotubes 101 are tangled and twisted with one another before they are blended with a resin. The carbon nanotubes are unraveled and straightened by blending them with a resin, as shown in **FIG. 1(b)**. **The unraveled and straightened carbon nanotubes 102 are expected to form an excellent conductive path in an electrode** even if the amount thereof is small.

Ohata ¶¶[0004]-[0005], [0016]

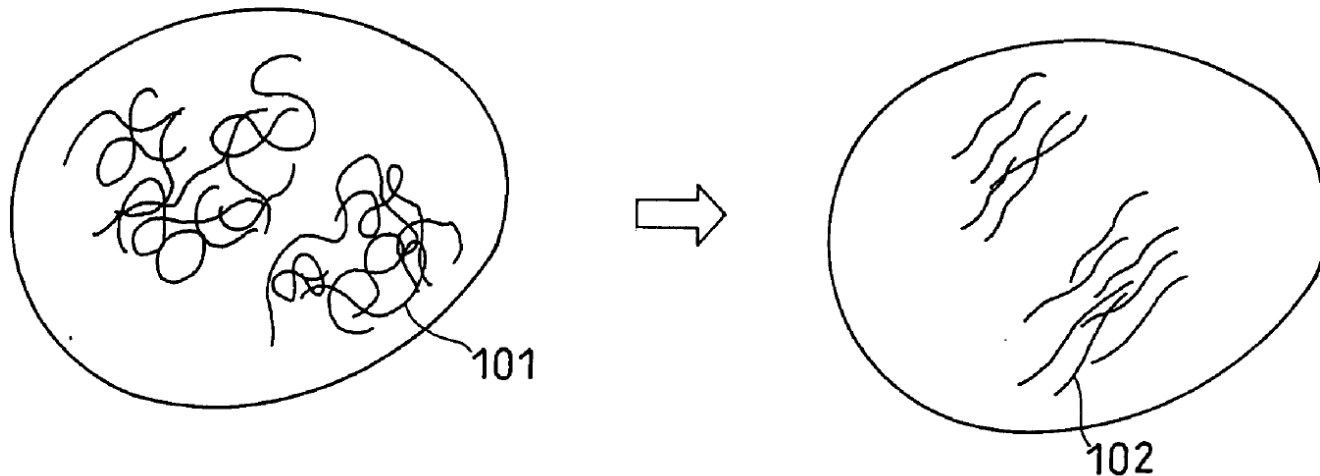
(a)

OHATA, FIG. 1

(b)

(CNTs “tangled and twisted with one another”)

(CNTs “unraveled and straightened”)



Ohata: Masterbatch CNT:PVDF Weight Ratios

[0006] The carbon nanotubes preferably have an average diameter (outer diameter) of not greater than $0.1\ \mu\text{m}$ and an aspect ratio of not less than 100 determined by dividing the average length of the carbon nanotubes by the average diameter. The proper amount of the carbon nanotubes contained in the masterbatch is 5 to 20 parts by weight per 100 parts by weight of the resin.

TABLE 1

	Amount of CNT Per 100 parts by weight of active material (parts by weight)	Amount of MB per 100 parts by weight of active material (parts by weight)	CNT content in MB (wt %)
Ex. 1	0.5	3.3	15
Ex. 2	0.2	1.3	15
Ex. 3	0.3	2	15
Ex. 4	0.8	5.3	15
Ex. 5	1.0	6.7	15
Ex. 6	1.5	7.5	20
Ex. 7	2.0	10	20
Ex. 8	3.0	15	20

CNT: Carbon nanotube
MB: Masterbatch

EXAMPLE 9

[0041] A positive electrode was produced in the same manner as in Example 1, except that a vinylidene fluoride-hexafluoropropylene copolymer was used as a resin used for the masterbatch instead of polyvinylidene fluoride. With the use of the thus obtained positive electrode, a prismatic non-aqueous electrolyte secondary battery analogous to the one in Example 1 was assembled.

Examples 10 to 14

[0042] Positive electrodes were produced in the same manner as in Example 1, except that carbon nanotubes having an average diameter and an aspect ratio shown in Table 2 were used. With the use of the thus-obtained positive

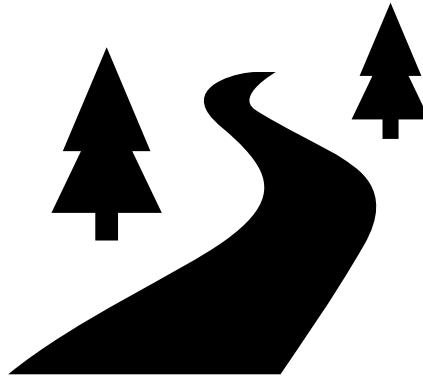
Ohata ¶¶[0006], [0040]-[0042]

Ohata: The Electrode Paste

[0023] An electrode material mixture paste can be prepared by blending an electrode material mixture containing the masterbatch and an electrode active material with a dispersion medium. During this process, a resin can be added to the electrode material mixture. The resin to be added may be the same resin as or a resin analogous to the one used during the production of the masterbatch, or a resin other than the above. In order to obtain a high-capacity electrode, the resin to be added is preferably particulate rubber that enables spot bonding between the active material particles.

[0024] The amount of the carbon nanotubes contained in the electrode material mixture is, for example, 0.2 to 3 parts by weight per 100 parts by weight of the electrode active material. In order to obtain a high-capacity electrode, the amount thereof is preferably not greater than 0.8 parts by weight. The dispersion medium should be selected according to the resin to be used. When polyvinylidene fluoride or a vinylidene fluoride-hexafluoropropylene copolymer is used, for example, N-methyl-2-pyrrolidone can be used.

Ohata ¶¶[0023]-[0024]



Overview of Kavan (LGES-1005)

Kavan's Method

Nanotubes were dispersed in water under short sonication in an ultrasonic bath. To this solution a powder material (TiO_2 , LiMnPO_4 or LiFePO_4) was added (in a proportion of 1–10 wt% of nanotubes in the mixture) and stirred overnight. The suspension was subsequently evaporated to dryness. The electrodes were prepared as follows: powder precursor material, either pure or carbon-containing (see above) was mixed with 5 wt% of polyvinylidene fluoride (PVDF) dissolved in N-methyl-2-pyrrolidone. The resulting slurry was diluted with N-methyl-2-pyrrolidone until a consistence of viscous paste, and then doctor-bladed onto F-doped conducting glass (FTO; TEC 15 from Libbey-Owens-Ford $15 \Omega \text{sq}^{-1}$). The

Kavan, 2

Kavan's Results

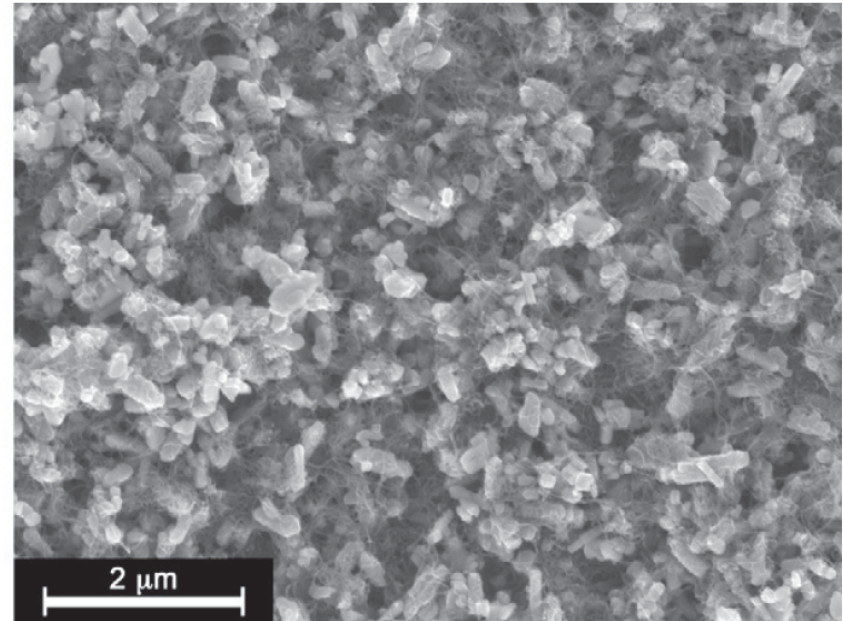
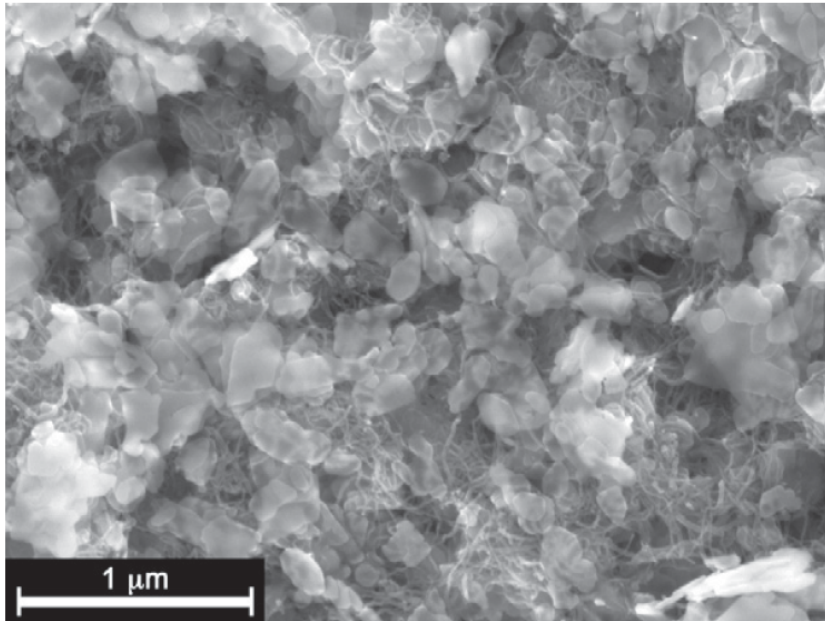
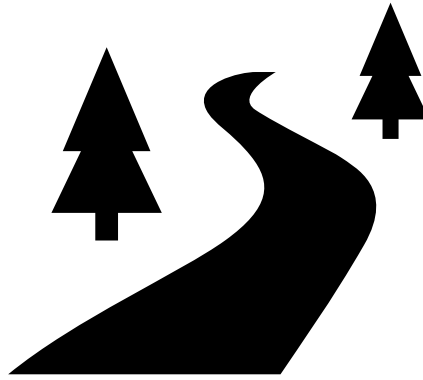


Fig. 3. Scanning electron microscopy images of thin film electrodes from phosphate olivines in a composite with 10 wt% of functionalized nanotubes, MWNT-ox. (Left) Composite with LiMnPO_4 , scale bar 1 μm . (Right) Composite with LiFePO_4 scale bar 2 μm .

the MWNT-bl surface. Obviously, and in contrast to the work of Zeng et al. [15] our data on nanotubes/ TiO_2 (P90) support the idea that oxidative functionalization through HNO_3 is crucial for proper anchoring of nanotubes towards TiO_2 . The second message of Fig. 2 is that industrial powder (P90) is a good source of nanocrystals which spontaneously self-assemble on the nanotube surface. This information is important, because previous studies related to nanoparticles grown by laboratory protocols [2-10,13-17].

Fig. 3 shows the morphological features of our olivine/MWNT-ox composites (10 wt% MWNT-ox). In contrast to the structure of P90/MWNT-ox (Fig. 2) the naked nanotubes are easily recognized between the individual crystals of LiMnPO_4 (Fig. 3, left chart) as well as between LiFePO_4 crystals (Fig. 3, right chart). Two reasons

Kavan, 3-4



**Whether Ohata-Kavan teaches
nanotubes with active material
“attached to their surface”
(all independent claims)**

Ohata-Kava's Masterbatch Process Embeds its ox-CNTs in PVDF resin to form a conductive binder

The Ohata-Kavan combination composition uses the teachings of Ohata as a starting point. LGES-1003, ¶38. For instance, “an electrode material mixture containing the masterbatch and an electrode active material” is blended with “a dispersion medium” to form “[a]n electrode material mixture paste.” LGES-1004, [0023]. The masterbatch contains carbon nanotubes dispersed in a resin, where the aspect ratio of the nanotubes is between 100 and 1000, as taught by Ohata. *See id.*, [0015], [0020].

Based on the teachings of Kavan, the carbon nanotubes used in the masterbatch of the combination are oxidized, as discussed in the next paragraph.
See LGES-1005, Abstract, 2, 4; LGES-1003, ¶39. Also based on the teachings of

’483 Pet. 18

Ground-Dispositive Issue

Whether the ox-CNTs of Ohata-Kavan's electrode paste would be fully coated by resin.

If the answer is **YES**, Petitioner does not dispute that it cannot prevail on the Ohata-Kavan grounds.

MRD-2013, 3

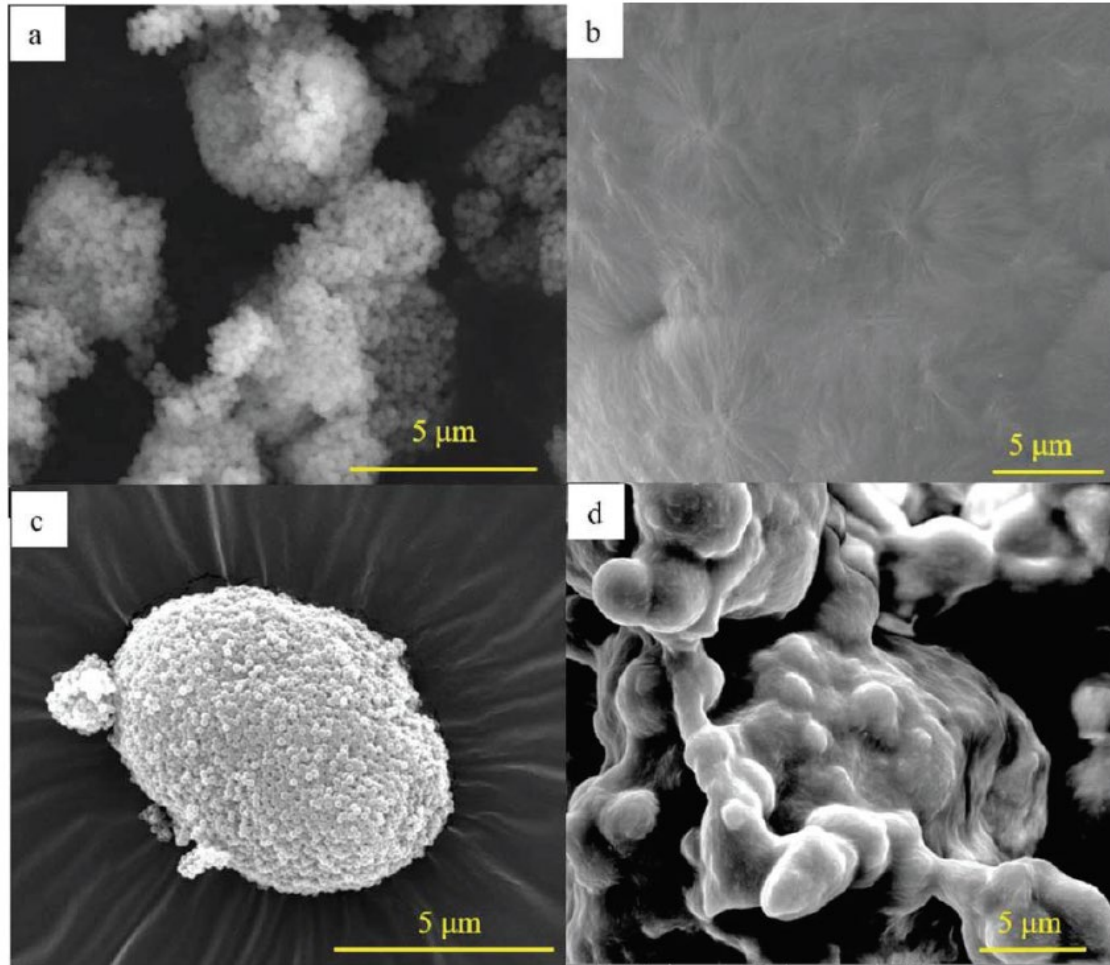
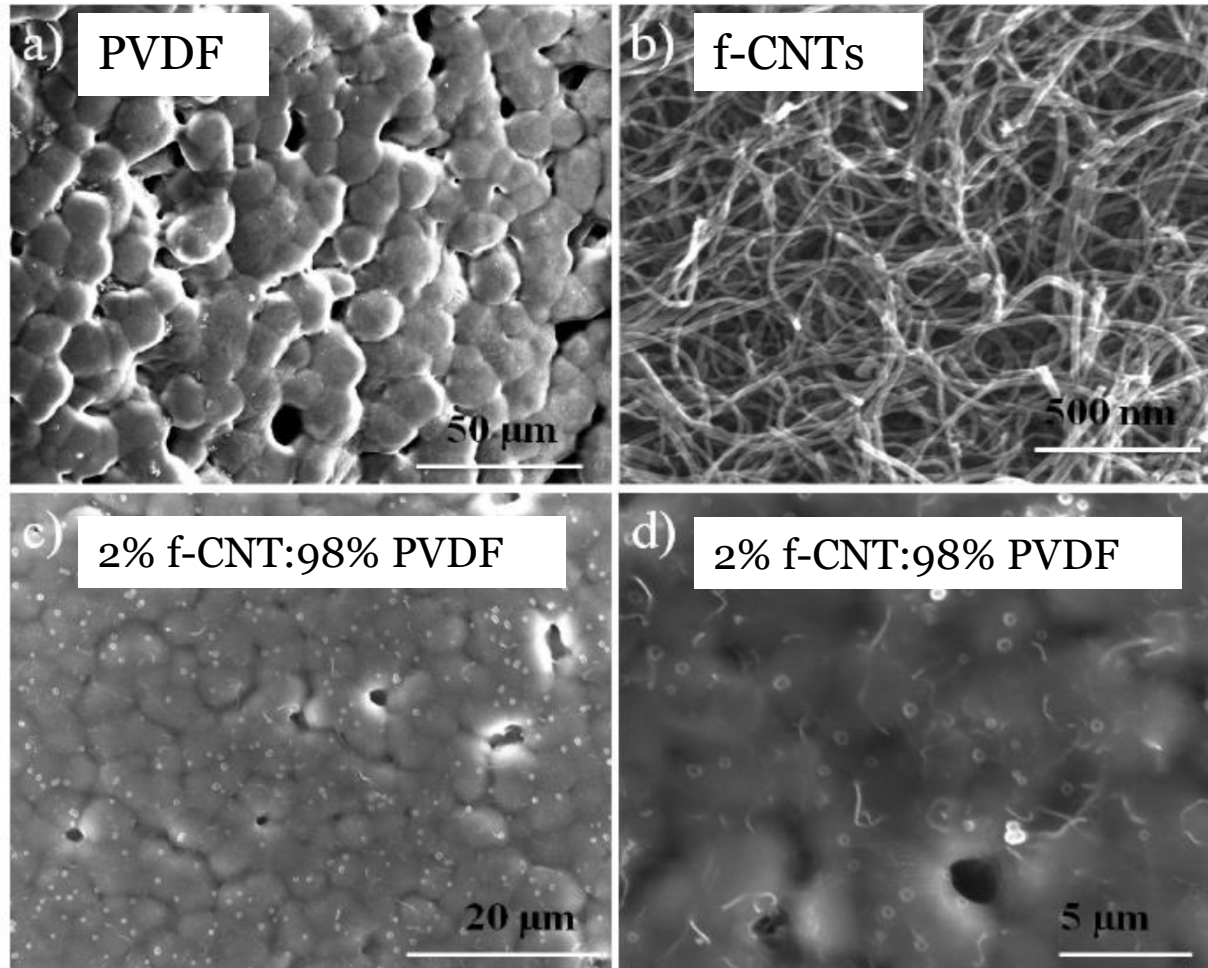


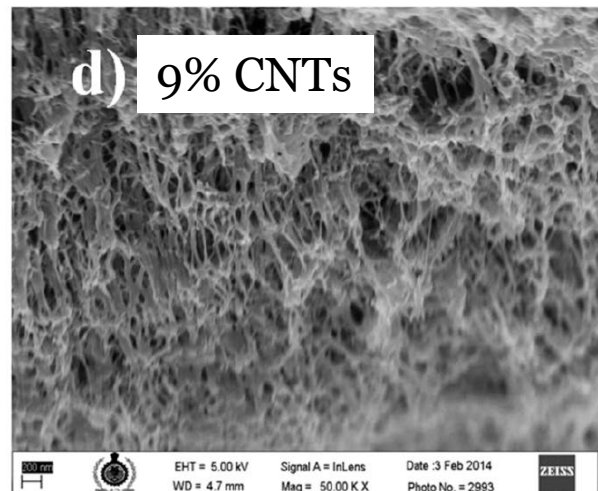
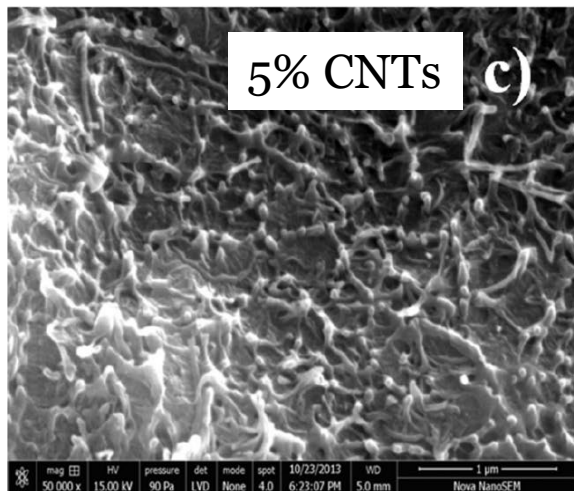
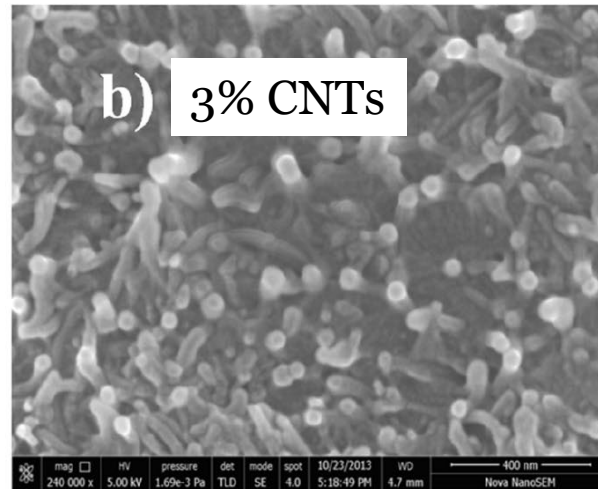
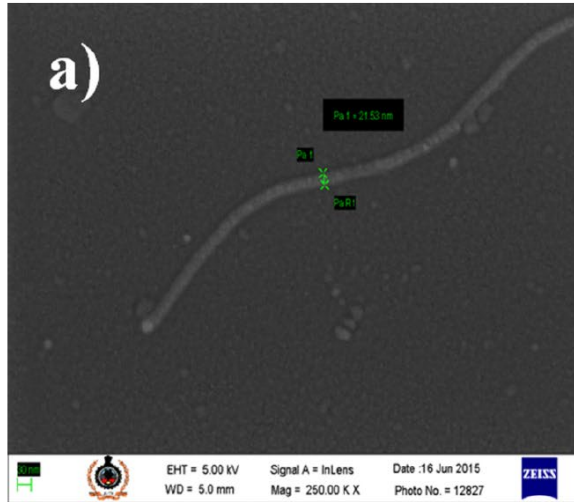
Figure 2. SEM images of PVDF powders before and after the thermal activation process: (a) and (b) are the Alfa Aesar; (c) and (d) are the HSV 900.

MRD-2010, 11-12, 15



“microspheres of polymer have been formed throughout the composites and carbon nanotubes are homogeneously dispersed within the polymer 3D scaffold”

MRD-2011, 3



“FIGURE 1 SHOWS THE FIELD-EMISSION SEM MICROGRAPHS OF THE MWCNTS IN PVDF MATRIX.”

The “strong interaction” between PVDF and CNT facilitates stronger bonding

MRD-2009, 2

In a CNT–PVDF–NMP suspension, PVDF molecular chains would coat onto CNT surface due to the strong interaction between PVDF and CNTs.^[23]

LGES-1014, 2

“The TEM micrographs (Fig. 3) of functionalised nanotubes ... shows **CNTs that are completely covered with matrix.**”

The Experts' Testimony

Dr. Chung's Testimony

115. A POSITA would understand that PVDF molecules likewise bond to oxidized CNTs. Specifically, PVDF and oxidized CNTs attract one another due to the dipole-dipole interaction between the highly polar fluorine-carbon covalent bonds of the PVDF molecules and the presence of functional groups (including functional groups with polar covalent bonds) on the surface of the oxidized CNTs.

MRD-2006 ¶115

Dr. Zhu's Testimony

Q When you mix resin with carbon nanotubes, would you agree that the resin covers the carbon nanotube surface?

A I would agree the resin cover significant amount of the surface of the carbon nanotube.

MRD-2024, 66:5-9

Ohata's "Dispersion Medium" Does Not Eliminate the PVDF Coating.

The Ohata-Kavan combination composition uses the teachings of Ohata as a starting point. LGES-1003, ¶38. For instance, "an electrode material mixture containing the masterbatch and an electrode active material" is blended with "a dispersion medium" to form "[a]n electrode material mixture paste." LGES-1004,

'483 Pet. 18

[0024] The amount of the carbon nanotubes contained in the electrode material mixture is, for example, 0.2 to 3 parts by weight per 100 parts by weight of the electrode active material. In order to obtain a high-capacity electrode, the amount thereof is preferably not greater than 0.8 parts by weight. The dispersion medium should be selected according to the resin to be used. When polyvinylidene fluoride or a vinylidene fluoride-hexafluoropropylene copolymer is used, for example, N-methyl-2-pyrrolidone can be used.

Ohata

Petitioner misrelies on Ohata's Reference to "easily dissolved" PVDF

[0032] A positive electrode material mixture paste was prepared by mixing 3.3 parts by weight of the resultant masterbatch, 100 parts by weight of lithium cobalt oxide (LiCoO_2) as an active material and 45 parts by weight of N-methyl-2-pyrrolidone with a mixer. The masterbatch was easily dissolved in N-methyl-2-pyrrolidone (60°C.). This positive electrode material mixture paste was applied onto both faces of a 20- μm -thick substrate made of aluminum foil, which was then dried and rolled. The electrode plate thus obtained was cut into a predetermined shape to give a positive electrode.

[0023] An electrode material mixture paste can be prepared by blending an electrode material mixture containing the masterbatch and an electrode active material with a dispersion medium. During this process, a resin can be added to the electrode material mixture. The resin to be added may be the same resin as or a resin analogous to the one used during the production of the masterbatch, or a resin other than the above. In order to obtain a high-capacity electrode, the resin to be added is preferably particulate rubber that enables spot bonding between the active material particles.

[0046] A negative electrode material mixture paste was produced by mixing 3.3 parts by weight of masterbatch, the same as the one used in the production of the positive electrode in Example 1, 100 parts by weight of flake graphite as an active material and 56 parts by weight of N-methyl-2-pyrrolidone as a dispersion medium with a mixer. This negative electrode material mixture paste was applied on both faces of a 10-mm-thick substrate made of copper foil, which was then dried and rolled. The electrode plate thus produced was cut into a predetermined shape to give a negative electrode.

MRD-2008, 7:55-62

In one embodiment, the mixing of the binder, and then adding the active material, rather than simultaneously mixing all ingredients has been found to improve the electrode interface. The detailed steps follow below.

Initially, acetylene black is mixed with NMP and PVDF at room temperature to form a macroscopically homogeneous slurry. Afterwards, active material is added, and the mixture again mixed until a macroscopically homogeneous slurry is again formed. It is understood that, although the slurry is macroscopically homogeneous, at the microscopic scale, the mixture is substantially nonhomogeneous. In this form the binder has previously and preferentially bonded to the CB.

MRD-2014, 8

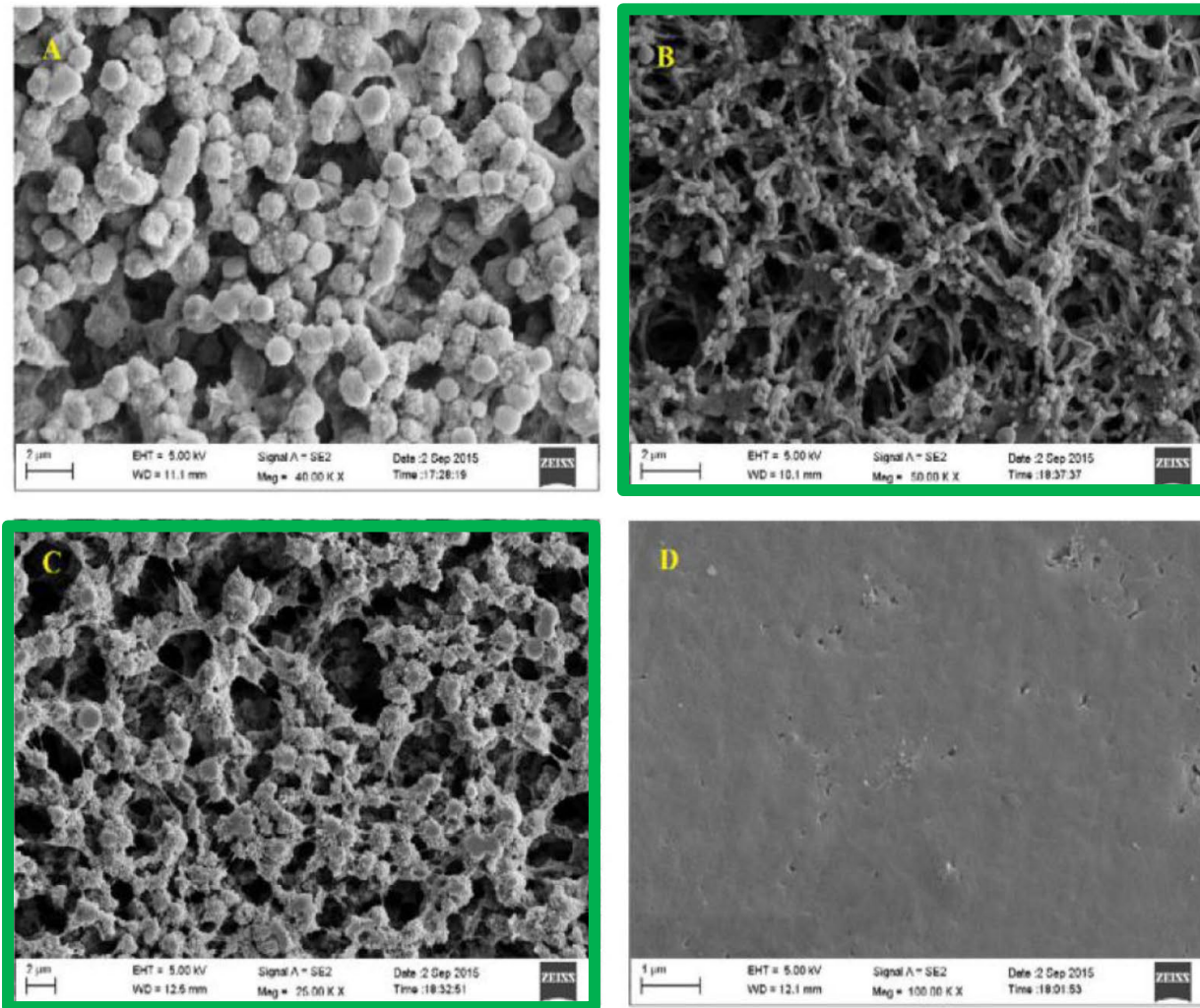


Fig. 2. (A–D): FESEM image of (A) PVDF, (B) PVDF with CNT, (C) PVDF with carbonic acid functionalised CNT and (D) PVDF with sulphonic acid functionalised CNT.

MRD-2014, 8 (cont.)

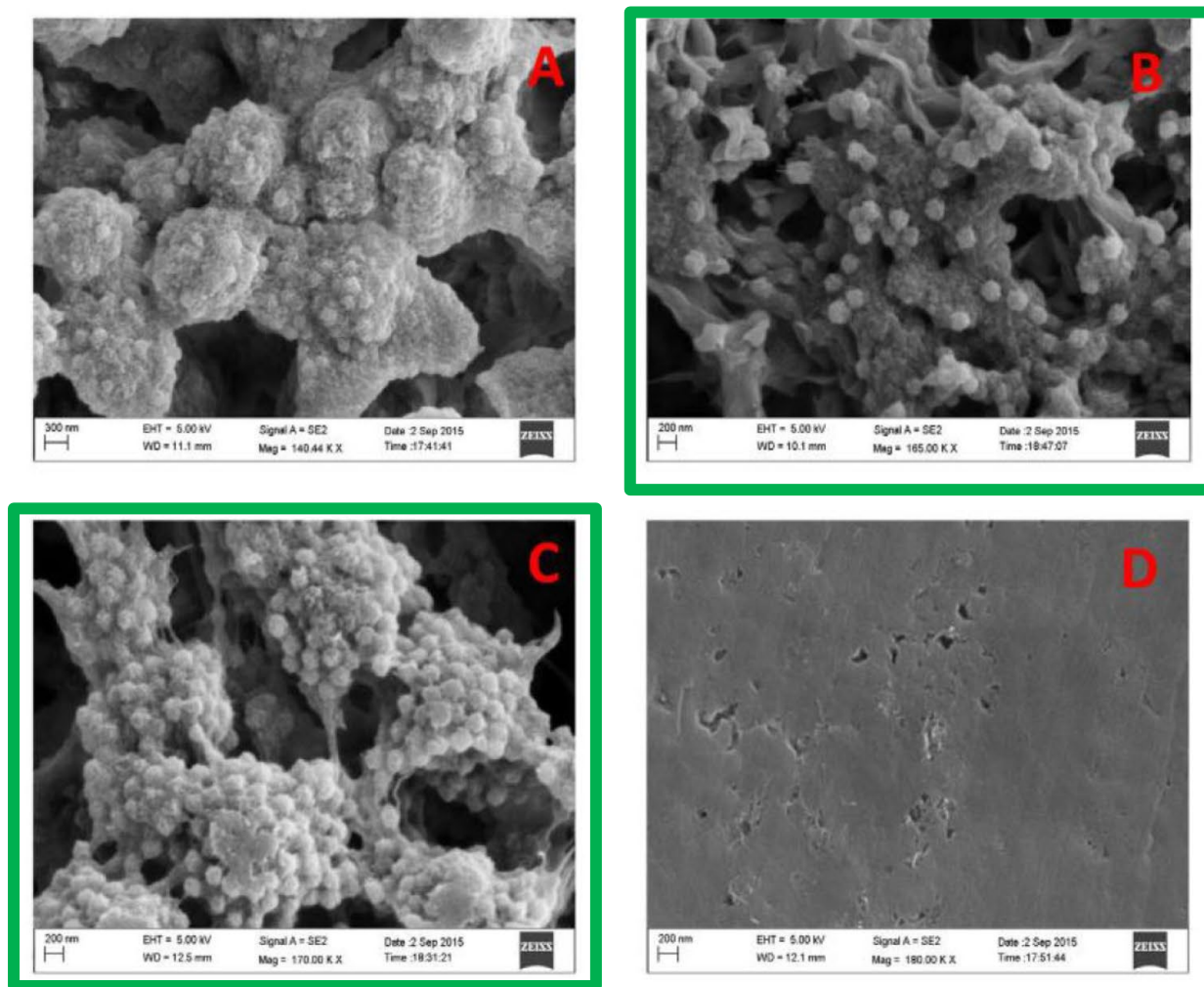


Fig. 3. (A–D) High Magnification FESEM image of (A) PVDF, (B) PVDF with CNT, (C) PVDF with carbonic acid functionalised CNT and (D) PVDF with sulphonic acid functionalised CNT.

MRD-2011, 3 Shows that Solvent Does Not Remove PVDF Coating From CNTs

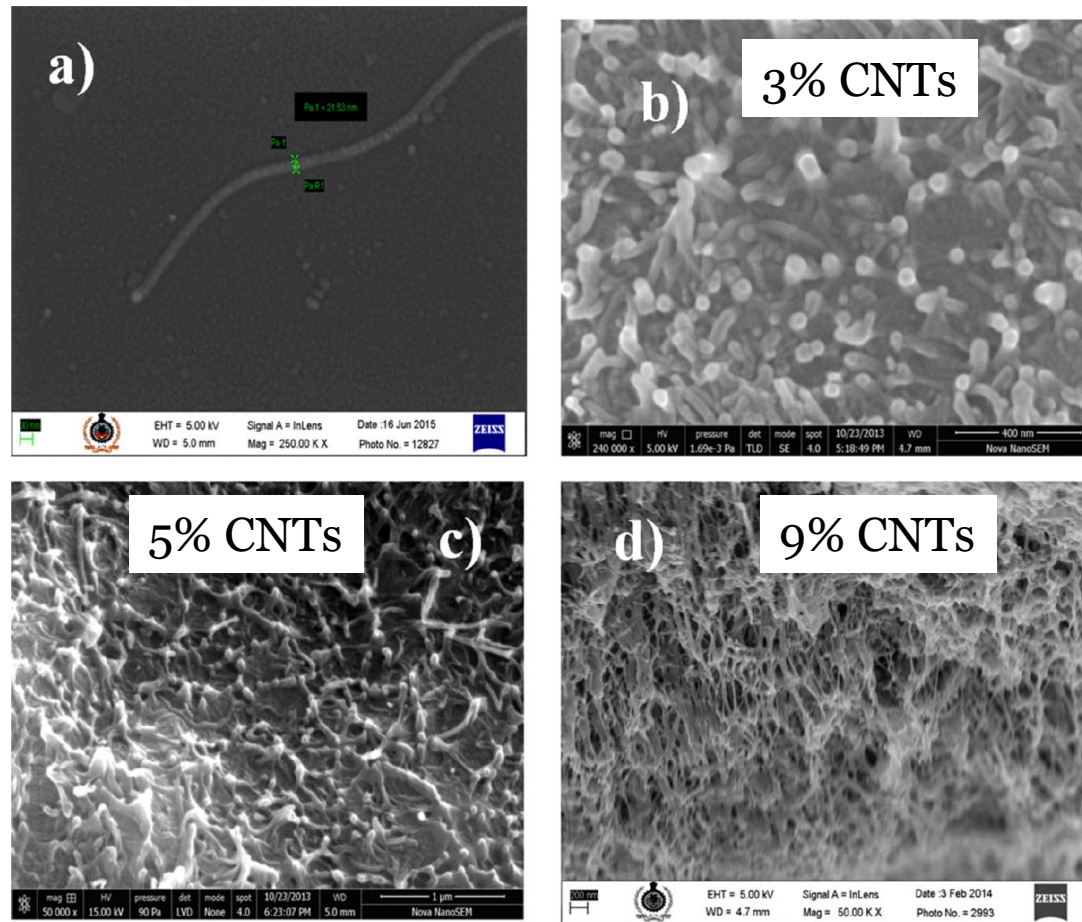


FIG. 1. FE-SEM images of MWCNT in the PVDF matrix. (a) shows a single MWCNT, and images of well dispersed MWCNTs for 3wt%, 5wt% and 9wt% samples in the PVDF matrix are shown in b), c) and d) respectively.

“FIGURE 1 SHOWS THE FIELD-EMISSION SEM MICROGRAPHS OF THE MWCNTS IN PVDF MATRIX.”

MRD-2020, 6-7

Teaches that mixing the carbon black with the binder results in a “gel formation” and “**PVDF polymer layer**” that “**prevents direct contact** of particle surfaces.”

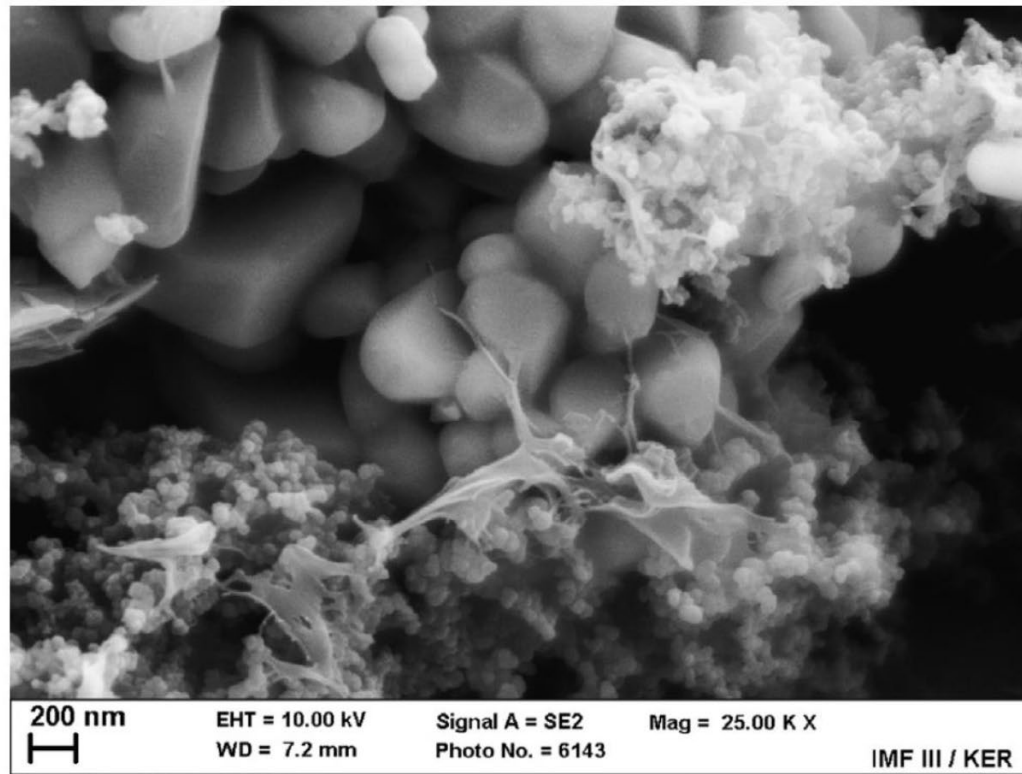
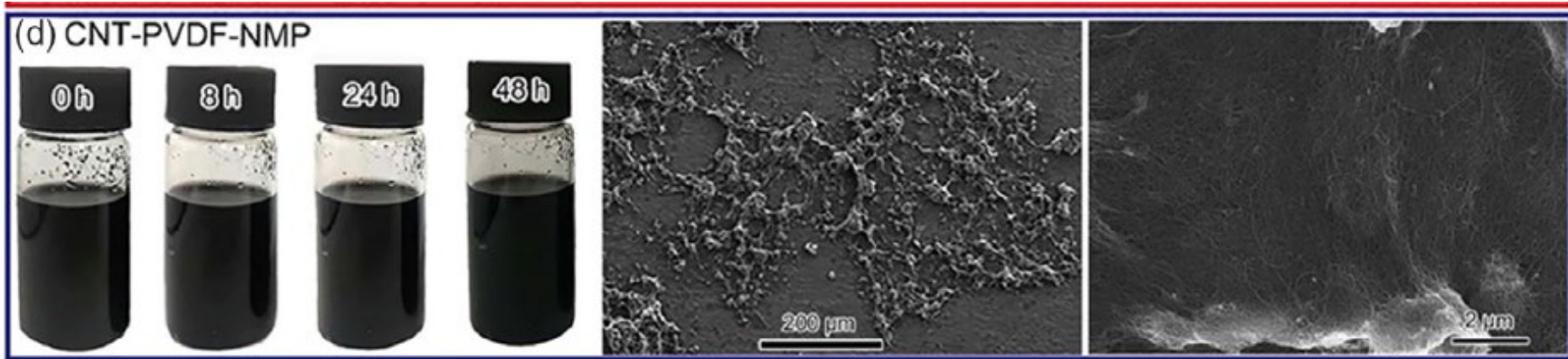


Fig. 13. SEM micrograph of a NMC cathode particles with adhering PVDF/carbon black clusters.

MRD-2009, 2



In a CNT–PVDF–NMP suspension, PVDF molecular chains would coat onto CNT surface due to the strong interaction between PVDF and CNTs.^[23]

Dr. Zhu's Testimony

8 Let me go back to that sentence. It
9 says, "5wt% of PVDF was dissolved in NMP."

10 A Yes.

11 Q That doesn't mean that the PVDF is
12 removed from the mixture, correct?

13 A No. It's not -- it doesn't mean the
14 PVDF was removed from -- from one mixture. So which mixture
15 are you referring to?

16 Q Well, it says, "The electrodes were
17 prepared as follows: Powder precursor material, either pure
18 carbon-containing (see above) was mixed with 5wt% of PVDF
19 dissolved in NMP."

20 Do you see that?

21 A Yes. So no, it was not removed from the
22 mixture. It was still inside the mixture. The mixture

MRD-2024, 41:5-22

Dr. Zhu's Unsubstantiated Testimony is Entitled to No Weight

14. The dissolution of PVDF usually takes some time, a POSITA would have understood the dissolution of the polymer is necessary step and may require some time to dissolve it. If not given enough time and not using the right approach, PVDF may not be completely dissolved and will lead to the aggregated undissolved particles in NMP solution. This is a strict condition that a POSITA would try to avoid. Therefore, the dissolution of PVDF in the solvent, followed by mixing with insoluble electrode active materials (cathode/anode) to form a slurry, is a general procedure and an established practice in the battery manufacturing industry. There are also dedicated procedures developed specifically for dissolving PVDF in NMP.

LGES-1056 ('483) ¶14

Dr. Zhu's Testimony On Mixing Orders

6 Q But can water be used as a binder?

7 A No. Water is not a binder material.

8 But as I said, the final -- the final slurry prepared will
9 have the content including active material, binder, carbon
10 fillers and dispersed medium. It's not necessary to mix in
11 a specific order. And this is where understood by other
12 people who do mixing. It's also where understood by
13 battery researchers who prepare the electrode slurry. So
14 you can have four components, A, B, C, D, right, and you can
15 mix A, B first and then add C, D, and eventually you have A,
16 B, C, D together. Or you can mix A and C first, and then
17 you add B and D, and then you will have eventually A, B, C,
18 D together to cause the slurry.

MRD-2024, 50:6-51:4

“Physical Contact” Contact is Not Required: Electron Tunneling

MRD-1010, 12: “electrons can easily travel from one tube to another tube without any obstacle (through tunneling)” when the “CNTs are touching each other or very close ($< 2\text{nm}$ [sic] in distance).”

MRD-1012, 5: describing “tunneling of electrons” as a “conduction mechanism” in PVDF-CNT

Dr. Chung (LGES-1055, 114:19-115:20): “for nanotube A and nanotube B to be in *electrical* contact, they have to be *extremely close* to one another.... I just mean *not exactly contacting*, just ... a tiny little microscopic *gap* at the point of so-called touching.”

“Physical Contact” Contact is Not Required: Percolation Threshold

MRD-2011, 7-8: “The metal-like negative permittivity behavior is also due to the percolation phenomenon with increasing conducting MWCNT content. Now the conductive component dominates the dielectric properties of the whole MWCNT-incorporated-PVDF matrix. In this case, the dielectric properties of composites transform from insulator-like to metal-like behavior.”

MRD-2014, 4: “The critical content of CNTs that characterizes a drastic increase in conductivity is commonly termed as the electrical percolation threshold (EPT).”

Dr. Chung (LGES-1054, 38:9-25): Testified that the CNT-PVDF masterbatch binder of Ohata-Kavan has to be viewed as “one unit” because it “is a composite material with the resin as the matrix and the nanotubes as a filler” such that the “electrical conductivity [of the composite] is much increased.”

The Percolation Threshold is Achieved at Ohata's Lowest CNT Percentage (5%)

MRD-2011, 7

material is still insulating. The permittivity and dielectric values are still low. However, at 5wt% loading, the sample approaches the percolation threshold, obtaining optimum conductivity and the loss parameter (obtained from $\tan \delta$) becomes >1 . Beyond the threshold, i.e. for 9 wt% sample,

MRD-2012, 5

Fig. 4 shows the AC electrical conductivity of the composites as a function of frequency. The electrical conductivity of insulating PVDF has increased to $1.7 \times 10^{-5} \text{ S cm}^{-1}$ with the addition of 3 wt% CNTs as observed from Fig. 4. The addition of $\text{Ni}_{40}\text{Co}_{60}$

Gan ¶[0027]

utilized. As illustrated herein, significant increases in the electrical conductivity of conductive polymer materials can be achieved utilizing functionalized CNTs in amounts of 5 wt% or less and even 1 wt % or less. Generally, the amount of

Dr. Chung Explicitly Testified Physical Contact is not Required

6 Q. Okay. And this network of nanotubes would
7 also need to make contact with the active particle
8 itself, right?

9 MR. ZINDA: Objection. Form.

10 THE WITNESS: No, because we had -- Ohata is
11 just trying to develop a more conductive form of binder.
12 Okay? And the nanotubes are inside this resin that
13 constitutes the binder. So the nanotubes are inside
14 the -- they are covered by the resin.

15 In spite of that, the nanotubes, if they form
16 the percolative network, is able to enhance the
17 conductivity of this binder, this lump of binder. So
18 the nanotubes would not be touching the active particle
19 because they are wrapped up -- I mean, embedded inside
20 this resin.

LGES-1054, 37:6-20

On Cross-Examination, Dr. Zhu Acknowledged Electron Tunnelling

8 Q. (BY MR. ZINDA) Have you ever heard of a
9 concept known as electron tunneling?

10 A. Yes.

11 Q. What is electron tunneling, in your opinion?

12 A. Electron tunneling is a microscopic phenomenon
13 and, typically, the electron can transport from one
14 place to another place to create empty spaces. A lot of
15 times it's vacuum and without any conducting materials
16 between them.

MRD-2043, 14:8-16

On Cross-Examination, Dr. Zhu Acknowledged the Percolation Threshold

Q Can you please provide me your understanding of what the percolation threshold is?

A Yeah. As I said yesterday, the percolation threshold is a percentage of a filler in composite materials, and usually there is a percentage -- threshold percentage is volume-based percentage. So if you have a filler which reach critical volume percentage in composite and then the filler can see it as a -- basically, a form of connected network, and that this usually called percolation threshold.

Q Correct. The composite -- yes. Does a -- does a percolation threshold allow a normally nonconductive composite material to act as a conductive material?

A If the percolation threshold is reached, in this context, the filler is a conductive filler and, as a matrix material, is nonconductive materials. And if you have enough filler in this matrix material and the filler is more than percentage, is above the threshold of percolation threshold, the -- then the composite, which is the conductive filler, and the nonconductive matrix materials will become effectively conductive medium as a whole.

MRD-2044, 8:15-9:18

Dr. Zhu's Belated Redirect Testimony is Unsubstantiated and Unexplained

Unsupported Expert "Say So"

Q Okay. So -- so that -- in the context of electrode materials of lithium-ion batteries, would you expect such tunneling effects to occur?

MR. ZINDA: Objection, form.

A If we consider a lithium battery, this is typically microscopic device, and I don't think the tunneling effect actually play any part in the role of these microscopic devices.

MRD-2044, 13:16-22

Petitioner Conflates “Electrical” Connections with “Physical” Connections

It is well-established that in battery electrode material, there must be electrical contact between the active material and the conductive additive, to enable current flow through the electrode. *See* LGES-1056, ¶10 (citing MRD-2017, [0094]; MRD-2018, [0092]).

’483 Reply, 6

Dr. Zhu's Testimony on Active Material Conductivity is Unsubstantiated

His Declaration Testimony

10. It is well-established that in a battery electrode material, there must be electrical contact between the active material and the conductive additive, due to the relatively low intrinsic electrical conductivity of the active material itself, to enable current flow through the electrode. MRD-2017,

LGES-1056 ('483) ¶ 10

His Cross-Examination Testimony

A. So are you referring to active material used in the lithium-ion battery?

Q. (BY MR. ZINDA) Correct.

A. So the intrinsic conductivity of acting materials in the lithium-ion battery, depending on what electrode you are referring to, some of them have good intrinsic conductivity, for example, the graphite materials, and some materials does not have good intrinsic electronic conductivity, such as some oxide materials. So this question is -- it's depending on which exactly electrode you are referring to.

MRD-2043, 15:12-22

Petitioner's Position is Inconsistent with its Other Invalidity Theory

Moreover, although no scale is provided for the Fig. 1(b) schematic illustration, a POSITA would have been able to estimate the scale based on Ohata's disclosure that the nanotubes "have an average length of 1 to 100 μm ." LGES-1004, [0019]; LGES-1003, ¶130. For instance, for nanotubes having a length of 100 μm , the space between the nanotubes is depicted as greater than 1 μm , e.g., the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**. LGES-1003, ¶130.

'483 Pet., 58

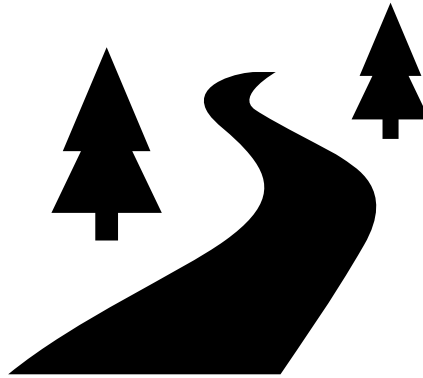
MRD-2008 Does Not Support Petitioner's "Physical Contact" Theory

Interpreting FIG. 1, at a discharge rate of about 20 C, the battery with an interface composition of CB:PVDF of 1:1 has capacity reduced to about 0.002 of the original capacity (a unitless 1). The battery with the CB:PVDF of 0.8:1 has capacity reduced to about 0.08 of the original capacity. Finally, the battery with the CB:PVDF of 0.5:1 has capacity reduced to about 0.2 of the original capacity. Thus, at the 20 C discharge rate, the compositional ratio of CB:PVDF of 0.5:1 appears to have about 100× the capacity of the battery with an interface composition of CB:PVDF of 1:1, illustrating the unexpected improvement in performance realized when using greater amounts of binder relative to CB.

It is believed that this behavior may be attributed to the improved interface electronic conductivity between the active material particles and the acetylene black conductive additives as shown in the impedance study of these electrodes. The interface impedance decreased dramatically with increasing amount of PVDF binder.

Conventional thinking is that when the ratio of non conductive binder is increased, at a given acetylene black concentration, the interface impedance will increase due to the lithium ion blocking effect from the binder. The findings shown here indicate the opposite of conventional thinking. Better integration of the interface between the carbon glue and the active material appears to significantly improve the kinetics of the lithium ion transport at the interface, therefore improving the battery performance at higher discharge rates.

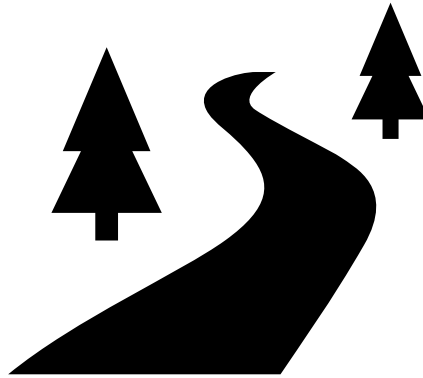
MRD-2008, 10:15-41



**Whether there was a motivation to
oxidize at 1-15% based on Ohata-Kavan
(all independent claims)**

Petitioner's Ohata-Kavan Motivations Fail

- A POSITA had no motivation to achieve the claimed invention in light of Ohata's unique "masterbatch" process.
- Petitioner's "dispersion" motivation is inconsistent with its assertion that Ohata's masterbatch already achieves "uniform dispersion."
- Petitioner's "purification" motivation is undermined by Kavan's teaching that its CNTs are *already* purified before oxidation.



**Whether Ohata-Kavan teaches
“discrete” carbon nanotubes
(all independent claims)**

Petitioner Does Not Dispute that it Fails to Carry its Burden under MRD's Proposed Construction

B. The nanotubes in the Ohata-Kavan composition are discrete

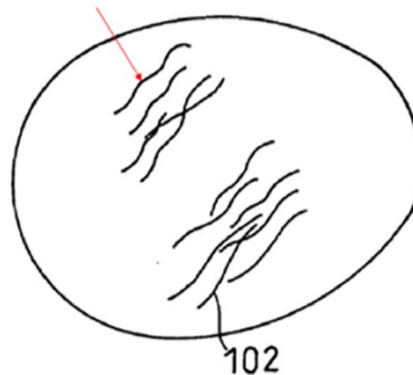
PO argues that the Ohata-Kavan composition would not include discrete nanotubes, but this argument relies on an overly restrictive construction for “discrete.” The term “discrete” does not preclude a network of nanotubes, *e.g.*, in point-to-point contact, providing a conductive pathway through the electrode material, as is the case in the Ohata-Kavan.

'483 Reply, 13-14

Petitioner Cannot Carry its Burden Even Under its Broader Construction

A POSITA would have recognized and/or otherwise found it obvious that—in view of Ohata’s description of its nanotubes as being “unraveled and straightened carbon nanotubes” and generally “dispersed” as individual nanotubes in the resin, and further in view of its illustrative schematic (below)—Ohata’s nanotubes are discrete, non-agglomerated, and exfoliated. LGES-1003, ¶83. Indeed, a POSITA would understand a carbon nanotube that is, as shown below, fully isolated from others to be discrete, non-agglomerated, and exfoliated. *Id.*

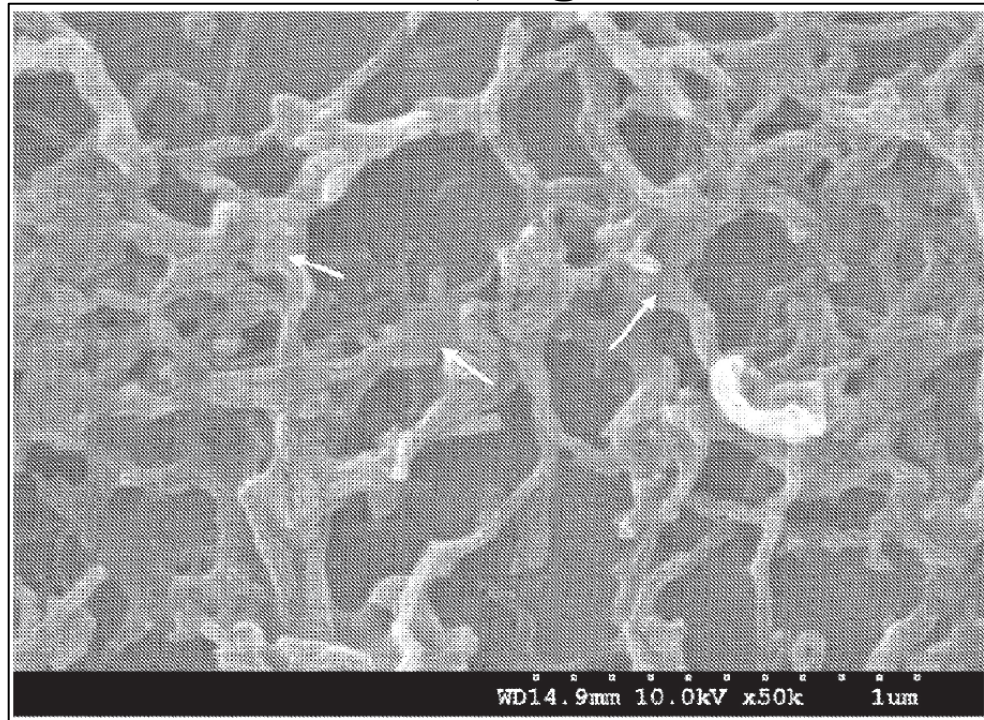
“discrete, non-agglomerated, and exfoliated carbon nanotubes”



LGES-1004, Fig. 1(b) (annotated)

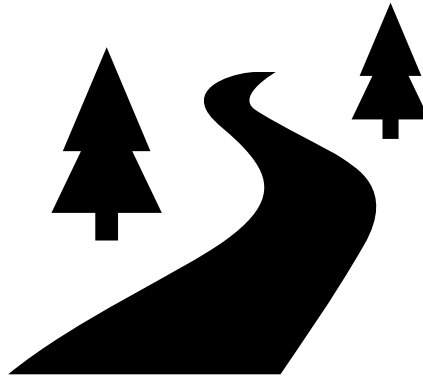
The Oxidized CNTs in the Masterbatch Would Be “Drawn Closer Together”

Gan, Fig. 5



Gan explains that functionalization of CNTs ...

[0020] 2. Reduces contact resistance between CNTs incorporated into the conductive polymer material by drawing the CNTs closer together; and



**Whether Ohata-Kavan teaches
discrete CNTs “uniformly dispersed
without clustering on a dimension scale
less than 1 micrometer”
(’909, Cl. 6 / ’483, Cl. 8)**

Claims 6 ('909 Patent) and 8 ('483 Patent)

6. The composition of claim 1, 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.

“uniformly dispersed”

In Ohata’s masterbatch process, “a conductive material” is *uniformly predispersed* in a binder.” LGES-1004, [0002]. In the combination, this “conductive material” is the oxidized carbon nanotubes, and via Ohata’s masterbatch process, the nanotubes are “*uniformly predispersed*” in the resin. *Id.*, [0002]; *see* [0005], [0015]; LGES-1003, ¶116. This masterbatch, with “uniformly predispersed” nanotubes, is then mixed with an electrode active material and a dispersion medium to form the combination paste. LGES-1004, [0002]; *see* [0023]; LGES-1003, ¶116.

In the combination, a POSITA would have understood, or at least found obvious, that the nanotubes remain **uniformly dispersed** in the combination paste. LGES-1003, ¶117.

’909 Pet., 33-34

Petitioner Misapprehended Ohata's Teachings

[0002] An electrode of non-aqueous electrolyte secondary batteries as typified by a lithium ion secondary battery and a lithium ion polymer secondary battery is typically produced by mixing an electrode active material with a conductive material and a binder to form a paste, which is then applied onto a current collector made of aluminum, titanium, stainless steel or the like, followed by rolling. A conductive material is used to secure the conductivity of an electrode. If the amount of conductive material is large, however, the battery capacity will be decreased. Accordingly, in order to reduce the amount of conductive material to be used, it is proposed to use a masterbatch process, whereby a **conductive material is uniformly predispersed in a binder**, in the production of a positive electrode (see Japanese Laid-Open Patent No. Hei 10-255844). As a conductive material, **carbon black such as furnace black or acetylene black**, and powdered graphite are used.

[0003] The primary particles of **carbon black** or powdered graphite are linked into a chain to form a conductive path. Such conductive path is likely to be damaged by shearing force. Shearing force is applied to the conductive material during the masterbatch process. For this reason, the use of the masterbatch process to uniformly disperse **the conductive material** in a binder does not yield an electrode superior in conductivity.

Gan undermines Petitioner's Position

In the combination, a POSITA would have understood, or at least found obvious, that the nanotubes remain **uniformly dispersed** in the combination paste. LGES-1003, ¶117.

'909 Pet., 33

Gan (MRD-2019) explains that functionalization of CNTs ...

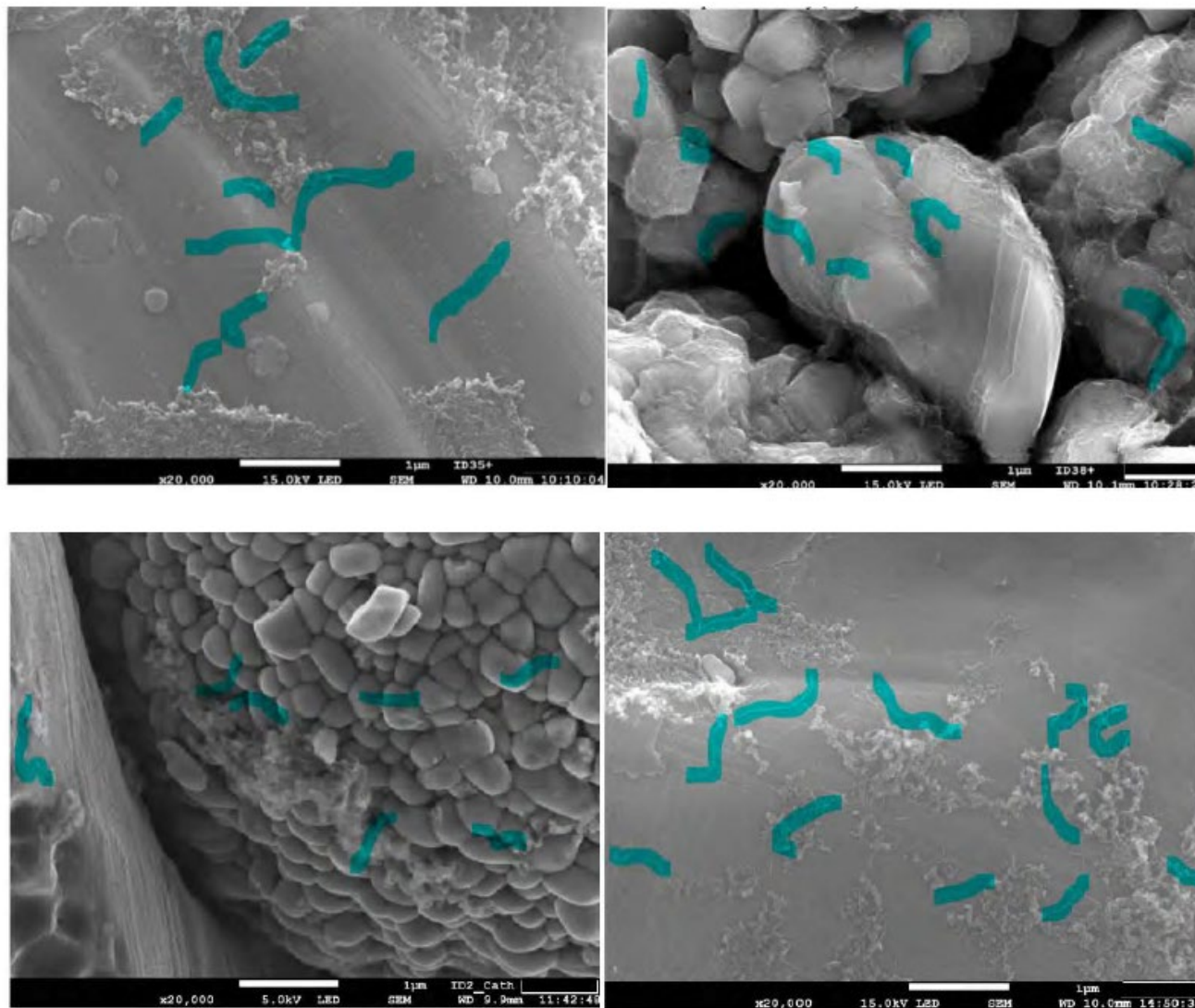
[0020] 2. Reduces contact resistance between CNTs incorporated into the conductive polymer material **by drawing the CNTs closer together;** and

Petitioner's Reply Attempts to Introduce a New Invalidity Theory

However, in Ohata's masterbatch process, carbon nanotubes are the additive (*see* LGES-1004, [0015]-[0016]), and thus, as explained in the Petition, a POSITA would have expected the nanotubes in Ohata's masterbatch to be uniformly dispersed. *See* Petition, 56-58. PO's objection to this position fails for two reasons. First, it overlooks that a masterbatch process is a conventional approach known to "uniformly disperse" an additive in a matrix material. LGES-1004, [0003]; *see* LGES-1063, 7:61-67. Second, its assertions misrepresent Ohata's disclosure. In

'483 Reply, 23

Petitioner Again Mischaracterizes MRD's Infringement Contentions



LGES-1050 ('909), 60, 61, 63, 65

Claims 6 ('909 Patent) and 8 ('483 Patent)

6. The composition of claim 1, 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.

“without clustering on a dimension scale of less than about 1 micrometer”

For instance, for nanotubes having a length of 100 μm , the space between the nanotubes is depicted as greater than 1 μm , *e.g.*, the nanotubes are dispersed without clustering on a dimension scale less than 1 micrometer.

'909 Pet., 35

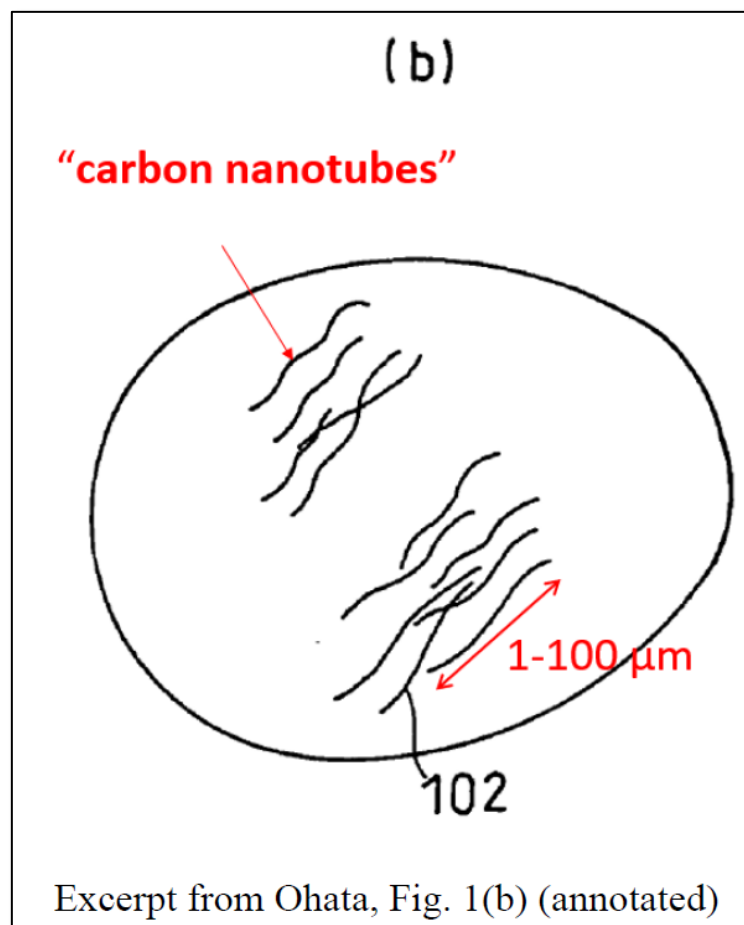
13 Q As a person of ordinary skill in the
14 art, what is your understanding of the meaning of that
15 phrase?
16 A My understanding is -- and also the
17 POSITA's understanding is that is a description of a status
18 that the nanotube is not bundled. So they have significant
19 distance between each other.

MRD-2024, 16:6-19

Petitioner's Entire Invalidity Theory is Based on Ohata's Figure 1 Having "Scale"

Moreover, although no scale is provided for the Fig. 1(b) schematic illustration, a POSITA would have been able to estimate the scale based on Ohata's disclosure that the nanotubes "have an average length of 1 to 100 μm ." LGES-1004, [0019]; LGES-1003, ¶118. For instance, for nanotubes having a length of 100 μm , the space between the nanotubes is depicted as greater than 1 μm , *e.g.*, the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**. LGES-1003, ¶118. Alternatively/additionally, as

'909 Pet., 35



LGES-1003 ('909) ¶[0118]

The “Scale” Theory Fails as a Matter of Law



“[I]t is well established that patent drawings do not define the precise proportions of the elements and may not be relied on to show particular sizes *if the specification is completely silent on the issue.*” See *White v. H.J. Heinz Co.*, 640 F. Appx 930 at 934 (quoting *Hockerson-Halberstadt, Inc. v. Avia Grp. Int’l*, 222 F.3d 951, 956 (Fed. Cir. 2000)); see also *Source Vagabond Sys. v. Hydrapak, Inc.*, 753 F.3d 1291, 1300 (Fed. Cir. 2014) (rejecting argument that the “relative sizes shown in the drawings rebut the Court’s finding” based on Hockerson); *Plantronics, Inc. v. Aliph, Inc.*, 724 F.3d 1343, 1351 (Fed. Cir. 2013) (rejecting argument that figures show “elongated” stabilizer members because specification was completely silent on whether the drawings were intended to be proportional or to scale).

Petitioner Misrepresents *Epstein*

⁶ The '909 Patent provides no substantive discussion of this feature. It is inappropriate to hold the prior art to a stricter standard of disclosure than what is disclosed in the specification of the patent itself. *See In re Epstein*, 32 F.3d 1559, 1568 (Fed. Cir. 1994) (“the Board’s observation that appellant did not provide the type of detail in his specification that he now argues is necessary in prior art references supports the Board’s finding that one skilled in the art would have known how to implement the features of the references and would have concluded that the reference disclosures would have been enabling”).

'909 Pet., 34, n.6

The PTAB has Already Rejected this Misapplication of *Epstein*



Trials and appeals

Google LLC. v. Nobots LLC, IPR2022-00940, Paper 38, 26-27 (P.T.A.B. Nov. 29, 2023) (rejecting petitioner’s argument, based on *Epstein*, that an alleged “lack of any description” in the specification of a claim feature alleviated petitioner of its burden of “showing that a particular claim limitation was ***recognized in the prior art***”).

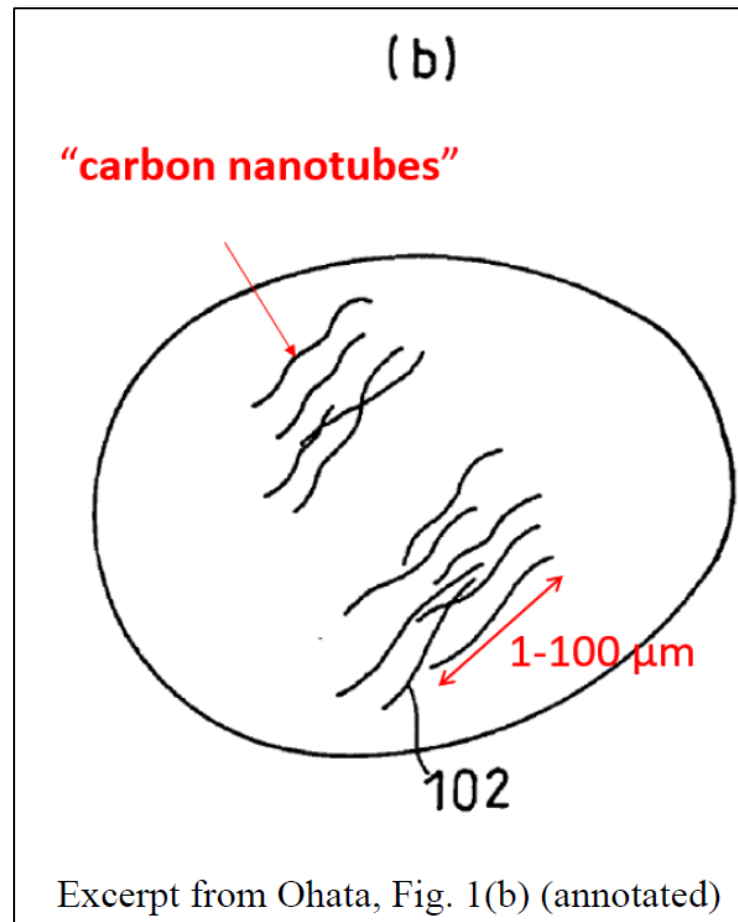
Petitioner's Reply Attempts to Improperly Gap-Fill the Petition

Moreover, PO's objection to the Petition's demonstration that the Ohata-Kavan nanotubes are dispersed "without clustering on a dimension scale less than 1 micrometer" overlooks key aspects of the Petition's application of Ohata to claim 6. *See* POR, 64-65. First, PO fails to address the Petition's explanation that a POSITA would have found this to be obvious given the size range of Ohata's nanotubes and Ohata's explicit disclosure that the nanotubes are "dispersed." *See* Petition, 34-35. And second, PO fails to contend with the role of Ohata's masterbatch process in enabling dispersion of nanotubes, which again, given the size range of Ohata's nanotubes, would have rendered obvious the claimed scale of dispersion. *See id.*; LGES-1056, ¶38.

'909 Reply, 19

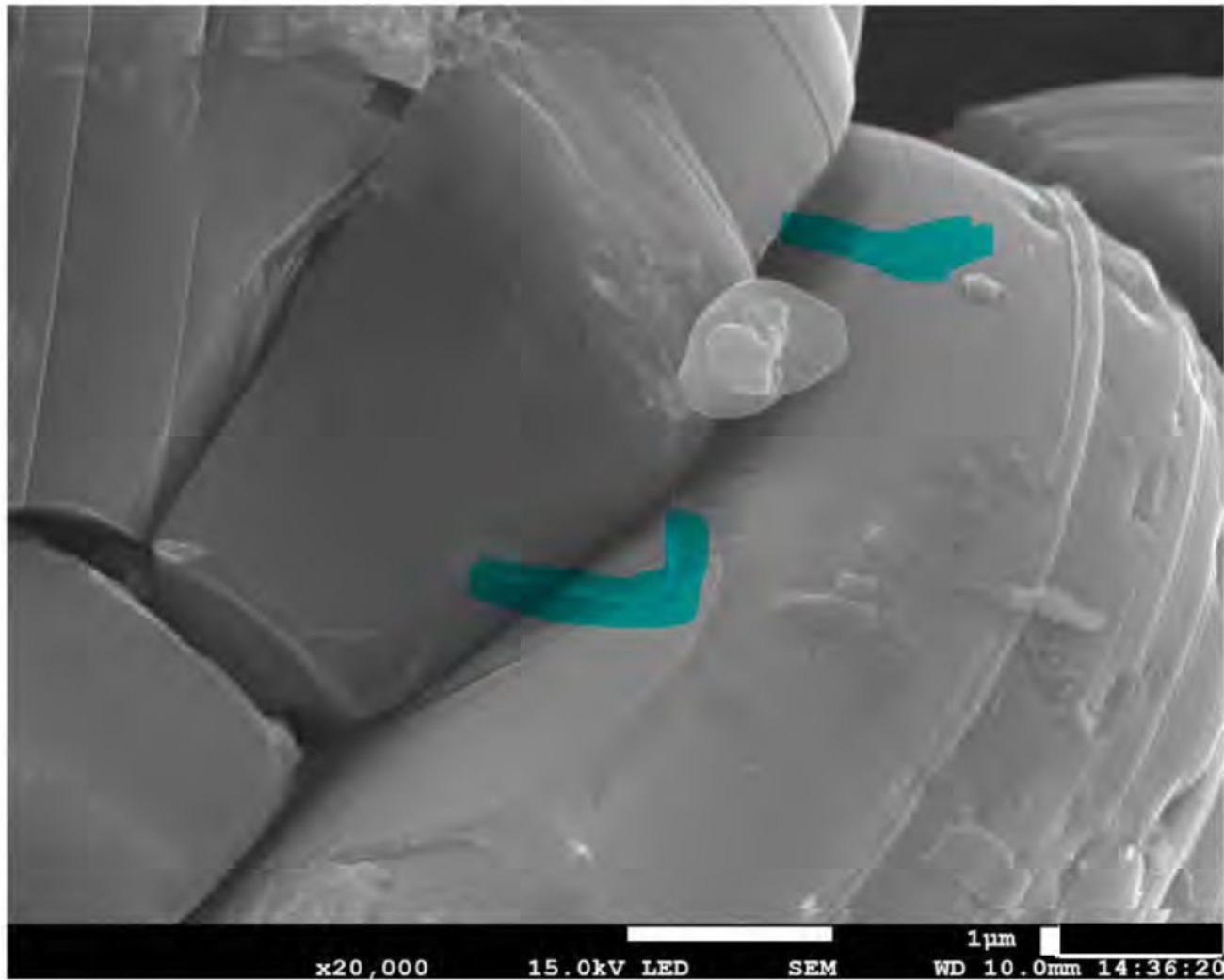
Even If “Scale” Could Be Used—Petitioner Estimated Scale for the Wrong Composition

“Masterbatch” – NOT “Electrode Material Mixture Paste”



LGES-1003 ('909) ¶[0118]

LGES-1050: LGES' Products

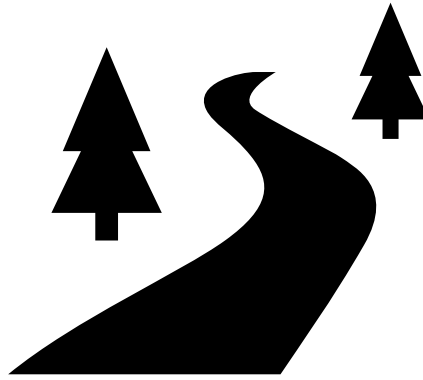


LGES-1050 ('909), 32

Cheng-Gan Grounds

'924 Petition

Ground	Claims	Basis for Rejection
1	1, 4, 5	§103: Ohata and Kavan
2A	1, 4, 5	§103: Cheng and Gan
2B	2, 3	§103: Cheng, Gan, and Zhao



Overview of Cheng (LGES-1007)

Cheng's Goal

SUMMARY

[0010] In view of the problems in the prior art, provided in the present invention is a high performance carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof, which can improve the high-power discharge capacity, cycle life, and compressibility of existing lithium iron phosphate cathode materials by fully utilizing properties of carbon nanotubes. Uniform dispersion of carbon nanotubes in the bulk phase and on surfaces of lithium iron phosphate is achieved by means of an in situ solid phase reaction process, thereby solving the problem in the prior art in which carbon nanotubes are distributed only on the surface of the electrode material and cannot form a continuously connected three-dimensional electrically conductive network structure both in the interior and on the surface of the material, and also solving the problem in which the properties of carbon nanotubes cannot be fully utilized because the carbon nanotubes cannot be fully dispersed.

Cheng ¶[0010]

Cheng's Three-Step Process

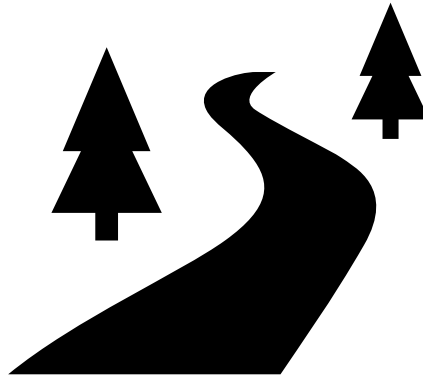
SUMMARY

First, taking unoxidized CNTs, acetylene black, and a dispersing agent and ultrasonically dispersing them in water or an organic solvent to obtain a mixture.

Second, making a precursor powder dispersion by mixing a Li source, an Fe, a phosphate salt, and an organic carbon source into the mixture of step 1, followed by adding a mixing and milling medium and perform mixing ball-milling and drying.

Third, performing an in situ solid phase reaction, specifically, a carbon thermal reduction, by taking the precursor powder dispersion from the second step and *calcining it to temperatures of “600 °C to 850 °C in an inert protective atmosphere for 4 to 48 hours*, pulverizing the burned materials, and then sieving.”

Cheng ¶[[0013]-[0019]

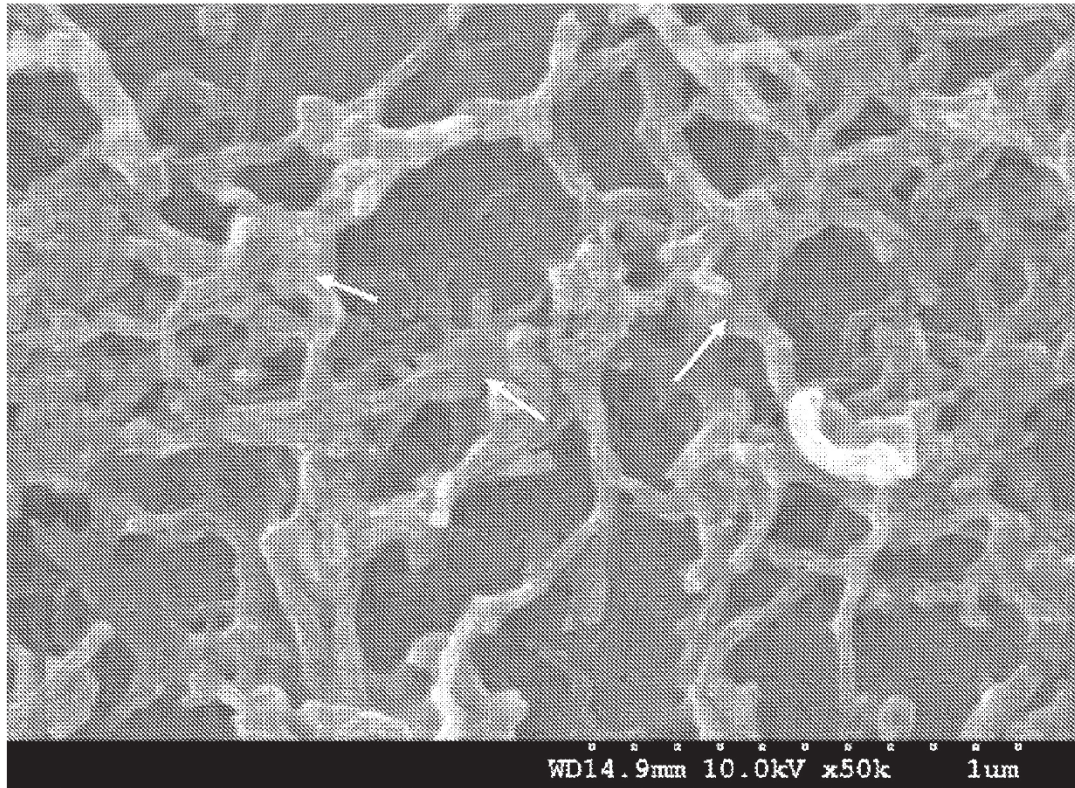


Overview of Gan (LGES-1021)

Gan's Goal

treatment. FIG. 5 illustrates the presence of a random interconnected network of MWNTs and PEDOT:PSS. The arrows in FIG. 5 identify locations where the PEDOT:PSS effectively wraps around and binds with the MWNTs to form a percolated network. A percolated network refers to the network of CNTs and conductive polymer that form a continuous conductive path extending through the composite. The

Gan ¶[[0041]

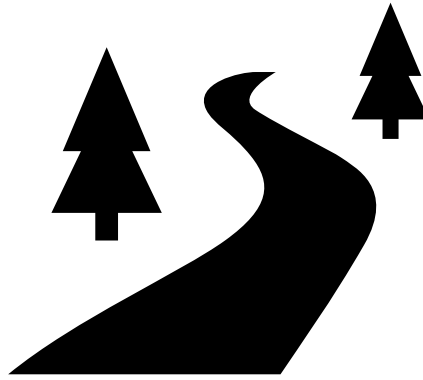


Gan's Functionalization

late (—COOAg) groups. The functionalization of the CNTs achieves at least one or more of the following objectives:

- [0019]** 1. Improves the solubility and dispersibility of the CNTs in water and conductive polymer materials into which the CNTs are to be mixed in accordance with the embodiments described herein;
- [0020]** 2. Reduces contact resistance between CNTs incorporated into the conductive polymer material by drawing the CNTs closer together; and
- [0021]** 3. Provides a conductive bridge between CNTs to reduce the electrical contact resistance therebetween.

Gan ¶¶[0018]-[0021]



**Whether Cheng-Gan teaches
“discrete” carbon nanotubes
(all independent claims)**

Petitioner Does Not Dispute it Loses Under MRD's Proposed Construction of "Discrete"

C. PO's assertion that Cheng-Gan does not achieve "discrete" CNTs is unsupported and contradicts the '924 Patent itself

PO's narrow interpretation of term "discrete" is inconsistent with both the '924 Patent and how a POSITA would understand the term.

'924 Reply, 25

Petitioner Mischaracterizes the Specification

102 mg of multi-wall carbon nanotubes (CNano, grade (-9000) oxidized to about 8 weight percent and aspect ratio about 60 are added to 100 mg copper sulfate, 640 mg sodium EDTA, 15 mg of polyethylene glycol, 568 mg of sodium sulfate and 60 mL of deionized water. The mixture is sonicated for 10 minutes and then heated to 40° C. 3 mL of formaldehyde (37% solution) and 500 mg of sodium hydroxide is added to bring the pH to 12.2. The mixture is stirred for 30 minutes at 85° C. and then filtered using a 5 micron PVDF filter and washed with 200 mL of deionized water. The resultant coated multiwall tubes show a coppery hue color. An electron micrograph shows copper oxide particles of diameter about 50 to 150 nm interconnected by well-dispersed carbon nanotubes.

LGES-1001, 5:15-28

Petitioner Mischaracterizes HOW the MRD Patents “Enhance Conductivity”

MRD’s Patents Prevent and Bridge Active Particle Cracking

200 micrometers. Also, the lithium nanocrystals are not ionically or covalently attached to the surface of the discrete carbon nanotubes to provide for intimate electron transfer and enhanced mechanical strength of the active material. During charging and discharging of conventional lithium ion batteries, the nanocrystals expand and contract. Over a number of cycles, this leads to microcrack formation in the layer of active material and hence to higher internal resistance and decline of the battery performance. With crystals bound to and interconnected with discrete carbon nanotubes the microcrack formation due to severe mechanical vibration, bending, or expansion and contraction will be retarded.

’924 Patent, 1:57-63

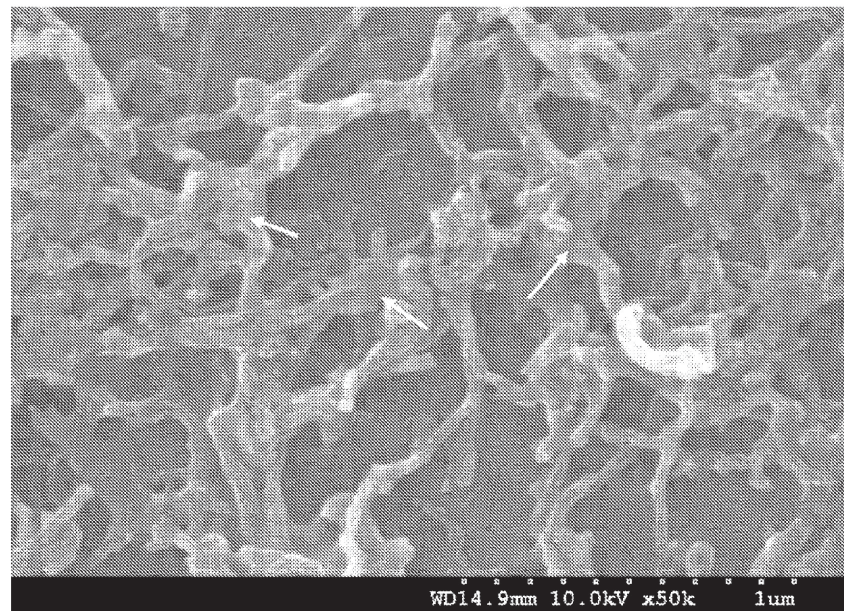
The present invention overcomes the difficulties of low electrical conduction, particularly due to expansion and contraction of the material during charging and discharging of the battery, improved lithium ion transport and mitigation of potentially damaging chemical side reactions by attaching nanosized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

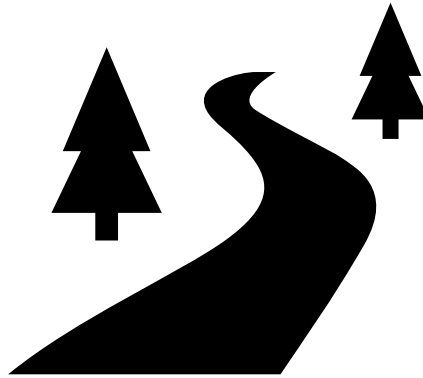
’924 Patent, 2:9-16

Cheng-Gan Enhance Conductivity via a “Percolated” / “Continuous” Network

treatment. FIG. 5 illustrates the presence of a random interconnected network of MWNTs and PEDOT:PSS. The arrows in FIG. 5 identify locations where the PEDOT:PSS effectively wraps around and binds with the MWNTs to form a percolated network. A percolated network refers to the network of CNTs and conductive polymer that form a continuous conductive path extending through the composite. The

Gan ¶[0041]





**Whether Cheng-Gan teaches or renders
obvious “oxidation levels from 1 % to
15%”**

Cheng-Gan's Calcining Conditions

SUMMARY

[0019] (3) In situ solid phase reaction: after the mixed precursor powder is dried in step (2), calcining the same at 600°C to 850°C in an inert protective atmosphere for 4 to 48 hours, pulverizing the burned material, and then sieving the same through a sieve of 150 to 400 meshes, thereby obtaining the carbon nanotube lithium iron phosphate composite cathode material.

Cheng ¶[0019]

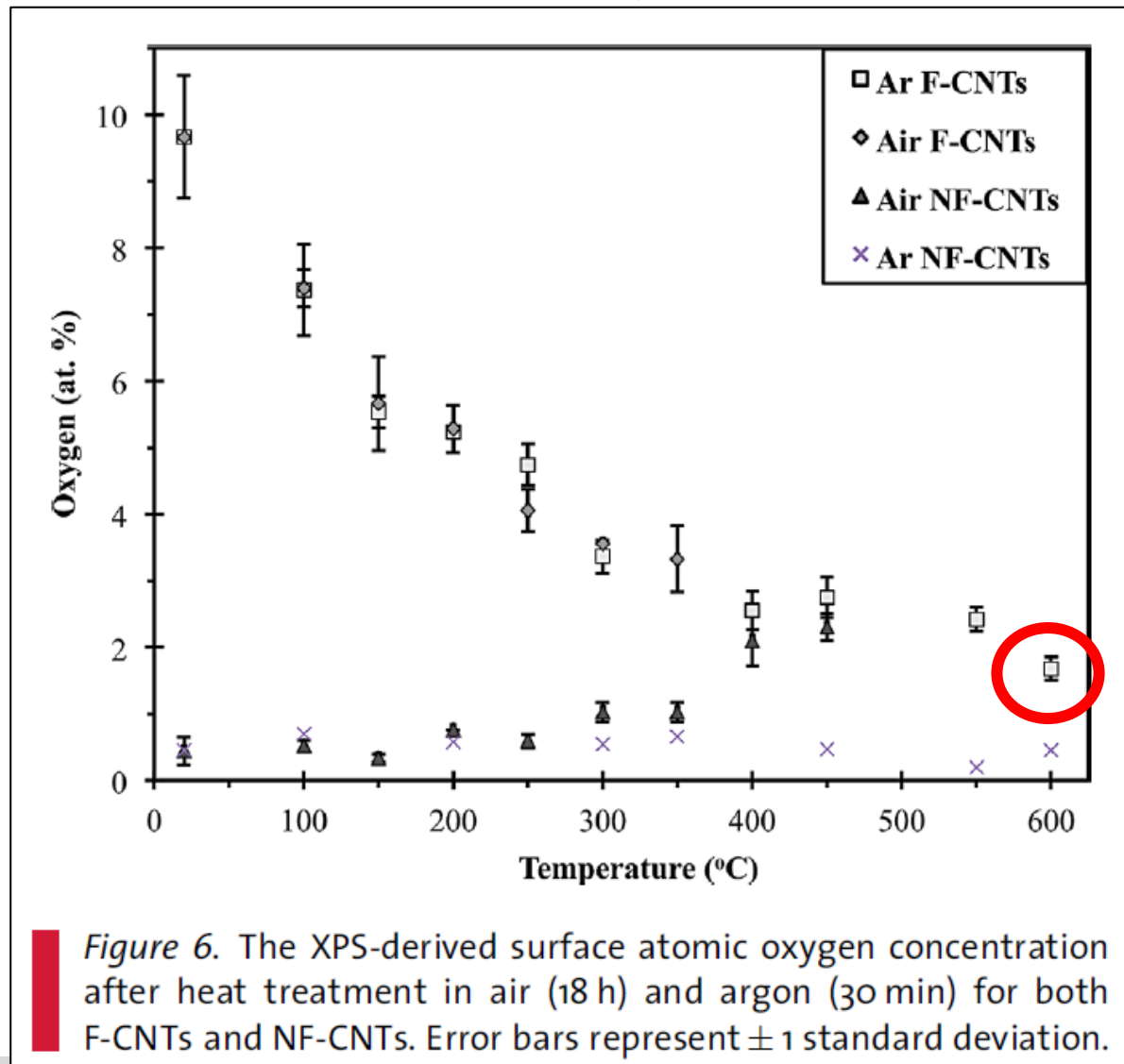
Cheng-Gan's Calcination Conditions Would Eliminate Essentially All ox-Functional Groups

MRD-2029, 4

The TG and DTG curves of CNT-O and CNT-A samples in helium are displayed in Figure 2. Several losses were observed in the TG curve of CNT-O. The first step, until 130 °C, corresponds to the loss of adsorbed water on the hydrophilic surface of oxidized carbon nanotubes [31,32]. The weight loss in the 150–350 °C range, with a maximum in the rate decomposition around 275 °C, can be assigned to the decarboxylation of the carboxylic groups [32]. The removal of the hydroxyl functionalities attached to the walls of carbon nanotubes is known to occur in the 350–550 °C interval [31]. Finally, the weight

Cheng-Gan's Calcination Conditions Would Eliminate Essentially All ox-Functional Groups

MRD-2032, 7



Petitioner Attempts to Improperly Change its Invalidity Theory

2. PO's assertion that Cheng's calcination conditions are limiting is unfounded

PO argues that specific calcination parameters disclosed in Cheng—"600°C+ for 4+ hours"—is a requirement. POR, 8, 62. However, neither Petition, nor Dr. Zhu's declaration, nor Cheng itself stated or implied that the specific temperature or time values disclosed in Cheng were limiting or required.

As Dr. Zhu explained, Cheng's conditions are disclosed as part of an example and are not presented as exclusive or limiting. MRD-2026, 40:11-41:4, 44:24-45:14). As noted by Dr. Zhu, these variations were within ordinary knowledge and skill of a POSITA. See MRD-2026, 40:4-42:14; 44:24-47:7.

In addition, Petition does not describe or suggest that a POSITA would have viewed Cheng's calcination conditions as a requirement. Rather, the

'924 Reply, 22-23

But the Petition Plainly Applied Cheng's ONLY Disclosed Calcining Conditions

The '924 Petition, 52

Cheng describes that the steps for

dispersing the carbon nanotube in “the precursor” include at least [1] mixing the above “mixture” with “a lithium source, an iron source, and a phosphate salt at an atom ratio of Li : Fe : P = 1 : 1 : 1” and “organic carbon source” (e.g., sucrose, glucose or phenolic resins) using “mixing medium” (e.g., least one of “deionized water, industrial alcohol or the absolute ethyl alcohol”) and [2] ball milling and drying. *See id.*, [0013]-[0018], claim 2.¹¹ After ball milling, the precursor powder is calcined and sieved. *See id.*, [0019], claim 2.

The '924 Petition, 57

[0012], [0021], claim 1, 2. For instance, Cheng claims and describes that the “**in situ solid phase reaction**” includes certain steps including [1] calcining the dried precursor powder at 600-850 °C for 4-49 hours, [2] pulverizing the burned material, and [3] then sieving the same through a sieve of 150-400 meshes (here, the dried precursor powder refers to a powder that is obtained by [a] mixing carbon nanotube, acetylene black, and dispersant mixture with lithium source, iron source, phosphate, and organic carbon source using the mixing medium, [b] ball milling the mixture, and [c] drying the mixture). *See id.*, [0019], claim 2. As a

Petitioner's Primary Rebuttal Evidence Does Not Disclose Oxidation Levels

Dr. Zhu's Testimony (MRD-2045, 6:1-20)

1	Q	Does LGES 1032 report the oxidation level
2		after the calcination step is performed?
3	A	In this specific paper, they did <u>not</u> report
4		the oxidation level.

Q Okay. And does LGES 1040 report the oxygen level -- I'm sorry. Does LGES 1040 report the oxidation level after the calcination step of LGES 1032?

A It does not report the oxidation level after the calcination, but it does use oxidized carbon nanotube, as I mentioned.

Petitioner's Rebuttal Evidence is Misleading

LGES-1042, 3-5

2 Experimental

2.1 Synthesis of porous CNT-foam composites

Multi-walled carbon nanotubes (CNT) are selected as a raw material for the foam synthesis. The carbon nanotubes were synthesized by a Chemical Vapour Deposition (CVD) process using $\text{Fe}/\gamma\text{-Al}_2\text{O}_3$ as a growth catalyst and a mixture of C_2H_6 and H_2 as a carbon source.^{24,25} The synthesis was carried out at

TGA analysis also indicates the presence of unburnt compounds which amount to about 5 to 8 wt% in the sample. Such species could be attributed to the residual iron catalyst which was buried under graphitic layers (2–3 wt%) and was not removed from the CNT during the purification process, and also to the SiO_2 phase, originating from the impurities that were present in the starting raw materials and were further converted into SiO_2 during the calcination process (see also XPS results below). It is worth noting that for the waste water treatment

Petitioner Attempts to Improperly Shift the Burden of Proof to Patent Owner

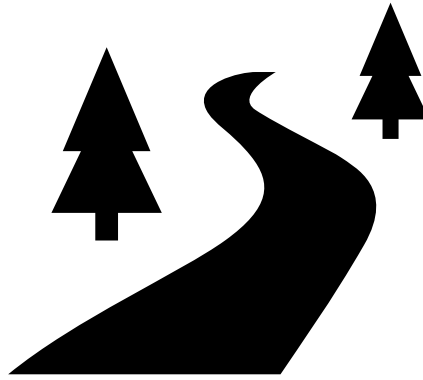
Petitioner does not dispute that essentially all oxidized functional groups can be removed under high temperature calcining conditions, but argues that in *some instances* the loss “can be minimal.” Reply, 18. Petitioner then criticizes PO for not demonstrating that essentially all oxidized functional groups would be removed in the LiFePO_4 electrode of Cheng-Gan. Reply, 18-20. However, it is the burden of **Petitioner** to demonstrate that Cheng-Gan would retain an oxidation level of 1% to 15% after the calcination treatment—it is not PO’s burden to prove that it would not. *See In re Magnum Oil Tools*, 829 F.3d 1364, 1380 (Fed. Cir. 2016) (“It is the petitioner that bears the burden of proof....”).

’924 Sur-Reply, 34

Lee-Wepasnick Grounds

'909 Petition

Ground	Claims	Basis for Rejection
1	1-2, 5-8	103: Ohata-Kavan
2A	1-2, 5-8	103: Lee-Wepasnick
2B	3-4	103: Lee-Wepasnick-Zhamu



Whether the Petition Adequately Explained its Evidence

The '909 Institution Decision, 33-34

#1

We have concerns about Petitioner's conclusions derived from Lee. Lee discloses that its "anode active material includes secondary particles formed of agglomerated primary nanoparticles," and that the "primary nanoparticles include a non-carbonaceous material bound with hollow carbon nanofibers." Ex. 1006, code (57), ¶ 14. Petitioner acknowledges these disclosures in Lee (*see* Pet. 42, 48, 52), but never sufficiently addresses them. For example, Petitioner fails to reconcile Lee's teachings regarding agglomeration of particles with its assertion that Figures 1 and 3 of Lee show discrete, non-agglomerated carbon nanotubes. Absent additional information, we decline to draw the same conclusion Petitioner does, particularly in view of Lee's disclosures regarding agglomeration. Petitioner cites to paragraphs 162 and 167 of the Zhu declaration to support the assertions in the Petition, but these paragraphs simply restate what is provided in the Petition, offering no additional support. Pet. 53–54, 56.

The '909 Institution Decision, 33-34

#2

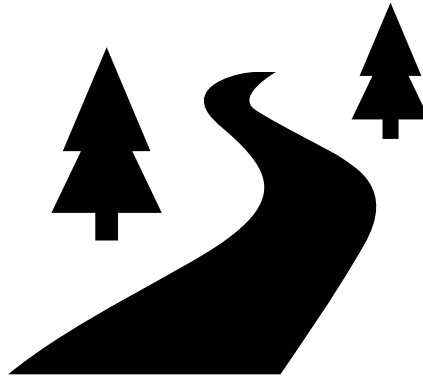
We have similar concerns about Petitioner's argument that the oxidation of nanotubes can help promote non-agglomeration of the nanotubes in the context of Lee. Pet. 54. We agree with Patent Owner that, on this record, "Petitioner's speculation about Lee somehow having 'discrete' carbon nanotubes [is] directly contrary to the teachings throughout Lee of 'agglomerated' nanoparticles." Prelim. Resp. 30. Moreover, Lee's teaching regarding agglomerated nanoparticles is particularly relevant considering Petitioner's qualification that oxidation "can" help promote non-agglomeration "in some instances." Pet. 54.

#3

Petitioner's argument regarding dispersal of nanofibers is similarly problematic. We have difficulty discerning, on this record, how a composition with agglomerated nanoparticles and including a polymer binder can provide discrete nanotubes. As before, Petitioner cites to paragraph 168 of the Zhu declaration, but that paragraph offers no more than the information presented in the Petition. Pet. 56–57.

The Petition's Failure to Adequately Explain its Evidence is Fatal

- “Arguments in a petition must be made ***with particularity***, not opacity.” *Microsoft Corp. v. FG SRC, LLC*, 860 Fed. Appx. 708, 713 (Fed. Cir. 2021).
- “Unlike district court litigation—where parties have greater freedom to revise and develop their arguments over time and in response to newly discovered material—***the expedited nature of IPRs bring with it an obligation for petitioners to make their case in their petition to institute.*** While the Board’s requirements are strict ones, they are requirements of which petitioners are aware when they seek to institute an IPR.” *Intelligent Bio-Sys.*, 821 F.3d at 1369.

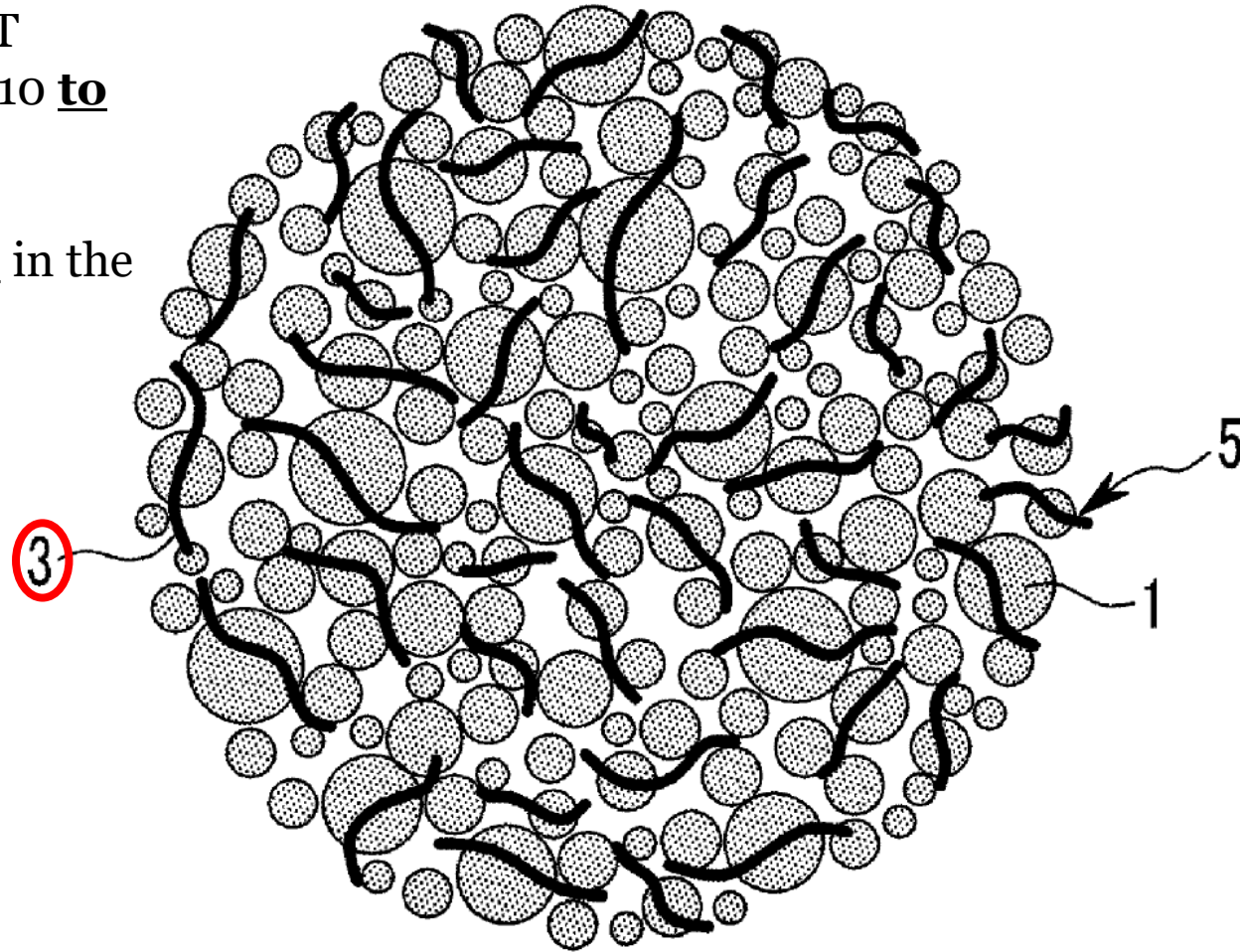


Whether the Lee-Wespasnick Teaches “Discrete” CNTs

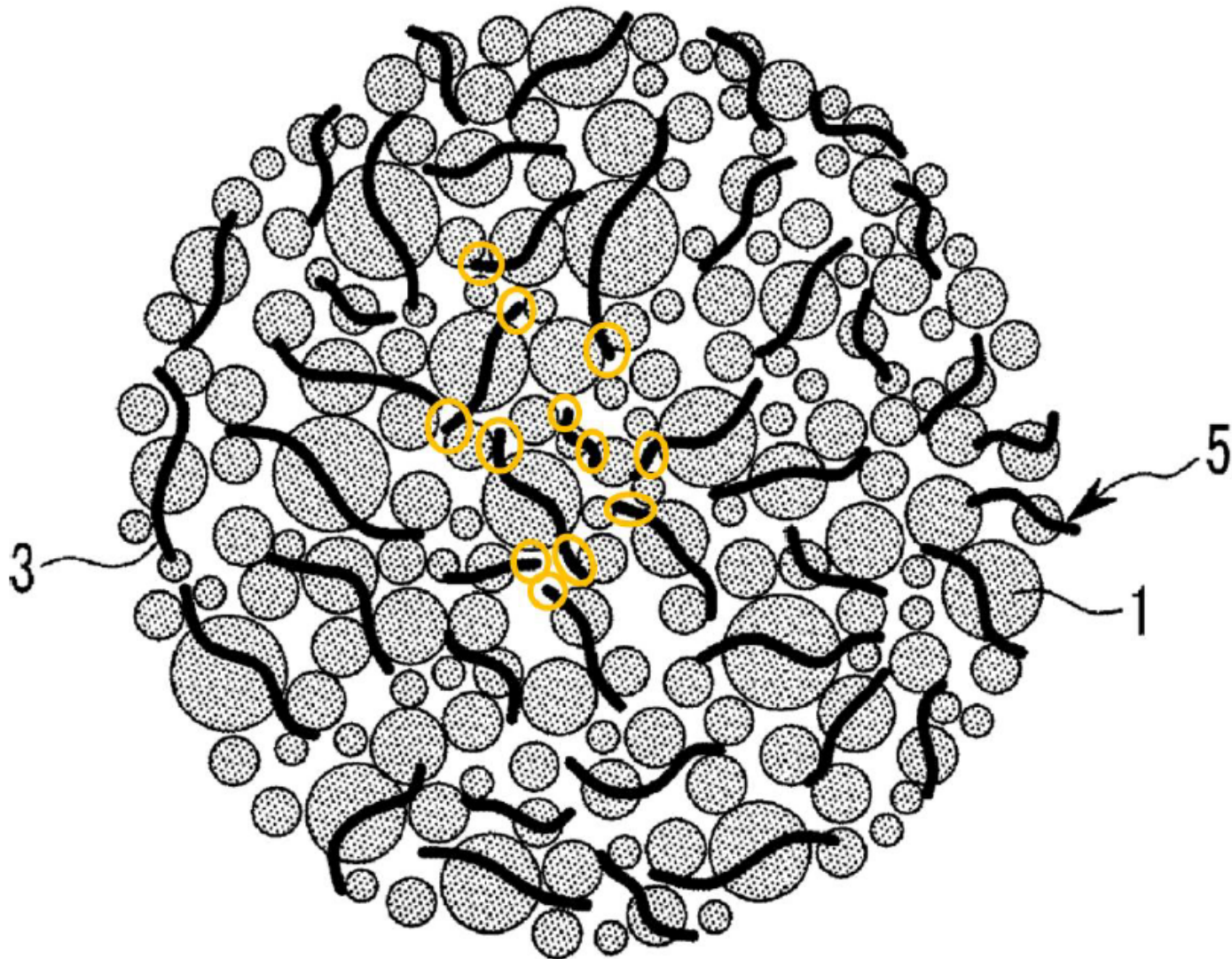
The Thick Squiggly Lines **3** in FIG. 1 of Lee Represent Intertwined CNTs

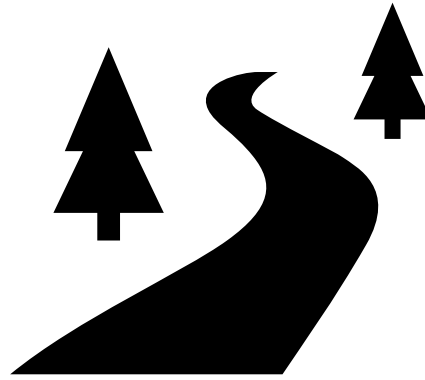
Reason #1: Lee Disclose a CNT Aspect Ratio Range of “about 10 to about 10,000”

Reason #2: CNTs are visible in the SEM image in Fig. 6 of Lee.



The “Bound” Nature of the CNTs Does Not Prevent Agglomeration, Particularly Due to Thermal Drying





Whether the Lee-Wespasnick Renders Obvious the Claimed 10-500 Aspect Ratio

Petitioner Fails to Present a *Prima Facie* Case of Obviousness

- Petitioner cites *In re Peterson* and relies solely on Lee's disclosure of an aspect ratio range of "about 10 to **about 10,000**" to argue for a *prima facie* case.



In re Peterson, 315 F.3d 1325, 1329 (Fed. Cir. 2003)
("A *prima facie* case of obviousness **typically** exists when the ranges of a claimed composition overlap the ranges disclosed in the prior art.")



Genetics Inst., LLC v. Novartis Vaccines & Diagnostics, Inc., 655 F.3d 1291, 1306 (Fed. Cir. 2011) ("The facts here present a case where the 'disclosed range is so broad as to encompass **a very large number of possible distinct compositions**' thus 'requir[ing] nonobvious invention,' not a case, as in *Peterson*, where prior art 'ranges that are **not especially broad** invite routine experimentation to discover optimum values.")

Petitioner Fails to Present a *Prima Facie* Case of Obviousness (Cont.)

- Patent Owner demonstrated that there are **key structural and behavioral differences** across Lee's remarkably broad range:
 - (1) differences in the number of bends;
 - (2) differences in the number of entanglements;
 - (3) differences in the number of CNT-CNT contact points;
 - (4) differences in the likelihood of achieving a percolated network;
 - (5) differences in electrical conductivity;
 - (6) differences in dispersibility, reducing the likelihood of achieving discrete or uniformly dispersed CNTs; and
 - (7) differences in the number of CNT defects.
 - (8) 9,500 different and larger aspect ratios than the narrower 10-500 range.

BACKUP SLIDES

MRD-2017 Shows that NMP does not remove PVDF coating from CNTs

[0123] Observations with an SEM (see FIG. 2) showed that the CNTs are well dispersed **around** the micrometric LiFePO_4/C particles. Furthermore, the electrical conductivity of the electrode obtained is equal to $2.8 \mu\text{S}/\text{cm}$.

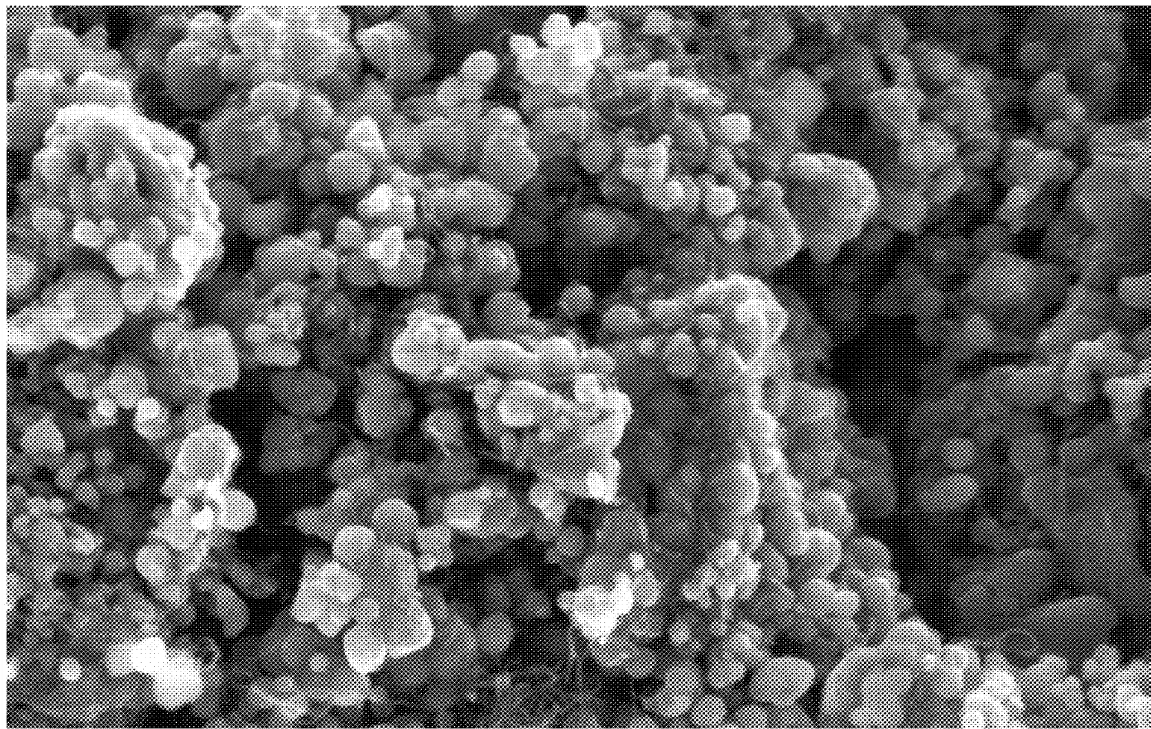


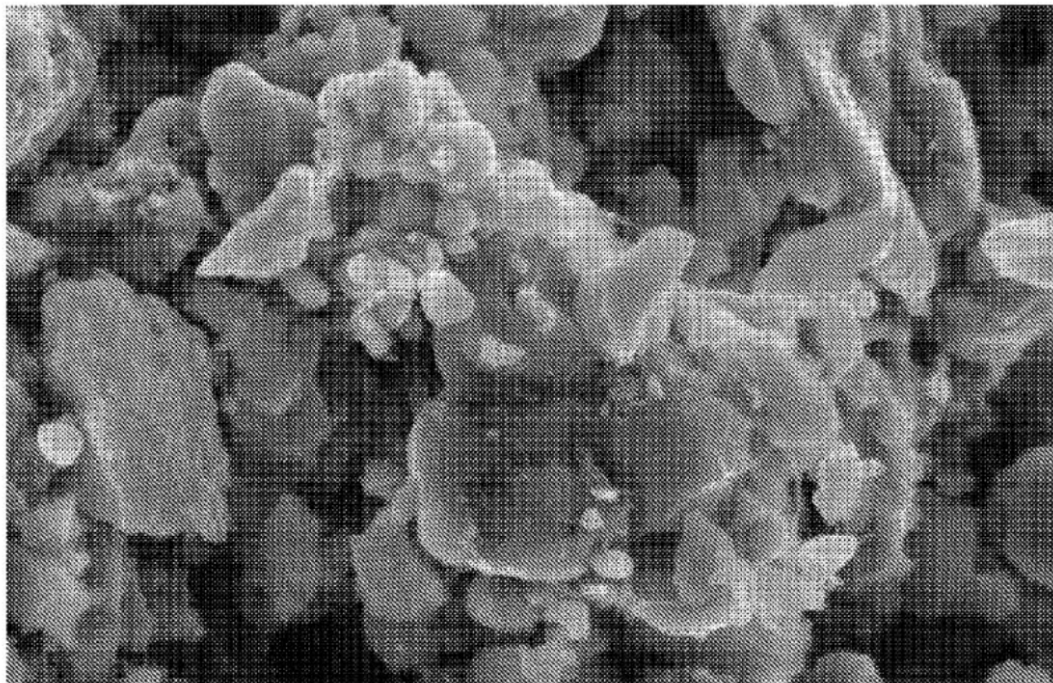
FIGURE 2

MRD-2018

“2% of CNTs; 5% of Kynarr HSV 900 [i.e., PVDF] and 93% of LiFePO₄/C with a solids content of 40% in the NMP Solvent.”

FIG. 2B

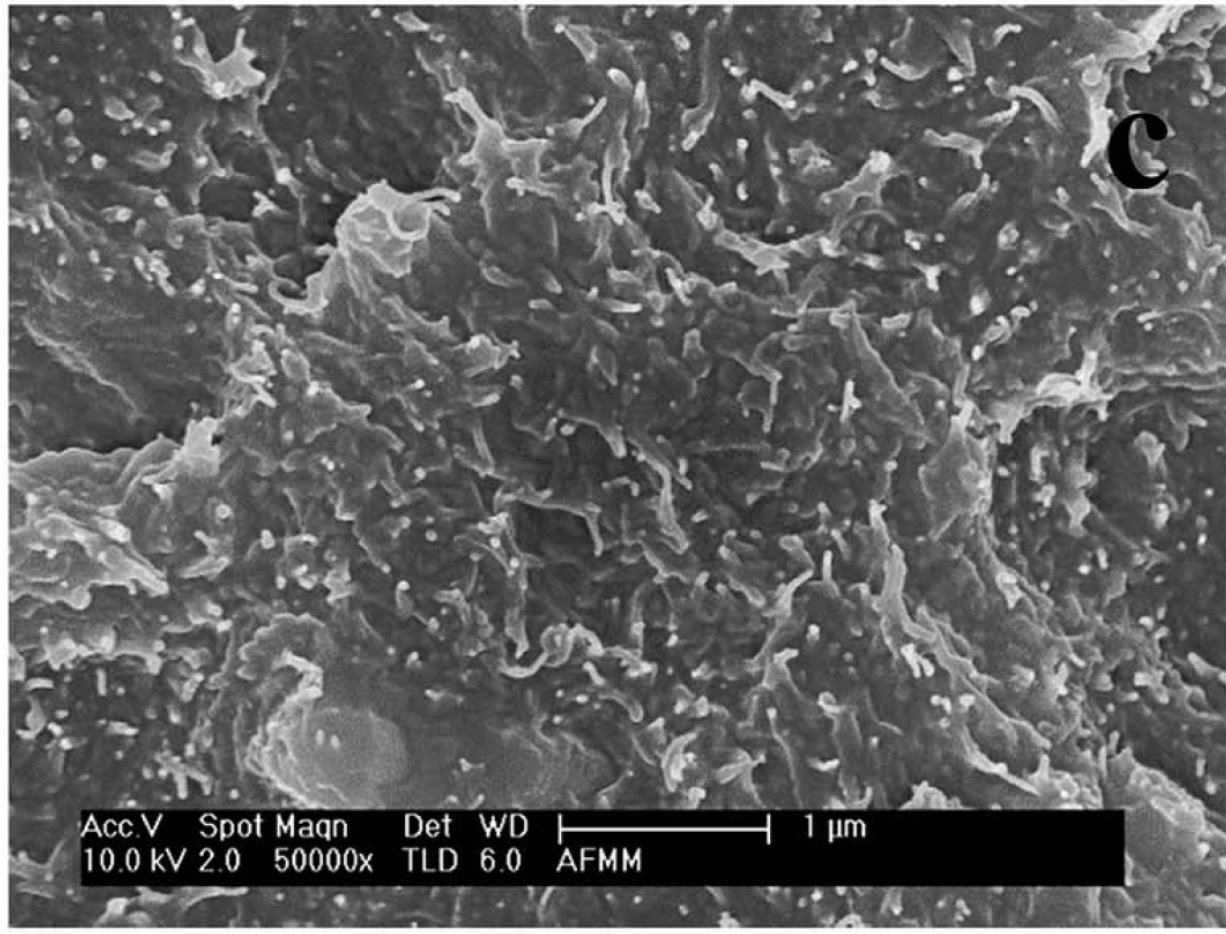
MRD-2018 ¶¶[0128]-[0131]



Mag = 50.00 KX $1\mu\text{ m}^*$ Sample ID= EHT = 5.00kV
Output To = Default Printer Date: 22 Feb 2010
File Name = YL-1035-015-minicer tranche-12.jpg GRL
WD = 4.1mm Aperture Size= 30.00 $\mu\text{ m}$ Detector = InLens User Name = PORTAIL

MRD-2012, 3, 6

“3 wt% CNTs in PVDF matrix”



MRD-2012, 3, 6