LG Energy Solution, Ltd. (Petitioner)
v.
Molecular Rebar Design, LLC (Patent Owner)

Petitioner's Demonstratives

Case No. IPR2024-01006 (Patent No. 8,808,909)
Case No. IPR2024-01011 (Patent No. 8,968,924)
Case No. IPR2024-01013 (Patent No. 10,153,483)[1]
Before Grace Obermann, Kristina M. Kalan, and Avelyn M. Ross
Administrative Patent Judges

FISH.

[1] Samsung Electronics Co., Ltd. and Samsung Electronics America, Inc., which filed petitions in IPR2025-00356, IPR2025-00357, and IPR2025-00359, have been joined as petitioners in these proceedings.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

LGES 1064
LGES v. MRD
IPR2024-01013

Asserted Grounds

IPR2024-01006 ('909 Patent)*

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

IPR2024-01011 ('924 Patent)

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

IPR2024-01013 ('483 Patent)

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

909 Pet., 2; 924 Pet., 1; 483 Pet., 1

FISH.

* The record from IPR2024-01006 is primarily referenced throughout when addressing same topics covered in all three IPRs.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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- Overview of the ’909, ’924, and ’483 Patents
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  - IPR2024-01006, -01011, -01013
- Issue 3: Obviousness over Lee and Wepasnick
  - IPR2024-01006 (’909 Patent) only
- Issue 4: Obviousness over Cheng and Gan
  - IPR2024-01011 (’924 Patent) only

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Overview of the ’909, ’924, and ’483 Patents

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Claim 1 of each patent

IPR2024-01006: The '909 Patent

What claimed is the following:

1. A composition useful for lithium ion batteries comprising:

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.

IPR2024-01011: The '924 Patent

What is claimed is:

1. A composition used in lithium ion batteries comprising: discrete carbon nanotubes having 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 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.

IPR2024-01013: The '483 Patent

What is claimed is the following:

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, 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 material 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.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 1

“Discrete” Carbon Nanotubes

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

“Discrete” Carbon Nanotubes

’909 Patent

1. A composition useful for lithium ion batteries comprising:

discrete carbon nanotubes having crystals or layers of lithium ion active material attached to their surface,

’924 Patent

1. A composition used in lithium ion batteries comprising:

discrete carbon nanotubes having ion active material attached to their surface, wherein the discrete carbon

’483 Patent

1. A composition useful for lithium ion batteries comprising: discrete, non-agglomerated, and exfoliated carbon nanotubes having a surface, and mixtures of ion active

Petitioner’s Reply

The Petition did not present a claim construction for the term “discrete.” Although PO similarly did not explicitly offer a construction, it contends—incorrectly—that Petitioner did, asserting that Petitioner’s position is that “discrete” nanotubes must be “‘fully isolated’ or ‘separated’ from one another.” POR, 51-52. PO then adopts this purported construction and applies it throughout the POR.

First, this is a blatant misrepresentation of Petitioner’s position. Second, it is inconsistent with the ’909 Patent itself, which provides no support for such a narrow interpretation.[1]

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's applied construction is inconsistent with the patents

Patent Owner's Response

Petitioner proposes that "discrete" CNTs means "generally individually distinct nanotubes" (Pet., 21-22) that are "fully isolated" or "separated" from one another. MRD-2023 ¶[0083]; MRD-2024, 11:4-8 (testifying that CNTs are "discrete" "because" they are "fully isolated" from one another), 12:17-13:22 (testifying that "discrete" CNTs are "separated from each other"). This is consistent with the plain and ordinary meaning of "discrete," as evidenced by its usage in the specification and the common meaning of "discrete." MRD-2006 ¶¶145-146 (citing LGES-1001, 1:40-44 ("discrete, i.e., individual, uniformly dispersed tubes"), 3:47-49 ("discrete" CNTs are "separated to give individual tubes"); MRD-2007, 8 (defining "discrete" as "separate and distinct; not attached to others; unrelated")).

'909 Patent

The term "carbon nanotubes" as used herein refers to allotropes of carbon with a cylindrical nanostructure. The nanotubes may be single, double or multiwall.

The term "discrete carbon nanotubes" as used herein refers to carbon nanotubes capable of being separated to give individual tubes.

Petitioner's Reply

The '909 Patent is unambiguous: "[t]he term 'discrete carbon nanotubes' as used herein refers to carbon nanotubes ***capable of being separated*** to give individual tubes." LGES-1001, 3:47-49 (emphasis added). Tellingly, PO quotes this very sentence, but only in part, stating that the '909 Patent confirms that "'discrete' CNTs are 'separated to give individual tubes.'" POR, 52. PO's stealthy omission of the key phrase "***capable of***" (LGES-1001, 3:47-49) unjustifiably changes the meaning of this statement, which, when read in its entirety, cannot support PO's narrow interpretation of the term "discrete."

Institution Decision

[1] The '909 patent defines "discrete carbon nanotubes" as "carbon nanotubes capable of being separated to give individual tubes." Ex. 1001, 3:47–49.

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IPR2024-01006: LGES-1001, 3:47-49; POR, 51-52; Pet. Reply, 3; ID, 4

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO misrepresents Petitioner's position

Patent Owner's Sur-Reply

Under Petitioner's new construction ***any*** CNT would qualify as "discrete", as all CNTs are "capable of" being separated. MRD-2043, 12:7-13, 13:13-20 (admitting the CNTs in Ohata's Figure 1(a) are not "discrete" but there "***is always a potential***"). This construction would not "giv[e] effect" to "discrete." *See Info-Hold v. Muzak LLC*, 783 F.3d 1365, 1373 (Fed. Cir. 2015).

Dr. Zhu's Deposition

Q. (BY MR. ZINDA) But are they capable of being separated from each other?
MR. IN: Objection, form.
A. Well, when you say they are capable or not separated each other, I think this is depending on how you treat other nanotubes and how the nanotube was processed. So there is no definite answer about whether entangled nanotube are capable to be separated. It depends on many things, like what is preparation procedure to separate those nanotubes. So --
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.

IPR2024-01006: Pet. Reply, 1; PO Sur-Reply, 10; MRD-2043, 13:3-20

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

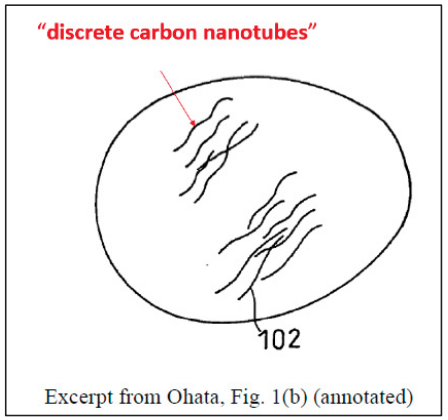
PO misrepresents Petitioner's position

Patent Owner's Response

Petitioner proposes that "discrete" CNTs means "generally individually distinct nanotubes" (Pet., 21-22) that are "fully isolated" or "separated" from one another. MRD-2023 ¶[0083]; MRD-2024, 11:4-8 (testifying that CNTs are "discrete" "because" they are "fully isolated" from one another), 12:17-13:22 (testifying that "discrete" CNTs are "separated from each other"). This is consistent with the plain and ordinary meaning of "discrete," as evidenced by its usage in the specification and the common meaning of "discrete." MRD-2006 ¶¶145-146 (citing LGES-1001, 1:40-44 ("discrete, i.e., individual, uniformly dispersed tubes"), 3:47-49 ("discrete" CNTs are "separated to give individual tubes"); MRD-2007, 8 (defining "discrete" as "separate and distinct; not attached to others; unrelated")).

Deposition of Dr Zhu

1	Q	Correct. And do you see the -- you
2	added a red arrow.	Do you see that?
3	A	Yes. I see that.
4	Q	And so you're identifying that nanotube
5	as discrete because it's fully isolated	from the other
6	nanotubes?	
7	A	Yeah. I say it is discrete,
8	non-agglomerated and exfoliated carbon nanotubes.	



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

IPR2024-01006: POR, 51-52; MRD-2024, 11:1-8;

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO misrepresents Petitioner's position

Petitioner's Reply

A. PO quotes Petitioner's expert out of context in attempting to substantiate its position

PO's citation to statements made by Petitioner's expert, Dr. Zhu, regarding the term "discrete" omits critical context. *See* POR, 51-52. Dr. Zhu's agreement that he was "identifying that nanotube [indicated in the figure *infra*] as discrete because it's fully isolated from the other nanotubes" (MRD-2024, 10:21-11:12) is not, as PO would like to believe, a definition requiring that a nanotube necessarily must be isolated to be discrete. Instead, refers to this particular example. In fact, while the term "discrete" surely encompasses individually isolated nanotubes, the term "discrete" is not limited to only individually isolated nanotubes. LGES-1056, ¶5; *see infra* §§I.B-C.

Supplemental Declaration of Dr. Zhu

5. As an initial matter, PO's reference to my statements at deposition regarding the term "discrete" omits critical context. *See* POR, 51-52. My agreement that I was "identifying that nanotube [marked with the red arrow in the figure *infra*] as discrete because it's fully isolated from the other nanotubes" (MRD-2024, 10:21-11:12) was not intended to be a definition requiring that a nanotube necessarily must be isolated to be discrete. Instead, it is a description of the particular illustrated example, as described below. *See* LGES-1058, 50 (my declaration in the '483 Patent IPR). As I explain in the next section, while "discrete" surely encompasses individually isolated nanotubes, it is not limited to only individually isolated nanotubes.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The patents contradict PO's interpretation of "discrete"

'909 Patent

BRIEF DESCRIPTION OF THE FIGURES

FIG. **1** depicts lithium salt coated as a nanolayer on discrete carbon nanotubes.

FIG. **2** shows $LiMnO_4$ with 5% oxidized carbon nanotubes of this invention.

FIG. **3** shows titanium dioxide crystals attached to discrete carbon nanotubes.

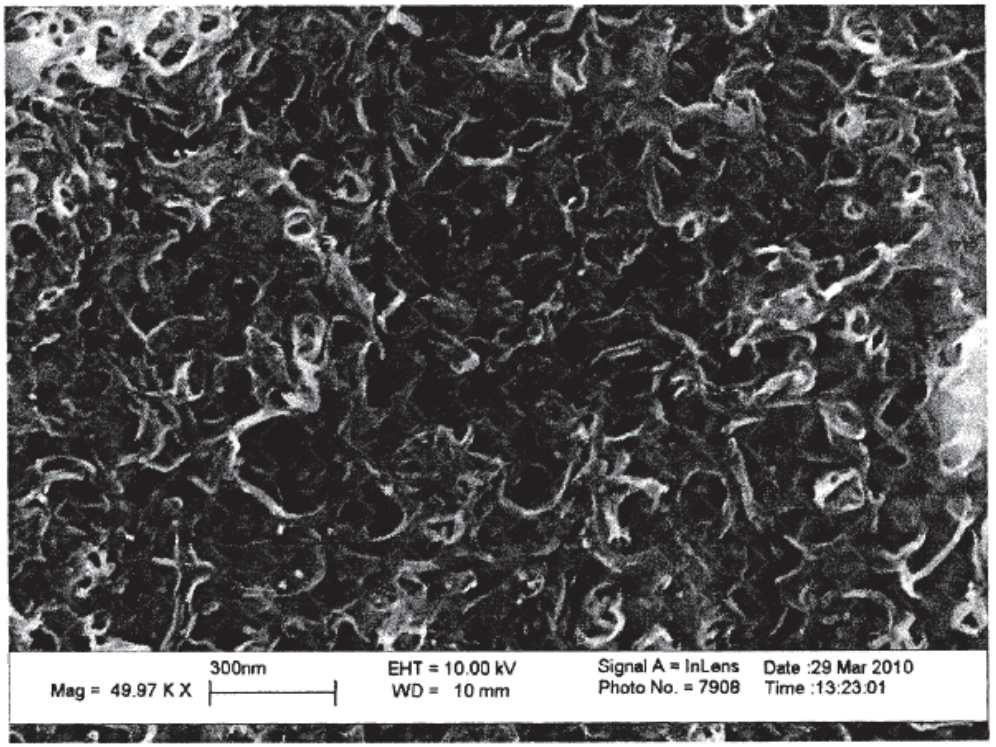


Figure 1

IPR2024-01006: LGES-1001, 3:35-40, FIG. 1

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The patents contradict PO's interpretation of "discrete"

'924 Patent

Example 1

Discrete Carbon Nanotubes with Attached Copper Oxide Nanoparticles Suitable for a Lithium Ion Battery Anode Material

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

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.

Attempts to overcome the above deficiencies have included synthesis of nanosized 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.

IPR2024-01011: Pet. Reply, 25-26; LGES-1056, ¶52; LGES-1001, 1:42-63, 2:17-29, 9-29

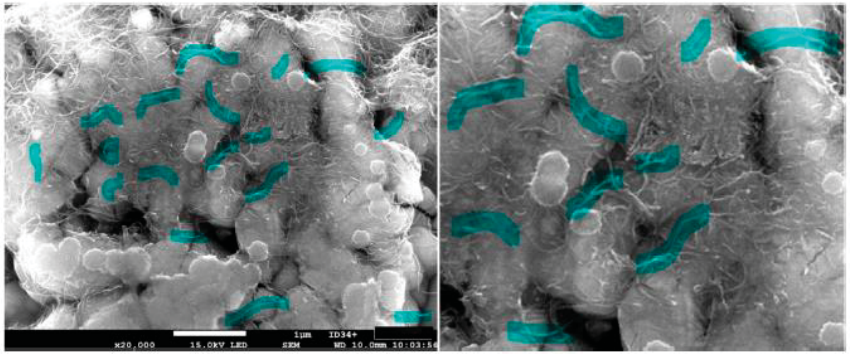
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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's position contradicts its own infringement contentions

Petitioner's Reply

PO's narrow construction is undermined by its own infringement contentions, in which PO alleged that the SEM images below depict "discrete" carbon nanotubes; the allegedly discrete nanotubes they highlight clearly depict nanotubes touching each other (original highlighting by PO). *See* LGES-1050, 33; LGES-1056, ¶6-7.



LGES-1050, 33 (original at left, cropped and magnified at right)

Patent Owner's Sur-Reply

SEM images are two-dimensional images of ***a three-dimensional space***, meaning that at lower-magnifications CNTs that *appear* to be attached because they overlap are not necessarily attached or touching. LGES-1055, 127:21-128:12. Petitioner "agree[s]" (*id.*, 128:3-9) and relies on this "commonsense principle" in IPR2024-01005 (Paper 23, 19-20).

FISH.

IPR2024-01006: Pet. Reply, 4; PO Sur-Reply, 10-11; MRD-2043, 16:7-17:10

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2

Obviousness over Ohata and Kavan ('909, '924, and '483 Patents)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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- Issue 2A: Active material is attached to the surface of carbon nanotubes in the Ohata-Kavan combination
- Issue 2B: The Ohata-Kavan nanotubes are discrete
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- Issue 2D: A POSITA would have expected success in combining Ohata and Kavan
- Issue 2E: Claim 6 of the '909 Patent and claim 8 of the '483 Patent are obvious over the Ohata-Kavan combination

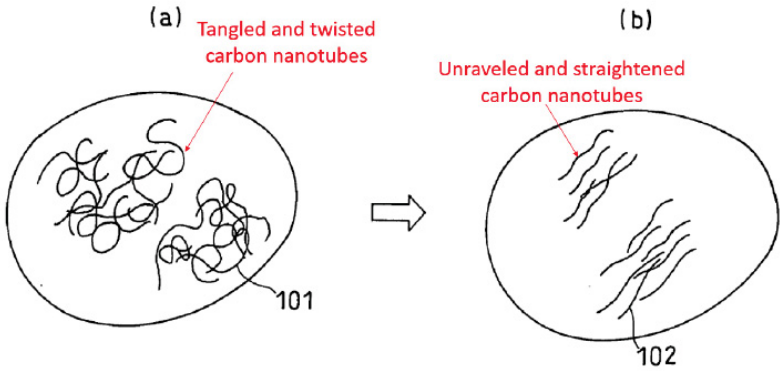
DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan Combination

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Ohata describes a CNT-containing paste for lithium ion batteries

Petition



Ohata, FIG. 1 (annotated to demonstrate a masterbatch (b) produced by blending carbon nanotubes with a resin)

Ohata prepares its composites by first "producing a masterbatch by blending at least carbon nanotubes with a resin." LGES-1004, [0015], [0022]. In this process, nanotubes that are initially "tangled and twisted with one another" (FIG. 1(a), *infra*) are "unraveled and straightened by blending them with a resin" such that they are "dispersed" in the resin (FIG. 1(b)). *Id.*, [0016]-[0017]; LGES-1003, ¶20.

An "electrode material mixture paste" is formed by mixing "the masterbatch and an electrode active material with a dispersion medium" and is applied "onto an electrode substrate" for use in a lithium secondary battery. LGES-1004, [0023], [0025]-[0027]; LGES-1003, ¶¶21-22.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Ohata describes a CNT-containing paste for lithium ion batteries

Ohata

[0015] A method for producing an electrode for a battery of the present invention has a step of producing a masterbatch by blending at least carbon nanotubes with a resin.

[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.

[0017] The resin with the unraveled and straightened carbon nanotubes dispersed therein also serves to increase the mechanical strength of an electrode plate. The 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.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Kavan discloses oxidized CNTs for use in lithium ion batteries

Petition

2. Kavan describes composite materials containing oxidized nanotubes for use in lithium ion batteries

Kavan describes composites of functionalized multi-walled carbon nanotubes (MWNTs) with electrode active materials, for use in lithium ion batteries. *See* LGES-1005, 1-2; LGES-1003, ¶23.

Kavan performs "oxidative functionalization" on the MWNTs to produce "MWNT-ox" ("material functionalized by oxidation with HNO_3. LGES-1005, 4; LGES-1003, ¶24. Oxidized nanotubes were mixed with "a powder material (TiO_2, $LiMnPO_4$ or $LiFePO_4$)," and pastes were prepared using the resulting material. LGES-1005, 2. The structure, composition, and electrochemical behavior of the resulting composites were characterized, showing performance advantages resulting from interactions between oxidized nanotubes and the positive or negative active material. *See* LGES-1004, 3-5, 7-9; LGES-1003, ¶¶25-32.

Kavan

A B S T R A C T

Multi-walled carbon nanotubes functionalized by carboxylic groups, exhibit better affinity towards TiO_2 (P90, Degussa) as compared to that of pristine nanotubes. Also the electrochemical performance of TiO_2 is improved by nanotube networking, but the Li-storage capacity of TiO_2 is unchanged. Whereas the composite of TiO_2 with non-functionalized nanotubes demonstrates simple superposition of the behavior of pure components, the composite with functionalized nanotubes shows unique faradaic pseudocapacitance which is specific for this composite only. The surface functionalization of nanotubes enhances charge storage capacity and reversibility of a composite with $LiMnPO_4$ (olivine), but mediates also the electrolyte breakdown at potentials >4.2 V. Whereas the electrochemical activation of $LiMnPO_4$ (olivine) by functionalized nanotubes is quite modest, excellent performance was found for $LiFePO_4$ (olivine) in composite materials containing only 2 wt% of functionalized nanotubes.

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IPR2024-01006: LGES-1005, 1; Pet., 6-7

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan combination

Petition

The Ohata-Kavan combination composition uses Ohata as a starting point. LGES-1003, ¶33. 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 nanotubes dispersed in a resin, where the aspect ratio of the nanotubes and the ratio of nanotubes to electrode active material are as taught by Ohata. *See id.*, [0015], [0020], [0024]; LGES-1003, ¶33.

In the combination, the nanotubes used in preparation of the masterbatch (*see* LGES-1004, [0015]) are functionalized, *e.g.*, "by carboxylic groups," as taught by Kavan, such that "[t]he amount of atomic oxygen present" on the nanotube surface is "7%." LGES-1005, Abstract, 2, 4. These oxidized nanotubes are then "unraveled and straightened by blending them with a resin" to form a masterbatch, based on Ohata. LGES-1004, [0016]; *see* [0021]; LGES-1003, ¶¶34-36.

IPR2024-01006: Pet., 7-8

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2A

Active material is attached to the surface of carbon nanotubes in the Ohata-Kavan combination

[1a-1b] discrete carbon nanotubes having crystals or layers of ***lithium ion active material attached to their surface*** *('909 Patent)*	*[1a-1b] discrete carbon nanotubes* having ***ion active material attached to their surface*** *('924 Patent)*	*[1d] wherein the discrete carbon nanotubes have crystals or layers of* ***lithium ion active material ionically attached to their surface*** *('483 Patent)*

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

In Ohata-Kavan, active material is attached to the surface of CNTs

Petition

Based on Kavan, the nanotubes in the combination electrode material paste have TiO_2 nanocrystals (**crystals of lithium ion active material**) **attached to their surface**. LGES-1003, ¶89. For instance, the TiO_2 nanocrystals are attached to the nanotube surface due at least in part to the surface oxidation of the nanotubes (*see* [1d], *infra*): "oxidative functionalization through HNO_3 is crucial for ***proper anchoring of nanotubes towards TiO_2***"). LGES-1005, 3-4; *see* 2 ("The surface oxidation of carbon nanotubes by HNO_3, H_2SO_4/HNO_3 or H_2O_2, producing among other groups also carboxyls, was sometimes mentioned to improve ***the anchoring of TiO_2***"), 4-5, 9, Abstract; LGES-1003, ¶89. For instance, as explained by Dr.

Based on Kavan, the nanotubes in the combination electrode material paste have phosphate olivine crystals (**crystals of lithium ion active material**) **attached to their surface**. LGES-1003, ¶98. The phosphate olivine crystals are attached to the nanotube surface due at least in part to the surface oxidation of the nanotubes (*see* [1d], *infra*): "The surface oxidation of carbon nanotubes … was sometimes mentioned to improve ***the anchoring of … $LiMnPO_4$***." LGES-1005, 2; *see id.* ("MWNT functionalization (by surface oxidation forming carboxyls) for ***optimum attachment of MWNT towards … olivine surfaces***"), 4; LGES-1003, ¶98.

Kavan

The surface oxidation of carbon nanotubes by HNO_3 [6,8,10,15,16,32,37,41,44], H_2SO_4/HNO_3 [4] or H_2O_2 [9], producing among other groups also carboxyls, was sometimes mentioned to improve the anchoring of TiO_2 [4,6,8–10,15,16,44] or $LiMPO_4$ [32,37,41]. In the first case, there is a clear analogy with the strong interaction of carboxyl-containing molecules (Ru-bipyridine dyes) with the TiO_2 surface, which is crucial in dye-sensitized solar cells [10]. However, there are also reports that the oxidative functionalization is not necessary for anchoring to TiO_2, because the pristine MWNT behave similarly [15]. The interaction of carboxyls with phosphate olivine surface is less understood, but certainly important, too [45,46]. To the best of our knowledge, there is no systematic study of these contradictory issues, although detailed understanding is desirable of the MWNT functionalization (by surface oxidation forming carboxyls) for optimum attachment of MWNT towards titania or olivine surfaces.

IPR2024-01006: Pet., 23-25; LGES-1005, 2

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

In Ohata-Kavan, active material is attached to the surface of CNTs

Dr. Zhu Declaration

90. Chemical attachment of TiO_2 to oxidized nanotubes has been confirmed elsewhere in the literature. For instance, X. Li demonstrated results indicating "that TiO_2 nanoparticles were chemically absorbed at SWNT defect sites via an ester-type linkage between carboxylic acid groups at the defect sites and hydroxyl groups at the surface of the TiO_2 nanoparticles." LGES-1030 (X. Li), Abstract. An ester-type linkage is a covalent bond between the carboxyl groups on the surface of the nanotubes and the hydroxyl groups on the surface of the TiO_2 particles. Additionally, J. Li reported XPS data demonstrating that "carboxylates groups on the CNTs will provide bonding to the TiO_2 … nanoparticles." LGES-1037 (J. Li), 4. Here, the XPS data support that there is covalent bonding between the TiO_2 particles and the carboxyl groups on the nanotubes.

98. Moreover, based on Kavan, the nanotubes in the combination electrode material paste have phosphate olivine crystals (**crystals of lithium ion active material**) **attached to their surface**. For instance, the phosphate olivine crystals are attached to the nanotube surface due at least in part to the surface oxidation of the nanotubes (*see* [1d], *infra*): "The surface oxidation of carbon nanotubes … was sometimes mentioned to improve ***the anchoring of … $LiMPO_4$.***" LGES-1005 (Kavan), 2; *see id.* ("detailed understanding is desirable of the MWNT functionalization (by surface oxidation forming carboxyls) for ***optimum attachment of MWNT towards … olivine surfaces***"), 4 ("the interaction of -COOH with phosphate surface"). For instance, the attachment between the phosphate olivine (*e.g.*, $LiFePO_4$ or $LiMnPO_4$) and the surface of the oxidized nanotubes can be a covalent bond, *e.g.*, between the carboxyl groups on the surface of the nanotubes and the transition metals (*e.g.*, Fe or Mn) in the $LiFePO_4$ or $LiMnPO_4$ particles.

IPR2024-01006: LGES-1003, ¶¶90, 98; Pet., 23-25

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

In Ohata-Kavan, active material is attached to the surface of CNTs

Dr. Zhu Declaration

92. Alternatively/additionally, there are Van der Waals forces between the TiO_2 and the oxygen-containing functionalities on the surface of the nanotubes. To the extent that Van der Waals bonding is considered an attachment, the TiO_2 particles are alternatively/additionally **attached** to the surface of the nanotubes through Van der Waals bonding.

93. Still alternatively/additionally, hydrogen bonds can arise between the TiO_2 particles and hydrogen atoms belonging to the oxygen-containing functionalities on the nanotube surface. *See* LGES-1037 (J. Li), 4 ("hydrogen bonding among the carboxylic, hydroxyl, and/or ether functional groups in both metal oxide and CNTs phases may also be partially responsible for the observed interconnectivity"). To the extent that hydrogen bonding is considered an attachment, the TiO_2 particles are alternatively/additionally **attached** to the surface of the nanotubes through hydrogen bonding.

IPR2024-1006: LGES-1003, ¶¶92-93; Pet., 23-24

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

In Ohata-Kavan, active material is attached to the surface of CNTs

Dr. Zhu Declaration

95. As a consequence, a POSITA would have recognized or otherwise found it obvious that one way by which the ion active materials of Kavan (e.g., $LiFePO_4$) become attached to the surface of Ohata-Kavan's nanotubes would be through ionic bonding/attachment. Indeed, it was well-known that the oxidized nanotubes, as in Ohata-Kavan, "have a negative electrostatic charge on the outer surface of the nanotube wall" and, thus, a POSITA would have recognized that such "negative electrostatic charge on the outer surface of the nanotube wall" would be used to enhance attachment of positively charged metal ions in the ion active material (e.g., $LiFePO_4$) to the nanotube surface via an ionic/electrostatic interaction. LGES-1024, [0057]; *see* LGES-1030 ("By oxidizing the nanotubes for various periods, different oxidation levels of the treated nanotubes can be easily prepared. This procedure is able to introduce carboxyl, carbonyl, and hydroxyl groups grown at any defects in the sidewalls of nanotubes. These oxygen groups ***show an affinity to couple with metal ions*** ... via the ***interaction of ionic exchange or surface polarity***."); *see also* LGES-1031, 2, FIG. 2g. In view of this, a POSITA would have recognized or otherwise found obvious that the negatively charged carboxyl group on the surface of the carbon nanotubes in Ohata-Kavan-Gong/Liu would be electrostatically attracted to and become capable of ionically attaching to the positively charged metal ions (*e.g.*, Fe++, Mn++, or the like) in the phosphate olivines such as $LiMnPO_4$ or $LiFePO_4$.

IPR2024-01013: LGES-1003, ¶95; Pet., 42-44

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

In Ohata-Kavan, active material is attached to the surface of CNTs

Petition

The Ohata-Kavan combination composition uses Ohata as a starting point. LGES-1003, ¶33. 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 nanotubes dispersed in a resin, where the aspect ratio of the nanotubes and the ratio of nanotubes to electrode active material are as taught by Ohata. *See id.*, [0015], [0020], [0024]; LGES-1003, ¶33.

Ohata

[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-metyl-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.

IPR2024-01006: Pet., 7; LGES-1004, [0032]

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO asserts that Ohata-Kavan CNTs are fully embedded within the resin matrix, preventing the attachment of active materials to the CNTs

Patent Owner's Response

First, the active materials of Ohata-Kavan cannot be "attached to the[] surface" of the oxidized CNTs as required by claim 1. *Infra* Section V.A.2. The masterbatch process of Ohata-Kavan results in CNTs ***fully embedded within, and coated by, the fluorocarbon resin matrix***, preventing the later mixed active material from attaching to the CNT surfaces. In fact, CNTs and fluorocarbon resin have an affinity toward one another, ensuring full coverage of the CNT surfaces with the resin. This design is purposeful in Ohata, because the masterbatch, with its high concentration of resin as compared to the concentration of CNTs, *is the binder* for the active material. As such, even assuming the active material is "attached" to the surface of the oxidized CNTs in Kavan *alone*, the same cannot be said of ***Ohata-Kavan***.

1. Petitioner overlooks the critical fact that the "masterbatch" process embeds the CNTs in resin to form the binder.

Petitioner vaguely describes Ohata as preparing "composites of carbon nanotubes and either positive or negative electrode active materials for use as electrodes in lithium ion batteries." Pet., 5. However, Ohata is doing something more specific and significant to the issues here—Ohata is creating an improved resin binder by ***embedding CNTs within the resin matrix*** to form its masterbatch. MRD-2006 ¶74 (citing LGES-1004 ¶¶[0005]-[0006], [0030]); MRD-2024, 58:5-10 (admitting Ohata is "embedding" conductive material "within the resin"). It is only *after* the CNTs are embedded in the masterbatch resin that they are *then* mixed with the active material to prepare the electrodes for the battery. *Id.* (citing LGES-1004 ¶¶[0005], [0032], [0046]). This is clear from the plain teachings of Ohata.

IPR2024-01006: POR, 19-21

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's assertions of nanotube embedding are incorrect

Petitioner's Reply

PO's theory that there is no contact between nanotubes and electrode active material is illogical and technically inaccurate

The solubility of Ohata's binder in NMP belies PO's argument that Ohata's nanotubes remain fully embedded in the binder matrix

IPR2024-01006: Pet. Reply, 5, 7

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The solubility of Ohata's binder in NMP belies PO's argument that Ohata's nanotubes remain fully embedded in the binder matrix

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Ohata's resin is dissolved during subsequent processing

Petitioner's Reply

Even to the extent that Ohata's nanotubes are fully embedded within the PVDF binder of the masterbatch, that embedding would not withstand processing to prepare the electrode material mixture paste. LGES-1056, ¶21. Ohata's masterbatch is mixed with active material particles and NMP, and "[t]he masterbatch ***was easily dissolved*** in [NMP]." LGES-1004, [0032]. Indeed, that the PVDF binder of Ohata's masterbatch is readily soluble in NMP was well-known. LGES-1056, ¶13-21 (citing LGES-1061, [0004]; LGES-1062, 2). The solubility of PVDF in NMP refutes PO's allegation that even after processing with NMP, the nanotubes remain "fully embedded within and coated by the resin matrix." POR, 43-44. And in fact, dissolution of PVDF makes sense given the need for physical contact between the nanotubes and active material discussed *supra*. LGES-1056, ¶22.

Ohata

[**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-metyl-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.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Ohata's resin is dissolved during subsequent processing

Dr. Zhu Supplemental Declaration

14. Dr. Chung offers no evidence that PVDF fails to dissolve under the relevant processing conditions. On the contrary, it is well known that PVDF is highly soluble in NMP. For instance, PVDF is fully dissolvable in NMP at concentrations greater than or equal to 10% by weight, indicating that all the example conditions disclosed in Ohata are within a fully dissolved range. *See, e.g.*, LGES-1062, 2; LGES-1004, [0032] (describing that "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 (LiCoO2) as an active material and 45 parts by weight of N-methyl-2-pyrrolidone with a mixer"); *see also id.*, [0040], [0044], [0046].

23. The high solubility of PVDF in NMP directly refutes PO's allegation that even after processing with NMP, the nanotubes remain "fully embedded within and coated by the resin matrix," thus "preventing the active material from attaching to the surface of the CNTs." POR, 43-44. And in fact, extensive dissolution of PVDF make sense given the need for physical contact between the nanotubes and active material discussed *supra*.

22. Even if Ohata's nanotubes are fully embedded within the PVDF binder (which is not true as the conductive paths have been established in the masterbatch) of the masterbatch formulation, that embedding would not withstand the further processing of the masterbatch to prepare the electrode material mixture paste. Ohata's masterbatch is mixed with active material particles and NMP, such that "[t]he masterbatch ***was easily dissolved*** in [NMP]." LGES-1004, [0032]. Moreover, it was well-known that PVDF (the binder of the Ohata-Kavan masterbatch) is readily soluble in NMP. *See* LGES-1061, [0004] ("Typically, a combination of polyvinylidene fluoride (PVDF) and N-methyl-2-pyrrolidone (NMP) is used for the binder and the solvent. Polyvinylidene fluoride is soluble in NMP and allows for the preparation of a paste having a proper viscosity"); LGES-1062, 2.

IPR2024-01006: LGES-1056, ¶¶14, 22, 23; Pet. Reply, 7-8

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Ohata's resin is dissolved during subsequent processing

Petitioner's Reply

Moreover, PO's position that "the PVDF coating would be more strongly bound to the oxidized CNTs" is inconsistent with literature reports of a weak interaction between functionalized nanotubes and PVDF. LGES-1056, ¶23 (citing LGES-1059, 5). Given this weak interaction, and the high solubility of PVDF in NMP, a POSITA would have recognized that NMP can in fact substantially remove PVDF from the CNT surface, enabling attachment of active material particles to the nanotubes. *Id.*

Dr. Zhu Supplemental Declaration

18. In asserting that CNTs remain embedded within PVDF so as to prevent active ion material from contacting the CNTs, Dr. Chung fails to account for the weak interaction between PVDF and oxidized CNTs. PVDF is highly hydrophobic, while oxidized CNTs carry many hydrophilic functiona groups, leading to poor interfacial adhesion. Hou (LGES-1059) is one of references that report this weak interaction between PVDF and CNTs. *See* LGES-1059, 5 ("[t]his result indicates that even though good dispersion of CNTs among the PVDF has been achieved, the interaction between CNTs and PVDF was relative weak").

19. Regarding pristine CNTs, although they have better interaction with PVDF than ox-CNTs, they (pristine CNTs) still have weak interaction with PVDF. Therefore, when NMP, PVDF, and CNTs are mixed, NMP will dissolve PVDF and substantially remove PVDF from CNT even if they were previously mixed. Again, as I explained above, a POSITA would understand the importance of dissolving PVDF for making good electrode slurry and this will be the pursuit for any regular handling of battery electrode preparation.

IPR2024-01006: Pet. Reply, 8; LGES-1056, ¶¶18-19, 24

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Patent Owner's Response

The subsequent steps of Ohata-Kavan do not change the fact the CNTs are coated by resin. After the masterbatch is prepared, it is used to form the electrode material mixture paste. *Id.* ¶118 (citing LGES-1004 ¶[0005]). This involves using the masterbatch as the binder by mixing it with a "dispersion medium" (e.g., NMP) and active material to form the electrode material mixture paste. *Id.* (citing LGES-1004 ¶¶[0023]-[0024], [0046]). For instance, in Example 1, Ohata discloses mixing 3.3 parts by weight of the masterbatch with 100 parts by weight of active material and 45 parts by weight of NMP. *Id.* (citing LGES-1004 ¶[0032]). NMP is a solvent that disperses the PVDF by dissolving it to break the matrix up into PVDF domains (or "islands") so that the domains can be dispersed throughout the electrode material mixture, providing local glue-like bonds between adjacent active material particles.

Id. In other words, smaller binder particles are dispersed throughout the electrode mixture, but the CNTs are still embedded within and coated by PVDF that is bonded to the CNTs. *Id.* ¶119. Indeed, the Ohata-Kavan masterbatch process does not teach or suggest filtering PVDF after creation of the electrode paste, which would be required to eliminate the PVDF domains. *Id.*; MRD-2024, 41:5-25. Thus, dispersing PVDF having an embedded conductive agent (e.g., CNTs) using organic solvent (e.g., NMP) still leaves the conductive agent "coat[ed]" with PVDF. *Id.* (citing MRD-2015, 1:31-34 ("PVDF dissolved in the organic solvent ***tends to coat*** particles of the active material and ***the conductive agent***, so that PVDF is not distributed effectively between these particles.")).

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Patent Owner's Response: MRD-2017

As another example, U.S. Patent Application Publication No. 2014/0319429 ("Nicolas-429") specifically identifies Ohata and drawbacks of its masterbatch process. *Id.* ¶123 (citing MRD-2017 ¶¶[0009]-[0010]). Nicolas-429 proposed its own "masterbatch" process. In Example 3, the process uses "5% by weight of CNTs, 1% by weight of PVP, 0.8% by weight of PVDF and 93.2% by weight of NMP" to form a paste, which is then mixed with LiFePO4. *Id.* (citing MRD-2017 ¶¶[0111]-[0118]). As shown in Figure 2 below, large domains of PVDF exist despite the use of a solvent (NMP) to disperse the PVDF:

Id. Nicolas-429 states that Figure 2 shows "CNTs are well dispersed ***around*** the micrometric $LiFePO_4/C$ particles." MRD-2017 ¶[0123]. A POSITA would understand that they are "around" the particles but not attached to the particles, because they are embedded within PVDF domains that prevents the active material from attaching to the surface of the CNTs. MRD-2006 ¶123.[3] A POSITA would expect the same result in Ohata-Kavan. *Id.*

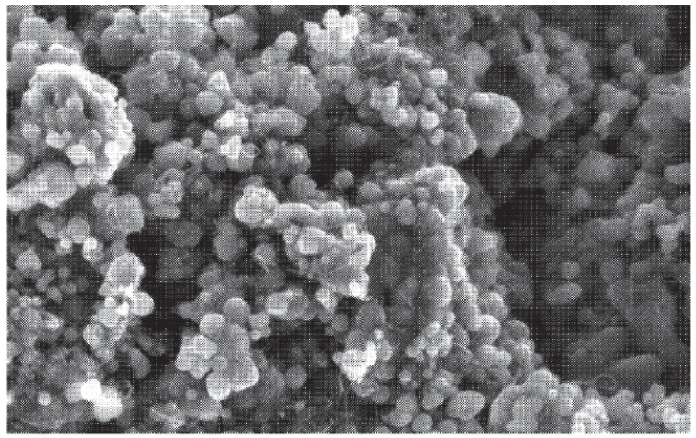


FIGURE 2

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Petitioner's Reply

Dr. Chung's own testimony confirms that PO's interpretation of MRD-2017 cannot be sustained. She admitted that Figure 2 is "not very clear," and indeed was unable to identify a single carbon nanotube in the image. LGES-1054, 53:7-57:2. Dr. Chung's statement that CNTs are "hidden inside these domains" is not grounded in what the figure shows, but rather on speculation that contradicts both the image and the specification. LGES-1054, 53:7-57:2. Nor does interpreting Figure 2 in light of the specification, as Dr. Chung proposes, save PO's position. *See* LGES-1054, 54:16-55:10. The specification of MRD-2017 confirms that CNTs are not embedded and isolated, but instead dispersed and connected to the active material. MRD-2017, [0094], [0123].

Dr. Chung's Deposition

Q. Okay. That wasn't quite my question, Dr. Chung. So let me try again.
Where do you see the LFP particles in Figure 2 of Nicolas?
MR. ZINDA: Objection. Form.
THE WITNESS: As I mentioned earlier in testimony, the image -- this image, as well as many other images, are not very clear. And it's often not totally sure as to the meaning of the individual features or the overall features in the image, that they are not very clear-cut.
And this image, it has that problem too. Okay. So just looking at the image, it's hard to see where is the lithium-ion phosphate particles.
BY MR. IN:
Q. Okay. These -- sorry. Go ahead.
A. One can look at the specification of the patent application, this Nicolas document, look at the specifications to get some information even though the image itself is not as clear as we would like.

FISH.

IPR2024-01006: Pet. Reply, 9-10; LGES-1054, 54:16-55:10

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Petitioner's Reply

For instance, PO cites MRD-2017's Figure 2 to argue that carbon nanotubes remain embedded in PVDF "domains," even after NMP dissolution, thereby preventing contact with active material. MRD-2006, ¶123. But PO's interpretation of this figure is speculative and contradicted by MRD-2017's own disclosure. Specifically, MRD-2017 explains that CNTs "form electrical bridges between the active material particles," and this explanation is supported by experimental results indicating that the CNTs are not, in fact, electrically isolated. MRD-2017, [0094], *see* [0123]-[0125]; LGES-1056, ¶25. That is, in MRD-2017, CNTs physically contact the active material, rather than being insulated from the active material, as PO contends. LGES-1056, ¶¶23-24; *see also* MRD-2018, [0092], [0131].

MRD-2017

[0094] By virtue of the process according to the invention, it is in particular possible to distribute the carbon nanotubes in such a way that they form a meshwork around the particles of active material and thus play a role both of conductive additive and also of mechanical maintenance, which is important in order to accommodate the variations in the volume during the charging/discharging stages. On the one hand, they provide for the delivery of the electrons to the active material particles and, on the other hand, due to their length and their flexibility, they form electrical bridges between the active material particles which move about as a result of their variation in volume. When they are used alone, the usual conductive additives (SP carbon, acetylene black and graphite), with their relatively low aspect ratio, are less effective in providing for the maintenance during the cycling of the transportation of the electrons from the current collector. This is because, with conductive additives of this type, the electrical pathways are formed by the juxtaposition of grains and the contacts between them are easily broken as a result of the expansion in volume of the particles of the active material.

[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 µS/cm.

IPR2024-01006: Pet. Reply, 9; MRD-2017, [0094], [0123]

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Petitioner's Reply

MRD-2018, another reference PO attempts, but fails, to use as corroborating its embedded nanotubes theory, is an application from the same inventors as MRD-2017 and discloses a similar composition for electrode manufacture, although with different proportions of constituent components. MRD-2018, [0121]-[0131]; LGES-1056, ¶26. Like MRD-2017, MRD-2018 states that "CNTs are well dispersed around the micron-sized particles of $LiFePO_4$/C (FIGS. 2A and 2B)," and that the CNTs "ensure the distribution of electrons to the particles of active material and . . . form electrical bridges between the particles of active material." MRD-2018, [0092], [0131]. This disclosure further undermines PO's assertion that the CNTs become embedded or isolated within the PVDF matrix.

MRD-2018

[0092] By virtue of the process according to the invention, it is especially possible to distribute the carbon nanofibers and nanotubes so that they form a mesh around the particles of active material and thus play both a conductive additive role and also a mechanical support role, important for accommodating the volume changes during charge-discharge steps. On the one hand, they ensure the distribution of electrons to the particles of active material and, on the other hand, owing to their length and their flexibility, they form electrical bridges between the particles of active material which shift following their change in volume. When they are used alone, standard conductive additives (SP carbon, acetylene black and graphite), with their relatively low aspect ratio, are less effective for ensuring the maintenance, during the cycling, of the transport of electrons from the current collector. Indeed, with conductive additives of this type, the electrical pathways are formed by the juxtaposition of grains and the contacts between them are easily broken following the volume expansion of the particles of active material.

[0131] SEM observations showed that the CNTs are well dispersed around the micron-sized particles of $LiFePO_4$/C (FIGS. **2**A and **2**B).

FISH.

IPR2024-01013: Pet. Reply, 11-12; MRD-2018, [0092], [0131]

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Patent Owner's Response: MRD-2008

Each of these references demonstrates that the masterbatch of Ohata-Kavan would result in oxidized CNTs heavily coated with binder resin to form a viscous "glue" like material, similar to the glue of Figure 6 of Liu-009. *Id.* ¶114. Indeed, Liu-009 states that, when carbon black is mixed with PVDF prior to adding the active material, the PVDF will be "bonded to" the carbon black. *Id.* (citing MRD-2008, 7:55-62) (explaining that "the binder has previously and preferentially ***bonded to the CB [i.e., carbon black]***"). A POSITA would understand that PVDF molecules likewise bond to oxidized CNTs due to the dipole-dipole interactions between the highly polar fluorine atoms of the PVDF molecules and the presence of functional groups on the surface of the oxidized CNTs. *Id.* ¶115. This interaction allows for significant interfacial adhesion between PVDF and oxidized CNTs. *Id.* In other words, the oxidized CNTs would not only be heavily embedded and coated with the PVDF matrix, but the PVDF coating would be more strongly bound to the oxidized CNTs due to these interactions. *Id.* (citing 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***."). Importantly, Dr. Zhu testified that he was unaware of this important interaction between PVDF and oxidized CNTs. MRD-2024, 45:16-46:14.

IPR2024-01006: POR, 33-34

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Petitioner's Reply

29. MRD-2008 clearly describes PVDF as an electrically insulating material. MRD-2008, 10:10, 5:28-32. If the conductive carbon black were truly embedded or encapsulated within PVDF to the point of being isolated from the active material—as PO suggests—the resulting structure would lack the necessary conductivity to function as an electrode. Indeed, MRD-2008 makes clear that conductivity improvements come not from embedding or insulating the conductive additive (carbon black), but from preserving carbon black's contact with the active material and maintaining conductive pathways.

31. Moreover, MRD-2008 contains no testing involving carbon nanotubes. Its findings are based entirely on carbon black, making PO's attempt to apply those findings to CNT-containing systems speculative at best. Even so, as discussed above, MRD-2008 directly contradicts PO's assertions that conductive additives become embedded and electrically isolated.

MRD-2008

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.

The acetylene black and PVDF composite film is nano-scale inhomogeneous, as show in the Transmission electron microscope (TEM) image of FIG. **4**, A. There are two distinct regions, an acetylene black rich region and a polymer rich region. The acetylene black rich region is coated with PVDF polymer providing mechanical integrity. The branch structure of the acetylene black is clearly preserved from the original acetylene black powder. (FIG. **4**, B) PVDF coatings integrate these conductive branches to form continuous electronic pathways.

IPR2024-01006: Pet. Reply, 10; LGES-1056, 29, 31; MRD-2008, 10:21-32, 11:13-17

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's own corroborating references refute its theory of complete embedding

Patent Owner's Sur-Reply

Petitioner's focus on MRD-2008 is an intentional distraction. Reply, 10. MRD-2008 was relied on solely as evidence that (1) mixing PVDF with conductive agent results in conductive glue even after mixing with NMP and (2) PVDF "bonds" to conductive agent. POR, 22, 34. PO did not rely on MRD-2008 to show the oxidized CNTs of the masterbatch would be fully embedded in the resin, because MRD-2008 uses ***significantly smaller*** ratios of PVDF:conductive-filler. MRD-2008, 9:6-9 (discussing CB:PVDF ratios of 0.5:1, 0.8:1, and 1:1). Regardless, Petitioner's argument that if the conductive carbon black were truly embedded it would be "isolated … from the active material" recycles the incorrect theory that direct **physical** contact is required for *electrical* conductivity. Reply, 10.

Patent Owner's Response

"glue" like material, similar to the glue of Figure 6 of Liu-009. *Id.* ¶114. Indeed, Liu-009 states that, when carbon black is mixed with PVDF prior to adding the active material, the PVDF will be "bonded to" the carbon black. *Id.* (citing MRD-2008, 7:55-62) (explaining that "the binder has previously and preferentially ***bonded to the CB [i.e., carbon black]***"). A POSITA would understand that PVDF molecules likewise bond to oxidized CNTs due to the dipole-dipole interactions between the highly polar fluorine atoms of the PVDF molecules and the presence of functional groups on the surface of the oxidized CNTs. *Id.* ¶115. This interaction allows for significant interfacial adhesion between PVDF and oxidized CNTs. *Id.* In other words, the oxidized CNTs would not only be heavily embedded and coated with the PVDF matrix, but the PVDF coating would be more strongly bound to the oxidized CNTs due to these interactions. *Id.* (citing MRD-2009, 2 ("In a CNT–PVDF–NMP suspension, PVDF molecular chains would ***coat onto CNT surface due to the strong***

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's theory that there is no contact between nanotubes and electrode active material is illogical and technically inaccurate

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Active material attached to CNTs is necessary for a viable battery

Petitioner's Reply

Given this basic physical operation of battery electrodes, the folly of PO's position becomes apparent. PO argues that a "resin layer separat[es] the active material from the surface of the CNTs" such that "it is impossible for the active material to attach to the surface of the CNTs." POR, 44. This makes no sense. PO acknowledges that PVDF binder is non-conductive. *See* LGES-1054, 37:22-25 ("PVDF is not conductive"); POR, 19. A configuration in which CNTs are fully embedded within a non-conductive resin and thus electrically isolated from the active material is not viable and is inconsistent with battery fabrication practices that are specifically intended to enhance conductivity. LGES-1056, ¶¶11, 15-16.

It is well-established that in battery electrode materials, 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]). Indeed, Dr. Chung readily acknowledged this to be the case, explaining at deposition that "the conductive additive ***needs to be in physical contact with the active material*** so that the charges can come out or go in of this active material particle." LGES-1054, 17:1-15. Said another way, without contact between the conductive additive (*e.g.*, nanotubes) and the active material, the electrode material would be inoperable in a battery. *See* LGES-1054, 16:2-12 , 36:18-37:5 ("you need to have a plurality of nanotubes coming together and making electrical contact between one another -- that is, touching one another … so as -- as a whole, the network would provide a continuous electrically conductive path.").

IPR2024-01006: Pet. Reply, 6-7

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Active material attached to CNTs is necessary for a viable battery

Deposition of Dr. Chung

Q. And in order to provide this electrically conducting path, does the conductive additive need to be in physical contact with the active material?

MR. ZINDA: Objection. Form.

THE WITNESS: Active material is very often not conductive as in this particular example, the lithium cobalt oxide. And so in order for this nonconductive species to be able to function so that when it undergoes the electrochemical reaction, whatever charges that come out of the win to this then needs to be a conduit for the charges to go in or to go out. Okay. Obvious active material. And that's why the conductive additive needs to be in physical contact with the active material so that the charges can come out or go in of this active material particle.

IPR2024-01006: LGES-1054, 17:1-15; Pet. Reply, 6

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Active material attached to CNTs is necessary for a viable battery

Petitioner's Reply

Moreover, PO's position is incompatible with Ohata's stated goal of improving electrical conductivity of electrode materials. Ohata aims to use nanotubes to "form an excellent conductive path in an electrode." LGES-1004, [0016]. Yet this could not happen in PO's hypothetical structure: nanotubes that are "fully embedded" within a non-conductive binder and thus not able to contact one another or the active material particles, as PO proposes (*see* POR, 19), would be unable to provide such a conductive path. LGES-1056, ¶12. Instead, as Dr. Zhu explains and as acknowledged by Dr. Chung, an electrode material without contact between nanotubes and active material would be non-functional, while Ohata's material has good electrochemical performance. *Id.*; LGES-1054, 14:15-18:16; LGES-1004, Table 3; LGES-1056, ¶12.

Ohata

[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.

IPR2024-01006; LGES-1004, [0016]; Pet. Reply, 7

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO argues that Petitioner ignores electron tunneling

Patent Owner's Sur-Reply

a. Petitioner ignores "electron tunneling."

MRD-2010 is a scientific publication involving functionalized CNTs (2 wt%) immersed in PVDF (98 wt%). MRD-2010, 9. Despite being fully embedded in PVDF, "electronics can ***easily*** travel from one tube to another tube without any obstacle (***through tunneling***)" when the "CNTs are touching each other ***or very close ([<]2nm [sic] in distance)***." *Id.*, 12; *see also* MRD-2012, 5 (describing "tunneling of electrons" as a "conduction mechanism" in PVDF-CNT mixtures); MRD-2043, 14:8-16 (explaining electron tunneling).

b. Petitioner ignores "the percolation threshold."

It is undisputed Ohata-Kavan embeds CNTs within PVDF to form a composite (PVDF-CNT binder) exhibiting increased electrical conductivity. POR, 20-23. This involves mixing a sufficient amount of CNTs (conductive filler) with PVDF (non-conductive binder) to reach the "percolation threshold"—*i.e.*, where conductivity is increased such that the ***overall*** composite exhibits "metal-like behavior" rather than the "insulator-like" property of PDVF. *See* MRD-2011, 2, 4, 7-8 ("This theory of percolation threshold is well documented"); MRD-2044, 8:15-9:18 (explaining percolation threshold); MRD-2014, 4; LGES-2021 ¶[0035]. This is achieved by small amounts of CNT. MRD-2011, 7 (threshold "approache[d]" at ***5wt% CNTs*** in PVDF); MRD-2012, 5 ("conductivity of insulating PVDF has increased" by magnitudes "with the addition of ***3wt% CNTs***"); LGES-2021 ¶[0027] (***5wt%*** achieves "significant increases in the electrical conductivity").

FISH.

IPR2024-01006; PO's Sur-Reply, 14-16

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Electron tunneling in lithium ion batteries

Dr. Zhu's Second Deposition

Q Okay. And also earlier today, do you recall Opposing Counsel asking you about the tunneling effect?

A Yes. I remember.

Q Okay. And do you recall responding that it is something that can happen at -- and I quote -- "short distances?"

A Yes. I remember.

Q Okay. Could you explain what you -- what you meant by "short distances?"

A Yeah. That short distance usually refers to the similar size of atoms. If we -- at one point, you can see the carbon atom's size, that is the short distance I am referring.

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.

Q And would you expect tunneling effects to occur in lithium-ion battery electrode materials that include carbon nanotubes in them?

MR. ZINDA: Objection, form.

A I -- I don't think tunneling effect is important for the electrode of lithium-ion battery. Same reason as I mentioned -- this is a microscopic device, and tunneling effect happen at a very short distance.

IPR2024-01006; MRD-2044, 12:23-14:3; Petitioner's Observations, 1

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

’483 Patent: Active materials are “ionically attached” to the surface of CNTs

Dr. Zhu

95. As a consequence, a POSITA would have recognized or otherwise found it obvious that one way by which the ion active materials of Kavan (e.g., $LiFePO_4$) become attached to the surface of Ohata-Kavan’s nanotubes would be through ionic bonding/attachment. Indeed, it was well-known that the oxidized nanotubes, as in Ohata-Kavan, “have a negative electrostatic charge on the outer surface of the nanotube wall” and, thus, a POSITA would have recognized that such “negative electrostatic charge on the outer surface of the nanotube wall” would be used to enhance attachment of positively charged metal ions in the ion active material (e.g., $LiFePO_4$) to the nanotube surface via an ionic/electrostatic interaction. LGES-1024, [0057]; *see* LGES-1030 (“By oxidizing the nanotubes for

Dr. Chung

Q. And same question for the first part of that sentence regarding ionically bonded.

What would cause the active material crystals to become ionically bonded to the surface of oxidized carbon nanotubes?

MR. ZINDA: Objection. Form.

THE WITNESS: Well, again, these bonds, such as the dangling bonds, kind of stick out from either party, and there can be a charge associated with the end of that bond. As a result, the bonds of the two parties can come together ionically to form an ionic bond.

THE WITNESS: If you have some metal ions kind of -- yeah, lying on the surface of this LFP, those ions would look -- let's say they're positive ions -- they would look for a negative charge somewhere in its vicinity to interact electrostatically.

IPR2024-01013: Pet. Reply, 22; LGES-1003. ¶95; LGES-1058, 15:9-19, 27:11-21

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2B

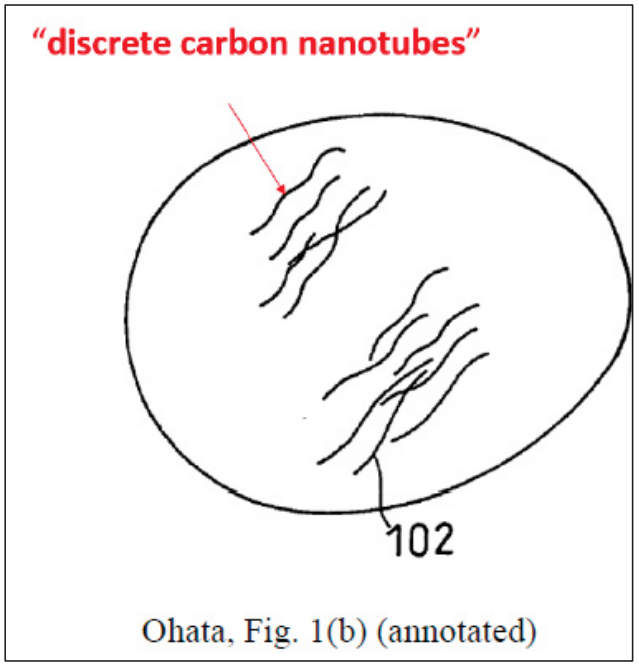
The Ohata-Kavan nanotubes are discrete

[1a] ***discrete carbon nanotubes*** *('909 Patent)*	*[1a]* ***discrete carbon nanotubes*** *('924 Patent)*	*[1a]* ***discrete,*** *non-agglomerated, and exfoliated carbon nanotubes having a surface* *('483 Patent)*

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete



Ohata, Fig. 1(b) (annotated)

Ohata

[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.

[0017] The resin with the unraveled and straightened carbon nanotubes dispersed therein also serves to increase the mechanical strength of an electrode plate. The electrode plate comprising such carbon nanotubes has a great strength against bending and therefore electrode damage is unlikely to occur during the production of an electrode group. In addition, even when the electrode material mixture layer repeatedly expands and contracts during charging and discharging, the electrode is unlikely to be degraded and thus it is possible to produce a secondary battery excellent in cycle characteristics.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Petition

The Ohata-Kavan **composition** includes the masterbatch, which contains **discrete** nanotubes, as taught by Ohata. LGES-1003, ¶80. Ohata describes "producing a masterbatch by blending at least carbon nanotubes with a resin." LGES-1004, [0015]. Prior to this blending, the nanotubes "are tangled and twisted with one another." *Id.*, [0016]; *see* FIG. 1(a). By "blending them with a resin" to form the masterbatch, "[t]he carbon nanotubes are ***unraveled and straightened***." *Id.*, FIG. 1(b), *infra*. The result is a "resin with the unraveled and straightened carbon nanotubes ***dispersed therein***," *e.g.*, a masterbatch containing **discrete carbon nanotubes**. *Id.*, [0017]; *see* [0016], [0022] ("As the resin in which ***the carbon nanotubes are dispersed***, a fluorocarbon resin can be used"), [0002] ("to reduce the amount of conductive material used, it is proposed to use a masterbatch process, whereby ***a conductive material is uniformly predispersed in a binder***"); LGES-1003, ¶80. For instance, to the extent **discrete** is interpreted as referring to nanotubes that are generally individually distinct, a POSITA would have found it obvious that at least some of these "unraveled and straightened carbon nanotubes" are **discrete** because they are generally "dispersed" as individual nanotubes in the resin, as described and illustrated by Ohata. LGES-1004, [0017]; *see* LGES-1001,

Ohata

[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.

[0017] The resin with the unraveled and straightened carbon nanotubes dispersed therein also serves to increase the mechanical strength of an electrode plate. The electrode plate comprising such carbon nanotubes has a great strength against bending and therefore electrode damage is unlikely to occur during the production of an electrode group. In addition, even when the electrode material mixture layer repeatedly expands and contracts during charging and discharging, the electrode is unlikely to be degraded and thus it is possible to produce a secondary battery excellent in cycle characteristics.

FISH.

IPR2024-01006: Pet., 19-20; LGES-1004, [0016]-[0017]

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Petition

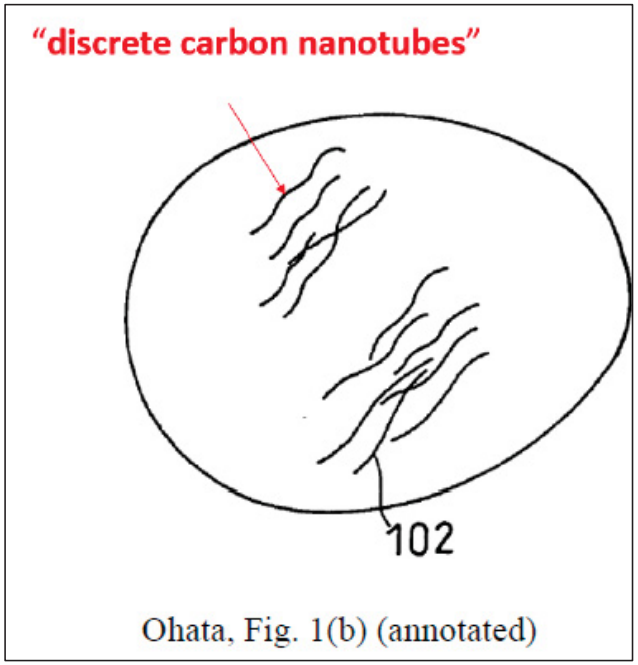
Alternatively/additionally, a POSITA would have understood or found obvious that the nanotubes in the combination composition are **discrete** because, at least in some instances, oxidation of nanotubes can help promote non-agglomeration of nanotubes. *See* [1d], *infra*; LGES-1003, ¶81. Oxidized nanotubes are functionalized by species such as carboxylic acid or hydroxyl groups. *See* LGES-1010, Abstract; LGES-1014, 821-822; LGES-1015, 4186-4187; LGES-1007, [0027]-[0028]; LGES-1003, ¶82. As indicated by these references and as explained by Dr. Zhu, the presence of these functionalities can contribute to physically and chemically inhibiting aggregation of nanotubes into clumps; instead, oxidized nanotubes are generally present as individually distinct nanotubes. LGES-1003, ¶82. In light of this behavior of oxidized nanotubes, a POSITA would have understood or found obvious that oxidized nanotubes in the combination can be present as generally individually distinct nanotubes, *e.g.*, that the nanotubes are **discrete**. LGES-1003, ¶83.

Dr. Zhu Declaration

the side walls"). As I explained above, the presence of these functionalities helps to physically and chemically inhibit the nanotubes from aggregating into clumps, and instead oxidized nanotubes are generally present as individually distinct nanotubes. *See* LGES-1014 (Gojny), 821-822 ("[t]he presence of carboxylic acid groups on the surface" "enables a better dispersion of CNTs" showing images of "oxidised [sic] MWCNTs … [that] are completely dispersed in the [epoxy resin] matrix due to the appearance of carboxylic groups on the surface"); LGES-1015 (Xing), 4186-4187 ("treated [oxidized] CNTs had better dispersion"); LGES-1007 (Farrugia), [0027] (describing that functionalization of carbon nanotubes "results in one or more chemical moieties attached to the carbon nanotubes," which "results in better dispersion of the nanotubes throughout the matrix").

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete



Ohata, Fig. 1(b) (annotated)

Patent Owner's Response

Petitioner fails to carry its burden of demonstrating that Ohata-Kavan results in "discrete" CNTs. As an initial matter, Petitioner's reliance on cartoon Figure 1 of Ohata is misplaced. Ohata itself calls Figure 1 a "schematic" diagram, which conceptually illustrates the ***concept*** of disentangling CNTs by shearing. MRD-2006 ¶¶147-148 (citing LGES-1004 ¶¶[0012]-[0013]). A POSITA would not rely on such a schematic as a real-world representation of actual masterbatch results. *Id.* ¶148. It would instead be understood for what it is—an illustration intended to conceptually depict the well-known concept of CNT disentanglement via shear forces. *Id.*

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Petitioner's Reply

PO takes issue with the Petition's "reliance on cartoon Figure 1 of Ohata." POR, 53. To be sure, Figure 1 is just a schematic diagram, but one that the Petition demonstrated would have been understood as representing the discrete nanotubes of Ohata's composition. *See* Petition, 20-22. Regardless, PO's myopic focus on Fig. 1 ignores what the Board already recognized at institution (Institution Decision, 17): the Petition relied upon not only Ohata's Fig. 1, but also numerous other citations, bolstered by explanation and expert corroboration, to demonstrate that the nanotubes in the combination are discrete. *See* Petition, 19-22; LGES-1003, ¶¶80-83. Indeed, PO's main objection to the Petition's showing of discreteness in the Ohata-Kavan nanotubes appears to be based not in any criticism of Ohata, but instead in its own excessively narrow interpretation of what constitutes a discrete nanotube.

Institution Decision

Patent Owner's arguments appear to be focused on the phrase "generally uniformly dispersed." Prelim. Resp. 23–24. We, however, do not discern any such term in Petitioner's arguments on this limitation. Instead, Petitioner directs us to Ohata's disclosure that its nanotubes are "unraveled and straightened" and then "dispersed" within the resin, and to Figure 1(b) showing isolated nanotubes, which support Petitioner's position.

Patent Owner also argues that Kavan Figure 2 shows its oxidized nanotubes are not discrete, but rather are highly entangled. Prelim. Resp. 23. This argument, however, fails to account for the combined teachings of Ohata and Kavan, in which Petitioner relies on Ohata's disclosure of dispersing discrete, unraveled, and straightened carbon nanotubes in resin. Pet. 19–20.

In view of the foregoing, we determine Petitioner has demonstrated sufficiently for purposes of institution that Ohata and Kavan disclose or suggest limitation [1a].

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Patent Owner's Response

> A POSITA would instead understand that the only reason Ohata seeks to "unravel" and "straighten" the CNTs is so that they can be better dispersed to form a continuous network better distributed throughout the binder. *Id.* ¶153. This is because it was well-known that heavier entanglement of CNTs "is undesirable because it results in formation of voids and aggregates in the composition which adversely effects the ***electrical conductivity properties*** of the composite...." *Id.* (quoting MRD-2019 ¶[0027]; citing MRD-2022 ¶[0073] (explaining CNT agglomeration makes it "difficult to form an effective conductive network")). Just as a heavily wound spiderweb is more difficult to spread apart, heavily entangled CNTs make it more difficult to disperse a CNT network. *Id.* Ohata seeks better dispersion of its CNTs so that it can provide a ***continuous network*** of CNTs throughout the entire binder, not to achieve discrete CNTs attached to the surface of active material. *Id.*

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Petitioner's Reply

PO argues that the Ohata-Kavan composition would not include discrete nanotubes because of the presence of a continuous network of nanotubes, but this argument relies on PO's 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 Ohata-Kavan. Indeed, the '909 Patent itself indicates that discrete nanotubes enhance conductivity, well-aligned with the purpose of the Ohata-Kavan nanotubes. LGES-1056, ¶31; *See* LGES-1001, 1:44-50, 2:24-28, 2:4-11, 2:12-17, Table 1.

'909 Patent

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

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.

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.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The term “discrete” does not preclude a network of nanotubes

’924 Patent

Example 1

Discrete Carbon Nanotubes with Attached Copper Oxide Nanoparticles Suitable for a Lithium Ion Battery Anode Material

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

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.

Attempts to overcome the above deficiencies have included synthesis of nanosized 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.

IPR2024-01011: Pet. Reply, 25-26; LGES-1056, ¶52; LGES-1001, 1:42-63, 2:17-29, 9-29

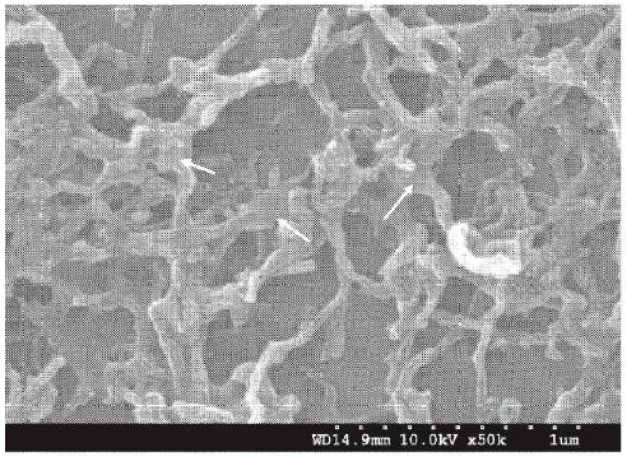
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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Patent Owner's Response

Even if the *masterbatch* of Ohata-Kavan would comprise discrete CNTs, Petitioner fails to demonstrate that the same can be said of the *electrode material mixture paste*, which is created by mixing the masterbatch with the active material and a dispersion medium. *Id.* ¶154 (citing LGES-1004 ¶[0023]). As evidenced by Figure 5 of Gan below, the random orientation of CNTs in a fluid facilitates the CNTs connecting to one another to forming a continuous conductive network. *Id.* (citing MRD-2019 ¶¶[0014], [0041]).



Petitioner's Reply

PO further attempts to argue against the discreteness of the Ohata-Kavan nanotubes fail in their reliance on Gan (MRD-2019)—an entirely different reference. For instance, in addressing the Petition's explanation that oxidation further promotes that the nanotubes are discrete, PO cites to Gan, which, it alleges, "teaches that functionalized CNTs within a polymer will be drawn closer together … facilitating the formation of a continuous network." POR, 58-59 (citing MRD-2019, [0018]-[0021]). Yet as noted *supra*, a continuous, conductive network of nanotubes is consistent with a population of discrete nanotubes, *e.g.*, nanotubes in point-to-point contact. Even to the extent that Gan is relevant to the structure of the Ohata-Kavan composition (which PO has not established), the morphology of Gan's nanotube network does not demonstrate absence of discrete nanotubes in Ohata-Kavan. LGES-1056, ¶32.

IPR2024-01006: POR, 56-57; Pet. Reply, 11-12

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are discrete

Patent Owner's Sur-Reply

Petitioner complains of "PO's myopic focus on Fig. 1" of Ohata (Reply, 11), but Petitioner relied almost exclusively on that figure as evidence of "discrete" CNTs. Petition, 19-22. Regardless, Petitioner conspicuously avoids responding to a key point—"Petitioner fails to demonstrate that [the masterbatch CNTs] would remain discrete after ***being further mixed*** with the active material to create the *electrode material mixture paste*." POR, 54-55. Petitioner cannot carry its burden because its arguments focus on the ***masterbatch*** of Ohata-Kavan, not the ***electrode mixture paste*** containing active material.

Recognizing this, Petitioner seeks to shift the burden to PO by arguing that Gan (MRD-2019) "does not demonstrate ***absence of*** discrete nanotubes" in the electrode material mixture. Reply, 12. However, it is Petitioner's burden to first prove ***the presence of*** discrete CNTs in the ***electrode mixture***.

Because Gan disproves Figure 1(b) as representative of how the CNTs of Ohata-Kavan would appear in the ***electrode mixture***, Petitioner attempts to dismiss it as an "entirely different reference." *Id.*, 11. But Petitioner relies on Gan to try and invalidate claims in IPR2024-01011 (Paper 2, 36-42), proving Gan is far more relevant than Petitioner alleges.

Petitioner argues the arrangement of CNTs in Gan's SEM images are irrelevant because they are not oxidized. Reply, 12. But a POSITA would have expected those CNTs to be *more interconnected* when oxidized because Gan teaches functionalization "draw[s] the ***CNTs closer together***." POR, 57.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2C

A POSITA would have been motivated to combine Ohata and Kavan

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Three of the motivations for oxidized nanotubes

First, when the combination paste is a negative electrode material mixture paste having TiO_2 as the active material (*see* LGES-1005, 2), oxidized nanotubes provide a "beneficial effect of nanotube networking." LGES-1005, 5; *see* Fig. 5; LGES-1003, ¶40. For instance, "the rate of electrochemical charge transfer is higher" in composites of TiO_2 and oxidized nanotubes as compared to composites with unfunctionalized nanotubes. LGES-1005, 9. A POSITA would have been motivated to use oxidized nanotubes together with TiO_2 such that the combination paste would enjoy fast electrochemical charge transfer. LGES-1003, ¶40.

Second, again when the combination paste is a negative electrode material mixture paste having TiO_2 as the active material (*see* LGES-1005, 2), oxidized nanotubes "exhibit better affinity towards TiO_2" than do unoxidized nanotubes. LGES-1005, Abstract. A POSITA would have been motivated to use oxidized nanotubes together with TiO_2 to obtain a combination paste with a robust interaction between the nanotubes and the electrode active material, which is beneficial for both electrochemical performance and mechanical stability, as explained by Dr. Zhu. LGES-1003, ¶41 (citing LGES-1037, 4).

Third, when the combination paste is instead a positive electrode material mixture paste having a phosphate olivine as the active material (*see* LGES-1005, 2), electrochemical performance of an electrode is improved by oxidized nanotubes. LGES-1003, ¶42. For instance, Kavan reports that "the $LiFePO_4$ olivine exhibits excellent performance in a composite with MWNT-ox," with "impressive values" for the capacity of the $LiFePO_4$/MWNT-ox composite. LGES-1005, 7; *see* 8 ("It is obvious that the functionalization of nanotubes is, indeed, crucial for enhancing the electrochemical activity of the composite materials with $LiFePO_4$"), 7, Figs. 8 and 10. A POSITA would have been motivated to incorporate oxidized nanotubes into the combination paste to achieve the apparent dramatic performance advantages taught in Kavan. LGES-1003, ¶42.

IPR2024-01006: Pet., 8-10

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Three of the motivations for oxidized nanotubes

Patent Owner's Response

Petitioner's "reasons for the combination" fail because they ignore the effects of the masterbatch process. *Id.* ¶131. The "first" reason is that "oxidized nanotubes provide a 'beneficial effect of nanotube networking'" when "the combination paste is a negative electrode material mixture paste having TiO_2 as the active material." Pet., 8-9. The "second" reason is that "oxidized nanotubes 'exhibit better affinity towards TiO_2.'" *Id.*, 9. And the "third" reason is that when a phosphate olivine is used as the active material, "electrochemical performance of an electrode is improved by oxidized nanotubes." *Id.* All three reasons are premised on the active material ***being able to "anchor" to the functional groups*** of the oxidized CNTs. MRD-2006 ¶132.

Petitioner's Reply

Indeed, PO specifically acknowledges that its sole objection to the "first three alleged reasons" for the combination advanced in the Petition is because "[a]ll three reasons are premised on the active material being able to 'anchor' to the functional groups of the oxidized CNTs." POR, 46. But as discussed *supra* (*see* §II.A), this is not correct: the active material is, in fact, able to anchor to the functional groups on the nanotubes. Accordingly, PO's objections amount to nothing more than a non-sequitur.

IPR2024-01006: POR, 45-46; Pet. Reply, 13

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fourth motivation for oxidized nanotubes

Fourth, a POSITA would have been motivated to use oxidized nanotubes in furtherance of Ohata's goal to have well-dispersed nanotubes in the composite. LGES-1003, ¶43. Ohata's masterbatch process is designed to generate an electrode material paste with dispersed nanotubes, *e.g.*, because of the electrical and mechanical advantages they may be able to provide: "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. The resin with the unraveled and straightened carbon nanotubes dispersed therein also serves to increase the mechanical strength of an electrode plate." LGES-1004, [0016]-[0017]; LGES-1003, ¶43. Oxidized nanotubes are consistent with this objective: they can be more readily dispersible than unfunctionalized nanotubes, as explained by Dr. Zhu. LGES-1003, ¶¶44-45 (citing LGES-1044, 1854; LGES-1014, 821-822; LGES-1015, 4186-4187; LGES-1007, [0027]. A POSITA would have been motivated to use oxidized nanotubes because, at least in some cases, doing so is consistent with, and can enhance, Ohata's goal of dispersed nanotubes in the masterbatch. LGES-1003, ¶46.

Ohata

[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.

[0017] The resin with the unraveled and straightened carbon nanotubes dispersed therein also serves to increase the mechanical strength of an electrode plate. The electrode plate comprising such carbon nanotubes has a great strength against bending and therefore electrode damage is unlikely to occur during the production of an electrode group. In addition, even when the electrode material mixture layer repeatedly expands and contracts during charging and discharging, the electrode is unlikely to be degraded and thus it is possible to produce a secondary battery excellent in cycle characteristics.

IPR2024-01006: LGES-1004, [0016]; Pet., 10-11

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fourth motivation for oxidized nanotubes

Patent Owner's Response

The "fourth" reason also lacks merit, because Petitioner contends that Ohata alone *already* achieves uniformly dispersed CNTs. Pet., 20 (arguing in Ohata "a conductive material is ***uniformly predispersed*** in a binder"') (quoting LGES-1004 ¶[0002])). Because Petitioner alleges that Ohata already achieves uniform dispersal without oxidation, there would have been no motivation to oxidize to improve dispersion. MRD-2006 ¶136. This is particularly so given that it was well-known that oxidation would adversely affect the intrinsic properties of CNTs. *Id.*

Petitioner's Reply

to Ohata's description of uniform dispersion of nanotubes in its masterbatch. Even if there were no room for improvement in the dispersion of nanotubes in the masterbatch (which is not necessarily true), PO ignores that the presence of oxidative functionalities on the nanotubes would still promote dispersion of the nanotubes in the later-formed NMP slurry. LGES-1056, ¶¶36-37.

Dr. Zhu Declaration

49. I note that oxidized nanotubes may have a lower conductivity than their unoxidized counterparts. However, use of oxidized nanotubes also brings a number of performance advantages, such as those I discussed above. I expect that a POSITA would have known how to balance the trade-offs between lower conductivity and the various advantages associated with oxidized nanotubes, and it is my opinion that given the strong advantages that are directly applicable to the context of the Ohata-Kavan combination, a POSITA would have chosen to implement oxidized nanotubes in the combination.

IPR2024-01006: LGES-1003, ¶49; Pet., 8; POR, 47-48; Pet. Reply, 15;

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fifth motivation for oxidized nanotubes

Fifth, a POSITA would have been motivated to use oxidized nanotubes in the combination paste because oxidative treatments can "remove amorphous carbon and metallic impurities," thus leading to a higher-purity starting material. LGES-1010, 2; LGES-1003, ¶47.

LGES-1010

The deliberate incorporation of surface oxygen into CNTs has been achieved through a variety of methods, chiefly wet chemical oxidation [14,19–21], plasma treatments [22,23], and rational functionalization strategies involving synthetic organic chemistry [24]. Among these various treatment methods, wet chemical techniques using different oxidizing acids (e.g., HNO_3) and strong oxidants (e.g., O_3) tend to be the most prevalent due to their easy implementation in laboratory and industrial settings. These same oxidative treatments are also used to help remove amorphous carbon and metallic impurities from as-produced CNTs [25–27]. Despite the fact that a wide variety of oxidizing conditions (defined by the oxidant and the reaction conditions) are used to treat and modify CNTs, the rationale behind the choice of a particular set of oxidizing conditions is rarely discussed, with few exceptions [28]. Furthermore, the effect of different oxidizing conditions on CNT surface chemistry or structure is seldom considered.

IPR2024-01006: LGES-1010, 2; Pet., 11

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fifth motivation for oxidized nanotubes

Patent Owner's Response

The "fifth" reason is that oxidation can help purify the CNTs. Pet., 11. But nothing in Ohata or Kavan teaches or suggests that the CNTs have impurities. MRD-2006 ¶137. To the contrary, Kavan teaches that it uses "purified" CNTs, which refers to CNTs lacking impurities. *Id.* (citing LGES-1005, 2 ("For oxidative functionalization, 1g of *purified* nanotubes was mixed….")). Thus, there is no evidence to support this "purification" motivation.

Petitioner's Reply

Regarding the Petition's explanation that oxidation can remove impurities (*see* Petition, 11), PO argues that "nothing in Ohata or Kavan teaches or suggests that the CNTs have impurities." POR, 48. This is not the correct analysis. Ohata need not have recognized that its nanotubes possess impurities for the reduction of impurities to motivate the combination. *See Intel Corp. v. PACT XPP Schweiz AG*, 61 F.4th 1373, 1380 (Fed. Cir. 2023) ("Additionally, 'universal' motivations known in a particular field to improve technology provide 'a motivation to combine prior art references *even absent any hint of suggestion* in the references themselves.'" (citing *Intel Corp. v. Qualcomm Inc.*, 21 F.4th 784, 797 (Fed. Cir. 2021)) (emphasis original)).

And as to PO's statement that impurities are not a concern because "Kavan teaches that it uses 'purified' CNTs" (POR, 48), this is beside the point: the Petition "uses Ohata as a starting point," not Kavan. Petition, 7.

IPR2024-01006: POR, 48; Pet. Reply, 15-16

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Motivations for oxidized nanotubes

Patent Owner's Response

Petitioner further contends that "a POSITA would have been motivated to use oxidized nanotubes in the Ohata-Kavan paste." Pet., 8. But the issue is not simply whether a POSITA would have been motivated "to use oxidized nanotubes," but whether there was a motivation to use oxidized CNTs ***at the claimed level of 1% to 15%***. Petitioner cannot rely on motivations to oxidize *generally*. The only basis for the assertion that ***the claimed range*** was obvious is based on Kavan's oxidation level—but that oxidation level is premised on the goal of "anchoring" to provide the described benefits. Because the Ohata-Kavan masterbatch process precludes these benefits from coming to fruition, Petitioner fails to provide any motivation for oxidizing at claimed level. Further, given the well-known disadvantages of oxidizing CNTs, and that a POSITA understood that the "anchoring" benefits of Kavan would not be achieved in Ohata-Kavan, a POSITA would have further lacked motivation to oxidize at Kavan's levels. MRD-2006 ¶138.

Petitioner's Reply

PO's argument that "Petitioner cannot rely on motivations to oxidize generally" without addressing "whether there was a motivation to use oxidized CNTs at the claimed level of 1% to 15%" (POR, 48) ignores the context underpinning the motivations advanced in the Petition. That is, PO overlooks that the first three reasons advanced in the Petition (*see* Petition, 8-10) are drawn specifically from Kavan's results, which indicate performance improvements achieved not when using generic oxidized nanotubes, but when using nanotubes oxidized at particular oxidation levels falling within the claimed range.

IPR2024-01006: POR, 48-49; Pet. Reply, 16

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation level of Kavan's nanotubes

A B S T R A C T

Multi-walled carbon nanotubes functionalized by carboxylic groups, exhibit better affinity towards TiO_2 (P90, Degussa) as compared to that of pristine nanotubes. Also the electrochemical performance of TiO_2 is improved by nanotube networking, but the Li-storage capacity of TiO_2 is unchanged. Whereas the composite of TiO_2 with non-functionalized nanotubes demonstrates simple superposition of the behavior of pure components, the composite with functionalized nanotubes shows unique faradaic pseudocapacitance which is specific for this composite only. The surface functionalization of nanotubes enhances charge storage capacity and reversibility of a composite with $LiMnPO_4$ (olivine), but mediates also the electrolyte breakdown at potentials >4.2 V. Whereas the electrochemical activation of $LiMnPO_4$ (olivine) by functionalized nanotubes is quite modest, excellent performance was found for $LiFePO_4$ (olivine) in composite materials containing only 2 wt% of functionalized nanotubes.

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Petition

Kavan's 7 atomic percent oxygen corresponds to an **oxidation level** of **9.1%** **by weight of the carbon nanotube**. LGES-1003, ¶¶104-106.

This oxidation level value (9.1% by weight) (*see* LGES-1005, 4) for nanotubes of the Ohata-Kavan combination falls within the recited range of oxidation levels (**1% to 15% by weight**). LGES-1003, ¶107. In the combination,

Kavan

a pH of 6, and dried at 120 °C for 48 h. The non-functionalized blank material is abbreviated MWNT-bl and the material functionalized by oxidation with HNO_3 is coded MWNT-ox. The extent of functionalization was determined by titration with a freshly prepared aqueous solution of NaOH (0.05 M) as in Ref. [50]. The amount of acid groups in the nanotubes was found to be 2.54×10^{-4} mol g^{-1} in MWNT-ox, and close to 0 in MWNT-bl.

• • •

tal in-plane vibration of multi-walled nanotube. While MWNT-bl was almost free from surface carboxyls, the sample MWNT-ox contained 2.54×10^{-4} mol g^{-1} acidic groups as determined by titration analysis (see Section 2). The amount of atomic oxygen present on the MWNT-ox surface was also analyzed by X-ray photoelectron spectroscopy (data not shown) that gave a value of 7%. These results are in accordance with those reported earlier for functionalized carbon nanotubes [50]. The sample MWNT-bl further exhibits strong

IPR2024-01006: LGES-1005, 1, 2, 4; Pet., 9, 28

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2D

A POSITA would have expected success in combining Ohata and Kavan

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Expectation of success

Petition

A POSITA would have expected success in incorporating oxidized nanotubes into the Ohata-Kavan paste. LGES-1003, ¶50-52.

First, use of oxidized nanotubes in compositions for lithium ion battery electrodes was conventional. *See* LGES-1003, ¶51 (citing LGES-1005, Abstract, 9; LGES-1032, 3:28-52).

Second, the composition of Kavan's slurry is generally similar to the composition of Ohata's electrode material paste, as explained by Dr. Zhu. *See* LGES-1004, [0022], [0024], [0032]; LGES-1005, 2, 7. These similarities would have provided the POSITA with a reasonable expectation that oxidized nanotubes, as taught in the context of Kavan's slurry, would be compatible with Ohata's electrode material paste. LGES-1003, ¶52.

IPR2024-01006: Pet., 11-12

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Expectation of success

Petition

A POSITA would have expected success in incorporating TiO_2 (for a negative electrode material mixture) and/or a phosphate olivine (for a positive electrode material mixture) into the Ohata-Kavan paste. LGES-1003, ¶¶68-71.

First, use of either TiO_2 or phosphate olivines in compositions for lithium ion battery electrodes was conventional, and these materials were well-characterized as electrode active materials. *See* LGES-1003, ¶69 (citing LGES-1033, 1; LGES-1034, [0004]; LGES-1041, Abstract).

Second, Ohata indicates that various electrode active material compositions can be used with its paste: "The negative electrode active material to be used also varies depending on the battery type." LGES-1004, [0027]; see [0026] ("The positive electrode active material to be used varies depending on the battery type."). Given this flexibility, and the conventional nature of TiO_2 and phosphate olivines as electrode active materials as noted *supra*, a POSITA would have expected these materials to be compatible with Ohata's paste. LGES-1003, ¶70.

Third, the composition of Kavan's slurry is generally similar to the composition of Ohata's electrode material paste, as previously discussed. *See* LGES-1004, [0022], [0032]; LGES-1005, 2; LGES-1003, ¶71. This similarity would have provided the POSITA with a reasonable expectation that either TiO_2 or phosphate olivines, as taught in the context of Kavan's slurry, could be used with Ohata's electrode material paste. LGES-1003, ¶71.

IPR2024-01006: Pet., 15-16

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Expectation of success: Patent Owner

Patent Owner's Response

The reasonable expectation of success requirement "refers to the likelihood of success in combining reference to meet the limitations ***of the claimed invention***." *Intelligent Bio-Systems, Inc. v. Illumina Cambridge, Ltd.*, 821 F.3d 1359, 1367 (Fed. Cir. 2016). A POSITA would have had no reasonable expectation of success in combining Ohata-Kavan such that the lithium ion active materials are "attached to th[e] surface" of the oxidized CNTs as claimed. MRD-2006 ¶143. Petitioner was required to demonstrate a reasonable expectation of success *in the Petition*, but instead simply ignored the fact that Ohata-Kavan embeds the oxidized CNTs in resin before mixing with the active material. Indeed, implicit in all of Petitioner's "reasons" for a reasonable expectation of success is the incorrect assumption that Ohata-Kavan exposes the CNTs to the active material like Kavan. *Id* ¶144. A POSITA would have had no expectation of success when considering that the oxidized CNTs would be covered in resin, preventing the attachment described by Kavan. *Id*.

Petitioner's Reply

PO argues that "implicit in all of Petitioner's 'reasons' for a reasonable expectation of success is the incorrect assumption that Ohata-Kavan exposes the CNTs to the active material." POR, 51. But because the nanotubes are, in fact, directly exposed to the active material, as discussed above, PO's objections are unjustified. And PO fails to provide any rebuttal to the Petition's proposition that a POSITA would have expected success in the context of oxidized nanotubes promoting better attachment with active material particles, as is the case in the properly framed Ohata-Kavan combination.

IPR2024-01006: POR, 51; Pet. Reply, 18

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 2E

Claim 6 of the ’909 Patent and Claim 8 of the ’483 Patent are obvious over Ohata-Kavan (IPR2024-01006 and IPR2024-01013)

[6b] wherein the carbon nanotubes are uniformly dispersed without clustering on a dimension scale less than 1 micrometer (’909 Patent)

[8e] wherein the carbon nanotubes are uniformly dispersed without clustering on a dimension scale of less than about 1 micrometer (’483 Patent)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are uniformly dispersed

Petition

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.

Dr. Zhu's Declaration

117. It is my opinion that a POSITA would have understood, or at least found obvious, that the nanotubes in the Ohata-Kavan combination remain **uniformly dispersed** in the combination paste. For instance, a POSITA would have recognized that the combination manufacturing process based on Ohata, in which the masterbatch is blended with "an electrode active material" and "a dispersion medium" to form an electrode material mixture paste (LGES-1004 (Ohata), [0023]), would allow the nanotubes in the resulting paste to remain **uniformly dispersed**, as they are in the masterbatch.

IPR2024-01006: Pet., 33-34; LGES-1003, 117
IPR2024-01013: Pet., 56-57; LGES-1003, 129

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are uniformly dispersed

Patent Owner's Response

> While Ohata does teach that carbon black can be "uniformly dispersed" as part of ***a prior art*** masterbatch, Ohata does not teach or suggest the same with respect to ***its*** masterbatch, which comprises CNTs (not carbon black) mixed with resin. *Id.* ¶164 (citing LGES-1004 ¶[0005] ("The present invention relates to ... a masterbatch comprising at least carbon nanotubes and a resin;")). To the contrary, Ohata describes the difficulty of dispersing CNTs in resin. *Id.* (citing LGES-1004 ¶¶[0021] ("the addition of the carbon nanotubes is difficult, taking the dispersibility of the carbon nanotubes in the resin into consideration"); [0019] ("the dispersibility of the carbon nanotubes in the resin may be slightly low")).

IPR2024-01006: POR, 61
IPR2024-01013: POR, 64-65

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Ohata-Kavan nanotubes are uniformly dispersed

Petitioner's Reply

¶38. Second, its assertions misrepresent Ohata. In particular, PO's citation to Ohata at [0021] to argue that "Ohata describes the difficulty of dispersing CNTs in resin" (POR, 61) fails to acknowledge that the indicated difficulty is contextualized to the context of "add[ing] a resin during the step of preparing an electrode material mixture paste," not in the context of a masterbatch. LGES-1004, [0021]. Furthermore, PO's citation to Ohata at [0019] (*see* POR, 61) omits that Ohata's reference to low dispersibility of nanotubes in resin is not a generic statement, but instead is specific to the case "when the length of the carbon nanotubes is large." LGES-1004, [0019].

Ohata

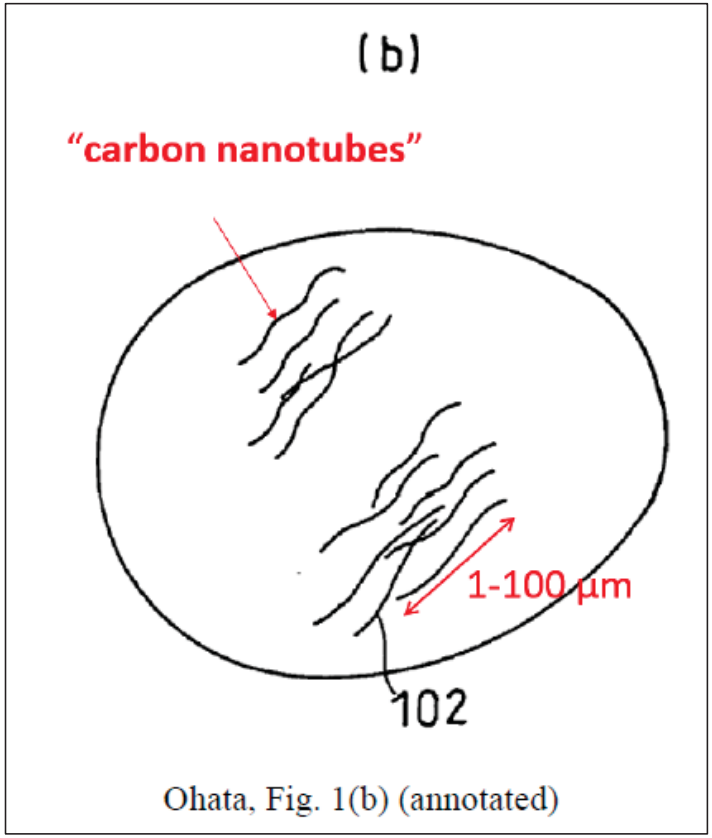
[0019] The carbon nanotubes preferably have an average length of 1 to 100 μm. When the carbon nanotubes are too short, excellent conductive paths may not be formed in some electrodes. No problem arises when the length of the carbon nanotubes is large; however, the dispersibility of the carbon nanotubes in the resin may be slightly low.

[0021] Although it is possible to add a resin during the step of preparing an electrode material mixture paste, the addition of the carbon nanotubes is difficult, taking the dispersibility of the carbon nanotubes in the resin into consideration. Therefore, the weight ratio of the carbon nanotubes to the resin contained in the masterbatch is preferably the same as or more than that of the carbon nanotubes to the resin contained in the electrode material mixture. 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.

IPR2024-01006: Pet. Reply, 18; LGES-1004, [0019], [0021]
IPR2024-01013: Pet. Reply, 23; LGES-1004, [0019], [0021]

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The dispersion is without clustering on a scale of < 1 micrometer



Ohata, Fig. 1(b) (annotated)

Petition

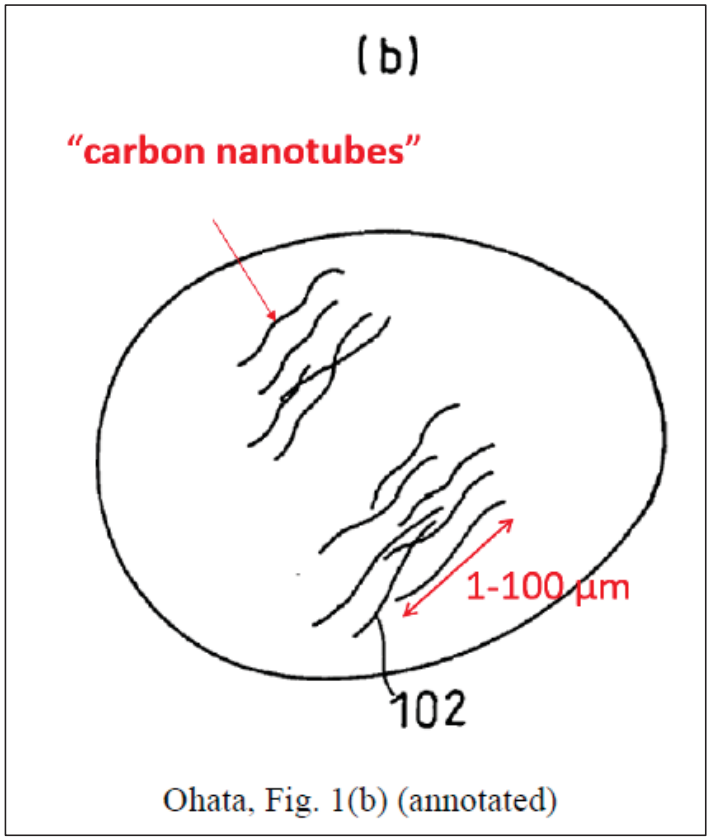
Moreover, a POSITA would have understood, or at least found obvious, that the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**.[6] Ohata's Fig. 1(b), again *infra*, which a POSITA would have understood as representative of the nanotube arrangement in the combination paste, as discussed in the previous paragraph, shows that the nanotubes are dispersed **without clustering**, *e.g.*, in that the nanotubes are present as generally individual nanotubes. LGES-1003, ¶118. Thus, because there is no clustering, a POSITA would have understood that the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**. *Id.*

IPR2024-01006: Pet., 34-36
IPR2024-01013: Pet., 56-58

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The dispersion is without clustering on a scale of < 1 micrometer



Ohata, Fig. 1(b) (annotated)

Petition

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 confirmed by Ohata's disclosure that the nanotubes are "dispersed," a POSITA further would have understood that the nanotubes in the Ohata-Kavan composition do not form clusters of greater than 1 micrometer in size, *e.g.*, the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**. LGES-1003, ¶118.

IPR2024-01006: Pet., 35-36
IPR2024-01013: Pet., 56-58

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The dispersion is without clustering on a scale of < 1 micrometer

Patent Owner's Response

Petitioner fails to provide any credible evidence that Ohata-Kavan teaches or suggests this limitation, which requires examining CNTs under a microscope *with a dimension scale*. *Id.* ¶169. Instead, Petitioner argues that Ohata's cartoon Figure 1(b) "shows that the nanotubes are dispersed ***without clustering***," and then leaps to the wholly unsupported conclusion that a POSITA would know this to mean that "the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**." Pet., 34-35. Petitioner fails to explain how a ***cartoon*** figure that is clearly not drawn to scale and does not represent *actual* results somehow meets this limitation. MRD-2006 ¶169. Petitioner instead improperly equates Figure 1(b) allegedly showing no clustering as automatically meaning the dimension scale requirement is met. *Id.*

Petitioner's Reply

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.

IPR2024-01006: POR, 64-65; Pet. Reply, 19
IPR2024-01013: POR, 68; Pet. Reply, 23-24

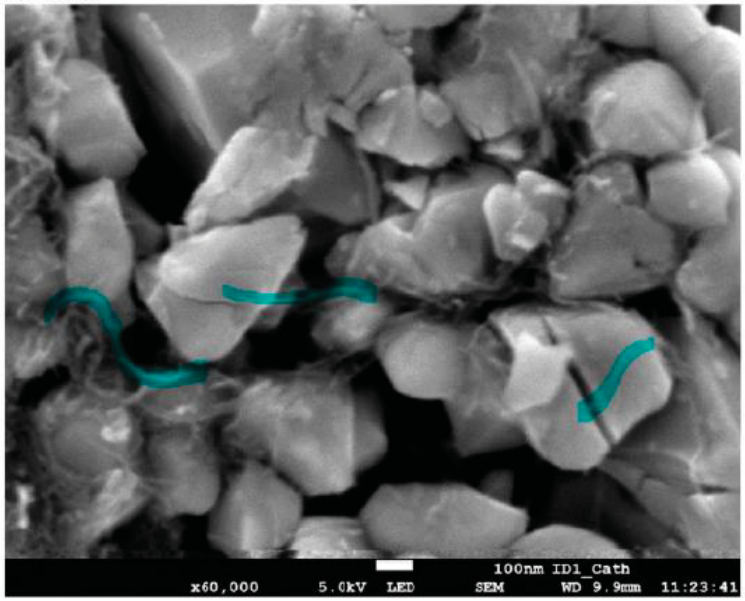
DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The dispersion is without clustering on a scale of < 1 micrometer

Petitioner's Reply

Notably, as mentioned in the Petition, the prior art cannot be held 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). PO appears to recognize this, pointing in its infringement contentions for claim 6 to nanotubes that are clearly not "uniformly" dispersed. This image highlights PO's inconsistencies on this claim: it looks to require theoretical uniformity for purposes of evading prior art while seeking relief from the same requirement when attempting to apply the claims to accused products.

Patent Owner's Infringement Contentions



IPR2024-01006: Pet. Reply, 19-20; LGES-1050, 55
IPR2024-01013: Pet. Reply, 24-24; LGES-1050, 55

FISH.
DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 3

Obviousness over Lee and Wepasnick (IPR2024-01006 only)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Table of Contents – Lee-Wepasnick

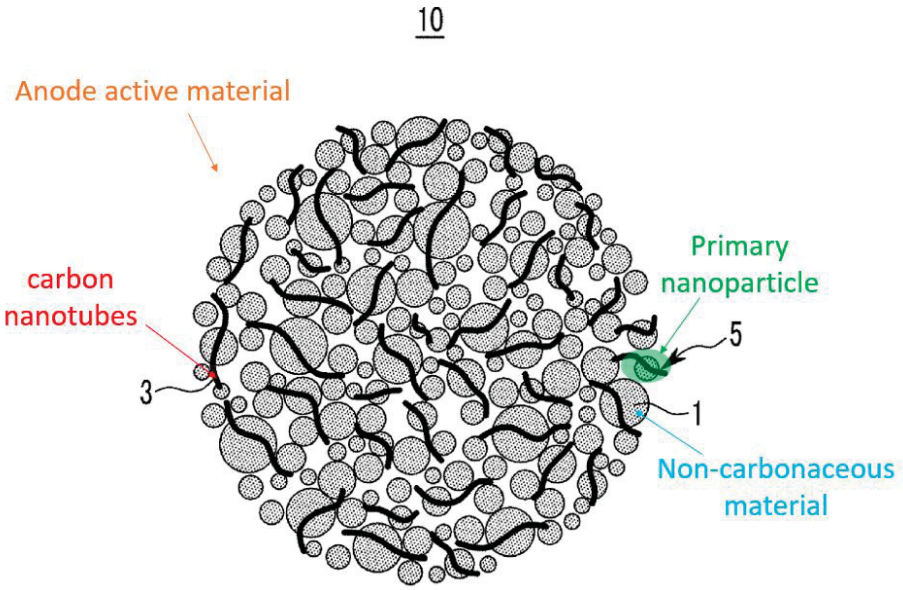
- Overview of the Lee-Wepasnick combination
- Issue 3A: The Lee-Wepasnick nanotubes are discrete
- Issue 3B: The claimed range of aspect ratios is obvious
- Issue 3C: Claim 6 is obvious over the Lee-Wepasnick combination

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lee-Wepasnick Combination

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Lee describes an anode active material including carbon nanotubes



Lee

[0040] An anode active material **10**, according to an exemplary embodiment of the present teachings, will be described with reference to FIG. **1**. Referring to FIG. **1**, the anode active material **10** includes secondary particles that are formed by the agglomeration of primary nanoparticles **5**. The primary nanoparticles **5** include a non-carbonaceous material **1** and hollow carbon nanofibers **3**, which are bound to the non-carbonaceous material by Van der Waals forces.

IPR2024-01006: LGES-1006, [0040], Fig. 1; Pet., 38; Pet. Reply, 21

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lee-Wepasnick combination

Petition

In Lee-Wepasnick, an "anode active material" includes carbon nanotubes and a non-carbonaceous material, as described by Lee. LGES-1006, [0040]. The carbon nanotubes are oxidized using one of the "commonly used wet chemical oxidants," as taught by Wepasnick, thereby functionalizing the nanotube surface with "carboxyl, carbonyl, and hydroxyl groups." LGES-1010, Abstract; LGES-1003, ¶137. In Lee-Wepasnick, a POSITA would have chosen an oxidative treatment based on Wepasnick's data about various oxidation treatments to achieve a desired "concentration and distribution of different oxygen functional groups on the CNT surface," to thereby control "the ultimate structure and properties" of the resulting material. LGES-1010, 2; LGES-1003, ¶137.

IPR2024-01006: Pet., 42

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 3A

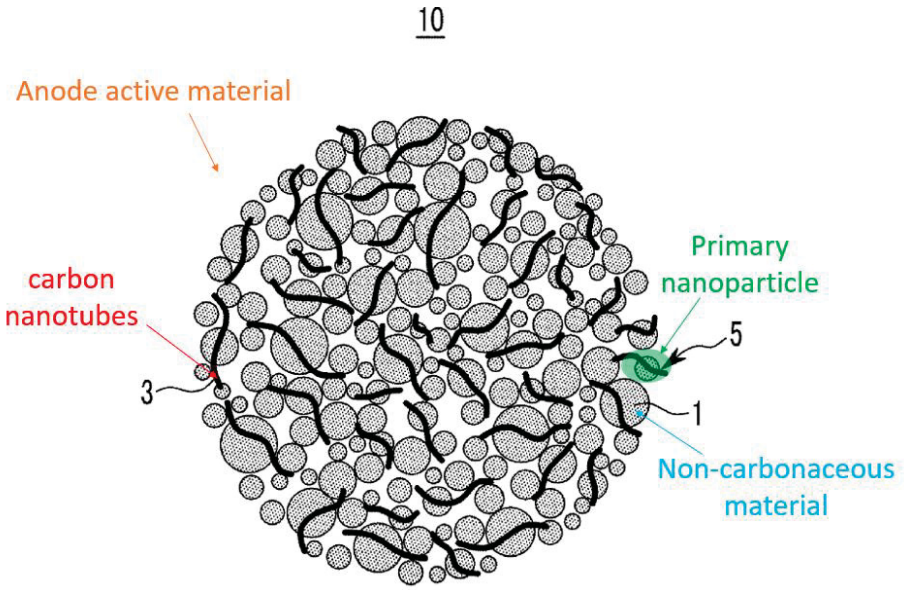
The Lee-Wepasnick nanotubes are discrete

[1a-1b] ***discrete carbon nanotubes*** *having crystals or layers of lithium ion active material attached to their surface*

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Structure of Lee's material



Lee

[0040] An anode active material **10**, according to an exemplary embodiment of the present teachings, will be described with reference to FIG. **1**. Referring to FIG. **1**, the anode active material **10** includes secondary particles that are formed by the agglomeration of primary nanoparticles **5**. The primary nanoparticles **5** include a non-carbonaceous material **1** and hollow carbon nanofibers **3**, which are bound to the non-carbonaceous material by Van der Waals forces.

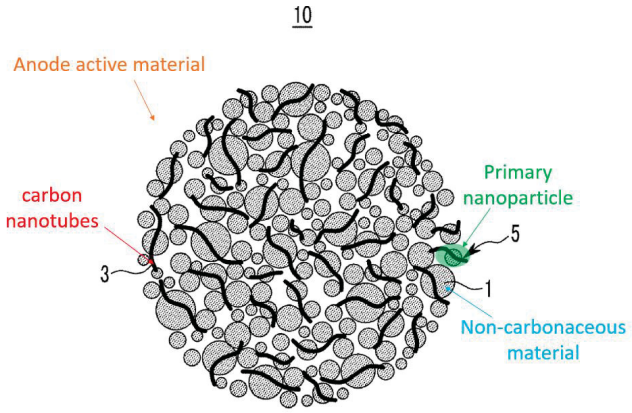
IPR2024-01006: LGES-1006, [0040], Fig. 1; Pet., 38; Pet. Reply, 21

FISH.
DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lee-Wepasnick nanotubes are discrete

Petition

As illustrated in Lee's FIG. 1, the carbon nanotubes in the combination anode active material (the **composition**) are **discrete**, *e.g.*, to the extent that discrete in this context is interpreted as referring to nanotubes present in the anode active material as generally individually distinct (*e.g.*, non-agglomerated) nanotubes.[8] LGES-1003, ¶162.



Alternatively/additionally, a POSITA would have understood or found obvious that the nanotubes in the combination composition are **discrete** because, at least in some instances, oxidization of nanotubes can help promote non-agglomeration of the nanotubes. *See* [1d], *infra*; LGES-1003, ¶163. A POSITA would have known that oxidized nanotubes are functionalized by species such as carboxylic acid or hydroxyl groups. *See* LGES-1010, Abstract; LGES-1014, 821-822; LGES-1015, 4186-4187; LGES-1007, [0027]-[0028]; LGES-1003, ¶163. As indicated by these references and as described by Dr. Zhu, the presence of these functionalities can contribute to physically and chemically inhibiting aggregation of nanotubes into clumps; instead, oxidized nanotubes generally can be present as individually distinct nanotubes. LGES-1003, ¶163. In light of this behavior of oxidized nanotubes, a POSITA would have understood or found obvious that oxidized nanotubes of the combination can be present as generally individually distinct nanotubes, *e.g.*, that the nanotubes are **discrete**. LGES-1003, ¶163.

FISH.

IPR2024-01006: Pet., 53-54; Pet. Reply, 21

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation promotes non-agglomeration

Dr. Zhu's Declaration

163. Alternatively/additionally, a POSITA would have understood or found obvious that the nanotubes in the combination composition are **discrete**. As I discuss further for element [1d], *infra*, the nanotubes in the Lee-Wepasnick combination are oxidized. At least in some instances, oxidization can help promote non-agglomeration of nanotubes, *e.g.*, oxidized nanotubes can be discrete. *See* [1d], *infra*. For instance, as I discussed above for element [1a] of the Ohata-Kavan ground, the sidewalls of oxidized nanotubes are functionalized by species such as carboxylic acid, carbonyl, or hydroxyl groups that are covalently bound to the nanotubes. The presence of these functional groups both physically and chemically prevent the nanotubes from aggregating into clumps and instead allow the nanotubes to be generally present as individually distinct nanotubes. *See* LGES-1005 (Kavan), Abstract; LGES-1010 (Wepasnick), Abstract; LGES-1014 (Gojny), 821-822; LGES-1015, 4186-4187; LGES-1007 (Farrugia), [0027]-[0028]. In light of this well-known behavior of oxidized nanotubes, a POSITA would have understood or found obvious that oxidized nanotubes of the combination composition can be present as generally individually distinct nanotubes, *e.g.*, that the nanotubes are **discrete**.

IPR2024-01006: LGES-1003, 163; Pet, 53

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation promotes non-agglomeration

LGES-1014

The presence of carboxylic acid groups on the surface enables a better dispersion of CNTs and reduces the amount of impurities. The nanotube/matrix interactions of oxidised MWCNTs and the epoxy resin seem to be still weak and as a consequence of this, the stress transfer from the matrix to the tube is low and pull-outs of the CNTs from the surrounding matrix were observed (compare Fig. 2).

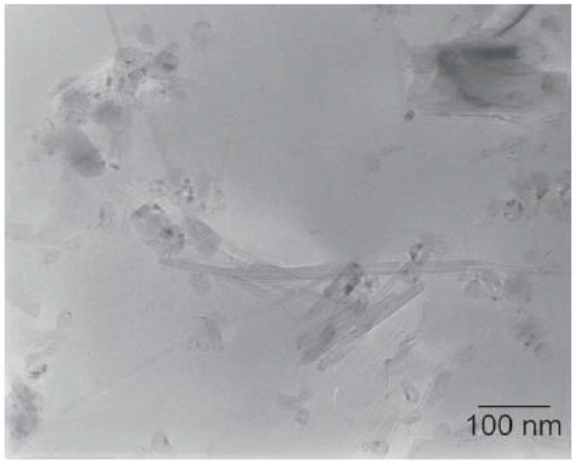


Fig. 1. TEM image of oxidised MWCNTs embedded in epoxy resin. The tubes are completely dispersed in the matrix due to the appearance of carboxylic groups on the surface.

LGES-1007

[0027] The carbon nanotubes can be functionalized by any means known in the art. For example, U.S. Pat. Nos. 5,698,175 and 5,203,814 and U.S. Patent Application Publication No. 2006/0249711, hereby incorporated entirely by reference, describe methods for functionalizing carbon nanotubes. The functionalization results in one or more chemical moieties attached to the carbon nanotubes. The chemical functionalization promotes direct covalent coupling between the carbon nanotubes and the polyester matrix, and results in better dispersion of the nanotubes throughout the matrix and increased interaction of the nanotube surface groups with the polymer. It also results in improvement in crystallinity due to the effect of the nanotubes on the resulting morphology of a semicrystalline matrix. CNT act as nucleation sites to promote increase in polymer crystallinity

FISH.

IPR2024-01006: LGES-1014, 821-822; LGES-1007, [0027]; Pet, 53

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lee-Wepasnick nanoparticles—not nanotubes—are agglomerated

Patent Owner's Response

Petitioner's failure to sufficiently explain or demonstrate how Lee can teach "discrete" CNTs given the repeated teachings of "agglomeration" is fatal to the Lee based grounds. Indeed, "[a]rguments in a petition must be made with particularity,

Lee

[0040] An anode active material **10**, according to an exemplary embodiment of the present teachings, will be described with reference to FIG. **1**. Referring to FIG. **1**, the anode active material **10** includes secondary particles that are formed by the agglomeration of primary nanoparticles **5**. The primary nanoparticles **5** include a non-carbonaceous material **1** and hollow carbon nanofibers **3**, which are bound to the non-carbonaceous material by Van der Waals forces.

Dr. Zhu's Supplemental Declaration

40. When Lee uses the term "agglomerated," this is a reference to the assembly of primary nanoparticles into secondary nanoparticles, not a reference to assembly of nanotubes with one another. For instance, this is apparent from paragraph [0040] of Lee, which states clearly that "the anode active material 10 includes secondary particles that are formed by ***the agglomeration of primary nanoparticles 5***." In fact, the nanotubes in Lee's primary particles are already bound to the non-carbonaceous material, and this prevents them from agglomerating with one another.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation promotes non-agglomeration

Patent Owner's Response

Figure 6. Moreover, Petitioner fails to explain how oxidation can help with dispersion of Lee's CNTs, which are "bounded" with agglomerated primary nanoparticles. *Id.*

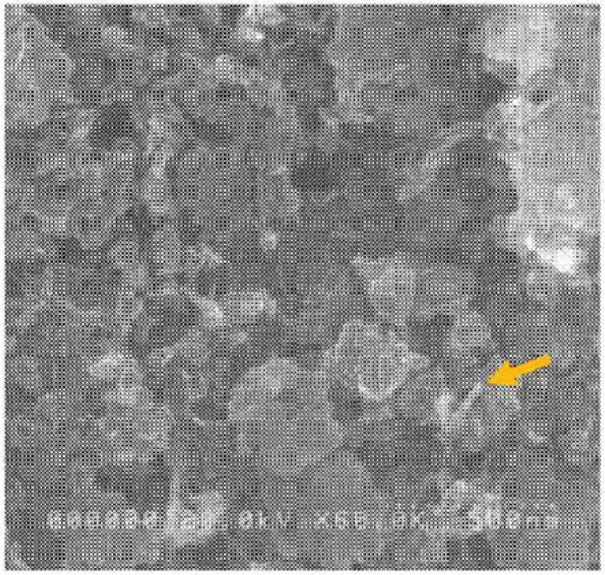
Dr. Zhu's Declaration

the side walls"). As I explained above, the presence of these functionalities helps to physically and chemically inhibit the nanotubes from aggregating into clumps, and instead oxidized nanotubes are generally present as individually distinct nanotubes. *See* LGES-1014 (Gojny), 821-822 ("[t]he presence of carboxylic acid groups on the surface" "enables a better dispersion of CNTs" showing images of "oxidised [sic] MWCNTs … [that] are completely dispersed in the [epoxy resin] matrix due to the appearance of carboxylic groups on the surface"); LGES-1015 (Xing), 4186-4187 ("treated [oxidized] CNTs had better dispersion"); LGES-1007 (Farrugia), [0027] (describing that functionalization of carbon nanotubes "results in one or more chemical moieties attached to the carbon nanotubes," which "results in better dispersion of the nanotubes throughout the matrix").

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's reliance on Lee's Fig. 6 SEM image is unfounded

FIG. 6



Patent Owner's Response

Although Petitioner alleges that these schematics demonstrate "discrete" CNTs, that is clearly untrue based on the SEM image in Figure 6 (annotated below), which is of the anode active material composition prepared in Example 1. *Id.* ¶180 (citing LGES-1006 ¶[0028]). At a 60,000x magnification level, it shows fibrous-looking material (e.g., orange arrow) associated with several clusters of silicon particles:

• • •

If there were discrete SWCNTs shown in Figure 6, ***each*** allegedly discrete SWCNTs would need to be 1/500th the size of the scale bar shown to see a distance representing a 1 nm diameter. *Id.* ¶182. However, a POSITA cannot see what a distance representing a 1 nm diameter looks, because the scale bar is much too coarse in connection with the magnification level. *Id.* The fact that SWCNTs are visible at all means they must be grouped together or intertwined with each other. *Id.*

IPR2024-01006: LGES-1006, Fig. 6; POR, 73-74

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's reliance on Lee's Fig. 6 SEM image is unfounded

Deposition of Patent Owner's expert, Dr. Chung

Q. How -- how many nanotubes do you think form that bundle that you've identified in Figure 6?

MR. ZINDA: Objection. Form.

THE WITNESS: It can be a lot. Depends on the diameter of one nanotube. If the diameter is 1 nanometer, then it will be a lot of nanotubes in that bundle.

BY MR. IN:

Q. Are there any other portions of that image where you believe a bundle of nanotubes are being shown?

A. As I said, the image is really low in quality. Very grainy. And it's hard to see the features there.

Petitioner's Reply

PO's reliance on Lee's Fig. 6 to conclude that Lee's nanotubes are "grouped together" (POR, 73-74) is undermined by Dr. Chung's admissions that this figure is unsuitable to support such a determination. At deposition, Dr. Chung conceded that "the image is really low in quality … and it's hard to see the features there." LGES-1054, 66:2567:1-. Moreover, even to the extent that this figure does show a bundle of "numerous CNTs intertwined with each other," as PO contends (POR, 74), its existence does not preclude that other, discrete nanotubes, consistent with the morphology of Lee's composition. Indeed, PO acknowledges that "POSITA cannot see what a distance representing a 1 nm diameter [nanotube] looks [like]" (POR, 74), meaning that discrete nanotubes would not necessarily even be visible. In fact, given the scale and admitted poor quality of this figure, it is simply not reliable as to the presence or absence of discrete nanotubes in the composition—and certainly not for conclusions that contradict Lee's teachings elsewhere regarding the structure of its composition.

FISH.

IPR2024-01006: LGES-1054, 66:15-67:1; POR, 73-74; Pet. Reply, 22-23

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's aspect ratio argument misrepresents the Petition

Patent Owner's Response

> Moreover, Figures 1 and 3 of Lee are unreliable given the incredibly broad CNT aspect ratio range of "about 10 to about 10,000" disclosed in Lee. *Id.* ¶183 (citing LGES-1006 ¶[0047]). A POSITA would have understood that the number of entanglements and degree of dispersibility of CNTs is affected by the aspect ratio and that an aspect ratio of at least 5,000 would almost certainly preclude the possibility of any discrete CNTs in Lee-Wespasnick. *Id.* Thus, a POSITA would know that distinct CNTs (as purportedly shown in FIGS. 1 and 3) would likely not be achievable at certain aspect ratios within Lee's very broad range. *Id.*[6]

IPR2024-01006: POR, 74-75

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's aspect ratio argument misrepresents the Petition

Petitioner's Reply

PO's argument that "an aspect ratio of at least 5,000 would almost certainly preclude the possibility of any discrete CNTs" (POR, 74) is a blatant misrepresentation of Petition. Nowhere does the Petition even mention the range "at least 5,000" (POR, 74). The Lee-Wepasnick nanotubes have an aspect ratio of 10-10,000. *See* Petition, 64. Even if larger aspect ratio nanotubes may be more subject to entanglement than smaller aspect ratio nanotubes, the Lee-Wepasnick nanotubes still encompass the claimed range of aspect ratios, which the '909 Patent confirms are capable of being discrete. Indeed, PO appears to acknowledges that its position lacks merit, qualifying its objection with the statement that distinct CNTs "would likely not be achievable ***at certain aspect ratios***"—but not at all aspect ratios—"within Lee's very broad range." POR, 75 (emphasis added).

Petition

The carbon nanotubes in the combination "have ***an aspect ratio of about 10 to about 10,000***," as taught by Lee. LGES-1006, [0047]; LGES-1003, ¶¶189-192 (citing LGES-1039, Abstract).

'909 Patent: Claim 1

1. A composition useful for lithium ion batteries comprising:

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.

IPR2024-01006: LGES-1001, claim 1; Pet., 64; Pet. Reply, 24-25

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's thermal treatment argument misunderstands the combination

Patent Owner's Response

> A POSITA would also understand that the process of making the anode active material in Lee-Wepasnick includes a mandatory thermal drying step that "may be performed at about 80°C., for about 24 hours." *Id.* ¶185 (quoting LGES-1006 ¶¶ [0061], [0083]). The purpose of this thermal drying step is to evaporate the organic solvent that was mixed with the non-carbonaceous material and CNTs in Lee-Wepasnick. *Id.* During and upon completion of Lee's thermal drying, the organic solvent evaporates and leaves the assembly of CNTs. *Id.* ¶186. This results in the collapse of the assembly of CNTs that draws the CNTs closer together, resulting in CNT agglomerates. *Id.* The agglomeration would make achieving discrete CNTs much more difficult, further calling Figures 1 and 3 into question. *Id.*

IPR2024-01006: POR, 75

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO's thermal treatment argument misunderstands the combination

Petitioner's Reply

PO's argument that as a result of Lee's thermal drying process, "the organic solvent evaporates … resulting in CNT agglomerates" (POR, 75) ignores that the thermal treatment is not applied to the nanotubes alone, but to the nanotubes together with the non-carbonaceous material. In Lee's process, prior to drying, "the non-carbonaceous material … bind with the hollow carbon nanofibers, thus forming the primary nanoparticles." LGES-1006, [0063]. Only then is the drying conducted. *Id.*, [0060]. In the context of drying a material that is "strongly bound together" (*id.*, [0063]), PO's error becomes apparent: there is no "collapse of the assembly of CNTs" (POR, 75), because there is no assembly of CNTs to begin with. Instead, the drying operates on primary nanoparticles—*i.e.*, nanotubes bound to non-carbonaceous material—that, through the drying process, agglomerate into secondary particles. *See* LGES-1006, [0061].

Lee

[0060] A method of manufacturing the anode **20**, according to an exemplary embodiment of the present teachings, will be described with reference to FIG. **4**. Referring to FIG. **4**, a non-carbonaceous material and hollow carbon nanofibers are mixed in an organic solvent, using a milling process. The resultant is then dried to prepare an anode active material (operation **S1**). The milling process may be bead milling or a ball milling.

[0063] As described above, as the anode active material is prepared, the non-carbonaceous material is pulverized into nanoparticles, which bind with the hollow carbon nanofibers, thus forming the primary nanoparticles. Thus, when the polymeric binder binds to the hollow carbon nanofibers, the primary nanoparticles are strongly bound together, so that the structure of the anode active material can be maintained.

IPR2024-01006: LGES-1006, [0060], [0063]; Pet. Reply, 25

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 3B

The claimed range of aspect ratios is obvious

[1c] wherein the discrete carbon nanotubes have an aspect ratio of 10 to 500

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Aspect ratios of the Lee-Wepasnick nanotubes

[1c] wherein the discrete carbon nanotubes have an aspect ratio of 10 to 500

Lee

[0047] Commercially available hollow carbon nanofibers may be used. Alternatively, hollow carbon nanofibers manufactured through a general manufacturing process, using a catalyst and a hydrocarbon gas may be used. The hollow carbon nanofibers **3** may have a diameter of about 1 to about 100 nm. The hollow carbon nanofibers **3** may have an aspect ratio of about 10 to about 10,000.

Petition

The claimed range of aspect ratios (**10-500**) overlaps with the range of aspect ratios (***10-10,000***) described by Lee and implemented in Lee-Wepasnick. LGES-1003, ¶190. The '909 Patent provides no discussion or evidence of unexpected results or criticality associated with nanotubes having an aspect ratio within the claimed range. Thus, based on the overlapping range disclosed by Lee, Lee-Wepasnick presents a *prima facie* case and renders obvious the recited range of aspect ratios. *See In re Peterson*, 315 F.3d at 1329; *In re Geisler*, 116 F.3d at 1469–70.

IPR2024-01006: LGES-1006, [0047]; Pet., 64-65

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Aspect ratios of the Lee-Wepasnick nanotubes

Patent Owner's Response

Here, with respect to the highest end of Lee's disclosed aspect ratio (10,000) compared to the highest end of the claimed aspect ratio (500)—***a key structural difference***—Lee teaches 9,500 more potential aspect ratios compared to the claimed aspect ratio. MRD-2006 ¶197 (citing LGES-1006 ¶[0047]). Additionally, the highest aspect ratio disclosed in Lee is 20x larger than the highest aspect ratio in the claimed invention. *Id.* These are significant differences resulting in materially different structure and behaviors. *Id.*

As in *Genetics*, due to the numerous significant differences in the structure and behavior, there would have been no motivation to try to optimize Lee-Wepasnick's remarkably broad range to identify the claimed range. *Id.* ¶202. The differences in structure and behavior are "nontrivial differences" that directly affect the resulting structure and behavior of the CNTs. *Id.* Lee cannot deprive the inventors of their invention by disclosing an excessively broad and unhelpful range that would require undue experimentation to identify the optimal claimed ranged.

IPR2024-01006: POR, 79-80

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Aspect ratios of the Lee-Wepasnick nanotubes

Petitioner's Reply

The *prima facie* obviousness of an overlapping range can be rebutted "by showing that the claimed range achieves unexpected results relative to the prior art range." *In re Woodruff*, 919 F.2d 1575, 1578 (Fed. Cir. 1990). But PO has not done this. Instead, PO has merely offered generic statements alleging "significant differences resulting in materially different structure and behaviors" (POR, 80), without providing any evidence that these effects (to the extent they exist at all) are specific to the particular claimed range.

PO's arguments that "due to the numerous significant differences in the structure and behavior, there would have been no motivation to try to optimize Lee-Wepasnick's remarkably broad range" are similarly deficient. Generic complaints about the breadth of the Lee-Wepasnick range is not factual evidence of unexpected results. *See* In re Geisler, 116 F.3d 1465, 1470 (Fed. Cir. 1997) ("it is well settled that unexpected results ***must be established by factual evidence***" (citing *In re Antonie*, 559 F.2d 618, 620, 195 USPQ 6, 8 (CCPA 1977)) (emphasis added)). PO has provided no such demonstration of criticality, nor provided any evidence tied to the claimed range. As such, PO's arguments are insufficient.

IPR2024-01006: Pet. Reply, 27-28

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 3C

Claim 6 of the ’909 Patent is obvious over Lee-Wepasnick

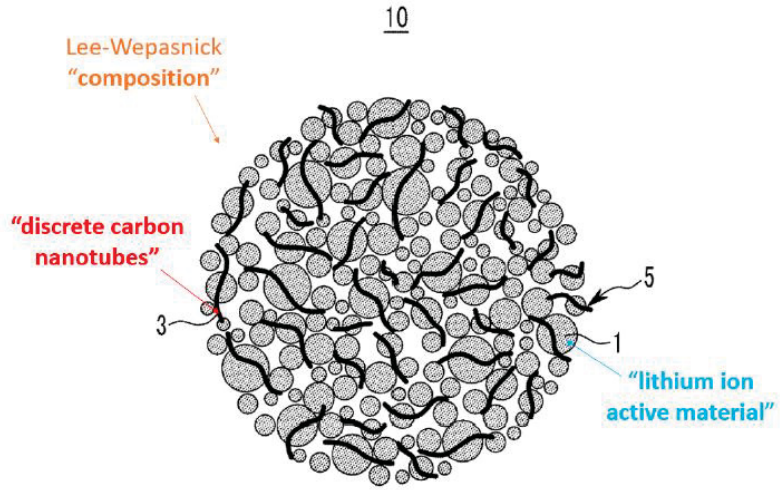
FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lee-Wepasnick nanotubes are uniformly dispersed

Petition

As discussed in [1a], the nanotubes in the combination anode active material (the **composition**) are present as generally individually distinct nanotubes, *e.g.*, they are **dispersed** in the composition. *See* [1a]; LGES-1003, ¶219. Based on Lee's FIG. 1, *infra*, which illustrates "the structure of an anode active material," and based on the dispersed nature of the nanotubes, a POSITA would have understood, or at least found obvious, that the nanotubes are **uniformly dispersed without clustering** in the combination anode active material. LGES-1003, ¶219. Thus, because there is substantially no clustering, a POSITA would have understood that the nanotubes are dispersed **without clustering on a dimension scale less than 1 micrometer**. LGES-1003, ¶219.



Lee, FIG. 1 (annotated)

IPR2024-01006: Pet., 76-77

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 4

Obviousness over Cheng and Gan (IPR2024-01011 only)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Table of Contents – Cheng-Gan

- Overview of the Cheng-Gan combination
- Issue 4A: The Cheng-Gan nanotubes have 1%-15% oxidation levels
- Issue 4B: A POSITA would have been motivated to combine Cheng and Gan and expected success
- Issue 4C: The Cheng-Gan nanotubes are discrete

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Cheng-Gan Combination

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng describes obtaining CNT/$LiFePO_4$ composite for lithium ion batteries

Petition

Cheng focuses on improving the dispersion of carbon nanotubes, both among the nanotubes themselves and in a precursor, by providing carbon nanotube/lithium iron phosphate composite material used for producing a positive electrode of the lithium ion battery. LGES-1007, [0010]. As discussed further *infra*, the composite material is prepared by at least making the carbon nanotubes, acetylene black (conducting agent), and dispersant mixture "in water or an organic solvent" based on ultrasonic dispersion, mixing the mixture with "a lithium source, an iron source and a phosphate salt," and performing further mixing and processing steps. *See id.*, [Abstract], [0012]–[0020], [0040], [0041], [0044], [0048], [0049], [0063], [0064], [0071], [0072], [0076], [0077], claim 2, claim 8; LGES-1003, ¶101. Cheng describes that the obtained carbon nanotube composite material [1] provides "carbon nanotube dispersion (dispersion of carbon nanotubes and dispersion in a precursor)," [2] exhibits a uniform distribution of carbon nanotubes both "on the surface of and in the lithium iron phosphate material," [3] forms "effective conductive network" or "a uniformly distributed network structure," thereby enhancing the electrical conductivity of the carbon nanotube composite material. *See* LGES-1007, [Abstract], [0012], [0021], [0023], [0030]–[0033]. LGES-1003, ¶101.

Cheng

(57) Abstract

The present invention belongs to the technical field of lithium ion battery materials, and in particular relates to a carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof. The method includes: ultrasonically dispersing carbon nanotubes, acetylene black, and a dispersing agent in water or an organic solvent to obtain a mixture; mixing a lithium source, an iron source, and a phosphate salt, simultaneously adding an organic carbon source and the mixture, and performing ball-milling and drying; and drying the same, then calcining the same in an inert protective atmosphere, and performing sieving to obtain a composite cathode material wherein carbon nanotubes are uniformly dispersed in a bulk phase and on surfaces of lithium iron phosphate, wherein the carbon nanotubes account for 0.5-15% by weight, and the lithium iron phosphate accounts for 85-99.5% by weight. When the obtained composite cathode material is used as a positive electrode of a lithium ion battery, the power output and the cycle characteristics can be significantly improved, and in addition, the capability of binding with an aluminum foil current collector is strong. An electrode sheet has high compacted density. The flexibility and winding property of the electrode sheet are good.

FISH.

IPR2024-01011: LGES-1007, Abstract; Pet., 36-37

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Gan describes benefits of functionalizing (oxidizing) CNT

Petition

Gan "relates to composites of carbon nanotubes and conductive polymer materials [corresponding to conducting agents]." LGES-1021, [0002], LGES-1003, ¶102. In particular, Gan describes "functionalized carbon nanotubes" that are mixed with conducting agents to obtain "composites that exhibit increased electrical conductivity." *Id.*, [Abstract]. "Generally such mixing can be achieved by adding the functionalized CNTs [carbon nanotubes] to the conductive polymer material directly and sonicating the mixture to uniformly disperse the functionalized CNTs in the conductive polymer material." LGES-1021, [0026].

Gan attributes this "increased electrical conductivity" of the functionalized carbon nanotube / conductive polymer composite to the improved dispersibility of nanotubes in water or polymer, resulting in a better constructed electrically conductive network structure (or "percolated network") of carbon nanotubes within the composite. *See id.*, Abstract, [0018]-[0024], [0041]; LGES-1003, ¶103. To

Gan

(57) **ABSTRACT**

Electrically conductive polymer materials, such as mixtures of poly(3,4-ethylenedioxythiophene) (PEDOT) and poly(styrenesulfonate) (PSS) are combined with functionalized carbon nanotubes to form composites that exhibit increased electrical conductivity. Functionalized or non-functionalized carbon nanotubes combined with the same electrically conductive polymer materials are combined with non-conductive polymers to increase the electrical conductivity of the non-conductive polymer. The functionalized carbon nanotubes are functionalized with carboxyl and/or hydroxyl groups. The resulting materials are useful in methods of forming electrically conductive films and electrically conductive features.

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.

FISH.

IPR2024-01011: LGES-1021, Abstract, [0018]-[0021]; Pet., 38-40

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Cheng-Gan combination

Petition

3. The Cheng-Gan combination

In the Cheng-Gan combination, the carbon nanotubes in Cheng's carbon nanotube/lithium iron phosphate composite would have carbon nanotubes that are functionalized or oxidized with carboxyl or hydroxyl groups at "about 2.5 to 10 wt%" based on Gan's disclosure.[10] LGES 1001, 3:60-64; LGES-1021, [0024]; LGES-1003, ¶107. As explained by Dr. Zhu, the term "functionalization" can be interchangeably used with "oxidation," at least in the instances relating to functionalizing the carbon nanotubes with functional groups having carboxylic, ketone or hydroxyl functionalities. LGES-1003, ¶107.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 4A

The Cheng-Gan nanotubes have 1%-15% oxidation levels

[1d] "[wherein the discrete carbon nanotubes have] oxidation levels from 1% to 15% by weight of the carbon nanotube"

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation level of Cheng-Gan's nanotubes

[1d] "[wherein the discrete carbon nanotubes have] oxidation levels from 1% to 15% by weight of the carbon nanotube"

Petition

3. The Cheng-Gan combination

In the Cheng-Gan combination, the carbon nanotubes in Cheng's carbon nanotube/lithium iron phosphate composite would have carbon nanotubes that are functionalized or oxidized with carboxyl or hydroxyl groups at "about 2.5 to 10 wt%" based on Gan's disclosure.[10] LGES 1001, 3:60-64; LGES-1021, [0024]; LGES-1003, ¶107. As explained by Dr. Zhu, the term "functionalization" can be interchangeably used with "oxidation," at least in the instances relating to functionalizing the carbon nanotubes with functional groups having carboxylic, ketone or hydroxyl functionalities. LGES-1003, ¶107.

Gan

[0024] The degree to which the CNTs are functionalized is a balance between introducing the desired functional groups into the CNTs to achieve one or more of the three objectives described above, while minimizing the effect on the desirable intrinsic properties of the CNTs. For example, while a greater degree of functionalization may improve the dispersibility of the CNTs, such improvement may come at a decrease in intrinsic properties of the pristine CNTs. In accordance with the embodiments described herein, suitable functionalization with carboxyl or hydroxyl groups ranged from about 2.5 to about 10 wt %.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation level of Cheng-Gan's nanotubes

Patent Owner's Response

Petitioner argues that a POSITA would have been motivated to functionalize the CNTs of Cheng to "about 2.5 to 10 wt%" per Gan. Pet., 41. However, Petitioner overlooks that Cheng-Gan performs a carbon thermal reduction that ***calcines the CNTs at 600°C to 850°C for 4 to 48 hours***. LGES-1007 ¶[0019]. Petitioner does not propose modifying Cheng-Gan to eliminate this step, which is a mandatory step in all embodiments of Cheng to (1) convert the carbon source into carbon and (2) form the lithium iron phosphate. MRD-2006 ¶¶168-170 (citing MRD-2038, 1; MRD-2034, 1); MRD-2026, 38:18-39:5.

The calcining would remove essentially all of the oxidized groups from the CNT surfaces, bringing the oxidation level below 1 wt%. *Id.* ¶171. For instance, a reference relied on by Petitioner teaches that at much lower temperatures of 150 °C to 350 °C, "***decarboxylation*** of the carboxylic groups present on the MWCNT walls" occurs. LGES-1009, 7. "Thermal degradation in the range between the 350 °C and 500 °C may be explained by the ***elimination of hydroxyl functionalities, attached to the MWCNT walls.***" *Id.* A POSITA would expect that at Cheng-Gan's much higher minimum calcination temperature of 600 °C, essentially all of the functional groups of Chen-Gang would be removed, particularly given the 4-hour minimum calcining time. MRD-2006 ¶171.

FISH.

IPR2024-01011: POR, 62-63

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Oxidation level of Cheng-Gan's nanotubes

Petitioner's reply

1. PO's cited references are inapplicable to the ox-CNT precursor mixture in Cheng-Gan, and expert testimony, corroborated by references, confirms functional group retention

All references relied on by PO regarding the thermal treatment of oxidized CNTs—including MRD-2029, 2030, 2031, 2032, and LGES-1009—pertain to pure ox-CNTs or plasma-treated CNTs that do not contain Li/Fe/PO_4 precursors. POR, 63-65; LGES-1056, ¶¶37-38.

Moreover, as noted above, none of PO's references involve precursor mixture of Cheng-Gan, which combines oxidized CNTs with Li/Fe/PO_4 precursors prior to thermal treatment. Petition, 53-54, 56-58. PO's reliance on the above-noted references is wrong, as they are directed to materially different systems which do not address the thermal behavior of Cheng-Gan's ox-CNT precursor mixture. LGES-1056, ¶40.

Dr. Zhu Supplemental Declaration

40. As I noted during my deposition, PO's comparison between isolated ox-CNTs and Cheng-Gan's ox-CNT mixture including Li/Fe/PO_4 precursors is inappropriate and technically incorrect. The thermal behavior of isolated ox-CNTs differs from that of the ox-CNT precursor mixture used in Cheng-Gan. *See* MRD-2026, 41:5-42:14; *see also id.*, 40:11-47:7. Moreover, as described above, the degree of inertness of the calcination atmosphere (e.g., inert gas type, flow rate), among other things, contributes to the retention of oxidized groups on CNTs—factors that are directly relevant to Cheng's in situ LFP-forming reactions, which require an inert protective atmosphere to maintain iron in the +2 oxidation state. LGES-1007, [0019] ("calcining . . . in an inert protective atmosphere").

41. For instance, as I explained in the deposition, reactions occur between the Li/Fe/PO_4 precursors and also other components present in the mixture. Specifically, lithium iron phosphate (LFP) forms from Li/Fe/PO_4 precursors and the functional groups on the CNTs become attached to the formed LFP, rather than being removed. *See* MRD-2026, 41:5-43:2, 45:15-47:7.

FISH.

IPR2024-01011: Pet. Reply, 18-20; LGES-1056, ¶¶40-41

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Even under PO's argument based on isolated ox-CNTs, PO's position cannot be sustained

Petitioner's Reply

> As an initial matter, regarding pure ox-CNTs, Dr. Zhu explains that loss of oxidized functional groups can be minimal, even under higher temperature thermal treatment conditions than those referred to by PO. POR, 62-64. For example, LGES-1042 discloses that pure oxidized CNTs with an initial oxygen content of 12.9 wt% exhibited only a slight reduction to 10.4 wt% after annealing at 900 °C for 2 hours under helium inert atmosphere. LGES-1056, ¶39 (citing LGES-1042, 7, 3, FIG. 6(e)). Here, Dr. Zhu demonstrates the

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Even under PO's argument based on isolated ox-CNTs, PO's position cannot be sustained

Dr. Zhu Supplemental Declaration

39. Prior to explaining the difference in thermal behavior between isolated ox-CNTs and ox-CNT mixture (including Li/Fe/PO_4) precursors, it is worth noting that even among isolated oxidized CNTs, minimal loss of oxidized functional groups during calcination can be achieved depending on experimental conditions. For instance, Liu (LGES-1042) describes oxidized multiwalled CNTs with an initial oxygen content of 10 atomic % (about 12.9 wt%), and reported that after annealing at 900 °C for 2 hours under helium inert atmosphere, the oxygen content decreased only slightly to 8 atomic % (about 10.4 wt%). LGES-1042, 7 ("[t]he oxygen content in the CNT sample is calculated to be about 10 at.% . . . After an annealing treatment under a helium atmosphere at 900 °C (CNT-900) [for 2 hours], the oxygen content in the CNT material slightly decreases, from 10 to 8 at.%"), 3 (disclosing experimental condition for "anneal[ing] in helium at 900 °C for 2h"), FIG. 6(e) (disclosing XPS spectra of CNT-900). This result directly contradicts PO's assertion that high-temperature calcination necessarily removes "essentially all" functional groups, and indicates that other factors, such as carbon structural order including the degree of carbon disorder (e.g., presence of disorderd carbon structure), chemical bonding environment, and the specific inertness of the calcination atmosphere (e.g., using helium as an inert gas), can significantly influence oxygen retention. *See id.* Therefore,

LGES-1042

The carbon and oxygen atomic ratios calculated by the high resolution C 1s and O 1s core level spectra are presented in Fig. 6b. It should be noted that reference SiO_2 samples were measured in order to subtract the contribution of SiO_2 impurities from the overall O 1s spectrum. Therefore the oxygen atomic ratio presented in Fig. 6b is exclusively due to oxygen species attached to carbon. The oxygen content in the CNT sample is calculated to be about 10 at.%. Such high oxygen content was attributed to the post-synthesis acid treatment of the sample. After an annealing treatment under a helium atmosphere at 900 °C (CNT-900), the oxygen content in the CNT material slightly decreases, from 10 to 8 at.%. On the other hand, the oxygen content in the CNT-foam-900, after a similar annealing treatment, is about 4 at.%, which is the lowest amount among the analyzed samples. The observed results indicate that oxygen desorption is easier from the foam-structured composite compared to the pristine CNT material. This phenomenon is attributed to the presence of a porous disordered carbon structure on the topmost surface of CNT which binds oxygen in a loose manner.

IPR2024-01011: LGES-1056, ¶39; Pet. Reply, 18-19; LGES-1042, 7

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

Petitioner's reply

> Importantly, Dr. Zhu further indicates oxidized functional groups on carbon nanotubes are retained and attached to LFP during high-temperature calcination when combined with Li/Fe/PO_4 precursors. LGES-1056, ¶¶43-46. First, LGES-1032 discloses that highly dispersed conductive network was formed based on oxidation. LGES-1032, 1-2; LGES-1056, ¶43. Second, LGES-1043 and LGES-1041 demonstrate that oxidized functional groups present on carbon atoms were retained and attached to the LFP after calcination of a mixture including carbon (bearing oxidized functional groups) and Li/Fe/PO_4 precursors at 650°C for 10 hours (for LGES-1043) and 600-800 °C for 6 hours (for LGES-1041), respectively, under inert protective atmosphere.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

Dr. Zhu Supplemental Declaration

43. Importantly, oxidized functional groups on carbon nanotubes are retained and attached to LFP during high-temperature calcination (under inert protective atmosphere) when combined with Li/Fe/PO_4 precursors. For example, LGES-1032 discloses that highly dispersed conductive network was formed based on functionalization (oxidation in this context). LGES-1032, 1 ("The carbon nanotubes were functionalized using a mixed acid method to ensure uniform dispersion into the aqueous sol"), 1 ("The nanotubes retained their dispersion and were incorporated directly into the porous $LiFePO_4$ particulates during the subsequent gelatinization and calcination steps resulting in an intimately interlaced 3D composite network"), 2 (explaining that CNTs retained dispersion in resulting $LiFePO_4$ / CNT composite). This demonstrates oxidized groups integrated into the interlayers and attached to active material, rather than being removed under calcination at 700°C for 10 hours under an inert protective atmosphere. LGES-1040, 2.

LGES-1032

The porous $LiFePO_4$–CNT composites were synthesized *via* a facile *in situ* sol–gel method (see details in the experimental section of ESI), where the carbon nanotubes were functionalized using a mixed acid method to ensure uniform dispersion into the aqueous sol. The nanotubes retained their dispersion and were incorporated directly into the porous $LiFePO_4$ particulates during the subsequent gelatinization and calcination steps resulting in an intimately interlaced 3D composite network. The formation of such a hierarchical composite was supported by the SEM and TEM investigations shown in Fig. 2. As shown in Fig. 2a, the "pristine" (non-CNT modified) $LiFePO_4$ particles are several microns in size and highly porous. This porous $LiFePO_4$ material was observed to be covered with a thin layer of carbon (around 1–2 nm) on the wall surface, attributed to decomposition residues from the citrate-containing starting gel. Overall weight loading of carbon in this porous $LiFePO_4$ was determined to be around 3 wt%,

LGES-1040 (Electronic Supplementary Information of LGES-1032)

mixed together and dried at 60 °C for 24 h. After thorough grinding with a mortar and pestle, the obtained material was fired in an inert (argon) atmosphere at 700 °C for 10 h with a heating rate of 10 °C min^{-1}. For the

FISH.

IPR2024-01011: LGES-1056, ¶43; Pet. Reply, 20; LGES-1032, 1, LGES-1040, 2

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

Dr. Zhu Supplemental Declaration

44. Moreover, LGES-1043 (R. Liu) confirms that oxidized functional groups present on carbon atoms were well retained and attached to the active material (LFP) after calcination at 650 °C for 10 hours under an inert protective atmosphere in the presence of Li/Fe/PO_4 precursors. LGES-1043, 2-3 ("Experimental Section" describing a powder mixture including glucose-derived carbon bearing oxidized functional groups and Li/Fe/PO_4 precursors subjected to calcination under inert atmosphere (e.g., argon) at 650 °C for 10 hours), 6 (explaining that Figure 4 shows XPS spectra of carbon/LFP samples), 6-7 ("The two shoulder peaks at 531.9 and 533.0 eV are attributed to the C-O and C=O bands arising from functional groups absorbed on the sample [LFP] surface . . . The additional peak at 288.2eV is the C=O band arising from functional groups absorbed on the sample [LFP] surface" and "[t]hese results confirm that the $LiFePO_4$/C composite was synthesized"), FIGS. 4(e), (f) (showing clear peaks of C-O and C=O). As shown in Figure 4(f), the C 1s XPS spectrum exhibits clear and distinct peaks at ~286 eV and ~288 eV, corresponding to C–O and C=O functional groups. *See* LGES-1043, 7, FIG. 4(f). Similarly, the O 1s spectrum (Figure 4(e)) shows clear peaks at ~531.9 eV and 533.0 eV, confirming the presence of oxidized oxygen containing species. *See* LGES-1043, 7, FIG. 4(e). These C-O and C=O bonds of functional groups are chemically analogous to those of functional groups found on oxidized CNTs.

LGES-1043

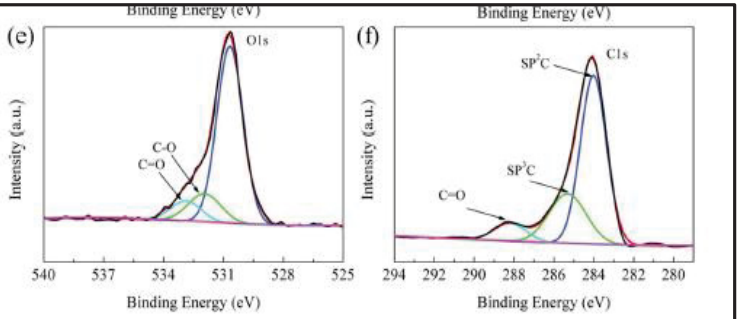


Figure 4. XPS survey of $LiFePO_4$/C composite (a); high resolution XPS spectrum of: Li 1s (b); Fe 2p (c); P 2p (d); O 1s (e); and C 1s (f) for $LiFePO_4$/C composite.

Figure 4 shows the high-resolution X-ray photoelectron spectroscopy (XPS) spectra of the Li 1s, Fe 3p, Fe 2p, P 2p, O 1s and C 1s core levels to determine the oxidation states of the elements in the $LiFePO_4$/C composite. The peak at 56.5 eV, corresponding to the lithium of the $LiFePO_4$/C composite, cannot be seen due to the superposed iron peak of Fe 3p [35,36]. The peak intensity of Fe 3p is higher than Li 1s because the Fe 3p has greater relative atomic sensitivity than that of Li 1 s [37,38]. The Fe 2p shows two peaks at 710.1 (2p3/2) and 724.1 eV (2p1/2) with a splitting energy of 14.0 eV, which is close to the standard splitting energy of 19.9 eV, demonstrating the oxidation state of Fe^{2+} [36,38]. Moreover, two small peaks at high binding energy of 713.9 and 728.5 eV are the characters of transition metal ions with partially filled-d orbits, which are assigned to the multiple splitting of the energy levels of Fe ion [37,38]. The peaks representing the other valence states of Fe ions cannot be seen, revealing that only Fe^{2+} ions exist in the $LiFePO_4$/C composite. The P 2p shows a peak at 132.9 eV, revealing that the valence state of P is 5+ [38]. The O 1s shows a peak at 531.0 eV, confirming that the valence state of O in the $LiFePO_4$/C composite is divalent. The two shoulder peaks at 531.9 and 533.0 eV are attributed to the C–O and C=O bands arising from functional groups absorbed on the sample surface [39]. The C 1s shows peaks at 284.0 and 284.4 eV, which correspond to the short-order sp2-coordinated and sp3-coordinated carbon atoms [38]. The additional peak at 288.2 eV is the C=O band arising from functional groups absorbed on the sample surface. These results confirm that the $LiFePO_4$/C composite was synthesized.

IPR2024-01011: LGES-1056, ¶44; Pet. Reply, 20-21; LGES-1043, 6-7, FIG. 4(e), (f)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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Dr. Zhu Supplemental Declaration

46. Moreover, LGES-1041 further demonstrates that oxidized functional groups present on carbon atoms were retained after calcination at 600-800 °C for 6 hours (under inert protective atmosphere) in the presence of Li/Fe/PO_4 precursors. LGES-1041, 2 (describing a powder including glucose-derived carbon bearing oxidized functional groups and Li/Fe/PO_4 precursors subjected to calcination under argon atmosphere at 600, 700, 800°C for 6 hours), 4-5 (explaining that Figure 4(d) shows C1s XPS spectra of carbon/LFP samples). As shown in Figure 4(d) of LGES-1041, the C1s XPS spectra of carbon/LFP samples exhibit strong peaks between 286-290 eV, corresponding to C-O and C=O bonds, where C-O and C=O bonds are chemically analogous to those found on oxidized CNTs. *Id.*, FIG. 4(d). These results confirm that such functional groups on the carbon atoms are not removed during calcination. As in Cheng-Gan and LGES-1032, LFP formation occurs alongside the retention of oxidized functional groups. *Id.*, (XRD analysis "point[s] out the orthorhombic structure without impurity phase and the high crystallinity of three synthesized olivines), 2, 4, FIG. 4(d). Thus, this directly contradicts the PO's core theory and supports that PO's cited references—focused on isolated pure ox-CNTs—are not applicable to ox-CNT/LFP precursor mixtures, where oxidized functional groups are retained in the presence of Li/Fe/PO_4 precursors under examples of Cheng's calcination conditions.

LGES-1041

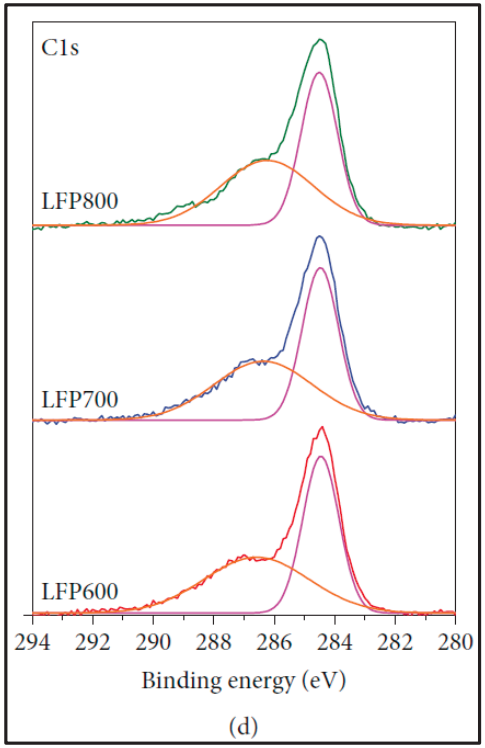


FIGURE 4: (a) Full-scan profiles of olivine samples; narrow-scan profile of Fe2p (b), O1s (c), and C1s (d).

IPR2024-01011: LGES-1056, ¶46; Pet. Reply, 20-21; LGES-1041, 5, FIG. 4(d)

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

Patent Owner's Response

Moreover, if the functional groups were truly protected because they are ***part*** of the reactions (as Dr. Zhu alleges), removal of oxidation would ***be accelerated***. *Id.* ¶181(explaining that it was known that the thermal tolerance of functional groups degrades in reactive environments). The functional groups would also adversely interfere with the formation of the lithium iron phosphate under Dr. Zhu's theory. *Id.* ¶¶ 182-184 (citing MRD-2042, 1).

Dr. Chung Declaration

182. Third, if the functional groups were part of the reaction as Dr. Zhu contends, it would fundamentally alter the resulting composition in undesired ways. Specifically, if the functional groups on the CNTs indeed react with the lithium salt precursors during the heating process (as Dr. Zhu alleges), the lithium salt precursors would be chemically modified through this reaction. The modification of the lithium salt precursors would negatively reduce the effectiveness of the precursors to form the desired lithium salt with the desired composition. The functional groups on the surface of a material tend to hinder a reaction that is intended to occur using reactants that coexist with the material with the functional groups (Gensch paper, i.e., MRD-2042). This phenomenon is known as functional group tolerance, i.e., the ability of a reaction to tolerate the presence of functional groups.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

Dr. Zhu Supplemental Declaration

48. However, this functional group tolerance theory is fundamentally flawed, as described above with respect to LGES-1032, LGES-1041, LGES-1043, and my testimony during deposition. During calcination, LFP forms from $Li/Fe/PO_4$ sources without issue and LFP attaches to the CNT during the calcination step. As I explained above, this behavior is distinct from the behavior of isolated ox-CNTs and shows that ox-CNT mixtures (including $Li/Fe/PO_4$ precursors) behave differently under thermal treatment.

49. Dr. Chung's reliance on "functional group tolerance" is additionally problematic and contradicts the '924 Patent itself. Specifically, her theory contradicts her own testimony and undermines the operability of the '924 patent's claimed system. For example, as acknowledged by Dr. Chung, Example 2 of the '924 patent—identified as representative of the claimed system—involves an ox-CNT precursor mixture (including $Li/Fe/PO_4$ precursors) undergoing in situ LFP formation under heat treatment. LGES-1053, 8:10-13:24. Despite the presence of functional groups on ox-CNT precursor mixture, Dr. Chung confirmed that "***all of the precursors*** have been converted to the final product [of LFP]" during the heat treatment phase. LGES-1053, 13:17-24; *see also id.*, 8:10-13-24. This admission directly contradicts her theory that functional groups would interfere with LFP formation or render the process inoperable. Moreover, MRD-2042 (the Gensch paper) only focuses on a few selected reaction as listed in its experimental section, and says nothing about CNTs, $Li/Fe/PO_4$, or inorganic solid-state reactions (e.g., formation of LFP). *See* MRD-2042, 1. Therefore, Dr. Chung's rebuttal fails both scientifically and factually, as it contradicts her own testimony and is inapplicable to the system in Cheng-Gan.

FISH.

IPR2024-01011: LGES-1056, ¶¶ 48-49; Pet. Reply, 21

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan's oxidized groups are retained and attached to LFP

'924 Patent

Example 2

Discrete Carbon Nanotubes with Lithium Iron Phosphate Coated on the Surface of the Tubes Suitable for a Lithium Ion Battery Cathode Material

0.2 grams of multi-wall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and aspect ratio about 60 are added to 23.5 grams of diethylene glycol and sonicated for 15 minutes. The solution is grey. 1.16 grams of iron acetate dissolved in 4 ml of deionized water is added and the mixture heated under nitrogen, stirring and sonication. After 1 hour at 140° C. a solution of 0.65 grams of lithium acetate and 0.74 grams of ammonium phosphate in 4.2 ml of deionized water is added. The temperature is raised to about 185° C. over 30 minutes, then held at 185° C. for 4 hours. The mixture is cooled to 110° C. and sonicated for 5 minutes followed by further cooling to ambient temperature while stirring. The solids are separated from the mixture by centrifugation followed by ethanol washing. The resultant powder is dried to constant weight vacuo.

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

Dr. Chung Deposition

Q. And that certainly wasn't what I was referring to. But those individual precursors, after the high temperature step, become LF -- LFP, correct?

MR. ZINDA: Objection. Form.

THE WITNESS: Yes. And it -- the -- after the reaction is complete so that all of the precursors have been converted to the final product, which is the lithium iron phosphate, then that's what you have.

FISH.

IPR2024-01011: LGES-1001, 5:30-53; LGES-1056, ¶¶ 48-49; Pet. Reply, 21; LGES-1053, 13

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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

Petitioner's reply

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 Petition relies on Cheng as disclosing a process in which an ox-CNT precursor mixture is subjected to calcination, without asserting that the exact time or temperature values are limiting. There is no indication in the Petition or the Declaration that a POSITA would have understood Cheng's process to preclude alternative or routine variations in calcination parameters.

Last but not least, the Petition notes that "it was well-known in the lithium ion battery industry that oxidized nanotube would improve the attachment of the ion active material (lithium iron phosphate or the like) to the carbon nanotube surface." Petition, 58 (citing LGES-1010, LGES-1005, LGES-1003). For example, the Petition explained that oxidized CNTs were well-known to provide a functional "foothold" for LFP attachment—supporting Dr. Zhu's position that a POSITA would have, if needed, adjusted calcination conditions (e.g., temperature, duration) based on the materials and synthesis objectives. *See* MRD-2026, 40:4-42:14; 44:24-47:7.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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

Petition

Cheng focuses on improving the dispersion of carbon nanotubes, both among the nanotubes themselves and in a precursor, by providing carbon nanotube/lithium iron phosphate composite material used for producing a positive electrode of the lithium ion battery. LGES-1007, [0010]. As discussed further *infra*, the composite material is prepared by at least making the carbon nanotubes, acetylene black (conducting agent), and dispersant mixture "in water or an organic solvent" based on ultrasonic dispersion, mixing the mixture with "a lithium source, an iron source and a phosphate salt," and performing further mixing and processing steps. *See id.*, [Abstract], [0012]–[0020], [0040], [0041], [0044], [0048], [0049], [0063], [0064], [0071], [0072], [0076], [0077], claim 2, claim 8; LGES-1003, ¶101. Cheng describes that the obtained carbon nanotube composite material [1]

Cheng

(57) Abstract

The present invention belongs to the technical field of lithium ion battery materials, and in particular relates to a carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof. The method includes: ultrasonically dispersing carbon nanotubes, acetylene black, and a dispersing agent in water or an organic solvent to obtain a mixture; mixing a lithium source, an iron source, and a phosphate salt, simultaneously adding an organic carbon source and the mixture, and performing ball-milling and drying; and drying the same, then calcining the same in an inert protective atmosphere, and performing sieving to obtain a composite cathode material wherein carbon nanotubes are uniformly dispersed in a bulk phase and on surfaces of lithium iron phosphate, wherein the carbon nanotubes account for 0.5-15% by weight, and the lithium iron phosphate accounts for 85-99.5% by weight. When the obtained composite cathode material is used as a positive electrode of a lithium ion battery, the power output and the cycle characteristics can be significantly improved, and in addition, the capability of binding with an aluminum foil current collector is strong. An electrode sheet has high compacted density. The flexibility and winding property of the electrode sheet are good.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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

Dr. Zhu Deposition

Q But in the specific process that we discussed about Cheng, the three-step process, he has to perform Step 3 to form the lithium iron phosphate, correct?

A Well, that's what I said. This is optional for him and the POSITA should know a number of different ways to form the lithium iron phosphate. And those are all well documented ways to produce lithium iron phosphate. When you prepare lithium iron phosphate, you always need a precursor: Lithium, iron and phosphorus. And how do you prepare them, actually what experimental condition. Like, what is the temperature you would like to use. What is the pressure you would like to use. And this is basically an option of the -- of the researchers, whether there are existing protocols, existing batteries by that time already and -- to forming the lithium iron phosphate.

And this is just one of the approach taken by Cheng in his example to do that. And he can certainly don't use that high temperature, use a lower temperature. He can still form lithium iron phosphate under the right condition.

Q So is it your opinion that you would use a lower temperature if you were using oxidized carbon nanotubes or would you do the same thing that Cheng did?

A I could use either way. I don't think it's necessary to have any specific difference in this case. You just needed to choose a way to forming lithium iron phosphate. And end of the day, you consider the cost of the raw materials and distribution of the carbon fillers and whether you would like to add any other ingredients in the lithium iron phosphate. And there are many considerations like that.

So sometimes you -- you can consider using higher temperature. Sometimes you can consider using lower temperature, yeah. So it's a wide range of selection depending on the exactly raw materials reaction conditions and the result you would like to achieve.

IPR2024-01011: MRD-2026, 40-41, 44-45; Pet. Reply, 22-23

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

PO fails to recognize that the objective of the combination is to yield a final composite with an oxidation level of about 2.5%-10% post-calcination

3. The Cheng-Gan combination

In the Cheng-Gan combination, the carbon nanotubes in Cheng's carbon nanotube/lithium iron phosphate composite would have carbon nanotubes that are functionalized or oxidized with carboxyl or hydroxyl groups at "about 2.5 to 10 wt%" based on Gan's disclosure.[10] LGES 1001, 3:60-64; LGES-1021, [0024]; LGES-1003, ¶107. As explained by Dr. Zhu, the term "functionalization" can be interchangeably used with "oxidation," at least in the instances relating to functionalizing the carbon nanotubes with functional groups having carboxylic, ketone or hydroxyl functionalities. LGES-1003, ¶107.

For instance, a POSITA would have recognized Gan's teachings about using carbon nanotubes that are functionalized with carboxyl or hydroxyl groups at "about 2.5 to 10 wt%," and would have applied this knowledge to the nanotubes in Cheng according to Gan's guidance. *See* LGES-1021, [0018]-[0024]; LGES-1003, ¶111. As described *supra*, Gan's guidance regarding "2.5 to 10 wt%" oxidation level is highly relevant to obtaining a better constructed electrically conductive network of the carbon nanotubes within the composite. *See* LGES-1021, Abstract, [0018]-[0024], [0041]; LGES-1003, ¶111.

Petition

As described *supra* with respect to the "Reasons for the combination" section (Part IV.B.4.), there would have been sufficient motivation for a POSITA to apply nanotubes having functionalization level (or oxidation level) such as those described by Gan to the Cheng's nanotube composite, and, at the same time, would have expected success. *See* discussion *supra* Part IV.B.4; LGES-1003, ¶150.

Accordingly, this range of oxidation level recited in Gan ("2.5 to 10%"), as applied to the nanotubes of the combination, renders obvious a composition in which the nanotubes have oxidation levels from 1% to 15% by the weight of the carbon nanotube . LGES-1021, [0024]; LGES-1003, ¶151. *See Ineos*, 783 F.3d at 869, *Titanium Metals*, 778 F.2d at 782.

Alternatively/additionally, a POSITA would understand that oxidized nanotubes—e.g., including those with oxidation levels that fall within the claimed range ("1% to 15%"), regardless of the method of measurement—were extensively used in various applications, including lithium ion batteries and further, the effect of oxidized nanotubes with oxidation level such as 9.1% was known to improve dispersibility of the carbon nanotubes and thus to enhance electrical conductivity of conductive network in the electrode composite in lithium ion battery industry. *See* discussion *supra* Part IV.A.5.(i).(1d); LGES-1003, ¶¶152-154.

IPR2024-01011: Pet. 41-42, 63-64

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 4B

A POSITA would have been motivated to combine Cheng and Gan and would have expected success

[1d] "[wherein the discrete carbon nanotubes have] oxidation levels from 1% to 15% by weight of the carbon nanotube"

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

First three motivations for oxidized nanotubes

Dr. Zhu's Declaration

110. The first reason for using oxidized nanotubes (or oxidized nanotubes having the functionalization levels (oxidation levels) such as those described in Gan) is that a POSITA would have recognized the importance of "about 2.5 to 10 wt%" oxidation levels in **obtaining a better constructed electrically conductive network of carbon nanotubes within the nanotube / lithium iron phosphate composite**, which is a design objective in Cheng. *See* LGES-1007 (Cheng), Abstract, [0003]-[0010], [0021], [0030]–[0033]. Bearing in mind the well-known role of **functionalization** of the carbon nanotubes in improving their dispersibility in an aqueous solution and the well-known fact that the **functionalized carbon nanotubes** can retain their dispersion and incorporate directly into the porous $LiFePO_4$ particulates, resulting in an intimately interlaced 3D composite network and promising electrode performance of the lithium ion batteries, a POSITA would have looked to Gan for guidance as to the functionalization level or oxidation level appropriate for this objective. LGES-1032 (Zhou), 1 ("**The porous LiFePO4–CNT composites were synthesized via a facile in situ sol–gel method** (see details in the experimental section of ESI), where **the carbon nanotubes were functionalized** using a mixed acid method **to ensure uniform dispersion** into the aqueous sol. The nanotubes **retained their dispersion** and were **incorporated directly into the porous LiFePO4 particulates during the subsequent gelatinization and calcination steps resulting in an intimately interlaced 3D composite network**"). For instance, Zhou describes that the nanotubes were incorporated throughout the $LiFePO_4$ particulates during the calcination steps and the functionalized nanotubes have "retained their dispersion" throughout the process and achieved the "**highly dispersed conductive networks**," where the "uniform dispersion of **CNT networks** into the $LiFePO_4$ is crucial for performance." *Id.*

IPR2024-01011: LGES-1003, ¶110; Pet. 42-43

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

First three motivations for oxidized nanotubes (Cont'd)

Dr. Zhu's Declaration

112. The second reason for using oxidized nanotubes (or oxidized nanotubes having the functionalization levels (oxidation levels) such as those described in Gan) is that a POSITA would have been motivated to enhance **dispersion of carbon nanotubes in water** and/or conducting agent in Cheng. For instance, enhancing dispersion of carbon nanotubes in water and/or conducting agent in Cheng would have facilitated the steps that contribute to a design objective in Cheng, which relates to "carbon nanotube dispersion (dispersion of carbon nanotubes and dispersion in a precursor)". *See* LGES-1007 (Cheng), Abstract, [0010]-[0014]. For instance, Cheng makes mixture of carbon nanotubes, acetylene black (conducting agent), and dispersant by "ultrasonically dispersing the same in **water,**" and after dispersion, mixes the mixture with "a lithium source, an iron source and a phosphate salt" and "organic carbon source" using the mixing medium such as "**deionized water**." *Id.* [0013]-[0017], claim 2. With regards to this context, Gan notes that the functional groups such as carboxyl groups or hydroxyl groups "are effective at increasing the solubility and dispersibility of the CNTs in **water,**" as well as in conducting agent such as conductive polymers. *See* LGES-1021 (Gan), [0018]-[0021]. Moreover, it was well-known to a POSITA that

113. The third reason for using oxidized nanotubes having the functionalization levels (or oxidation levels) such as those described by Gan is that, a POSITA would have recognized Gan's guidance of oxidation levels at "**about 2.5 to 10 wt%**" **remain within a safe threshold that preserves "the desirable intrinsic properties of the CNTs."** *Id.*, [0024]. For instance, Gan emphasizes that, when finding a suitable level for functionalization, the goal is to "**minimiz[e] the effect on the desirable intrinsic properties** of the CNTs." For instance, Gan teaches the **suitable level of oxidation at "about 2.5 to 10 wt%" and that "while a greater degree of functionalization may improve the dispersibility of the CNTs, such improvement may come at a decrease in intrinsic properties of the pristine CNTs.**" *Id.* This indicates a safe oxidation level range that preserves the intrinsic properties of the carbon nanotubes, including electrical conductivity of pristine nanotubes, while maximizing their dispersibility in water or polymer.

IPR2024-01011: LGES-1003, ¶¶112-113; Pet. 43-45

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

First three motivations for oxidized nanotubes (Cont'd)

Patent Owner's Response

Petitioner's specific motivations to oxidize at Gan's disclosed levels also fail. The first "motivation" is "to achieve a better constructed electrically conductive network of nanotubes." Pet., 42. The second "motivation" offered is "to enhance dispersion of carbon nanotubes." *Id.*, 43-44. Petitioner acknowledges that both motivations are premised on the benefits provided by the functional groups associated with oxidation. *Id.* 42-44. However, those functional groups would be essentially all removed during calcining, undermining both motivations. MRD-2006 ¶193.

Petitioner's reply

In particular, PO asserts that several motivations are premised on the benefits provided by functional groups associated with oxidation. POR, 66-68. PO then asserts that these benefits would be eliminated by calcination. *See id.* However, as explained above, this conclusion is not supported, and is inconsistent with the chemistry of the Cheng-Gan precursor mixture.

Further, PO overlooks the first motivation discussed in the Petition, which cites LGES-1032. LGES-1032 describes LFP synthesis and calcination process similar to that of Cheng-Gan, in which LGES-1032 describes functionalized CNTs were uniformly dispersed and attached into $LiFePO_4$ during calcination (e.g., 700 °C for 10 hours), forming "highly dispersed conductive networks," while retaining their dispersion. Petition, 43; LGES-1003, ¶110; LGES-1032, 1; LGES-1040, 2. As the Declaration explains, "uniform dispersion of CNT networks into the $LiFePO_4$ is crucial for performance." LGES-1003, ¶110

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fourth motivation for oxidized nanotubes

Dr. Zhu's Declaration

114. The fourth reason of using oxidized carbon nanotubes in Cheng's design, as described in Gan, is that extensive applications of oxidized or functionalized carbon nanotubes were well-known in various fields including lithium ion battery industry. *See* LGES-1023 (Bosnyak), Abstract, [0005], [0014], [0032], [0037]; LGES-1024 (Sue), [0006] [0056] [0101]; *see supra* Part IV.A.2. & A.5.(i).(1d). For instance, Gan identifies "commercial sources such as CheapTubes, Inc . . . and Carbon Solutions, Inc.," where oxidized nanotubes having specific oxidation levels can be obtained. These sources highlight their broad applications, particularly in "[e]nergy [s]torage" components, and more particularly, in "lithium-ion batteries." LGES-1021 (Gan), [0018]; LGES-1011 (stating "CNTs have the intrinsic characteristics desired in material used as electrodes in batteries and capacitors"). Further, for instance, the Patent Owner of the '924 patent describe, in a patent application that is a prior art herein, use of exfoliated oxidized carbon nanotube in electrodes for lithium ion batteries. *See* LGES-1023 (Bosnyak), Abstract, [0005], [0014], [0032], [0037], [0074], [0077], [0078].

Patent Owner's Response

The fourth "motivation" is that oxidized CNTs were well-known and commercially available. Pet., 45. The mere fact that oxidized CNTs were used in the industry and available for purchase says nothing about whether there is a *reason* to use them in the hypothetical Cheng-Gan combination. MRD-2006 ¶195. *PersonalWeb Techs., LLC v. Apple, Inc.*, 848 F.3d 987, 994 (Fed. Cir. 2017).

IPR2024-01011: LGES-1003, ¶114; Pet. 45; POR, 68

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Fifth motivation for oxidized nanotubes

Dr. Zhu's Declaration

115. The fifth reason of using oxidized carbon nanotubes in Cheng's design, as described in Gan, is that a POSITA would have recognized well-known facts that the [1] "role of oxidation is to create 'foothold' functional groups, such as carboxyl groups, which then serve as attachment points to anchor . . . nanoparticles" such as $LiFePO_4$ and [2] oxidized carbon nanotubes improve the "colloidal stability of CNTs," ensuring their uniform dispersion. *See* LGES-1010 (Wepasnick), 2 ("In functionalization strategies, the role of oxidation is to create ''foothold'' functional groups, such as carboxyl groups, which then serve as attachment points to anchor either larger biomolecular structures [31,32] or nanoparticles [19,28–32]"), 2 ("surface oxidation has been shown to exert a pronounced effect on both the sorption properties and colloidal stability of CNTs [12,13,29,30]"). Based on this knowledge, along with teachings of Cheng and Gan, a POSITA would have understood that the functional groups on oxidized nanotubes [1] facilitate the bonding between carbon nanotubes and nanoparticles (such as lithium iron phosphate) and [2] improve the "colloidal stability of CNTs." *Id.* This supports Cheng's design objective of achieving better dispersion of both the carbon nanotubes themselves and throughout the lithium iron phosphate. *See* LGES-1007 (Cheng), [0010]–[0014]. Further, as described above, Gan specifies a suitable degree of oxidation level (e.g., "2.5–10 wt%"), which would have motivated a POSITA, as they would have understood that this level of oxidation

Patent Owner's Response

The fifth "motivation" is that it was known that oxidation creates "anchors" and improve "colloidal stability of CNTs." Pet., 46. Again, however, the alleged benefits here are tied to the functional groups associated with the oxidation. MRD-2006 ¶196. Cheng-Gan's calcination would remove these "anchors," eliminating the purported benefits. *Id.*

FISH.

IPR2024-01011: LGES-1003, ¶115; Pet. 46; POR, 68

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Reasonable expectation of success

Dr. Zhu's Declaration

118. First, as described above, the oxidized carbon nanotubes or the oxidation levels taught in Gan: [1] remain within the safe threshold that preserves (or does not harm) the desirable intrinsic properties of the carbon nanotubes; [2] help producing better constructed electrically conductive network of carbon nanotubes within the composite; [3] help reduce contact resistance between carbon nanotubes to thereby enhance conductivity of the nanotube composite; [4] improve the dispersibility of carbon nanotubes; [5] improve the "colloidal stability of CNTs" to further ensure uniform dispersion of carbon nanotubes; and [6] provide enhanced "sorption properties," as they would offer further "attachment points to anchor" lithium iron phosphate. LGES-1010 (Wepasnick), 1, 2; LGES-1021 (Gan), [0018]-[0024], [0029], [0030]-[0033]; LGES-1032 (Zhou), 1.

119. Second, oxidation (or functionalization) of carbon nanotube with carboxyl or hydroxyl groups, or effect of such oxidation in enhancing electrical conductivity of the electrode or nanotube composite and improving dispersibility of the carbon nanotubes were well-known in lithium ion battery industry. *See* discussion *supra* Part IV.A.2. As described above, Kavan states that "the LiFePO olivine exhibits excellent performance in a composite with MWNT-ox" and that "the improvement" in electrochemical activation of $LiFePO_4$ "caused by surface oxidation of nanotubes is dramatic" and discloses cyclic voltammograms with results for electrodes composed of a composite of $LiFePO_4$ and non-oxidized nanotubes and electrodes composed of a composite of $LiMnPO_4$ and oxidized nanotubes. *Id.* Again, Kavan characterizes the results for the oxidized nanotube based composite as "impressive" in terms of both "capacity" and "charge/discharge reversibility." *Id.*

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Reasonable expectation of success

Dr. Zhu's Declaration

120. Third, enhancing "sorption properties" between carbon nanotubes and ion active materials (or transition metal oxide) based on functionalization of carbon nanotubes was well-known in the lithium ion battery industry. *See* LGES-1010 (Wepasnick), 2; LGES-1030 (Xiang), 2; LGES-1027 (Yu), 6. For instance, it was well-known that carboxyl or hydroxyl groups on functionalized nanotubes offer further "attachment points to anchor either larger biomolecular structures or nanoparticles." *See* LGES-1010 (Wepasnick), 2. Further, it was demonstrated that ion active materials or transition metal oxide, such as copper oxide or titanium dioxide precursors or nanoparticles that are formed on the carbon nanotubes, interact or react with the carboxyl or hydroxyl groups of the functionalized carbon nanotubes to form attachment or chemical bonding. LGES-1027 (Yu), 6 ("Such surface functional groups provide active sites for directed deposition of anatase nanocrystals around the surface of MWCNTs. For example, the –OH groups on the Ti precursor possibly react with the –OH and –COOH groups on the MWCNTs surface through esterification to form C–O–Ti or O=C–O–Ti bonds"); LGES-1030 (Xiang), 2 ("Due to the electrostatic effects, some Cu (II) ions added in the solution are absorbed in the outward end of the micelles while some are still dispersed in the solution (Step B). Once NaOH solution is dropped in, $Cu(OH)_2$

121. Fourth, oxidation levels that fall within the range disclosed in Gan (2.5–10 wt%) were well-known to be used extensively in various technical fields, including lithium ion battery. *See supra* Part IV.A.5.(i).(1d); *see also* LGES-1011.

122. Fifth, oxidation levels that fall within the range disclosed in Gan (2.5–10 wt%) were well-known to enhance electrochemical activity of the nanotube composite materials with LiFePO4." *See supra* Part IV.A.2 & A.5.(i).(1d). For instance, Kavan demonstrates an **oxidation level of 9.1%** and states that in the $LiFePO_4$/MWNT-ox composite "[t]he improvement caused by surface oxidation of nanotubes is dramatic" and "[i]t is obvious that the functionalization of nanotubes is, indeed, crucial for enhancing the electrochemical activity of the composite materials with $LiFePO_4$." LGES-1005 (Kavan), 7, 8. For instance, Kavan indicates good electrochemical performance in composites of oxidized nanotubes and $LiMnPO_4$, stating that "the oxidative treatment of nanotubes improves the reversibility of $LiMnPO_4$ charge/discharge." *Id.*, 7.

IPR2024-01011: LGES-1003, ¶¶120-122; Pet. 49-50

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Reasonable expectation of success

Patent Owner's Response

3. Petitioner fails to demonstrate a reasonable expectation of success in light of the calcining step.

A POSITA would have had no reasonable expectation of success in combining Cheng-Gan such that the CNTs maintain (1) Gan's required minimum level of 2.5 wt% or (2) the minimum level of 1 wt% required by the claims. MRD-2006 ¶200. Petitioner was required to demonstrate a reasonable expectation of success *in the Petition*, but instead simply ignored the calcination step of Cheng-Gan. Indeed, implicit in all of Petitioner's "reasons" for a reasonable expectation of success is the incorrect assumption that the oxidation levels in Cheng-Gan would "fall within the range disclosed in Gan (2.5–10 wt%)." Pet., 48-50. However, a POSITA would have had a reasonable expectation of success of achieving oxidation above either level ***when considering the effects of calcination***. *Id.* ¶¶199-200.

Petitioner's reply

In particular, PO asserts that several motivations are premised on the benefits provided by functional groups associated with oxidation. POR, 66-68. PO then asserts that these benefits would be eliminated by calcination. *See id.* However, as explained above, this conclusion is not supported, and is inconsistent with the chemistry of the Cheng-Gan precursor mixture.

Further, PO overlooks the first motivation discussed in the Petition, which cites LGES-1032. LGES-1032 describes LFP synthesis and calcination process similar to that of Cheng-Gan, in which LGES-1032 describes functionalized CNTs were uniformly dispersed and attached into $LiFePO_4$ during calcination (e.g., 700 °C for 10 hours), forming "highly dispersed conductive networks," while retaining their dispersion. Petition, 43; LGES-1003, ¶110; LGES-1032, 1; LGES-1040, 2. As the Declaration explains, "uniform dispersion of CNT networks into the $LiFePO_4$ is crucial for performance." LGES-1003, ¶110

In sum, PO's position fails to account for the nature of the Cheng-Gan precursor mixture and the in situ synthesis involved. At least for the reasons set forth above, a POSITA would not be discouraged from relying on Gan's oxidized CNTs, and the technical objective of the combination would have been achieved. Petition, 41-44, 63. That is, as conceded by PO, "a POSITA would have had a reasonable expectation of success of achieving oxidation above either level [claimed level or Gan's level] when considering the effects of calcination." POR, 68-69.

IPR2024-01011: POR, 69-70; Pet. Reply, 24

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

ISSUE 4C

The Cheng-Gan nanotubes are discrete

[1a] "[A composition used in lithium ion batteries comprising] discrete carbon nanotubes"

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Petition

First, in the combination, Cheng describes and claims methods of "carbon nanotube dispersion," in which the carbon nanotube disperses [1] themselves and [2] in a "precursor" that includes lithium iron phosphate. *See* LGES-1007, [Abstract], [0010], [0012]-[0015], claim 2. Cheng states that these methods address issues such as [1] carbon nanotubes not being "fully dispersed" [2] carbon nanotubes "being distributed only on the surface of the electrode material," [3] carbon nanotubes not "form[ing] a continuously connected three-dimensional electrically conductive network structure both in the interior and on the surface of the material." *See id.*, [0010]; LGES-1003, ¶127. Accordingly, Cheng focuses on solving the issues of nanotube dispersion and states that resulting composite material contains nanotubes that are "**uniformly dispersed**." *See id.*, Abstract, [0010], [0012], [0041], claim 1; LGES-1003, ¶127.

Cheng

(57) Abstract

The present invention belongs to the technical field of lithium ion battery materials, and in particular relates to a carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof. The method includes: ultrasonically dispersing carbon nanotubes, acetylene black, and a dispersing agent in water or an organic solvent to obtain a mixture; mixing a lithium source, an iron source, and a phosphate salt, simultaneously adding an organic carbon source and the mixture, and performing ball-milling and drying; and drying the same, then calcining the same in an inert protective atmosphere, and performing sieving to obtain a composite cathode material wherein carbon nanotubes are uniformly dispersed in a bulk phase and on surfaces of lithium iron phosphate, wherein the carbon nanotubes account for 0.5-15% by weight, and the lithium iron phosphate accounts for 85-99.5% by weight. When the obtained composite

[0012] An in situ preparation method of a carbon nanotube/lithium iron phosphate composite cathode material comprises carbon nanotube dispersion (dispersion of carbon nanotubes and dispersion in a precursor) and an in situ solid phase reaction process. A specific preparation process comprises:

2. An in situ preparation method of the carbon nanotube/lithium iron phosphate composite cathode material according to claim 1, characterized by comprising carbon nanotube dispersion and an in situ solid phase reaction process, a specific preparation process comprising:

(1) carbon nanotube dispersion: weighing carbon nanotubes, acetylene black, and a dispersing agent, ultrasonically dispersing the same in water or an organic solvent to obtain a mixture, a low-concentration mother liquor containing 0.5-8% of carbon nanotubes and acetylene black by weight, the ratio of the weight of carbon to the weight of the dispersing agent being (20 to 5) : 1, the weight of carbon being the sum of the weight of the carbon nanotubes and the weight of the acetylene black, and the weight ratio of the carbon nanotubes to the acetylene black being (0.1 to 10) : 1;

IPR2024-01011: Pet. 51; LGES-1007, Abstract, [0012], claim 2
DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Petition

Second, in the combination, Cheng emphasizes that the carbon nanotube composite material obtained based on the disclosed methods result in **non-aggregated** and **uniform dispersion** of nanotubes. *See* LGES-1007, Abstract, [0007], [0010], [0021]. In addition to emphasizing that the nanotubes of composite are uniformly dispersed and form an uniformly distributed network structure, Cheng emphasizes that the nanotubes are in **non-aggregated state**. For instance, Cheng describes that when there is an "agglomerat[ion]," it is difficult to form an "effective conductive network" or an "uniformly distributed network structure." *Id.*, Abstract, [0007], [0010], [0021], [0023], [0073; LGES-1003, ¶129.

Third, in the combination, Cheng describes use of diameter and length that contribute to **lesser aggregation** and **better dispersion** of carbon nanotubes, thereby contributing to the **uniform dispersion** and **non-aggregated state** of **nanotubes**. For instance, Cheng states that the described ranges of diameter (e.g., 10 to 200 nm) and length (e.g., 5 to 20 nm) contribute to forming of "effective conductive network," where "the carbon nanotubes are **less entangled** with each other and are **more easily dispersed**." LGES-1007, [0021]; LGES-1003, ¶130.

Cheng

(57) Abstract

The present invention belongs to the technical field of lithium ion battery materials, and in particular relates to a carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof. The method includes: ultrasonically dispersing carbon nanotubes, acetylene black, and a dispersing agent in water or an organic solvent to obtain a mixture; mixing a lithium source, an iron source, and a phosphate salt, simultaneously adding an organic carbon source and the mixture, and performing ball-milling and drying; and drying the same, then calcining the same in an inert protective atmosphere, and performing sieving to obtain a composite cathode material wherein carbon nanotubes are uniformly dispersed in a bulk phase and on surfaces of lithium iron phosphate, wherein the carbon nanotubes account for 0.5-15% by weight, and the lithium iron phosphate accounts for 85-99.5% by weight. When the obtained composite

[0073] In the present comparative example, since the tube diameter of the carbon nanotubes was too small and the length was greater, mutual entanglement was severe. The carbon nanotubes and the acetylene black were agglomerated, and it was difficult to form an effective conductive network.

[0021] The high-performance carbon nanotube lithium iron phosphate composite material obtained in the present invention has the following features: carbon nanotubes or carbon nanofibers are uniformly distributed on the surface of and in the lithium iron phosphate material by means of an in situ solid phase reaction. In the present invention, it has been found that the tube diameter and length of the carbon nanotubes have a considerable effect on utilization of the properties of the composite electrode material. Thus, the carbon nanotubes used in the present invention need to have the tube diameter of 10 to 200 nm and the length of 5 to 20 μm, and the optimal effect is achieved when the carbon nanotubes have the tube diameter of 50 to 150 nm and the length of 10 to 15 μm. Compared with carbon nanotubes having a smaller tube diameter and a greater length, the carbon nanotubes are less entangled with each other and are more easily dispersed. Therefore, formation of an effective conductive network is facilitated. Only when the effective conductive network is formed, the conductive network and the acetylene black can act on each other to achieve a synergistic effect.

IPR2024-01011: Pet. 52-53; LGES-1007, Abstract, [0021], [0073]

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Petition

Fourth, in the combination, Cheng is modified to use **oxidized nanotubes** as in Gan , and the method of carbon nanotube dispersion (dispersion of carbon nanotubes and dispersion in a precursor) is similar to a dispersion method disclosed in the '924 patent. For instance, the '924 patent has two examples (Examples 2 & 3), which allegedly describe "Discrete Carbon Nanotubes with Lithium Iron Phosphate Coated on the Surface of the Tubes Suitable for a Lithium Ion Battery Cathode Material." LGES-1001, 5:30-67, 6:1-4. Here, the Example 2 of the '924 patent describes sequential steps of [1] forming oxidized carbon nanotube / solvent mixture based on ultrasonic dispersion, [2] mixing the mixture with lithium source, iron source and phosphate source with mixing medium (e.g. deionized water), and [3] heat treatment. Similarly, Cheng, modified to use oxidized nanotubes as in Gan, disclose each and all of the above-noted steps of Example 2 in sequential order. *See* LGES-1007, Abstract, [0013]-[0019], claim 2. As explained by Dr. Zhu, this suggests that similar steps in forming the composite would have likely yielded similar dispersion results. LGES-1003, ¶131.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Cheng

(57) Abstract

The present invention belongs to the technical field of lithium ion battery materials, and in particular relates to a carbon nanotube/lithium iron phosphate composite cathode material and an in situ preparation method thereof. The method includes: ultrasonically dispersing carbon nanotubes, acetylene black, and a dispersing agent in water or an organic solvent to obtain a mixture; mixing a lithium source, an iron source, and a phosphate salt, simultaneously adding an organic carbon source and the mixture, and performing ball-milling and drying; and drying the same, then calcining the same in an inert protective atmosphere, and performing sieving to obtain a composite cathode material wherein carbon nanotubes are uniformly dispersed in a bulk phase and on surfaces of lithium iron phosphate, wherein the carbon nanotubes

[0013] (1) Carbon nanotube dispersion: weighing a certain quantity of carbon nanotubes, acetylene black, and a dispersing agent, ultrasonically dispersing the same in water or an organic solvent to obtain a mixture, a low-concentration mother liquor containing totally 0.5-8% of carbon nanotubes and acetylene black by weight, the ratio of the weight of carbon (the sum of the weight of the carbon nanotubes and the weight of the acetylene black) to the weight of the dispersing agent being (20 to 5) : 1, the weight ratio of the carbon nanotubes to the acetylene black being (0.1 to 10) : 1, and the dispersing agent being polyvinylpyrrolidone (PVP) or polyvinyl alcohol (PVA).

[0014] (2) Precursor dispersion: according to a method of preparing a lithium iron phosphate material, selecting a raw material, mixing the raw material with the mixture from step (1), and simultaneously adding an appropriate organic carbon source, pyrolytic carbon formed by the organic carbon source in a subsequent heat treatment process being capable of acting synergistically with the carbon nanotubes in performance to improve performance of the electrode material.

'924 Patent

Example 2

Discrete Carbon Nanotubes with Lithium Iron Phosphate Coated on the Surface of the Tubes Suitable for a Lithium Ion Battery Cathode Material

0.2 grams of multi-wall carbon nanotubes (CNano, grade C9000) oxidized to about 8 weight percent and aspect ratio about 60 are added to 23.5 grams of diethylene glycol and sonicated for 15 minutes. The solution is grey. 1.16 grams of iron acetate dissolved in 4 ml of deionized water is added and the mixture heated under nitrogen, stirring and sonication. After 1 hour at 140° C. a solution of 0.65 grams of lithium acetate and 0.74 grams of ammonium phosphate in 4.2 ml of deionized water is added. The temperature is raised to about 185° C. over 30 minutes, then held at 185° C. for 4 hours. The mixture is cooled to 110° C. and sonicated for 5 minutes followed by further cooling to ambient temperature while stirring. The solids are separated from the mixture by centrifugation followed by ethanol washing. The resultant powder is dried to constant weight vacuo.

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

IPR2024-01011: LGES-1007, Abstract, [0013]-[0014]; LGES-1001. 5:31-52; Pet. 53-54

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Petition

Alternatively/additionally, a POSITA would have understood or found obvious that the nanotubes in the combination composition are **discrete** because, at least in some instances, oxidation of nanotubes can help promote **non-agglomeration of nanotubes**. LGES-1003, ¶132. Oxidized nanotubes are functionalized by species such as carboxylic acid or hydroxyl groups. *See* LGES-1010, Abstract; LGES-1014, 821-822; LGES-1015, 4186-4187; LGES-1008, [0027]-[0028]; LGES-1003, ¶132. As indicated by these references and as explained by Dr. Zhu, the presence of these functionalities can contribute to physically and chemically inhibiting aggregation of nanotubes into clumps; instead, oxidized nanotubes are generally present as individually distinct nanotubes. LGES-1003, ¶71. In light of this behavior of oxidized nanotubes, a POSITA would have understood or found obvious that oxidized nanotubes in the combination can be present as generally individually distinct nanotubes, *e.g.*, that the nanotubes are **discrete**. LGES-1003, ¶132.

Patent Owner's Response

v. Petitioner's "alternative" argument

Petitioner also offers the "alternative" argument that "a POSITA would have understood or found obvious that the nanotubes in the combination composition are **discrete** because, at least in some instances, oxidation of nanotubes can help promote **non-agglomeration of nanotubes**." Pet., 54 (emphasis in original). However, the only evidence offered by Petitioner is that oxidation can help "inhibit[] aggregation of nanotubes into clumps." *Id.* At best, this merely evidences that oxidation can help *disperse* CNTs—not that it results in *discrete* CNTs. *Id.* Indeed, CNTs can be uniformly dispersed throughout a composition to form a continuous network, but that does not mean they are "discrete." MRD-2006 ¶244. Petitioner's leap in logic—that "behavior of oxidized nanotubes" necessarily achieves discrete CNTs—is entirely unsupported. *Id.*; *See also* LGES-1005, 3 (top right of Fig. 2 showing oxidized CNTs that are still highly entangled).

FISH.

IPR2024-01011: Pet. 54-55; POR, 83

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Dr. Zhu Declaration

71. The sidewalls and/or ends of oxidized nanotubes are functionalized by species such as carboxyl, carbonyl, or hydroxyl groups. *See* LGES-1005 (Kavan), Abstract ("Multi-walled carbon nanotubes functionalized by carboxylic groups"); LGES-1010 (Wepasnick), Abstract ("Six commonly used wet chemical oxidants … were evaluated in terms of their effects on the surface chemistry and structure of MWCNTs … X-ray photoelectron spectroscopy (XPS) and energy dispersive spectroscopy (EDX) were used to characterize the extent of surface oxidation, while chemical derivatization techniques used in conjunction with XPS allowed the concentration of carboxyl, carbonyl, and hydroxyl groups at the surface to be quantified"); LGES-1014 (Gojny), 821-822 (describing that oxidation of carbon nanotubes resulted in in "[t]he presence of carboxylic acid groups on the surface"); LGES-1008 (Farrugia), [0028] ("oxidation is carried out by acid-treatment, wherein oxidation chemistry may be used to open the end caps of both single and multi-walled nanotubes to produce carboxyl, carbonyl and hydroxyl groups at the opened ends and defects on the side walls"). As I explained above, by the presence of these functionalities, oxidized nanotubes are physically and chemically inhibited from aggregation into clumps, and instead are generally present as individually distinct nanotubes. *See* LGES-1014 (Gojny), 821-822 ("[t]he presence of carboxylic acid groups on the surface" "enables a better dispersion of CNTs" showing images of "oxidised [sic] MWCNTs … [that] are completely dispersed in the [epoxy resin] matrix due to the appearance of carboxylic groups on the surface"); LGES-1015 (Xing), 4186-4187 ("treated [oxidized] CNTs had better dispersion"); LGES-1008 (Farrugia), [0027] (describing that functionalization of carbon nanotubes "results in one or more chemical moieties attached to the carbon nanotubes," which "results in better dispersion of the nanotubes throughout the matrix").

IPR2024-01011: LGES-1003, ¶71; Pet. 54-55

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Patent Owner's Response

In summary, Cheng and Gan both seek a "continuous" or "percolated" network of CNTs, whereby the CNTs are non-agglomerated and better dispersed throughout a composition. *Id.* ¶210. Neither reference teaches or suggests that the CNTs are discrete or that having discrete CNTs is a goal. *Id.* Indeed, having discrete CNTs in Cheng and Gan would run contrary to the purpose of both references, which is to maintain a *continuous* or *percolated* network of CNTs throughout. *Id.* There is no indication that either Cheng or Gan recognized that having discrete CNTs attached to the surface of the active material provides any benefit, such as retardation of active material cracking. *Id.*

b. Petitioner fails to show "discrete" CNTs.

Despite Cheng and Gan both seeking a continuous network of CNTs, Petitioner argues that Cheng-Gan would result in "discrete" CNTs. Pet., 51-55. However, Petitioner's arguments are premised on incorrectly equating "discrete" CNTs with "uniformly dispersed" or "non-agglomerated" CNTs. Pet., 51-55. CNTs can be "non-agglomerated" and "uniformly dispersed" without being "discrete." MRD-2006 ¶211. For example, a continuous network of CNTs can be uniformly distributed throughout an electrode composition, in which case the CNTs may be "non-agglomerated" and "uniformly dispersed." *Id.* However, those CNTs are not "discrete" because they are continuously connected (i.e., each CNT is connected to another CNT). *Id.* This is precisely the type of CNT network that Cheng and Gan

5. Cheng and Gan teach away from "discrete" CNTs.

A POSITA "would be led in a direction divergent from the path that was taken by the" inventors, because Cheng and Gan seek a continuous or percolated network of CNTs to improve conductivity—the opposite objective of achieving discrete CNTs having active material attached to their surface (e.g., to retard cracking). *In re Gurley*, 27 F.3d at 553. Discrete CNTs could not form part of the "continuous" network of CNTs sought by Cheng or Gan. Indeed, Gan teaches that two goals of CNT functionalization is to "draw the CNTs ***closer together***" and "provide[]s a ***conductive bridge between CNTs*** to reduce the electrical contact resistance therebetween" MRD-2006 ¶¶245-246 (citing LGES-1021 ¶¶[0020], [0022]). These goals run contrary to the achieving "discrete" CNTs. *Id.* A POSITA would be discouraged from functionalizing CNTs as taught by Gan, because Gan teaches that functionalization would be less likely to result in discrete CNTs. *Id.* ¶247.

FISH.

IPR2024-01011: POR, 73, 84

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cheng-Gan nanotubes are discrete

Petitioner's reply

Importantly, while PO asserts that a POSITA "would be led in a direction divergent from the path that was taken by the" inventors, because "Cheng and Gan seek a continuous or percolated network of CNTs to improve conductivity—the opposite objective of ['926 patent] achieving discrete CNTs ... to retard cracking"—this argument is directly contradicted by the '924 Patent itself. POR, 84. As Dr. Zhu explains, the '924 patent repeatedly associates "discrete" CNTs with enhanced electrical conductivity. LGES-1001, 1:49-55, 2:29-33, 2:9-16, 2:17-21, 5:51-53; Table 1; LGES-1056, ¶¶50-51.

The disclosures in the '924 Patent, noted by Dr. Zhu, are irreconcilable with PO's argument that "discrete" CNTs must be electrically isolated. The specification of the '924 Patent does not define "discrete" as excluding all contact between CNTs. To the contrary, it emphasizes that discrete CNTs enhance electrical conductivity, which requires at least some degree of interconnection of nanotubes. For instance, the '924 Patent states that the "crystals [are] bound to and interconnected with discrete carbon nanotubes," which indicates that the discrete CNTs form a conductive network—i.e., they are not agglomerated, but still make point-to-point contact sufficient to interconnect active material particles. LGES-1001, 1:60-61, 2:22; LGES-1056, ¶52.

Dr. Zhu Supplemental declaration

The '924 patent repeatedly associates "discrete" CNTs with enhanced electrical conductivity. For example:

- "Carbon nanotubes as conventionally made . . . result in a **tangled bundle of tubes** . . . [a]lso lithium nanocrystals are not ionically or covalently attached to the surface of the **discrete carbon nanotubes** to provide for **intimate electron transfer**." LGES-1001, 1:49-55.
- "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." LGES-1001, 2:29-33.
- "The present invention **overcomes the difficulties of low electrical conduction** . . . by attaching nanosized crystals or nanosized layers of lithium ion active material to the surface of **discrete**, functionalized **and well dispersed** carbon nanotubes." LGES-1001, 2:9-16.
- "Likewise, for the lithium battery anode materials, active anode materials, such as carbon particles, tin oxide or silicon, can be attached to the discrete carbon nanotube surfaces to provide numerous benefits such as improved capacity, **electron and ion conductivity** and mechanical strength." LGES-1001, 2:17-21.
- "Example 2 . . . The resultant dried powder is determined to have a **[reduced electrical] resistance** of 2,000 ohm-cm **compared to a commercially obtained** sample determined as 19,000 ohm-cm." LGES-1001, 5:51-53.
- As shown in Table 1 of the '924 Patent, the '924 Patent focuses on the conductivity improvement, with Table 1 comparing conductivity/resistance. LGES-1001, Table 1.

IPR2024-01011: Pet. Reply, 25-26; LGES-1056, ¶51

FISH.

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE