UNITED STATES PATENT AND TRADEMARK OFFICE

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BEFORE THE PATENT TRIAL AND APPEAL BOARD

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LG ENERGY SOLUTION, LTD.,
Petitioner

v.

MOLECULAR REBAR DESIGN, LLC,
Patent Owner

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Case IPR2024-01005
U.S. Patent 9,636,649

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PATENT OWNER'S RESPONSE

LGES 1057
LGES v. MRD
IPR2024-01011

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EXHIBIT LIST

Exhibit No.	Description
MRD-2006	Declaration of Dr. Deborah D.L. Chung, Ph.D.
MRD-2007	U.S. Patent Publication No. 2012/0058255 ("Gan")
MRD-2008	PCT Publication WO 2011/163129 ("Bosnyak-129")
MRD-2009	PCT Publication WO 2010/117392 ("Bosnyak-392")
MRD-2010	Deposition Transcript of Dr. Samuel P. Gido in IPR2024-01005, dated March 13, 2025
MRD-2011	Deposition Transcript of Dr. Samuel P. Gido in IPR2024-01012, dated March 20, 2025
MRD-2012	WEBSTER'S NEW WORLD COLLEGE DICTIONARY (4th ed. 1999), definition of discrete
MRD-2013	Declaration of Dr. Yu Zhu in IPR2024-01013, dated June 12, 2024

I. Introduction

Patent Owner presents substantial new evidence and arguments demonstrating that Petitioner fails to carry its burden of proving unpatentability of any claim.

Ground 1 (Sun alone and in combination with Bureatea, Kaji, and Matushita) is fundamentally flawed. First, Petitioner overlooks the fact that Sun does not teach the actual generation of a dispersion of discrete, *multiwall* carbon nanotubes ("MWCNTs). Sun tries to, but while Sun is successful in creating a dispersion with fully exfoliated single wall carbon nanotubes ("SWCNTs"), Sun is forced to admit it has not done it for MWCNTs. *Infra* Section V.B. Second, Sun teaches a vast range of potential aspect ratios for its CNTs (over 10,000) in contrast to the claimed narrowly tailored range. A POSITA would not see experimenting to "optimize" within Sun's range of aspect ratios as routine, because A POSITA would understand that the immense range of aspect ratios in Sun encompass compositions of carbon nanotubes ("CNTs") that have significant structural and behavioral differences—requiring experimentation of over thousands of potential values of aspect ratios—would be far from obvious. *Infra* Section V.C. Third, because each of the Ground 1 combinations relies on Sun *alone* to teach these limitations, the Petition has failed to show enablement for the sections of Sun it has relied upon. Since Sun fails to

generate a dispersion of discrete, *multiwall* carbon nanotubes, a POSITA would be forced either to experiment unduly with the parameters of Sun's disclosed methodology or to experiment unduly with a whole new one. *Infra* Section V.D. Fourth, Petitioner cannot plausibly carry its burden with respect to claims 2, 3, 6, and 8. *Infra* Sections V.E.1 and V.E.2. Since the Petition fails to demonstrate Sun successfully generating a dispersion of discrete, multiwall carbon nanotubes at all, much less at the weight percentage of claim 1, it has also failed to demonstrate any teaching of the higher weight percentage of claim 2, the narrowed weight percentage ranges of claims 6 and 8, and that they are in the form of free flowing particles as described by claim 3.

Ground 2 (Farrugia and Narayan) is also deeply flawed. First, Petitioner fails to adequately explain how merely mentioning "discrete carbon nanotubes" one time in Farrugia, can teach a dispersion of discrete, multiwall carbon nanotubes. *Infra* Section VI.B. The generation of such dispersions is difficult, as demonstrated by Sun's failure, and neither Farrugia or Narayan even try. Second, this absence of any teaching of any kind leaves a POSITA without any enabling content from which to even begin what would clearly be a daunting level of experimentation. *Infra* Section VI.C. Third, the range of aspect ratio values a POSITA would learn from the combination of Farrugia and Narayan would preferably be in excess of 1000,

teaching away from the clamed range of 25-500. Fourth, with respect to claims 2 and 3, the Petition's failure to demonstrate that discrete, multiwall carbon nanotubes are taught by the combination, it cannot be said to teach a dispersion where 70% of them by weight are discrete, or that they are free flowing particles.

II. Level of Ordinary Skill

Patent Owner applies Petitioner's proposed definition of a POSITA for purposes of this proceeding only. Patent Owner's technical expert, Dr. Deborah Chung, has a Bachelor's Degree in Engineering and Applied Science and a Master's Degree in Engineering Science from the California Institute of Technology. MRD-2006 ¶5. Dr. Chung also has a Master's Degree in Materials Science and a Doctorate in Materials Science from the Massachusetts Institute of Technology. *Id.* A central focus of Dr. Chung's decades of technical experience has been studying carbon nanotubes ("CNTs"). *Id.* ¶¶14-17. Thus, Dr. Chung qualifies as a POSITA.

III. Overview of the '649 Patent

United States Patent No. 9,636,649 (the '649 Patent) is directed to a dispersion of oxidized, discrete, MWCNTs. LGES-1001, 13:50-55. At the time of the invention of the '649 Patent, there was significant interest in using CNTs in polymer composites as a reinforcing agent. *Id.*, 1:31-33. "However, utilization of carbon nanotubes in these applications [had] been hampered due to the general inability to

reliably produce individualized carbon nanotubes." *Id.*, 1:33-36 In order to reach their full potential in these materials, it was believed that the CNTs needed to have aspect ratios substantially greater than 40, and it was known that the maximum aspect ratio for a given tube length is reached when each tube is fully separated from another. *Id.*, 1:36-39; MRD-2006 ¶62.

The '649 Patent describes its inventors' method of creating discrete MWCNTs. LGES-1001, 6:3:25. The claimed dispersion has MWCNTs with aspect ratios between 25-500 that comprise less than about 30% of the weight of the dispersion. *Id.,* 13:50-55. The dispersion is intended to be used in elastomer composite blends, and the claimed dispersion is the result of a better process of creating individualized MWCNTs with sufficient values of the aspect ratio. *Id.*, 1:20-23, 1:36-39. It overcomes prior art processes that disentangle using aggressive oxidative means that result in aspect ratios that are too small, as well as using MWCNTs that have small enough values of the aspect ratio to avoid the increase in viscosity that make the suspension unusable. *Id.*, 1:46-49, 1:54-62; MRD-2006 ¶63. The methods described in the '649 Patent came after experimenting with less successful methodologies such as those in PCT Publication WO 2011/163129 (MRD-2008, "Bosnyak-129") and PCT Publication WO 2010/117392 (MRD-2009, "Bosnyak-392").

IV. Claim Construction

The Board is required to construe "'only those terms … that are in controversy, and only to the extent necessary to resolve the controversy.'" *Realtime Data, LLC v. Iancu*, 912 F.3d 1368, 1375 (Fed. Cir. 2019) (quoting *Vivid Techs., Inc. v. Am. Sci. Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999)). Because there is a controversy regarding the meaning of "discrete carbon nanotube fibers" (Claim 1) and that controversy is material to whether Petitioner has carried its burden of showing unpatentability, claim construction of that term is necessary.

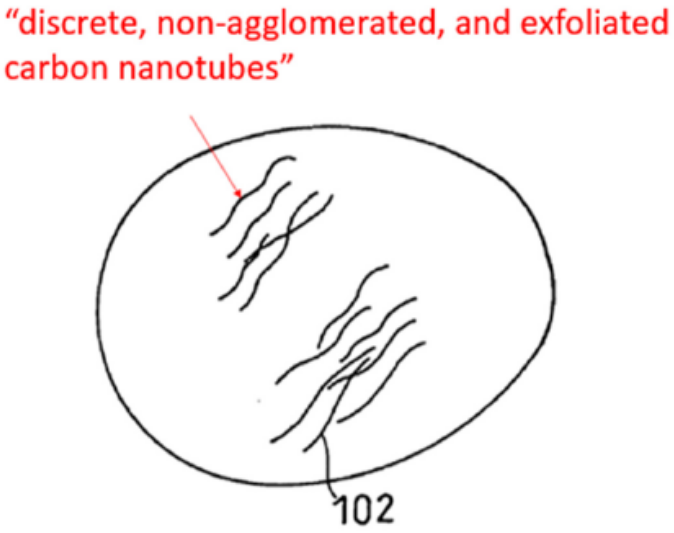
A POSITA would thus understand that the plain and ordinary of meaning of "discrete carbon nanotubes" are carbon nanotubes that are separate and distinct from other nanotubes; not attached to other nanotubes. MRD-2006 ¶54. The face of the patent teaches this. *Id*. For example, the specification teaches that an "embodiment of the invention is a method for obtaining *individually* dispersed carbon nanotubes." LGES-1001, 3:8-9. The background discusses hampered utilization of CNTs because of the "inability to reliably produce individualized carbon nanotubes." *Id.*, 1:34-36. The methodology of the specification uses the fact that "carbon nanotube fibers can have oxidation species comprising of carboxylic acid or derivative carbonyl containing species and are essentially discrete individual fibers." *Id.*, 5:36-39. And the specification goes on to describe mixtures of master batches where the

individually dispersed nanotubes are distributed whether uniformly or non-uniformly in each rubber domain." *Id.*, 5:65-67. The specification also teaches that the carbon nanotube fibers are "exfoliated," which a POSITA would understand that "exfoliation" refers to the untangling of carbon nanotubes from one another, allowing the CNTs to be separated from one another to become "discrete." LGES-1001, 4:33-44; MRD-2006 ¶54.

This understanding of "discrete carbon nanotubes" is consistent with teachings from the specification above and the common meaning of discrete. MRD-2006 ¶54; (citing MRD-2012, 4 (defining "discrete" to mean "separate and distinct; not attached to others; unrelated")). This construction is also consistent with the plain and ordinary meaning of "discrete carbon nanotubes" offered by Petitioner's expert, Dr. Yu Zhu, in co-pending IPRs challenging three other CNT-related patents of Patent Owner. *Id*. ¶55. For example, in reference to the annotated figure below, Dr. Zhu testified:

> And looking again to Ohata's illustrative example, provided below, it can be seen that ***most*** of the nanotubes are depicted as being discrete, non-agglomerated, and exfoliated. Indeed, a POSITA would have understood a carbon nanotube that is, as shown below, ***fully isolated*** from each other to be discrete, non-agglomerated, and exfoliated.[1]

[1]All emphasis is added unless otherwise indicated.



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

MRD-2013 ¶83. Dr. Zhu thus considers "most" (but not all) of the CNTs to be discrete because they are individual, distinct and separate/not attached (i.e., "fully isolated") from the other CNTs. This is consistent with his deposition testimony, where he testified that the nanotube pointed to by the red arrow above was identified as "discrete" by him "*because* it's fully isolated from the other nanotubes." MRD-2010, 10:21-11:6; *id.*, 12:17-13 (testifying that discrete CNTs "have been *separated from each other*"); *id.*, 13:13-22 (responding "yeah" to being asked "Your testimony was that discrete carbon nanotubes are non-bundled nanotubes that are *separate from each other*, correct?"); MRD-2006 ¶56.

Although Petitioner does not expressly propose this term for construction,

Petitioner and its expert at times suggest "discrete carbon nanotube fibers" means carbon nanotubes that are "lacking direct association with other nanotubes ***along substantially the length of the nanotubes***." Pet., 12 (citing LGES-1003 ¶¶42-44) and 55 (citing LGES-1003 ¶¶215-216. Petitioner's unusually specific and subjective construction should be rejected for several reasons.

For one, it is inconsistent with the common meaning of discrete. Petitioner's own expert, Dr. Zhu, testified about the ordinary meaning of "discrete carbon nanotubes," and he explained that they are CNTs which are "fully isolated" or "separate" from one another. MRD-2006 ¶¶56-57; MRD-2010, 10:21-11:6; *see also id.*, 12:17-13 (testifying that discrete CNTs "have been *separated from each other*"); *id.*, 13:13-22 (responding "yeah" to being asked "Your testimony was that discrete carbon nanotubes are non-bundled nanotubes that are *separate from each other*, correct?"). Petitioner cites no evidence to support deviating from the ordinary meaning of discrete, as support by the teachings of the specification, the common usage of the term, and Dr. Zhu's testimony.

Petitioner suggest that the specification defines "discrete" to mean "exfoliated." *See, e.g.*, Pet., 12-13. However, as explained above, discrete CNTs are necessarily exfoliated because such CNTs have to be disentangled from the raw bundle of CNTs to be fully isolated or detached from one another. MRD-2006 ¶58.

Consequently, discrete CNTs are necessarily exfoliated but exfoliated CNTs are not necessarily discrete, as exfoliated CNTs can remain attached to another CNT (e.g., via Van der Waals forces). *Id*.

Additionally, Petitioner provides no explanation as to how or why its proposed construction allows for *some* "direct association" with other CNTs but not "along substantially the length of the nanotube." Pet., 12 (citing LGES-1003 ¶¶42-44), 55 (citing LGES-1003 ¶¶215-216); MRD-2006 ¶57. There is no support in the specification for this construction, which appears to have contrived by Petitioner to try and ensnare CNTs from the prior art that are clearly not discrete as claimed. Indeed, Dr. Gido struggled mightily during his deposition to explain how a POSITA would even determine whether CNTs qualify as "discrete" under his unsubstantiated, subjective interpretation. MRD-2011, 8:11-19 ("Q. And what is a substantial portion of its length? A. That is not well, you know, defined, necessarily. ***You just have to look at these things*** and see whether they look like they're aggregating or associating, and, you know, it's not along a substantial or most of its length or a significant part of its length."); *id.*, 9:1-11 ("Q. 50 percent. Would that be substantial? A. I didn't really think -- I mean I was not asked to think about and attach a number to a particular, you know, a particular amount of association. I would say that 50 percent is a fair amount of association.").

In fact, Dr. Gido even testified that under his interpretation ***entangled*** CNTs could be "discrete." *Id.*, 9:24-10:10 ("Q. Does a discrete carbon nanotube fiber have to be disentangled from another carbon nanotube? To the extent that entanglement might involve them being associated with one another along a substantial part of their length, you know, they shouldn't be, although certainly in some cases, you know, particularly long fibers might loop around one another without actually being stuck together.").

Petitioner and Dr. Gido's proposed construction is inconsistent with the common meaning of "discrete," the usage of "discrete" throughout the specification, and the understanding of "discrete" offered by Dr. Zhu regarding other CNT patents. Petitioner's proposal should be rejected, and the plain and ordinary meaning—separate and distinct; not attached to others—should apply. MRD-2012, 4; MRD-2006 ¶55.

V. <u>Grounds 1A-1D</u>: The Sun Combinations Do Not Render Any Claims Obvious

Each claim of the '649 Patent requires discrete ***MWCNTs***, not discrete single wall carbon nanotubes ("SWCNTs"). LGES-1001, claims 1-23. The Petition glosses over this requirement, stating that the discrete carbon nanotubes ("CNTs") of Sun's aqueous solution and nanocomposite are multiwall. Pet., 22. They are not. To reach

this mistaken conclusion, Petitioner incorrectly reads Sun's experimental statements and findings as applying to MWCNTs, even though Sun admits that they do not. Sun performs its experiment on SWCNTs separately from MWCNTs and gets fundamentally different results. *Infra* Section V.A. and V.B. Indeed, while Sun's procedure does successfully produce fully exfoliated and individualized SWCNTs, Sun ***admits that it was unable to achieve that same result for MWCNTs***. LGES-1004 ¶[0069]. Because Sun itself does not teach discrete MWCNTs—and in fact, admits it was unable to achieve discrete MWCNTs via experimentation—Sun alone does not meet every element of the claims. *Id.*

In addition to Sun failing to achieve discrete MWCNTs, Sun also fails to render obvious the claimed aspect ratio range of 25 to 500. *Infra* Section V.C. Sun only discloses the meaningless aspect ratio range of "at least about 10" that "may be greater than about 10,000." LGES-1004 ¶[0038]. This is effectively a range of 10 to essentially infinity. *Id.* However, Federal Circuit precedent dictates that when the claimed range is narrow (i.e., 25 to 500) and the prior art range is overlapping and broad (i.e., "at least about 10" to "greater than about 10,000"), such broad range is insufficient to show prima facie obviousness, if the structure associated with the narrow range is different than the structure of associated with the broad range. *ee Genetics Inst., LLC v. Novartis Vaccines & Diagnostics, Inc.*, 655 F. 3d 1291, 1304-

07 (Fed. Cir. 2011). "Our case law is clear that the structure of a claimed compound and its properties are inseparable for purposes of § 103." *Id.* at 1307 (citing *Sanofi-Synthelabo v. Apotex, Inc*., 550 F.3d 1075, 1086 (Fed. Cir. 2008) ("For chemical compounds, the structure of the compound and its properties are inseparable considerations in the obviousness determination.")).

Sun also fails to enable a POSITA to create discrete MWCNTs. *Infra* Section V.D. The only guidance provided by Sun regarding its CNTs is that they have an aspect ratio range from about 10 to greater than about10,000, and Sun's failed results is indisputable evidence of how difficult it is to achieve discrete MWCNTS—as opposed to SWCNTs. *Id.*

A. Overview of Sun

Sun is directed to dispersing CNTs and creating polymer nanocomposites with those nanotubes. MRD-2006 ¶64 (citing LGES-1004, Abstract). Sun sought to overcome previous methods that would irreparably damage the polymers and teaches that its methods would be useful in electrical, thermal, and mechanical applications. *Id.* (citing LGES-1004 ¶¶[0006], [0009]).

Sun experiments (starting at LGES-1004 ¶[0054]) separately on SWCNTs and MWCNTs and reports the results separately. *Id.* ¶65 (*compare e.g.,* LGES-1004 ¶[0054] *with* ¶[0056]; *id.* ¶[0057] *with* ¶[0058]; *id.* ¶¶[0063]-[0066], Table 1 *with*

¶[0066], Table 2; *id.*, FIGURE 6 *with* FIGURE 7). Since the claims of the '649 Patent require MWCNTs, it is important to differentiate what Sun teaches with respect to MWCNTs and SWCNTs. *Id.*

While Sun discusses both SWCNTs and MWCNTs, it usually generalizes its description of nanotube parameters to "carbon nanotubes" without specifying whether it is talking about single wall or multiwall CNTs. MRD-2006 ¶66. Sun also gives parameter ranges without differentiating what portion of those ranges refer to SWCNTs or MWCNTs. *Id* (citing LGES-1004 ¶¶[0037] and [0038]). While a POSITA would understand and appreciate there is some overlap between SWCNTs and MWCNTs for many parameters, a POSITA would also understand SWCNTs and MWCNTs can have significant differences in other characteristics. *Id.* Moreover, a POSITA would understand that within either group—single or multiwall—there are substantial differences in structure and behavior between CNTs that have significantly different parameters. *Id.* For example, a MWCNT with a very large aspect ratio (e.g., greater than 1,000) will have significantly different structure and behavior than a MWCNT with a small aspect ratio (e.g., less than 50). *Id.*

Carbon nanotubes are often described using aspect ratio (length divided by diameter). *Id.* ¶67. This parameter significantly influences the properties of CNTs, such as reinforcement, conductivity, and dispersibility within materials. *Id.* Sun

teaches that its CNTs may have aspect ratios that are "at least about 10" but that can range to "at least about 20," "at least about 1,000," "greater than about 1,500," and even "greater than about 10,000." *Id.* (quoting LGES-1004 ¶[0038]). In other words, Sun discloses that its CNTs may have aspect ratios ranging from 10 to essentially infinity in practice. *Id.*

A POSITA would understand that while these aspect ratios could apply to single wall or multiwall CNTs, the structure and behavior of CNTs will differ significantly between CNTs with substantially different aspect ratios. *Id.* ¶68; *see Infra* IV.C. Further, even CNTs with similar aspect ratios can exhibit different properties depending on the number of walls the individual nanotube may have. *Id.* For example, MWCNTs can have 2 walls (referred to as "double-walled") or even beyond 14 walls. *Id.*

A POSITA would also understand that diameters and lengths can also differ significantly among different CNTs, depending, for example, on the number of walls the CNTs have, even among CNTs that have the same aspect ratio. *Id.* ¶69. For example, a CNT with aspect ratio 100 may have diameter 10 nm and length 1000 nm (1 μm), or have diameter 5 nm and length 500 nm (0.5 μm); these are very different values of the diameter and length. The greater is the number of walls, the larger tends to be the diameter. *Id.*

Sun describes the diameters of the CNTs in general as being "between about 1 nm and 20 nm." LGES-1004 ¶[0037]. A POSITA would understand that small diameters are less likely to be associated with MWCNTs, with diameters at 1 nm or below almost never being multiwall. MRD-2006 ¶70. Petitioner's expert concedes that it would not be reasonable to apply a 1 nm diameter to a MWCNT—even claiming he "certainly wouldn't do it." MRD-2010, 66:25–67:5. Sun also acknowledges this obliquely, "[a]s may be understood by one skilled in the art, a MWCNT may have a diameter that is greater than about 50 nm." LGES-1004 ¶[0037]; MRD-2006 ¶70.

The length of CNTs is another physical parameter a POSITA would understand can be quite different between SWCNTs and MWCNTs, even if those CNTs have similar aspect ratios. *Id.* ¶71. Similar to diameters, a POSITA would understand that larger CNT lengths are more likely to correspond to MWCNTs, due to the greater fragility of SWCNTs with only a single wall) compared to MWCNTs (with multiple walls). *Id.* Further, a POSITA would understand that in chemically or mechanically aggressive environments, very long CNTs are only suitable if they are MWCNTs, as the SWCNT are much more susceptible to breakup under those environments. *Id.*

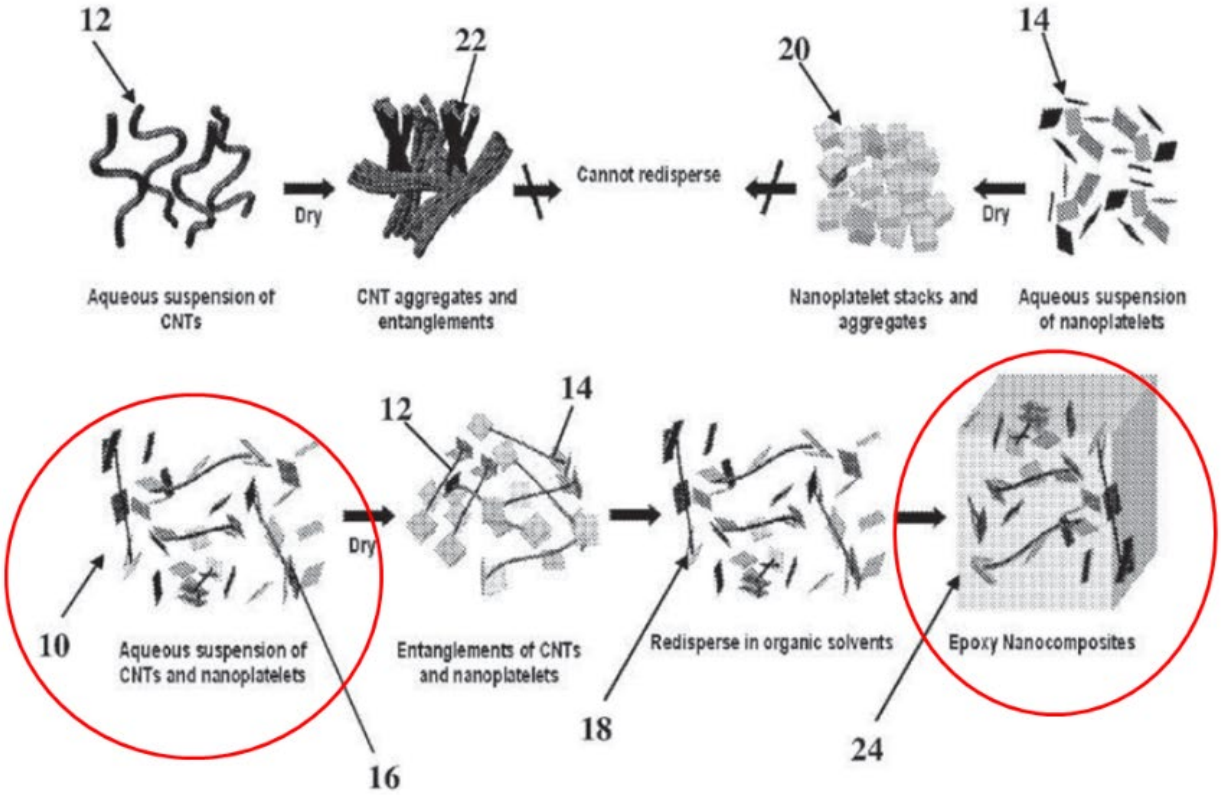
Sun describes its CNTs as having a length that "is at least about 100 nm; alternately, at least about 1000 nm, and in certain instances, the length of the nanotubes may exceed 1 μm." LGES-1004 ¶[0038]. A POSITA would understand that because Sun is using both SWCNTs and MWCNTs, the lengths in the upper ends of his ranges are most likely MWCNTs. MRD-2006 ¶72.

Sun again tacitly concedes this understanding by noting that:

> In certain instances, MWNTs may comprise a slightly longer length, and have a higher aspect ratio, compared to the SWNTs. In certain instances, MWNTs are preferred for their length and aspect ratio. Alternately, the SWNTs may be preferred as they have unique properties compared to the MWNTs.

LGES-1004 ¶[0038]).

In Sun, the CNTs (multiwall or single wall) are functionalized using acids and put into an aqueous suspension (numeral 10 in FIG. 1, annotated below) with nanoplatelets. LGES-1004 ¶¶[0039-][0040]). According to Sun, FIG. 1 is a "***conceptual scheme***" containing "individually dispersed CNTs and exfoliated nanoplatelets":



Sun, FIG. 1 (annotated by Patent Owner)

Id. ¶[0014]. Sun's process eventually results in the creation of an epoxy nanocomposite 24. MRD-2006 ¶74 (citing LGES-1004 ¶[0046, FIG. 1). Petitioner alleges that both the suspension 10 and the nanocomposite 24 qualify as the dispersion of claims 1 and 20. Pet., 11-16; MRD-2006 ¶74.

While Sun describes its conceptual scheme as applicable to CNTs generally, Sun's actual experimental results show that they clearly only apply to SWCNTs—and not MWCNTs. MRD-2006 ¶75. As explained further below, when taken as a

whole, Sun can only be said to teach achieving a dispersion (consistent with FIGURE 1) using SWCNTs. *Id.* Indeed, Sun admits that it was unable to achieve discrete MWCNTs, which makes sense when the fundamental differences between SWCNTs and MWCNTs are considered. *Id.* (citing LGES-1004 ¶[0069].

B. Sun Does Not Teach Discrete MWCNTs as Claimed

To meet the elements of all claims of the '649 Patent, a reference or combination of references, must teach a dispersion of discrete, "***multiwall***" CNTs. LGES-1001, claims 1-23. Sun does not. While Sun's experiment successfully generates a dispersion of fully exfoliated SWCNTs, Sun expressly teaches that it was unable to achieve the same results with MWCNTs. MRD-2006 ¶¶75-76.

The Petition relies heavily on Sun's FIG. 1 as evidence that Sun's process results in the dispersion of discrete, multiwall carbon nanotubes as required of the '649 Patent. *Id.* ¶77 (citing Pet., 11-20). However, FIG. 1 is a generalized "***conceptual scheme***" illustration of what Sun hypothesizes is the carbon nanotube behavior through its experimental process. *Id.* (citing LGES-1004 ¶¶[0014], [0038]). The illustrated figure, and accompanying text, appear to generalize this behavior across "single walled nanotubes, few walled nanotubes, multi-walled nanotubes or combinations thereof," *Id.* (citing LGES ¶[0038]), even though a POSITA would understand that various types of CNTs—e.g., single-walled, double-

walled nanotubes, multi-walled nanotubes or combinations thereof—would be expected to behave differently under similar conditions. *Id.*

Moreover, a POSITA would understand that there will be significant structural and behavioral differences between CNTs of vastly different aspect ratios regardless of CNT walls and types, and thus, will lead to CNTs of significantly different aspect ratios behaving differently in Sun's experimental scheme. *Id.* ¶78. Thus, a POSITA would be skeptical that CNT behavior in such a scheme could be uniformly generalized across a remarkably large range of aspect ratios and all conceivable numbers of CNT walls and types. *Id.*

A POSITA would understand that Sun's experiments (starting at LGES-1004 ¶[0054]) are an attempt to produce results that validate the "***conceptual scheme***" depicted in the illustration of FIG. 1 and take particular note of the *differences* in the results for MWCNTs as compared to SWCNTs. *Id.* ¶79. Sun is unambiguous about its success in "demonstrate[ing] that [its] approach can **fully exfoliate SWNT** bundles into individual tubes." *Id.* (quoting LGES-1004 ¶[0065]).

Sun also includes actual TEM images of said success in the epoxy nanocomposite:

> Further, the effectiveness of the above approach to exfoliate bundled SWNTs in epoxy is shown in FIG. 7. The pretreated-SWNTs are still in a bundled state with significant bundle-to-bundle entanglements

> (FIG. 7 a). After mixing with exfoliated ZrP nanoplatelets using the above drying-redispersion method, SWNTs can be fully debundled and well dispersed in epoxy matrix as in FIGS. 7*b* and 7*c*.

Id. ¶80 (quoting LGES-1004 ¶[0070]).

But noticeably, when discussing the MWCNT results, Sun states that the experiment was only able to "obtain dispersed MWNTs and **fully exfoliated <u>ZrP nanoplatelets</u>**." *Id.* ¶81 (quoting LGES-1004 ¶[0068]). Sun does not state that it achieved fully exfoliated MWCNTs; Sun only claims that the ***nanoplatelets*** are fully exfoliated. *Id.*

Similarly, Sun's description of the TEM images of MWCNT results differ markedly from those of SWCNT results. *Id.* ¶82. Sun describes FIG. 6 (the images of MWCNT experiment) as follows:

> FIG. 6 shows the TEM images of epoxy nanocomposites with 0.2 and 0.4 wt. % of MWNTs. The concentrations of exfoliated nano platelets are 1.0 and 2.0 wt%, respectively. Well-dispersed MWNTs and **full exfoliation of nanoplatelets** can be observed.

Id. (quoting LGES-1004 ¶[0069]).

Again, there is a clear difference between the observed results of MWCNTs (shown in FIG. 6) and SWCNTs (shown in FIG. 7). *Id.* ¶83. For SWCNTs, Sun touts "the effectiveness of the above approach to **exfoliate bundled SWNTs** in epoxy is shown in FIG. 7." *Id.* (quoting LGES-1004 ¶[0070]).

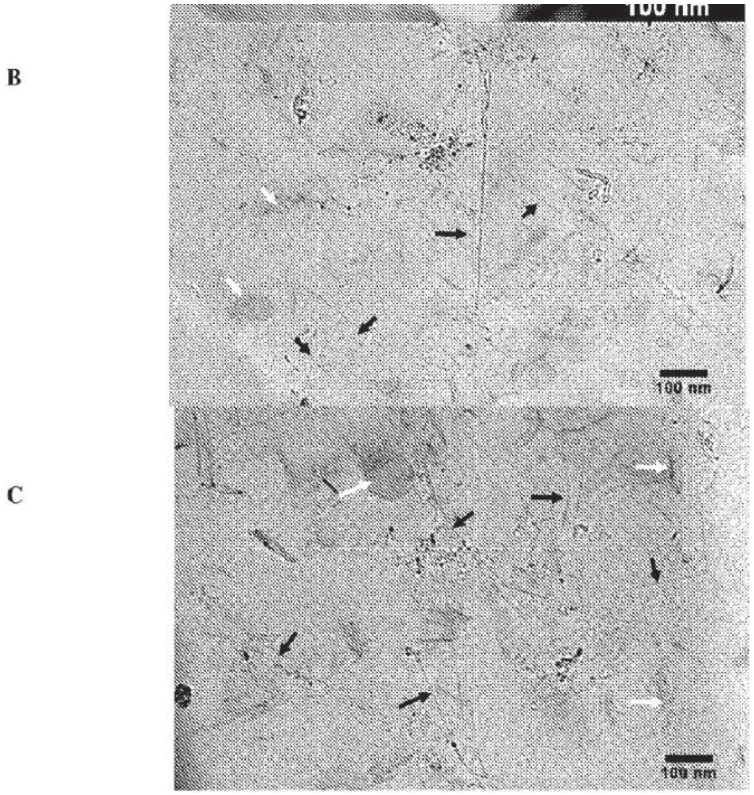
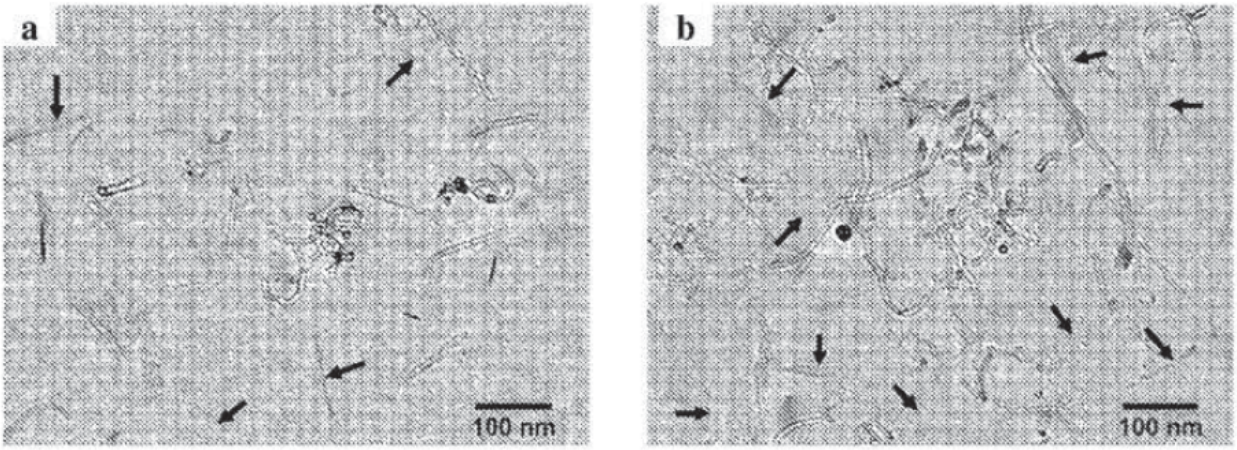


FIGURE 7

LGES-1004, FIG. 7.

In FIG. 7B, Sun's annotations indicate that "black arrows are individually dispersed and straight **SWNTs**." LGES-1004 ¶[0025]. Likewise, in FIG. 7C, Sun's annotations indicate that "red arrows are individually dispersed and straight **SWNTs**." LGES-1004 ¶[0026]. Notably, because there are only two example images of these results, and the reproduction of those images in the Sun reference is of poor quality, the SWNTs identified by the arrows are very difficult to discern and it is impossible to tell whether they are actually "fully exfoliated." MRD-2006 ¶84.

For the results of its MWCNT experiment, however, Sun only has "arrows indicating **exfoliated <u>ZrP nanoplatelets</u>**" in FIGs. 6a and 6b, and no arrows calling out CNTs at all:



LGES-1001, FIGs. 6a, 6b

Id. ¶85 (quoting LGES-1004 ¶¶[0022]–[0023]).

Similar to FIGs. 7B and 7C, FIGs. 6a and 6b are of very poor quality and it is very difficult to discern what are, and are not, MWCNTs. *Id.* ¶86. However, these images clearly do not show discrete MWCNTs. *Id.* In fact, and most importantly, Sun does not proclaim to show successful experimental results of fully exfoliating MWCNTs for this process, as he unhesitatingly does for SWCNTs. *Id.*

To the contrary, Sun expressly concedes that he was unable to achieve fully exfoliated MWCNTs:

> The TEM images shown here clearly **suggest** that through the aqueous dispersion, organic redispersion approach, both MWNTs and nanoplatelets **can be** fully dispersed down to individual level in epoxy matrices.

Id. ¶87 (quoting LGES-1004 ¶[0069]). Rather than stating that the experiment "shows" that it has been fully dispersed down to individual level, Sun's experiment merely "suggests" that it could be. *Id.* A POSITA would thus understand that Sun did not achieve fully exfoliated MWCNTs, but at most that Sun ***believed*** his experimental results merely suggest that such a result ***could possibly be*** achieved under different, unspecified conditions. *Id.* However, Sun does not disclose those specific conditions—it merely discloses an aspect ratio of "at least 20" to "greater than about 10,000." *Id.* (quoting LGES-1004 ¶[0038]).

From these results (success with SWCNTs and failure with MWCNTs), a POSITA would understand that Sun's "conceptual scheme" in FIG. 1 is a teaching applicable to SWCNTs, but is *not* a teaching applicable to MWCNTs. *Id.* ¶88. Further, a POSITA would see Sun's failure to create a dispersion of discrete MWCNTs understandable despite its success creating discrete SWCNTs. *Id.*

To take one example, a POSITA would understand that even under optimal conditions, nanoplatelets would likely be less successful at affecting bundles of MWCNTs than they would be at affecting bundles of SWCNTs. *Id.* ¶89. The surface

area of a CNT increases in proportion to its radius, but its volume increases by the *square* of its radius. *Id.* Therefore, as the CNT diameter increases, the volume of a CNT will increase *much faster* than the surface area, and hence the volume will increase much faster than the number of sites on the CNT for the nanoplatelets to attach. *Id.* A POSITA would understand that the specific surface area (surface area per unit mass) of a SWCNT is much higher than that of a MWCNT. *Id.* Thus, a POSITA would not be surprised that a procedure leveraging specific surface area, such as one counting on the mechanical forces of nanoplatelets attached to the outside surface of CNTs for disentangling the CNTs, would not be as successful with CNTs having low specific surface area (MWCNTs) as it might be for CNTs having high specific surface area (SWCNTs). *Id.*

And this is what Sun's results show. *Id.* ¶90. Sun creates a dispersion of discrete SWCNTs but fails to create a dispersion of discrete ***MWCNTs***. *Id.* Accordingly, a POSITA having read the entirety of Sun—including the results of the SWCNT and MWCNT experiments—would understand the conceptual illustration of FIG. 1 to only be applicable to SWCNTs. *Id.* Because Sun as a whole conveys to a POSITA, that FIG. 1 is only applicable to SWCNTs, a POSITA would not understand it to disclose discrete ***MWCNTs*** as claimed. *Id.* Petitioner thus fails to carry its burden of demonstrating unpatentability based on Sun.

C. Sun Does Not Render Obvious the Claimed Aspect Ratio Range of 25 to 500

Petitioner argues that "Sun presents a *prima facie* case and renders obvious the recited range of aspect ratios" because "aspect ratios recited in element [1c] ('25 to 500') overlaps with Sun's disclosed range of aspect ratios ('at least about 10' or 'at least about 20,' LGES-1004 [0038]), and at least one specific instance of Sun's aspect ratio (100) falls within the claimed range." Pet., 21; MRD-2006 ¶91.

While "the at least one specific instance" of a CNT having an aspect ratio of 100 is within the generalized range Sun discloses, Sun never expressly discloses MWCNTs having an aspect ratio of 100. MRD-2006 ¶92 (citing LGES-1004 ¶[0038]. This "at least one specific instance" is an inference made by Petitioner from assuming a CNT of 1 nm diameter and 100 nm length (to arrive at a 100 aspect ratio). *Id*. (citing Pet., 20). But Petitioner's expert admits that this assumption is inappropriate for MWCNTs. *Id*.; MRD-2010, 66:25-67:5, 63:17–64:4.

But more importantly, Petitioner cannot rely on Sun's incredibly broad aspect ratio range to render the Challenged Claims as obvious. Despite the Petition's selectively pairing down Sun's disclosed aspect ratio range to "at least about 10" or "at least about 20," (Pet., 21), the actual range of aspect ratios disclosed by Sun is

enormous—from at least 10 to "greater than 10,000." MRD-2006 ¶93 (citing LGES-1004 ¶[0038]). Effectively, an infinite upper limit of the range. *Id.*

If the claimed range is narrow (i.e., 25 to 500 in the '649 Patent) and the prior art range is overlapping and broad (i.e., about 10 to greater than about ***10,000*** in Sun), such broad range is insufficient to show *prima facie* obviousness if the *structure* of a CNT with an aspect ratio of about 10 to about greater than 10,000 is different from the *structure* of a CNT with an aspect ratio of 25 to 500. *See Genetics Inst., LLC.,* 655 F. 3d at 1304-07. "Our case law is clear that the structure of a claimed compound and its properties are inseparable for purposes of § 103." *Id.* at 1307 (citing *Sanofi-Synthelabo*, 550 F.3d at 1086 ("For chemical compounds, the structure of the compound and its properties are inseparable considerations in the obviousness determination.")).

An overly broad range is insufficient to show obviousness, because such a range does not provide an ordinary motivation to optimize the broad range. *Id.* at 1306-07 (holding that ordinary motivation to optimize did not apply where disclosure was 68,000 truncated protein variants, comprising 2,332 amino acids, which were structural differences); *contra In re Peterson*, 315 F.3d 1325, 1330 n1 (Fed. Cir. 2003) ("Although ranges that are not especially broad invite routine experimentation to discover optimum values, rather than require nonobvious

invention, we do not have here any assertion that the disclosed range is so broad as to encompass a very large number of possible distinct compositions.”).

In *Genetics*, the Federal Circuit found that proteins in the claimed patent versus the prior art patent, differed *structurally* in terms of the size of the permitted amino acid deletions, the location of the permitted amino acid deletions, and the degree of allowable amino acid substitutions. *Genetics*, 655 F.3d at 1306. Because of such *structural* differences in the claimed invention and the prior art patent, and considering that the prior art patent disclosed a broad range of 68,000 protein variants comprising 2,332 amino acids, there was no motivation for a POSITA to be invited to perform routine experimentation to discover an optimum range. *Id.* In affirming the district court, the Federal Circuit found that where the disclosed range was so broad as to encompass a very large number of possible distinct compositions, *prima facie* obvious was not satisfied. *Id.*, 1306-07.

Here, with respect to Sun’s largest disclosed aspect ratio (greater than about 10,000) compared to the highest end of the claimed aspect ratio (500)—***a key structural difference***—Sun teaches at least 9,500 more potential aspect ratios compared to the claimed aspect ratio (i.e., 10,000 – 500 = 9,500). MRD-2006 ¶95 (citing LGES-1004 ¶[0038]; citing LGES-1001, 13:50-55). Additionally, the highest aspect ratio disclosed in Sun is at least 20x larger than the highest aspect ratio in the

claimed invention (i.e., 10,000 / 500 = 20). *Id.* The at least 9,500 more potential aspect ratios and the at least 20x larger difference in aspect ratios, are significant differences to create several additional differences relating to the structure and/or behavior of the oxidized CNTs of Sun versus the oxidized CNTs in the claimed invention. *Id.*

In general, a POSITA would understand that even at a single given CNT diameter, having a ***larger aspect ratio*** CNT versus a smaller aspect ratio CNT, is not only a key structural difference in the CNTs themselves, but may lead to the following *additional* differences in the structure and behavior of the CNTs: (1) increase in the number of bends, including increase in the likelihood of bending; (2) increase in the number of entanglements, (3) increase in the number of CNT-CNT contact points; (4) increase in the likelihood of reaching percolation (i.e., the establishment of a continuous conductive path due to the CNT-CNT contact points in the network); (5) increase in electrical conductivity of the CNT network due to increased approach to percolation; (6) decrease in dispersibility, thereby reducing the likelihood of having discrete CNTs and uniform CNT dispersion; and (7) increase in the number of defects (e.g., nanoscale indentations of the wall of the CNT). MRD-2006 ¶96. A POSITA would understand that a composition comprising CNTs with an aspect ratio of greater than 10,000 (as disclosed by the range of Sun)

would experience the foregoing effects to a significant degree, whereas a composition with an aspect ratio of 500 (as claimed) would not. *Id.*

Further, if the diameters and the lengths of the CNTs vary unknowably throughout the range of aspect ratios, as they almost certainly do in Sun, the differences resulting from disparate lengths remain, but are then scattered unpredictably across the entire range, making it even more difficult to account for their effects. *Id.* ¶97.

But even if we take the easier to conceptualize fictional case and assume the CNTs have the same diameter, a composition that comprises CNTs with an aspect ratio of greater than about10,000 as in Sun (versus a composition that comprises CNTs with an aspect ratio of 500 as in the claimed invention) would be expected to have an ***increase*** in: the number of bends, likelihood of bending, number of entanglements, likelihood of entangling, number of CNT-CNT contact points, likelihood of having CNT-CNT contact points, likelihood of reaching percolation, electrical conductivity of the CNT network due to the increased approach to percolation, number of defects, and likelihood of having defects. *Id.* ¶98.

In addition, taking the easier to conceptualize fictional case and assuming the CNTs have the same diameter, the composition of Sun would also have a ***decrease*** in dispersibility, reducing the likelihood of having discrete CNTs or uniformly

dispersed CNTs—due to the increase in the number of entanglements of the CNTs, which causes the formation of agglomerates of CNTs. *Id.* ¶99. In the case of Sun, where the diameters and length vary unpredictably across the aspect ratio range, there will be unpredictable increases and decreases in these structures and behaviors throughout the range. *Id.*

Further, taking the easier to conceptualize fictional case and assuming the CNTs have the same diameter, the CNTs in the composition of Sun have a ***higher*** chance of being in contact with adjacent CNTs, making the composition have a higher electrical conductivity compared to the claimed composition comprising the 500 aspect ratio CNT. *Id.* ¶100. This increase in electrical conductivity is due to the greater number of CNT-CNT contact points, which increases the likelihood of percolation in the CNT network. *Id.*

Finally, again taking the easier to conceptualize fictional case and assuming the CNTs have the same diameter, the composition of Sun has a longer length of CNTs compared to the length of the CNTs in the claimed invention. *Id.* ¶101. A CNT that has a longer length, as in Sun, would tend to have an ***increase*** in the number of defects and also a higher likelihood of having at least one defect along its length compared to a CNT that has a shorter length as in the claimed invention. *Id.* Depending on the nature and severity of the defects, the defects on a CNT can lead

to the loss of the CNT's structural integrity (i.e., mechanical property degradation) and also a reduction in its electrical conductivity. *Id.* The extent of the mechanical and electrical degradation depends on the number of walls in the CNT, which affects the diameter of the CNT. *Id.* For example, if a defect occurs at the outermost wall of a CNT, there is a more pronounced effect of the defect when the number of walls is smaller. *Id.*

As in *Genetics*, due to the numerous and significant differences in the structure and behavior of a greater than about 10,000 aspect ratio CNT composition (i.e., Sun) compared to a 500 aspect ratio CNT composition (i.e., the claimed invention), there is no motivation for a POSITA to try to optimize to find a narrower aspect ratio within Sun's remarkably broad range. MRD-2006 ¶102. Sun's teaching of up to 9,500 more aspect ratios and up to a 20x larger aspect ratio, when compared to the claimed aspect ratio, means that there are a large number of possible distinct compositions comprising CNTs (of various types) with various aspect ratios—similar to the broad range that *Genetics* found cannot support *prima facie* obviousness. Indeed, just as in *Genetics*, the numerous differences in structure and behavior of CNTs at these widely different aspect ratios are "nontrivial differences" that directly affect the resulting structure and behavior of the CNTs. *Id.*; *Genetics*, 655 F.3d at 1306 ("On the facts of this case, the nontrivial differences in the proteins

at issue compel the requirement of identifying a reason for the chemical modification."). Accordingly, Petitioner fails to make obvious the claimed aspect ratio of 25 to 500, because it relies solely on Sun's overly broad teaching of about 10 to greater than about10,000 aspect ratio.

D. Sun is Not Enabling

Given Sun's own failed results with MWCNTs, it is indisputable that a POSITA would need to conduct further testing and alterations to Sun's disclosed methods in order to actually achieve a dispersion of discrete, MWCNTs. MRD-2006 ¶105. Indeed, at best, Sun admits that its SWCNT results "suggest" that MWCNTs could potentially be fully exfoliated, but Sun does not disclose *how* to achieve those results. *Id.* (citing LGES-1004 ¶[0069]).

Sun does not disclose what additional testing would look like, including what alterations would need to be made to achieve the same SWCNT result with the fundamentally different MWCNTs. *Id*. ¶106. In Petitioner's Ground 1A, Sun *alone* is relied upon for arguing Claim 1 obvious. *Id*. (citing Pet., 2). For Petitioner's Ground 1D, Sun is placed in combination with Kaji and Matsushita for claim 20. *Id.* However, in arguing the combination's application to claim 20, the Petition appears to rely solely on Sun for teaching claim elements [20pre]-[20a] and [20d]. *Id*. (citing Pet., 46). Since the aspects of the '649 Patent's claimed dispersion including a

plurality of oxidized, discrete, multiwall carbon nanotubes are part of claim limitations [20a] and [20d], Sun is again, used *alone* by Petitioner for teaching these limitations as they apply to claim 20. *Id.*

"In the absence of such other supporting evidence to enable a skilled artisan to make the claimed invention, a standalone § 103 reference must enable the portions of its disclosure being relied upon." *Raytheon Techs. Corp. v. GE*, 993 F.3d 1374, 1377, 1380-1381 (Fed. Cir. 2021) (rejecting a standalone prior art reference [Knip] asserted in § 103 concerning a "futuristic turbine engine" as not enabled). "[I]f an obvious case is based on a non-self-enabled references and no other prior art reference or evidence would have enabled a skilled artisan to make the claimed invention, then the invention cannot be said to have been obvious." *Id.*, 1376. "Thus, Knip's self-enablement (or lack thereof) is not only relevant to the enablement analysis, in this case it is ***dispositive***." *Id.* at 1382.

But where would a POSITA even start? Sun discloses an aspect ratio range (about 10 to greater than about 10,000) that is practically ***infinite***, and its failure to provide what aspect ratio it used in either its successful SWCNT experiment or failed MWCNT experiment does not give a POSITA a place to even begin. MRD-2006 ¶107. Structure and behavior vary across different aspect ratios and vary *significantly* between greatly different aspect ratios. *Supra* Section V.C. A POSITA

reading Sun is left with experimenting over a large number (perhaps *thousands*) of aspect ratios. MRD-2006 ¶107.

Beyond the infinitely large number of aspect ratios disclosed in Sun, is the widely ranging experimental methodologies available to a POSITA, and the dozens of parameter choices that can potentially be modified to change the outcome. *Id.* ¶108. Sun describes one methodology, but there is also the methodology disclosed in Bosnyak-129, which uses acid treatments in combination with sonification. *Id.* ¶108 (citing MRD-2008 ¶[0076]). But methodologies such as these will require significant experimentation with the test parameters, such as residual metal concentration allowance (MRD-2008 ¶[0008]), filtration methods (MRD-2008 ¶[0011]), the weight concentration of acids (MRD-2008 ¶[0012]), the energy input levels for the sonic treatments (MRD-2008 ¶[0014]), the temperature environment duration for the acid treatment (MRD-2008 ¶¶[0015], [0017]), the weight concentration of nanotubes (MRD-2008 ¶¶[0018]-[0019]), the surface modification level (MRD-2008 ¶¶[0020]-[0023]), the various additives the nanotubes are blended with (MRD-2008 ¶¶[0024]-[0030]), and many others. MRD-2006 ¶108.

In other words, it is not even clear to a POSITA what particular methodology to begin with and enabling the unspecific teachings of Sun would require significantly more than simply plugging in any one of the thousands of potential

aspect ratios within Sun's range. *Id.* ¶109. The foregoing is evidence that Sun, asserted as a standalone § 103 reference by Petitioner, is not self-enabled and dispositive—it does not teach a POSITA how to make and use its disclosed compositions, and therefore, cannot be used to establish obviousness. *Raytheon Techs. Corp.*, 93 F.3d at 1377, 1382.

And even when starting with a similar methodology as the one disclosed in Sun, like in Bosnyak-392, there are multiple experimental parameters that need to be accounted for and potentially adjusted by a POSITA, such as the nature of an acid and filtering solution used and the specific composition of the nanocrystalline material. MRD-2006 ¶110 (citing MRD-2009 ¶¶[0011]-[0013]). Sun uses alpha-zirconium phosphate (ZrP), but even Sun itself says "nanoplatelets [can] comprise any suitable material as known to one skilled in the art without limitation, such as clay, nanoclay, graphite, inorganic crystal, organic crystal combinations thereof." *Id.* (quoting LGES-1004 ¶[0040]). Redispersion of the nanotubes can be "into different mediums, materials, matrices, plastics, polymers, composites, and the like without limitation," and include "any known ionic or nonionic surfactant." *Id.* (quoting LGES-1004 ¶[0052]).

Further, as taught in Bosnyak-392, the choice of nanoparticle composition is known to produce different outcomes for MWCNTs than it does for SWCNTs and

specifically states that "it is difficult to achieve separation of zirconium phosphate from the exfoliated carbon nanotubes when multi-wall carbon nanotubes are used." *Id.* ¶111. (quoting MRD-2009 ¶[0050]). That a POSITA would have to undertake undue experimentation to figure out what testing parameters would be needed to reach the claim limitations of the '649 Patent is dispositive here. *Raytheon Techs. Corp*., 993 F.3d at 1382 (stating that "Knip's self-enablement (or lack thereof) is not only relevant to the enablement analysis, in this case it is dispositive.").

Having to test over potentially thousands of different aspect ratios where structure and behavior of the MWCNTs vary widely, and having to choose between multiple methodologies with multiple experimental parameters whose adjustment could be outcome-determinable *is* undue experimentation. MRD-2006 ¶112. Indeed, Sun itself was unable to successfully achieve discrete MWCNTs, and presumably Sun used the testing parameters and MWCNT aspect ratios it believed to be best suited for that purpose. *Id*. This only further evidences that undue experimentation would be required to enable discrete MWCNTs as claimed based on the disclosures of Sun.

E. None of the Dependent Claims Are Rendered Obvious By the Combinations Based on Sun

Since neither independent claims 1 or 20[2] have been rendered obvious by the Petition's Grounds 1A-1D, none of the dependent claims are rendered obvious. However, the Petition also independently fails to demonstrate that dependent claims 2, 3, 6 and 7 are obvious.

1. Claims 2 and 3 are not rendered obvious under Ground 1A

Claim 2 of the '649 Patent requires that the dispersion of claim 1, wherein at least 70 percent by weight of the nanotubes are discrete. LGES-1004, claim 2. The Petition contends that a "POSITA would have found it obvious that given Sun's functionalization treatment, substantially all of the nanotubes are functionalized such that they are discrete, *e.g.*, **at least 70% by weight of the nanotubes are discrete**." MRD-2006 ¶113 (quoting Pet., 25). However, as shown previously, Sun does not

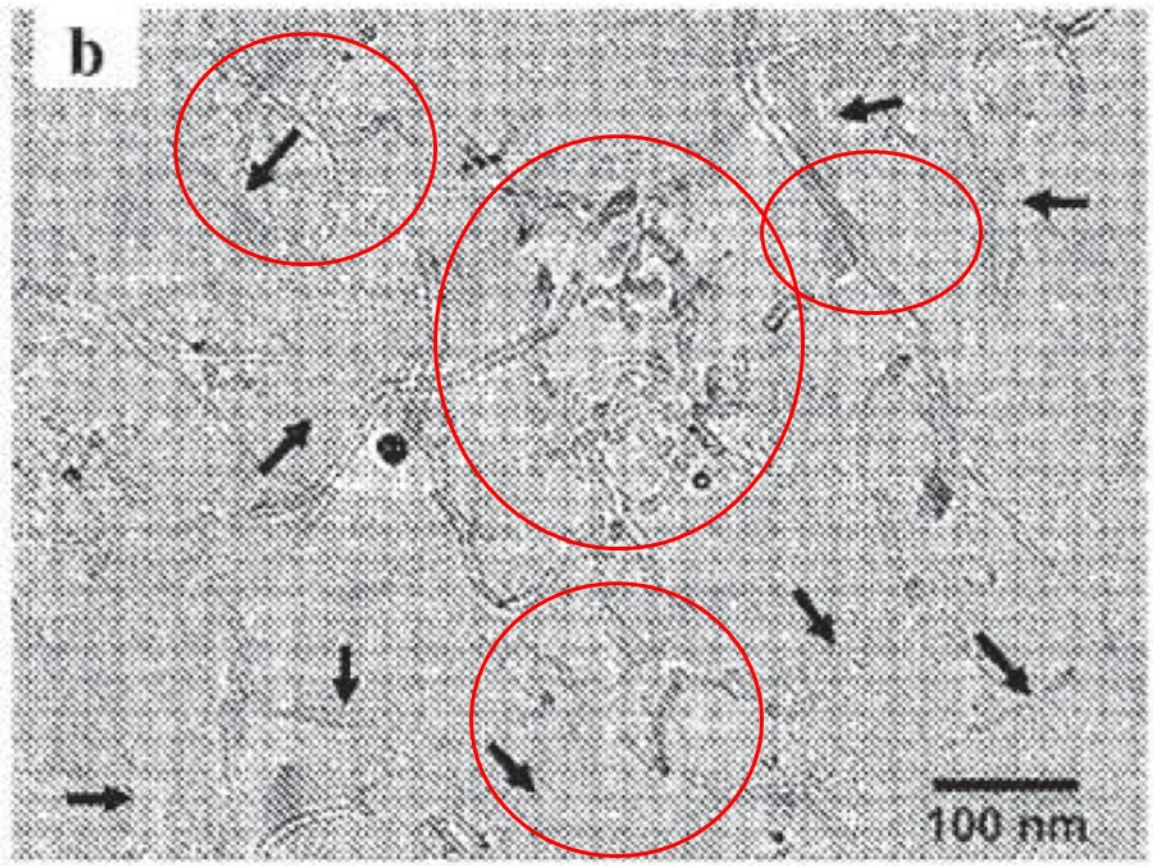
[2] Independent claim 20 has similar limitations as independent claim 1, such as requiring "multiwall," "discrete carbon nanotubes" having an "aspect ratio of 25 to 500." LGES-1001, claim 20. Thus, the Petition fails to render claim 20 as obvious by the combinations based on Sun for the same reasons. *See supra* Sections V.A. – V.D.

actually teach that *any* of the MWCNTs it experiments are discrete. *Supra* Sections V.A. and V.B. Far from rendering obvious a full 70% by weight level of discreteness, a POSITA would go into an interpretation of Sun knowing that following its procedures will not produce *any* discrete MWCNTs without undue experimentation. MRD-2006 ¶113.

Just as the Petition fails to demonstrate that Sun renders claim 1 obvious, claim 2's more stringent requirement of "at least 70% by weight" of the MWCNTs being discrete, cannot be rendered obvious over a prior art reference that fails to demonstrate the generation of even 1% by weight of discrete MWCNTs. *Id.* ¶114. And even if Sun could be read to actually disclose some discrete MWCNTs, nothing in Sun discloses the percentage of overall CNTs that would qualify or enable a POSITA to actually meet this high "discreteness" threshold. *Id.* Indeed, Petitioner's assertion that at least 70% of the MWCNTs would be discrete is pure speculation. It is not based on any actual technical analysis or data, such as an SEM or TEM image of a sample prepared according to Sun. *Id.*

Similarly, the Petition fails to demonstrate that claim 3's requirement that the dispersion be in the form of "free flowing particles" is obvious under Ground 1A. Pet., 26; MRD-2006 ¶115. Petitioner's sole rational is again, because a "POSITA would have understood or found obvious that the nanotubes in Sun's dispersion are

discrete, *e.g.*, that they are generally individual, separated nanotubes." Pet., 26. As demonstrated above, Sun does not generate a dispersion of discrete, ***multiwall*** carbon nanotubes. *Supra* Sections V.A. and V.B. Further, Sun's TEM images of the MWCNT experiment results demonstrates that they are *not* free flowing particles:



LGES-1004, FIG 6b (annotated by Patent Owner)

MRD-2006 ¶115 (citing LGES-1004, FIG. 6b). This image shows MWCNTs entangled with each other, which are <u>*not*</u> able to "flow freely relative to another

nanotube in the dispersion," as the Petition erroneously contends. *Id.* (quoting Pet., 26).

2. Claims 6 and 7 are not rendered obvious under Ground 1A

Similar to the failings of Sun's ever expanding aspect ratio range, Sun's weight percentage ranges do not render obvious the narrow weight percentage ranges required by claims 6 and 7. Claim 6 requires a weight percentage range of 15% to about 25% and claim 7 requires an even narrower weight percentage range of 18% to about 22%. LGES-1001, 14:7-12. In arguing that these claims are obvious over Sun, the Petition relies on Sun's disclosure of a weight percentage for its aqueous dispersion of "at least 1 wt%." Pet., 28. For the epoxy nanocomposite, the Petition relies on Sun's disclosure of a weight percentage range between "0.01 wt% and about 50 wt%." *Id* (citing LGES-1004 ¶[0045]). However, arguing that at least 1 wt% necessarily teaches every wt% above 1wt% is essentially arguing that Sun teaches 99% of all physically possible weight percentages. MRD-2006 ¶116.

The only concrete weight percentages for MWCNTs actually given by Sun are the "final MWNT concentration of 0.2 and 0.4 wt % in epoxy nanocomposites." MRD-2006 ¶117 (quoting LGES-1004 ¶[0058]). The structural and behavioral differences between concentrations of 0.4 wt% and 15 wt% or 18 wt% are

substantial. *Id.* The greater the wt% of the carbon nanotubes, whether single wall or multiwall CNTs, the lower is the likelihood of generating and maintaining discreteness among the CNTs. *Id.* Further, a jump from 0.4 wt% to 15 wt% or 18 wt% increases the viscosity of the resulting suspension, thereby degrading the processability of the suspension. *Id.* This degradation may necessitate substantial alteration of the composition and/or processing parameters, which a POSITA would need to consider. *Id.*; *see also supra* Section V.D.

Where the disclosed range is so broad as to encompass a very large number of possible distinct compositions, *prima facie* obvious is not satisfied. *See Genetics*, 655 F.3d at 1306-7 (finding 68,000 truncated variants of a protein made up of 2,332 amino acids is a situation where the prior art's disclosed range is so broad as to encompass a very large number of possible distinct compositions). Here, where the prior art's range is so broad as to be 99% of *all possible* concentrations, Sun cannot be said to have rendered the '649 Patent's narrow ranges obvious. This is particularly clear in light of the significant structural and behavioral differences between a dispersion of 0.4 wt% and one of 15 or 18%. MRD-2006 ¶118.

VI. Grounds 2A-2B: The Combinations Based on Farrugia and Narayan Do Not Render Any Claim Obvious.

As with Sun, the combinations of Farrugia and Narayan fail to disclose a dispersion of oxidized, discrete MWCNTs as required by the claims. Unlike Sun, which actually discloses a proposed methodology for generating and maintaining SWCNT discreteness (although one that does not enable a POSITA to successfully do so for MWCNTs), neither Farrugia or Narayan disclose any methodology that would generate or preserve a dispersion of discrete CNTs. This is unsurprising, since maintaining a dispersion of discrete carbon nanotubes would be counter to the shared purpose of Farrugia and Narayan—maximizing electrical conductivity via a network of interconnected CNTs.

A. Overview of Farrugia and Narayan

Farrugia describes a polymer-based printer toner that includes carbon nanotubes for increased conductivity. LGES-1007 ¶[0018]; MRD-2006 ¶119. "Because carbon nanotubes have a high aspect ratio, only small amounts of carbon nanotubes need to be present in a toner in order to achieve the same conductivity as compared to conventional conductive additives." LGES-1007 ¶[0018].

As the Petition concedes, Farrugia's objective for the inclusion of CNTs into its toner polymers is primarily to increase the electrical properties, namely the

conductivity of the resulting polymer-based composites. Pet., 46 (citing LGES-1007 ¶¶[0018]-[0019]); MRD-2006 ¶120. This enhanced electrical conductivity is "'[b]ecause carbon nanotubes have **a high aspect ratio**, [and] only small amounts of carbon nanotubes need to be present in a toner to achieve the same conductivity as compared to conventional conductive additives.'" Pet., 47 (citing LGES-1007 ¶[0018]); MRD-2006 ¶120.

Narayan is also seeking to increase the electrical conductivity of structures using high aspect ratio CNTs. *Id*. ¶121 (citing LGES-1008 ¶¶[0007], [0014]). In fact, [t]he amount and characteristics of the carbon nanotubes are 'selected **as to provide electrical** conductivity' to the composite." Pet., 48 (citing LGES-1008 ¶[0008]); MRD-2006 ¶120.

For Narayan, this conductivity is needed for foams and elastomers used in gaskets, and seals to provide electromagnetic shielding and electrostatic dissipation in microelectronic applications. *Id*. ¶122 (citing LGES-1008 ¶[0003]). To be effective in electromagnetic shielding and dissipation, one of the most common approaches involves providing high electrical conductivity via a network of electrically connected conductive units, such as a network of CNTs. *Id.* As with Farrugia, conductivity is key, and **high aspect ratios** CNTs are selected because

longer CNTs tend to provide conductivity in the composite even when the CNTs are present in relatively low proportions. *Id.*

B. The Farrugia-Narayan Combination Does Not Teach a Dispersion of Discrete, Multiwall Carbon Nanotubes

The only argument in the Petition for the combination of Farrugia and Narayan disclosing discrete CNTs is a single sentence from Farrugia: "Discrete nanotubes, aggregates of nanotubes, or both discrete and aggregate nanotubes may be suitable for use according to the present disclosure." Pet., 55 (quoting LGES-1008 ¶[0024]). But just because Farrugia starts with discrete CNTs, does not mean that that the final composition ends up with discrete CNTs. MRD-2006 ¶123.

Farrugia functionalizes its CNTs to, in part, "promote 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." MRD-2006 ¶124 (quoting LGES-1007 ¶[0027]). However, it is also well known that functionalization "[r]educes the contact resistance between CNTs incorporated into [a] conductive polymer material by *drawing the CNTs closer together*" and "[p]rovides a conductive bridge between CNTs to reduce the electrical contact resistance therebetween." *Id.* (quoting MRD-2007 ¶¶[0020]-[0021]). Further, preventing the reaggregation or re-bundling of

individualized CNTs needs to be purposefully hindered if one wishes to preserve discreteness, as Sun attempts to do with nanoplatelets—"[t]hus, each nano-particle interferes with re-aggregation, or re-bundling of the nanotubes by a steric, or physical, hinderance effect." *Id.* (quoting LGES-1004 ¶[0044]).

Farrugia is silent about creating, preserving, or maintaining any individualization of its CNTs other than its indifference to the state of the CNTs at the beginning. *Id*. ¶125 (citing LGES-1008 ¶[0024] ("Discrete nanotubes, aggregates of nanotubes, or both discrete and aggregate nanotubes may be suitable for use according to the present disclosure.")). Eliminating, or not preserving, any CNT individualization would even be understood by a POSITA as desirable in Farrugia's applications because Farrugia, like Narayan, has the objective of increasing electrical conductivity (Pet., 51) and a dispersion of individualized CNTs would have been expected to be *less conductive* for Farrugia's stated purposes than a dispersion of CNTs that have interconnectivity. *Id.*

Gan is one such example of increasing electrical conductivity in polymers by creating a continuous conductive network of CNTs. MRD-2006 ¶126 (citing LGES-2007). Gan harnesses the known tendency of oxidized CNTs to be drawn closer together and illustrates how and why this tendency aids in increased electrical conductivity:

> A percolated network refers to the network of CNTs and conductive polymer that form a continuous conductive path extending through the composite. The presence of such continuous conductive path through the composite is believed to be a reason why the electrical conductivity of the conductive polymer material increases when CNTs are present.

Id. (quoting MRD-2007 ¶[0041]).

This type of electrical connectivity (illustrated in FIG. 5, below), is typical and expected of CNTs dispersed into polymers, unless some intentional means of creating and/or maintaining discrete, individualization of the dispersed CNTs is used:

MRD-2007, FIG. 5

Id. ¶127 (citing MRD-2007, FIG. 5). While the above image is an example of an interconnected mass of non-functionalized CNTs, Gan's experimental example uses functionalized CNTs expressly because they will increase the electrical conductivity of the resulting CNT/polymer composition and is known to draw the CNTs closer together. *Id.* (citing MRD-2007, ¶¶[0020], [0023]). Further the amount of functionalization needs to be optimal, as excessiveness it can lead to agglomeration within the polymer matrix. *Id.* (citing MRD-2007 ¶[0027]). Thus, functionalization would lead to an increase in the interconnectivity seen in the above FIG. 5. *Id.*

While Farrugia vaguely teaches starting with discrete carbon nanotubes (without any explanation as to how it achieved that result), Farrugia does not teach creating a dispersion of the discrete carbon nanotubes, maintaining that discreteness, or in any way utilizing discreteness to its advantage. MRD-2006 ¶128 (citing LGES-1007 ¶[0024]). Nor would a POSITA read Farrugia as attempting to do so. *Id.* A POSITA would understand that Farrugia, like Narayan and Gan, is utilizing dispersed but interconnected CNTs in a polymer, because it is the interconnectivity that provides the increased electrical conductivity all three seek to increase. *Id.* In other words, even if Farrugia did somehow begin with "discrete" CNTs as generally stated, the CNTs would not be discrete in the final composition because the goal of

Farrugia-Narayan is to form a continuous network of interconnected CNTs to provide improved electrical conductivity. *Id.*

C. Nothing in Farrugia or Narayan Enables a POSITA to Make a Dispersion of Discrete, Oxidized, Multiwall Carbon Nanotubes

Even if it could be argued that a POSITA would read Farrugia as trying to use a dispersion of intentionally discrete CNTs, nothing in either Farrugia or Narayan would teach the POSITA how to do this. MRD-2006 ¶129. To render a claim obvious, the prior art, taken as a whole, must enable a skilled artisan to make and use the claimed invention. *See In re Kumar*, 418 F.3d 1361, 1368 (Fed. Cir. 2005).

Making CNTs discrete, particularly MWCNTs, as the claims require, is challenging. MRD-2006 ¶130. This is evident not only from Sun's own failure to do so, but from the abundance of prior art cited by Petitioner in an *effort* to show discrete MWCNTs. *Id*. But Farrugia teaches *nothing* about how discreteness can be created or maintained. *Id*. In fact, any discreteness in Farrugia's nanotubes (again the concept of discreteness in the Petition's combination comes *solely* from Farrugia) may have had in the beginning (e.g., if Farrugia could have somehow simply bought discrete MWCNTs) would be expected to be lost by the end of Farrugia's process. *Id.*

Farrugia's entire purpose is to generate a toner with improved conductivity, and it accomplishes this by taking the created nanotube/polymer composite and incorporating it into an emulsion aggregation toner. MRD-2006 ¶131 (citing LGES-1007 ¶[0055]). The emulsion/aggregation process will tend to bunch and entangle the CNTs. *Id.* The resulting emulsion/aggregation is then coagulated, which will further bunch and entangle the CNTs. *Id.* (citing LGES-1007 ¶[0057]). This is a perfectly acceptable outcome for Farrugia—the desired outcome in fact, since it seeks conductivity enhancement. *Id.* In fact, Farrugia's final product would have been expected to have a lower conductivity if the CNTs somehow maintained discreteness, since the electrical connectivity among the CNTs is an important factor to providing high conductivity. *Id.*

Far from seeing discreteness of MWCNTs as a feature taught and enabled by the combination of Farrugia and Narayan, a POSITA would understand that any discreteness of MWCNTs present in this combination, came from somewhere else—perhaps such as Farrugia who starts with discrete CNTs that were apparently acquired from another source (though Farrugia does not say). MRD-2006 ¶132. Further, a POSITA would find no teaching in either reference for maintaining that discreteness and would not expect to, as it would be *directly counter to their explicitly stated purpose*, which is to enhance conductivity through percolation. *Id.*

Conductivity through the length of a polymer by means of CNTs relies on the CNTs themselves, representing a largely unbroken electrical pathway from one end to the other, precisely like a traditional electrical circuit of wire. MRD-2006 ¶133. In fact, because of the nanometer scale of CNTs and the millimeter scale of the applications in Farrugia and Narayan, having each nanotube be in electrical contact with numerous others helps ensure that an electrical pathway is maintained, or, even more effectively, multiple simultaneous electrical pathways are created. *Id.* Thus, having any discreteness of the CNTs that may have existed in the beginning, would have only been expected to hurt the goal that Farrugia and Narayan are trying to achieve. *Id*

All claims of the ’649 Patent require a dispersion of discrete, multiwall carbon nanotubes. LGES-1001, claims 1-23. While Farrugia may teach starting with discrete CNTs, it does not teach how to generate them. MRD-2006 ¶134. And even if it did, it does not teach how to generate a dispersion that maintains that discreteness. *Id.* “[E]ven though a non-enabling reference can play a role in an obviousness analysis, the evidence of record must still establish that a skilled artisan could have made the claimed invention.” *Raytheon Techs. Corp.*, 993 F.3d at 1381. The combination of Farrugia and Narayan do not teach a POSITA how to make the claimed invention, and the combination would be counter to their shared purpose.

MRD-2006 ¶134. Indeed, a POSITA would understand that the use of CNTs with an aspect ratio of greater than 1,000, which is the most preferable aspect ratio disclosed by Narayan for increased electrical conductivity, would make it even more difficult to achieve "discrete" MWCNTs. *Id.* (citing LGES-1008 ¶[0014].

D. Farrugia and Narayan Teach Away From Using the Aspect Ratio Range of the Claims

All of the claims of the '649 Patent require a plurality of carbon nanotubes with a range of aspect ratios between 25 and 500. LGES-1001, claims 1-23. Farrugia gives no ranges for aspect ratios, only that ***large*** aspect ratios are better "[b]ecause carbon nanotubes have a high aspect ratio, only small amounts of carbon nanotubes need to be present in a toner in order to achieve the same conductivity as compared to conventional conductive additives." LGES-1007 ¶0018]. This focus on conductivity is acknowledged by Petitioner, who points out that "Farrugia attributes this enhanced electrical conductivity to the aspect ratio of nanotubes." Pet., 47; MRD-2006 ¶355.

Petitioner argues that the Farrugia-Narayan combination would have "carbon nanotubes in Farrugia's nanotube/polymer composite, and in the toner incorporating that composite, have 'aspect ratios greater than or equal to about 5,' 'greater than or equal to about 10,' or 'greater than or equal to about 100,' based on Narayan." Pet.,

48 (citing LGES-1008 ¶[0014]). The Farrugia-Narayan combination that Petitioner proposes is only obvious if the POSITA is *motivated by hindsight to match the claim limitations*. MRD-2006 ¶136. Without that impermissible hindsight, the Farrugia-Narayan combination would be viewed quite differently. *Id.*

Farrugia explicitly teaches away from using small aspect ratios—explicitly teaching that it wants its CNTs to have a "high aspect ratio" to provide better interconnectivity throughout its composition. MRD-2006 ¶137 (citing LGES-1007 ¶[0018]). This is consistent with conventional thinking at the time that high aspect ratio CNTs were best suited to provide a continuous network of conductivity through a composite material. *Id.* (citing MRD-2011, 30:23-31:16). In making its proposed combination, the Petition omits a salient statement that directly follows its cherry-picked quote: "Preferably, the carbon nanotubes have an aspect ratio greater than or equal to about 10, more preferably greater than or equal to about 100, and ***even more preferably greater than or equal to about 1000***." LGES-1008 ¶[0014]. Thus, not only does Farrugia *teach away* from using smaller aspect ratios CNTs, Narayan also teaches aspect ratios larger than the claimed ranges are desired. MRD-2006 ¶137.

Even assuming a POSITA had a motivation to combine Farrugia and Narayan, it would not be to use a small aspect ratio range (25 to 500) that Farrugia expressly teaches as sub-optimal. MRD-2006 ¶138. Rather, a POSITA would be motivated to

use a large aspect ratio as taught by Narayan (e.g., preferably greater than 1,000) for the purpose of increased conductivity, as desired by both Farrugia and Narayan. *Id.*

Like Sun in Petitioner's Ground 1 combination, the range of aspect ratios "taught" by Narayan (e.g., "and even more preferably greater than or equal to about 1000") is so large that it is effectively potentially infinite in practice. MRD-2006 ¶139 (quoting LGES-1008 ¶[0014]). And when such a large range of options is disclosed, there is no motivation for a POSITA to be invited to perform routine experimentation to discover an optimum range. *Id.; see also Genetics*, 655 F.3d at 1306. And it is far from it here. MRD-2006 ¶139. All of the teachings of Farrugia and Narayan motivate a POSITA to use aspect ratios *greater than or equal to about 1000*. *Id.* The only motivation a POSITA would have to do otherwise would be if the POSITA were looking at the claims of the '649 Patent and trying to meet its limitations. *Id*.

Consequently, other than via impermissible hindsight-based reasoning, a POSITA would not find it obvious to modify Farrugia to rely on a small aspect ratio disclosed by Narayan as less than preferrable. *Id.* ¶140. Such a modification is directly contrary to Farrugia's express use of "high aspect ratio" carbon nanotubes to achieve improved conductivity. *Id.* (quoting LGES-1007 ¶[0018]).

E. None of the dependent claims are rendered obvious by the combinations based on Farrugia an Narayan

Since independent claim 1 has not been rendered obvious by the Petition's Ground 2, none of the dependent claims are rendered obvious. However, the Petition also independently fails to demonstrate that dependent claims 2 and 3 are obvious.

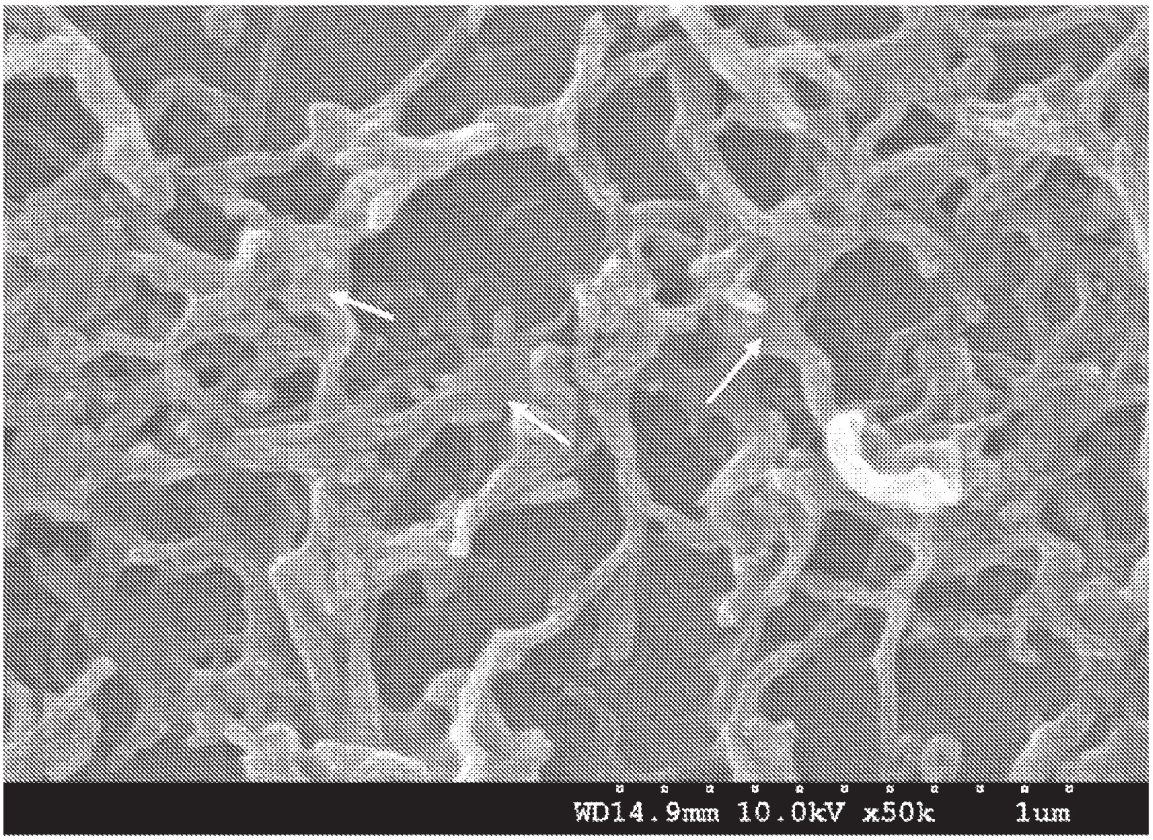
Dependent claim 2 of the '649 Patent requires that the dispersion of claim 1 wherein at least 70 percent by weight of the nanotubes are discrete. LGES-1001, 13:56-57. Petition again relies on the single sentence of Farrugia, "[d]iscrete nanotubes, aggregates of nanotubes, or both discrete and aggregate nanotubes may be suitable for use according to the present disclosure," as teaching this limitation. Pet., 63. Believing that "the particular weight percent of nanotubes that are discrete in a given composition is a straightforward design choice," then the choice of having only discrete nanotubes would necessarily be above 70 percent by weight of the nanotubes. *Id.*, 64; MRD-2006 ¶141.

However, as shown above, neither Farrugia nor Narayan teach any mechanism for generating discrete CNTs, do not teach any mechanism for maintaining that discreteness in as dispersion, and neither would benefit from having done so, particular since both references are not enabled for those teachings. MRD-2006 ¶142; *supra* Sections VI.A. and VI.B. Far from rendering obvious a full 70%

by weight of discreteness, a POSITA would go into an interpretation of Farrugia and Narayan knowing that this level of discreteness would be *detrimental* to both of their intended results. MRD-2006 ¶142.

Just as Petitioner fails to demonstrate that Sun renders claim 1 obvious, claim 2's more stringent requirement of "at least 70% by weight" of the MWCNTs being discrete, cannot be rendered obvious over a prior art reference that fails to teach the generation or maintenance of any percent weight of discrete MWCNTs in a dispersion. *Id*. ¶143.

Similarly, Petitioner fails to demonstrate that dependent claim 3's requirement that the dispersion be in the form of free-flowing particles is obvious under Ground 2A. Pet., 65; MRD-2006 ¶144. Petitioner's sole rational is again, the single sentence of Farrugia. Pet., 74. But a POSITA would not see either Farrugia or Narayan as teaching "free flowing particles" of CNTs. MRD-2006 ¶144 (quoting LGES-1001, 13:58-60). Instead, a POSITA would note that both Farrugia and Narayan are trying to achieve a network of interconnected and electrically conductive CNTs, that would be expected to result in a percolated network such as what was depicted in Figure 5 of Gan:



MRD-2006 ¶144 (citing MRD-2007, FIG. 5).[3]

The CNTs of Farrugia, no matter how discrete they may have started initially, most certainly would not be discrete after (1) the emulsion/aggregation process; and

[3] FIG. 5 shows the interconnected structure of non-functionalized CNTs. Functionalization would likely lead to an even greater degree of interconnection. *See supra* Section X.B.

(2) the coagulation of the emulsion/aggregation, both of which will tend to bunch and entangle the CNTs. *Id*. ¶155 (citing LGES-1007 ¶¶[0055]-[0057]).

VII. CONCLUSION

For the reasons above, the Petition fails to demonstrate beyond a preponderance of the evidence that any Challenged Claim is unpatentable.

Dated: March 31, 2025

Respectfully submitted,

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CERTIFICATE OF COMPLIANCE

The undersigned certifies that this document complies with the type-volume limitations set forth in 37 C.F.R. § 42.24 *et seq*. This document, excluding those portions excluded under § 42.24(a)(1), contains 11,365 words as calculated by the program used in creating it.

/James A. Glenn/
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CERTIFICATE OF SERVICE

Pursuant to 37 C.F.R. §§ 42.6(e)(4), the undersigned certifies that on March 31, 2025, a copy of the foregoing document (*Patent Owner's Response*, including all Exhibits), was served via electronic mail on all attorneys of record at the email addresses provided for electronic service listed in Petitioner's mandatory notices, as set forth below:

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