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DECLARATION OF SAMUEL P. GIDO

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IPR2025-00355

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I, Samuel P. Gido, declare the following:

I. QUALIFICATIONS AND BACKGROUND INFORMATION

1. My education and experience are described more fully in the attached curriculum vitae. For ease of reference, I have highlighted certain information below.

A. Educational Background

2. I first became interested in polymers as an undergraduate student in chemical engineering at Princeton University. At Princeton I took an introductory polymer course, an advanced undergraduate polymer course, and a graduate level polymer course. I did research for my bachelor's thesis at the Textile Research Institute, a nearby independent research lab associated with Princeton, on coating processes for porous polymeric materials. I received my BSE (bachelor of science in engineering) degree in chemical engineering, with high honors (magna cum laude) from Princeton in 1988.

3. From there I moved on to the Massachusetts Institute of Technology (MIT) for a doctoral program in chemical engineering. At MIT, I also participated in the interdisciplinary Program in Polymer Science and Technology (PPST, which has been renamed the Program in Polymers and Soft Matter (PPSM), http://polymerscience.mit.edu/). This program required that, in addition to

my normal chemical engineering curriculum, I take two semesters of graduate level polymer courses encompassing polymer synthesis, polymer processing, polymer composites, polymer characterization, and polymer properties testing. I did my doctoral research under Prof. Edwin L. Thomas, who was in the Materials Science and Engineering Department at MIT. Prof. Thomas is a world renowned expert in the study of polymer structure using electron microscopy. Therefore, my training and research experience at MIT focused on the use of electron microscopy, SEM and TEM, to study a range of different types of polymers including plastics, rubbers, thermosets, and polymer composites with added fillers. I received my Ph.D. in 1993,

B. Professional Experience

4. After my Ph.D., I moved on to begin my career as a professor in the Polymer Science and Engineering Department at the University of Massachusetts at Amherst. Over my academic career I have received millions of dollars of research funding from government (U.S. Army, National Science Foundation, U.S. Department of Education), foundations (Bill and Melinda Gates Foundation, W. M. Keck Foundation), and industry.

5. I have focused on a bottom-up concept of new materials design for advanced applications. In particular, I have worked on development of new molecular structures for the control of nanometer and micrometer length scale structure

and morphology, directed toward improving properties such as: mechanical strength, rubber elasticity, transport (diffusion) of gases and liquids in porous materials, electrical and magnetic properties, and adhesion. A large part of this research is on controlling the phase separation behavior of materials to form unique, functional structures. In my research I use electron microscopy (SEM and TEM), X-ray scattering, Atomic Force Microscopy (AFM), mechanical (tensile) testing, diffusion studies, and electrochemical cell studies to characterize the properties of new polymeric and composite materials.

6. I have nearly 100 peer reviewed publications and 14 issued patents (11 US, 1 EU, 2 Chinese), including numerous publications on elastomers, thermosets, fluoropolymers including fluoroelastomers, and polymer materials with inorganic fillers. I am an inventor on US Patent 7,619,036, which teaches novel molecular structures and properties for fluoropolymers and fluoroelastomers.

7. I have done research on materials for batteries and fuel cells, including fluorinated polymers and polymer composites containing carbon nanotubes as well as other inorganic fillers. This work was funded by Kureaha (Japan), and the U.S. Army. In these studies, we developed polymer chemistries, nanometer and micrometer scale composite structures, porous structures, and

coating processes. I was the director of a 5 year, $3.75M Collaborative Research Center between the University and the U.S. Army Research Laboratory on Flexible Protective Materials to be worn by soldiers. Carbon nanotubes and polymer-carbon nanotube composites contributed unique properties, such as high strength, high modulus and electrical conductivity to materials we developed.

8. Additionally, I have worked and continue to work as a consultant for W. L. Gore, a leading company in fluorinated polymers. I have consulted for industry on the topic of coating processes for polymer-filler composite layers, particularly for Rexam Graphics and Scapa Tape. I was also a co-founder of a startup company, Smart Surfaces LLC, that developed environmentally friendly, non-stick coatings for boats (and other structures in a marine environment) to prevent fouling with marine organisms. These coating use crosslinked silicone rubbers with various additives including inorganic particles and silicone oil plasticizer. Smart Surfaces was sold to FujiFilm in 2006, and I continued to consult for the resultant company: FujiFilm Smart Surfaces LLC.

9. I did a research project for Loctite Corporation in which I used electron microscopy to study crosslinked, adhesive, thermoset polymer layers filled with inorganic reinforcing particles. And I did research for Boston

Scientific, which resulted in a U.S. Patent, on polymer coatings for heart stents, using a number of techniques including electron microscopy. These coatings contained filler particles, a particulate drug, and we studied the diffusion of this drug through the coating layer.

10. One of the best ways to really understand a topic is to teach it to someone else. I have taught and continue to teach topics, relevant to this matter, to our advanced graduate students (doctoral candidates) in the Polymer Science and Engineering Department at the University of Massachusetts at Amherst. In past years I have taught PSE 608 Introduction to Polymer Engineering, which includes topics such as mechanical properties, polymer processing, and composite structures. I have also taught in the past and am currently teaching PSE 602 Polymer Characterization Laboratory, in which the students gain practical laboratory experience with techniques including SEM, X-ray scattering, and mechanical testing. I teach PSE 608 Physical Chemistry of Polymers I and PSE 789 Physical Chemistry of Polymers II. In this sequence of classes, I cover, among other things, phase behavior and phase separations (coagulation), and the theory of X-ray scattering. I also teach PSE 721 Polymer Morphology. This class focuses on nanometer and micrometer size structure in polymers and composites, as well as the use of electron microscopy to study materials structure. In PSE 608 and also in PSE

721, silicone rubbers and silicone composites and dispersions are discussed. I also teach a seminar class for freshman, FFYS 191POLY The Polymer Age, which focuses on popular and societal impacts of polymers. In this class, I present information on carbon nanotubes, silicones, composites, and materials used in lithium ion batteries.

11. I have been the primary research (thesis) advisor to 21 granted Ph.D.s, three of whom have gone on to become professors in their own right. My other students have gone on to careers at major companies or national laboratories including: 3M, Dow Chemical, Eastman Chemical, General Electric, DuPont, Intel, and the U.S. Army Research Laboratory.

12. I have been retained on behalf of LG Energy Solution, Ltd., to offer technical opinions relating to U.S. Patent No. 9,636,649 ("the '649 Patent," LGES-1001) and prior art references related to its subject matter. I have reviewed the '649 Patent and the following references.

- LGES-1004 U.S. Patent Pub. No. 2010/0197832 ("Sun")
- LGES-1005 U.S. Patent No. 7,091,120 ("Buretea")
- LGES-1006 U.S. Patent Pub. No. 2010/0016473 ("Kaji")
- LGES-1007 U.S. Patent Pub. No. 2010/0124713 ("Farrugia")
- LGES-1008 U.S. Patent Pub. No. 2003/0213939 ("Narayan")
- LGES-1009 U.S. Patent No. 5,079,123 ("Nanya")

- LGES-1013 Japanese Unexamined Patent Application No. 2009-235650 ("Matsushita")

13. I have also reviewed various supporting references, including the following, and other documentation as further noted in my opinions below.

- LGES-1010 U.S. Patent Pub. No. 2010/0009165 ("Patel")
- LGES-1011 U.S. Patent Pub. No. 2011/0272856 ("Rasmussen")
- LGES-1012 U.S. Patent Pub. No. 2008/0315453 ("Molitor")
- LGES-1014 "Surface modified multi-walled carbon nanotubes in CNT/epoxy-composites." Florian H. Gojny, Jacek Nastalczyk, Zbigniew Roslaniec, Karl Schulte. Chemical Physics Letters 370 (2003) 820–824 ("Gojny")
- LGES-1015 "Sonochemical Oxidation of Multiwalled Carbon Nanotubes." Yangchuan Xing, Liang Li, Charles C. Chusuei, and Robert V. Hull. Langmuir 2005, 21, 4185-4190 ("Xing")
- LGES-1016 U.S. Patent No. 7,807,127 ("Forohar")
- LGES-1019 U.S. Patent Pub. No. 2010/0006152 ("Hatton")
- LGES-1020 U.S. Patent No. 9,327,054 ("Sundara")
- LGES-1021 U.S. Patent Pub. No. 2012/0058255A1 ("Gan")
- LGES-1022 U.S. Patent Pub. No. 2007/0259994 ("Tour")

- LGES-1024 EP Patent No. EP 2 139 630 ("Bergmark")
- LGES-1025 U.S. Patent No. 7,163,973 ("Ahsan")
- LGES-1026 U.S. Patent No. 7,846,998 ("Akagi")
- LGES-1027 U.S. Patent Pub. No. 2006/0014873 ("Ikezawa")
- LGES-1028 U.S. Patent Pub. No. 2011/0183253 ("Aga")
- LGES-1029 U.S. Patent No. 3,471,395 ("Sumner")
- LGES-1030 U.S. Patent No. 4,625,761 ("Uchida")
- LGES-1031 "Characterization of Zirconium Phosphate/Polycation Thin Films Grown by Sequential Adsorption Reactions" Hyuk-Nyun Kim, Steven W. Keller, Thomas E. Mallouk, Johannes Schmitt, and Gero Decher. *Chem. Mater.,* Vol. 9, No. 6, 1997
- LGES-1032 "The IUPAC Rules for Naming Organic Molecules" Stanislaw Skonieczny. *Journal of Chemical Education*. Vol. 83 No. 11, pp. 1633-1637 (2006) ("Skonieczny")
- LGES-1033 U.S. Patent No. 8,282,725 ("Shimomura")
- LGES-1034 U.S. Patent No. 6,569,231("Mathias")
- LGES-1036 U.S. Patent No. 7,749,682 ("Miyabe")
- LGES-1037 U.S. Patent No. 7,959,280 ("Koike")
- LGES-1038 U.S. Patent No. 6,544,326 ("Itou")

- LGES-1039 U.S. Patent No. 5,458,681 ("Hasegawa")
- LGES-1040 U.S. Patent No. 5,750,304 ("Yamaguchi")
- LGES-1041 A. Hirsch. "Functionalization of Single-Walled Carbon Nanotubes" *Angew. Chem. Int. Ed.* 2002, 41, No. 11, 1853-1859 ("Hirsch")
- LGES-1042 N. Canter. "Mold-release agents and coatings." *Tribology & Lubrication Technology* 2008, 31-38 ("Canter")
- LGES-1043 J.E. Plevyak and L.A. Sobieski. "Improved RIM Processing with Silicone Internal Mold Release Technology." *Journal of Cellular Plastics* 1984, 363-368 ("Plevyak")
- LGES-1045 B.D. Cullity. "Elements of X-Ray Diffraction." Addison-Wesley Publishing Company, Inc. 1978. P. 86-87. ("Cullity")
- LGES-1046 U.S. Patent Pub. No. 2009/0200517 ("El Bounia")
- LGES-1047 P. C. Hiemenz, T.P. Lodge. "Polymer Chemistry, Second Edition." CRC Press 2007. Chapters 2 and 10 ("Hiemenz")
- LGES-1048 J. Mijovic, E. M. Pearce, and C.-C. Foun. "Fluoroelastomer-Modified Thermoset Resins." *Advances in Chemistry*; American Chemical Society 1984, 293-307 ("Mijovic")

- LGES-1049 U.S. Patent No. 4,421,878 ("Close")
- LGES-1051 U.S. Patent Pub. No. 2006/0124028 ("Huang")

14. Counsel has informed me that I should consider these materials through the lens of one of ordinary skill in the art related to the '649 Patent at the time of the earliest possible priority date of the '649 Patent, and I have done so during my review of these materials. The '649 Patent claims the benefit of priority to an earliest date of December 14, 2010 ("the Critical Date"). Counsel has informed me that the Critical Date represents the earliest possible priority date to which the challenged claims of '649 Patent are entitled, and I have therefore used that Critical Date in my analysis below.

15. I have no financial interest in the parties or in the outcome of this proceeding. I am being compensated for my work as an expert on an hourly basis. My compensation is not dependent on the outcome of these proceedings or the content of my opinions.

16. In writing this declaration, I have considered the following: my own knowledge and experience, *e.g.*, as I discussed above. In addition, I have analyzed various publications and materials, in addition to other materials I cite in my declaration.

17. My opinions, as explained below, are based on my education, experience, and expertise in the fields relating to the '649 Patent. Unless otherwise

stated, my testimony below refers to the knowledge of one of ordinary skill in the art as of the Critical Date, or before. Any figures that appear within this document have been prepared with the assistance of Counsel and reflect my understanding of the ’649 Patent and the prior art discussed below.

II. OVERVIEW OF CONCLUSIONS FORMED

18. This declaration explains the conclusions that I have formed based on my analysis. To summarize those conclusions, based upon my knowledge and experience and my review of the prior art publications listed above, I believe that:

- Claims 1-7, 11, 13-14, 17-19 are obvious over Sun
- Claim 8 is obvious over Sun and Buretea
- Claims 9-10, 15, and 21-23 are obvious over Sun and Kaji
- Claims 16 and 20 are obvious over Sun, Kaji, and Matsushita
- Claims 1-8, 11-15, 17-19, and 21 are obvious over Farrugia and Narayan
- Claims 22-23 are obvious over Farrugia, Narayan, and Nanya

III. LEVEL OF ORDINARY SKILL IN THE ART

19. In my opinion, one of ordinary skill in the art relating to, and at the time of, the invention of the ’649 Patent would have had a Master’s degree in materials science, chemistry, chemical engineering, a field relevant to

polymer science and engineering, or the equivalent, and at least three years of experience in nanomaterials, electrochemistry, batteries, or polymer related activities. Additional work experience can compensate for less formal education, and vice versa.

20. Based on my experiences, I have a good understanding of the capabilities of one of ordinary skill. Indeed, I have participated in organizations and worked closely with many such persons over the course of my career. Based on my knowledge, skill, and experience, I have an understanding of the capabilities of one of ordinary skill.

21. Having undergraduate and graduate degrees in polymer related fields, having taken and taught numerous college level and graduate level polymer classes, and having over 30 years of relevant experience, I meet and exceed the qualifications as a POSITA in this matter.

IV. SUMMARY OF THE ’649 PATENT

A. Overview of the ’649 Patent

22. The ’649 Patent states that its “invention” relates to “carbon nanotubes as composites with materials such as elastomers, thermosets and thermoplastics or aqueous dispersions of open-ended carbon nanotubes with additives.” LGES-1001, Abstract. However, this is a specific and narrow statement, which is at odds with the broad and relatively generic claims of the’649

Patent. The claims reciting merely that a dispersion includes oxidized, discrete, and multiwall carbon nanotubes and an additive. *Id.*, 13:50-55. As I demonstrate in my analysis below, a dispersion having these constituent components was well-known in the art as of the Critical Date.

23. I note that the ’649 claims do recite certain characteristics of the dispersion, relating to the aspect ratio of the nanotubes, and their weight percentages in the dispersion. LGES-1001, 13:50-55. But these characteristics also were not new, as my analysis below demonstrates. Moreover, the claims of the ’649 Patent provide particular ranges of values for these characteristics, while the patent itself provides no indication of whether, or why, these ranges are important. *See* LGES-1001, 5:18-24, 8:19-28.

V. PRIOR ART ANALYSIS

A. [GROUND 1] – CLAIMS 1-7, 11, 13-14, AND 17-19 ARE OBVIOUS OVER SUN

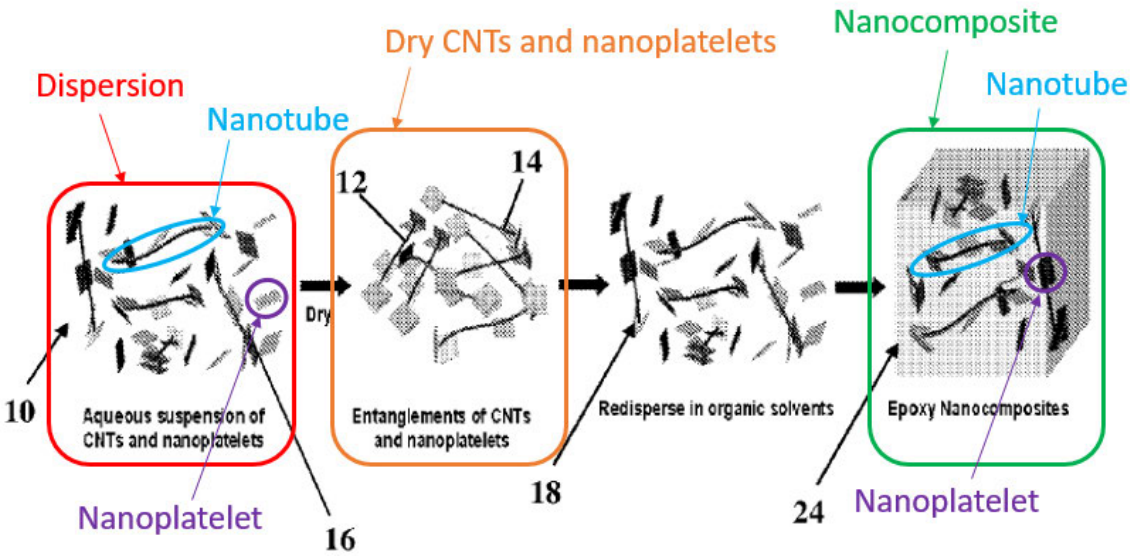
24. It is my opinion that a POSITA would have found claims 1-7, 11, 13-14, and 17-19 to be obvious over Sun, as I discuss in the following sections.

1. Sun describes both an aqueous dispersion of carbon nanotubes and an epoxy-nanotube composite

25. Sun describes a “drying-redispersion method to disperse carbon nanotubes and inorganic nanoparticles into epoxy to form exfoliated epoxy

nanocomposites" "containing … well-dispersed carbon nanotubes." LGES-1004 (Sun), [0072].

26. As shown in Fig. 1, which I have reproduced below, Sun's process involves the initial generation of a "dispersion 10," which I have outlined in red, by mixing "[o]xidized, suspended carbon nanotubes (CNTs) 12" with "aqueous solutions of nanoplatelets 14." LGES-1004 (Sun), [0036]. I have circled one example nanotube in blue, and one example nanoplatelet in purple. I note that Sun sometimes refers to the mixture 10 as a suspension (*see*, *e.g.*, *id.*, [0068], Fig. 1). I understand Sun to be using the terms dispersion and suspension interchangeably to refer to the same item: a mixture of nanotubes and nanoplatelets dispersed in a solvent, such as water. Sun describes that the nanotubes can be "single walled carbon nanotubes (SWNTs) or multi-walled carbon nanotubes (MWNTs)." *Id.*, [0038].



Excerpt from Sun, Fig. 1 (annotated)

27. Sun explains that the "dispersion of nanotubes is attributed to the presence of the nanoplatelets." LGES-1004 (Sun), [0044]. Specifically, Sun explains that "the negatively charged surface of nanotubes … attracts the positively charged surface of the nanoplatelets," and as a result, "the nanoplatelets attach to the nanotube walls, and at least partially enter between nanotubes of the bundles." *Id.* By this mechanism, "[t]he nanoplatelets force the nanotube bundles into individual tubes," where "[t]he positively charged nanoplatelets are electrostatically tethered to the negatively charged nanotubes" such that "the nanoplatelets have individual, exfoliated, or separated tubes attached, or tethered to the surfaces of the nanoplatelets." *Id.* In this configuration, "each nanoparticle," *e.g.*, each nanoplatelet,

"interferes with the re-aggregation, or re-bundling of the nanotubes by a steric, or physical, hindrance effect" that "comes from the two-dimensional heterogeneous shapes of the nanoplatelets interfering with other nanoplatelets associated with nearby nanotubes." *Id.* That is, "[t]he surface charge and two-dimensional nature of exfoliated nanoplatelets disperse the nanotubes and hinder the re-aggregation thereof." *Id.*

28. The aqueous dispersion of nanotubes and nanoplatelets is dried, and the dried material is suitable for "further dispersion 18 in solvents." LGES-1004 (Sun), [0068]; *see* [0047]-[0048]. For instance, the nanotubes and nanoplatelets, which is referred to as a "nanofiller," "may be dispersed in a medium such as, but not limited to, epoxy, plastic, or polymer, to form a nanocomposite 24," outlined in green. *Id.*, [0045]; *see* [0052]. "In an embodiment, the nanofiller is re-suspended in acetone prior to mixing in an epoxy." *Id.*, [0045]. "The solvent is removed from the nanocomposite," and "a curing agent is added to the epoxy and nanotubes, and heated to polymerize, crosslink, vulcanize, or otherwise cure … the epoxy nanocomposite." *Id.*, [0046]; *see* [0052]. The resulting nanocomposite contains "[w]ell-dispersed" nanotubes and nanoplatelets, both of which "can be fully dispersed down to individual level in epoxy matrices." *Id.*, [0069].

29. The claims of the ’649 Patent relate to dispersions. It is my opinion that a POSITA would have found the claimed dispersions of the ’649 Patent to be obvious over Sun’s aqueous dispersion (*e.g.*, the aqueous dispersion 10, which is outlined in red in FIG. 1, above). *See*, for instance, LGES-1004 (Sun), [0043]. It is also my opinion that a POSITA, alternatively or additionally, would have found the claimed dispersions of the ’649 Patent to be obvious over Sun’s nanocomposite (*e.g.*, the nanocomposite 24, which is outlined in green in FIG. 1, above)—in both its uncured and cured form. *See*, for instance, LGES-1004 (Sun), [0045]-[0046]. I discuss both of these alternatives in the following analysis, and provide specific citations to Sun’s description and experimental examples to demonstrate why Sun renders obvious the claimed dispersion. I also note, and discuss further below, that certain of the dependent claims are obvious over only one of these alternatives—*e.g.*, over only Sun’s aqueous dispersion or over only Sun’s nanocomposite.

2. Claim-by-Claim Analysis

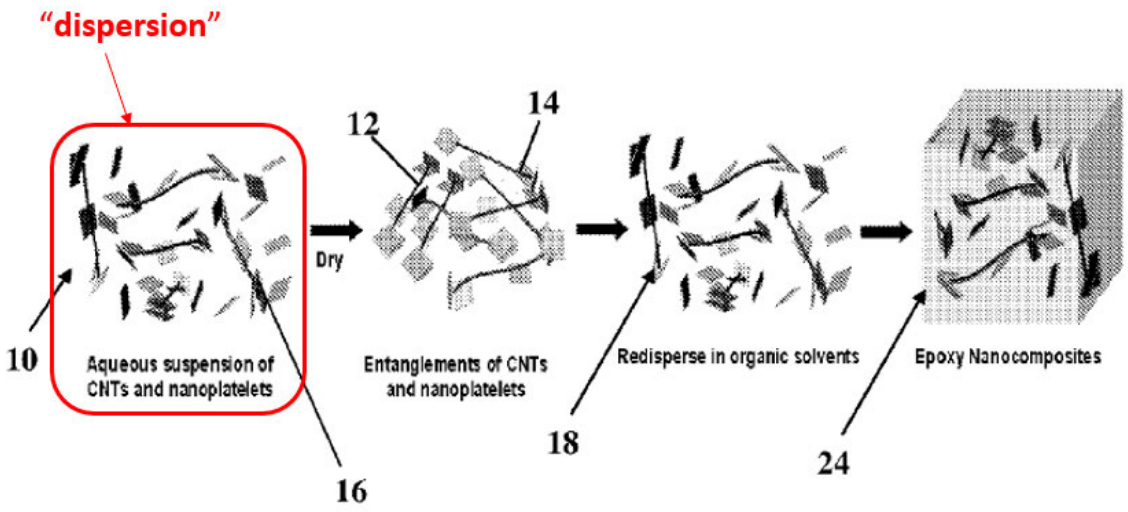
(1) Claim 1

[1pre] A dispersion comprising

30. To the extent the preamble is deemed to be limiting, it is my opinion that Sun renders it obvious.

First, Sun's aqueous dispersion is a dispersion

31. Sun describes an "aqueous nanotube-nanoplatelet ***dispersion*** 10," which is "a substantially homogeneous solution of dispersed nanotubes 12 and associated nanoplatelets 14." LGES-1004 (Sun), [0043]. This dispersion 10 is outlined in red in FIG. 1, which I have reproduced below. Sun's aqueous dispersion (*e.g.*, the dispersion 10) is **a dispersion**. *See also* the following citations from Sun, all of which describe dispersions, *e.g.*, aqueous dispersions of nanotubes and nanoplatelets: [0036] ("the electrostatic association of nanotubes 12 and nanoplatelets 14 in solutions or solvents 18 form ***a dispersion 10***"), [0042] ("the aqueous-oxidized nanotube solution, and aqueous-exfoliated nanoplatelet solution are admixed, directly, and agitated to form ***a dispersion 10***"), [0044] ("The exfoliation and ***dispersion*** of nanotubes [in the dispersion 10] is attributed to the presence of nanoplatelets"), [0068] ("the aqueous suspension 10, containing dispersed CNTs 12 and exfoliated nanoplatelets 14"), Abstract ("a nanotube and nanoplatelet dispersion in a solvent"), [0036] ("the present disclosure presents novel methods for the ***dispersion of nanotubes***"), [0032] ("as used herein, the terms 'disperse,' 'de-rope,' or 'de-bundle' refer to the substantial separation or disentanglement of individual nanotubes"), [0011].



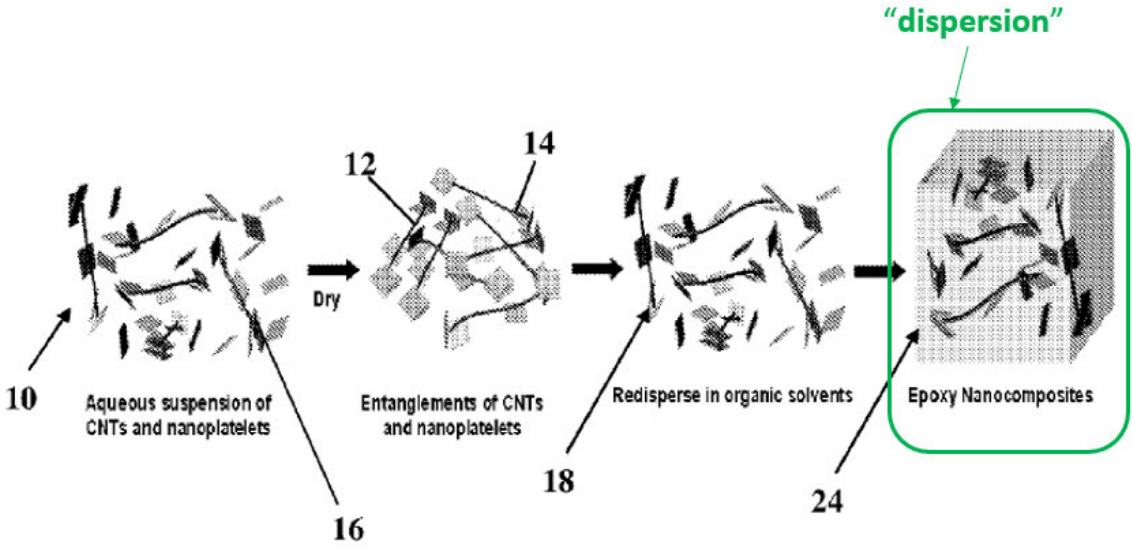
Excerpt from Sun, Fig. 1 (annotated)

32. Sun describes an experimental example that further confirms that its aqueous nanotube solution is **a dispersion**. Specifically, Sun describes that in its Example 1, "[t]wo Aqueous solutions containing oxidized MWNTs and fully exfoliated ZrP nanoplatelets were directly mixed. … ***After dispersion***, the aqueous mixtures were dried." LGES-1004 (Sun), [0056]; *see* [0069] ("aqueous dispersion" shown by electron microscope (TEM) imaging.).

Alternatively or additionally, Sun's nanocomposite is a dispersion

33. Sun describes forming "a ***nanocomposite*** 24" by "dispers[ing]" nanofiller containing nanotubes and nanoplatelets "in a medium such as … epoxy, plastic, or polymer." LGES-1004 (Sun), [0045]-[0046]. This nanocomposite, which I have outlined in green in FIG. 1, reproduced below, is **a dispersion**. For instance, the nanocomposite is a dispersion because the

nanomaterial components (*e.g.*, nanotubes and nanoplatelets) are dispersed in the matrix medium of the nanocomposite. This is described in Sun at least as follows: [0014] ("FIG. 1 illustrates a conceptual scheme for the preparation of ***epoxy nanocomposites containing individual dispersed CNTs*** and exfoliated nanoplatelets"), [0068] ("The concept for ***re-dispersing*** exfoliated nanoplatelets and carbon nanotubes in polymers is summarized in FIG. 1"), [0036] ("the present disclosure presents novel methods for the ***dispersion of nanotubes***"), [0032] ("as used herein, the terms 'disperse,' 'de-rope,' or 'de-bundle' refer to the substantial separation or disentanglement of individual nanotubes"); *see also* [0052] ("The nanotubes are ***redispersed*** into different mediums" for formation of a nanocomposite).



Excerpt from Sun, Fig. 1 (annotated)

34. Sun provides additional details about the formation of its nanocomposite which further confirm that the nanocomposite is **a dispersion**. Specifically, Sun describes that to make the nanocomposite, nanofiller (*e.g.*, nanotubes and nanoplatelets) "is re-suspended in acetone." LGES-1004 (Sun), [0045]. A precursor, such as "an epoxy," then is mixed into the suspension and cured in the presence of a "curing agent" to form "a nanocomposite 24." *Id.*, [0045]-[0046]; *see also* [0052] ("nanotubes are dispersed in liquid precursors," which are "any curable matrix or medium, such as epoxies, plastics, alloys, composites, and other materials without limitation"). The nanocomposite is cured by removing the solvent, adding a curing agent, and "heat[ing] to polymerize, crosslink, vulcanize, or otherwise cure … the epoxy nanocomposite." *Id.*, [0046]; *see also* [0052].

35. The uncured mixture of nanofiller (*e.g.*, nanotubes and nanoplatelets) in the precursor (*e.g.*, epoxy)—with or without the solvent—is **a dispersion** at least because the nanofiller is dispersed in the uncured precursor. The cured nanocomposite (*e.g.*, nanotubes and nanoplatelets in the cured epoxy matrix) is also **a dispersion** at least because the nanofiller is dispersed in the cured matrix material. *See* LGES-1004 (Sun), [0014] ("***epoxy nanocomposites containing individual dispersed CNTs*** and exfoliated nanoplatelets"), Abstract ("the method comprises ***redispersing*** at least one nanotube ***in a***

matrix precursor solution"), claims 30-31 ("an epoxy matrix … wherein the exfoliated ***carbon nanotubes are dispersed*** in the first state of the epoxy matrix"), [0045]-[0046] ("The nanofiller and epoxy mixture comprises a nanocomposite"), [0052].

36. Sun's experimental example further confirms that its nanocomposite is **a dispersion**. Specifically, Sun's procedure includes "improved steps for ***redispersing MWNTs*** and exfoliated ZrP ***into epoxy*** to form exfoliated nanocomposites." LGES-1004 (Sun), [0062]; *see also* [0069] ("The TEM [Transmission Electron Microscopy] 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***"), FIG. 6 (showing "***[w]ell-dispersed MWNTs*** and full exfoliation of nanoplatelets" ***in an epoxy nanocomposite***," [0069]).

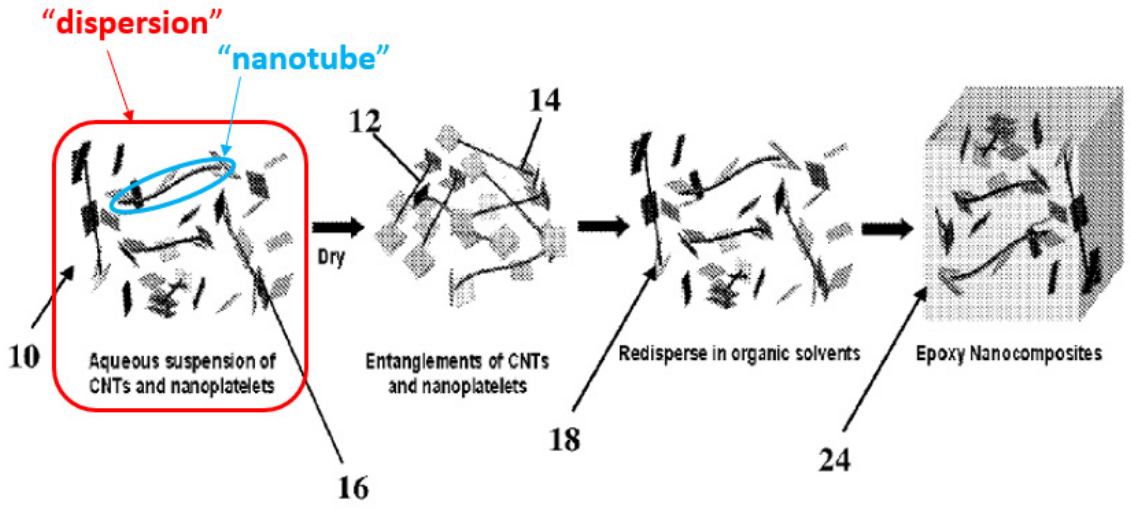
[1a] a plurality of oxidized, discrete carbon nanotubes and

37. It is my opinion that a POSITA would have found obvious that Sun's aqueous suspension and Sun's epoxy nanocomposite (both dispersions, as I discussed above) include a plurality of oxidized, discrete carbon nanotubes. I discuss each of these elements in turn in the following paragraphs.

<u>*Sun's aqueous dispersion includes oxidized, discrete carbon nanotubes*</u>

a. The nanotubes are oxidized carbon nanotubes

38. Sun's "aqueous nanotube-nanoplatelet dispersion," which corresponds to the dispersion of claim 1, as I discussed above, includes **a plurality of carbon nanotubes**. LGES-1004 (Sun), [0043]. For instance, Sun describes that its aqueous dispersion includes "[o]xidized, suspended ***carbon nanotubes (CNTs)*** 12." LGES-1004 (Sun), [0036]. I have circled one such carbon nanotube in blue in FIG. 1, reproduced below. The following citations further confirm that Sun's aqueous dispersion includes **carbon nanotubes**: LGES-1004 (Sun), [0037]-[0038] ("The nanotubes 12, illustrated for example in FIG. 1 … comprise ***carbon nanotubes***" such as "multiwalled carbon nanotubes (MWNTs)"), [0068] (an "aqueous suspension 10, containing ***dispersed CNTs 12***"), claims 13-14 ("admixing the functionalized nanotube solution and exfoliated nanoplatelet solution to form a nanofiller solution," "wherein the ***carbon nanotube*** is selected from the group consisting of multi-walled carbon nanotubes, single walled carbon nanotubes, and combinations thereof"), [0031], [0042]-[0044], claims 20 and 27.



Except from Sun, FIG. 1 (annotated)

39. Additionally, the carbon nanotubes in Sun's aqueous dispersion are **oxidized**: Sun describes that the nanotubes in the dispersion 10 are "***[o]xidized***, suspended carbon nanotubes." LGES-1004 (Sun), [0036]. That the nanotubes in Sun's aqueous dispersion are oxidized is further confirmed by the following citations, which indicate an oxidation process that can result in oxygen-containing species being covalently bound to the nanotubes: [0039] ("The nanotubes are chemically modified, or surface modified, for instance functionalized. In certain instances, ***the nanotubes are oxidized*** by pre-treatment in at least one acid."), [0042] ("the aqueous-***oxidized nanotube*** solution, and aqueous-exfoliated nanoplatelet solution are admixed … to form a dispersion 10"), [0044] ("the negatively charged surface of nanotubes, for instance example ***oxidation***, attracts the positively

charged surface of the nanoplatelets"), claim 16 ("wherein the nanofiller solution comprises, at least one exfoliated nanoplatelet associated with at least one ***oxidized nanotube***"), claim 20 ("adding at least one nanotube to a first solution, wherein the first solution ***oxidizes the at least one nanotube*** … admixing ***the oxidized nanotube solution*** and exfoliated nanoplatelet solution to form a nanofiller solution").

40. Sun's experimental example also confirm that the aqueous suspension includes **oxidized carbon nanotubes**. For instance, Sun describes that the experimental "Preparation of Carbon Nanotubes" (including multi-wall carbon nanotubes, or MWNTs), stating: "The CNTs were first oxidized in a mixture of concentrated sulfuric acid and concentrated nitric acid." LGES-1004 (Sun), [0054]; *see* FIG. 2A, showing "the TEM [transmission electron microscopy] image of ***oxidized MWNTs***." Sun then describes that an "[a]queous solution[] containing ***oxidized MWNTs***" was mixed with nanoplatelets to form an aqueous dispersion. *Id.*, [0056]-[0057].

b. The nanotubes are discrete

41. Claim element [1a] also recites that the nanotubes in the dispersion are **discrete**. It is my opinion that a POSITA would have understood, or at least found obvious, that the oxidized nanotubes in Sun's aqueous dispersion are **discrete** nanotubes, based on Sun's description of the structure and

mechanics of its aqueous dispersion, as I discuss in the following paragraphs.

42. The first reason why a POSITA would have understood or found obvious that Sun describes oxidized nanotubes in Sun's aqueous dispersion are discrete is based on Sun's description of the structure of the aqueous dispersion. Sun states that in the aqueous dispersion, "the nanoplatelets attach to the nanotube walls, and at least partially enter between nanotubes of the bundles." LGES-1004 (Sun), [0044]. Then, "[t]he nanoplatelets ***force the nanotube bundles into individual tubes*** with mechanical agitation, for example during ultrasonication." *Id.* Following the mechanical agitation, "the nanoplatelets have ***individual, exfoliated, or separated tubes*** attached, or tethered to the surfaces of the nanoplatelets." *Id.* I note that Sun defines "exfoliated" as referring to "***individually dispersed***, or monodisperse nanotubes." *Id.*, [0033]. *See also id.* ("The nanotubes dispersed by the nanoplatelets are ***exfoliated***"), [0042] ("In order to ***exfoliate and disperse*** the oxidized nanotubes"), FIG. 5. That is, Sun describes that the role of the nanoplatelets in the aqueous dispersion is to "force the nanotube bundles into individual tubes," *e.g.*, "to exfoliate and disperse the oxidized nanotubes." LGES-1004 (Sun), [0042], [0044]; *see* [0068] ("the presence of

nanoplatelets also prevents CNTs from re-aggregation and re-entanglement"), FIG. 5.

43. In light of this described role of the nanoplatelets in separating the nanotubes in the aqueous dispersion, it is my opinion that a POSITA would have understood or found obvious that the dispersion includes **discrete** nanotubes: at least some of the nanotubes in the dispersion are generally individually distinct, *e.g.*, that the nanotubes in the dispersion lack direct association with other nanotubes along substantially the length of the nanotubes. (I note that the '649 Patent describes "carbon nanotube fibers" that "are essentially discrete individual fibers, not entangled as a mass." LGES-1001, 5:37-40.)

44. Additionally, this dispersion of discrete nanotubes in the aqueous dispersion is depicted schematically in FIG. 1 (reproduced above), which shows discrete (*e.g.*, individually distinct) nanotubes in the aqueous dispersion.

45. A second reason why a POSITA would have understood or found obvious that nanotubes in Sun's aqueous dispersion are **discrete** is based on the nanotube oxidation process described by Sun, as I discuss in the following paragraphs.

46. Sun's nanotubes are oxidized in "a mixture of sulfuric acid and nitric acid," where "the nitric acid to sulfuric acid ratio is approximately 3:1." LGES-1004 (Sun), [0039]; *see* [0054] ("The CNTs were first oxidized in a mixture

of concentrated sulfuric acid and concentrated nitric acid with a volume ratio of 3:1 by ultrasonication in a sonication bath for 2 hours.”). It was well-known that oxidation of carbon nanotubes under these conditions can result in surface functionalization with species such as carboxylic acid or hydroxyl groups, as I discuss below.

47. Unfunctionalized nanotubes stick together along their lengths, thus forming clumps, because of an interaction between the graphene layers that make up the walls of the nanotubes: this interaction is known as π-π stacking, as shown schematically below (*see* LGES-1041 (Hirsch), 1854):

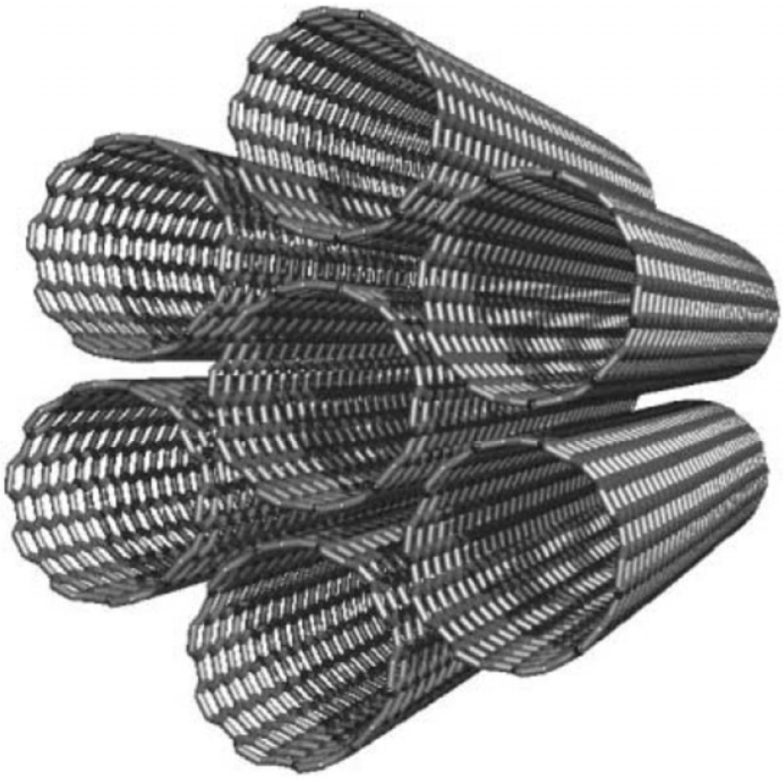
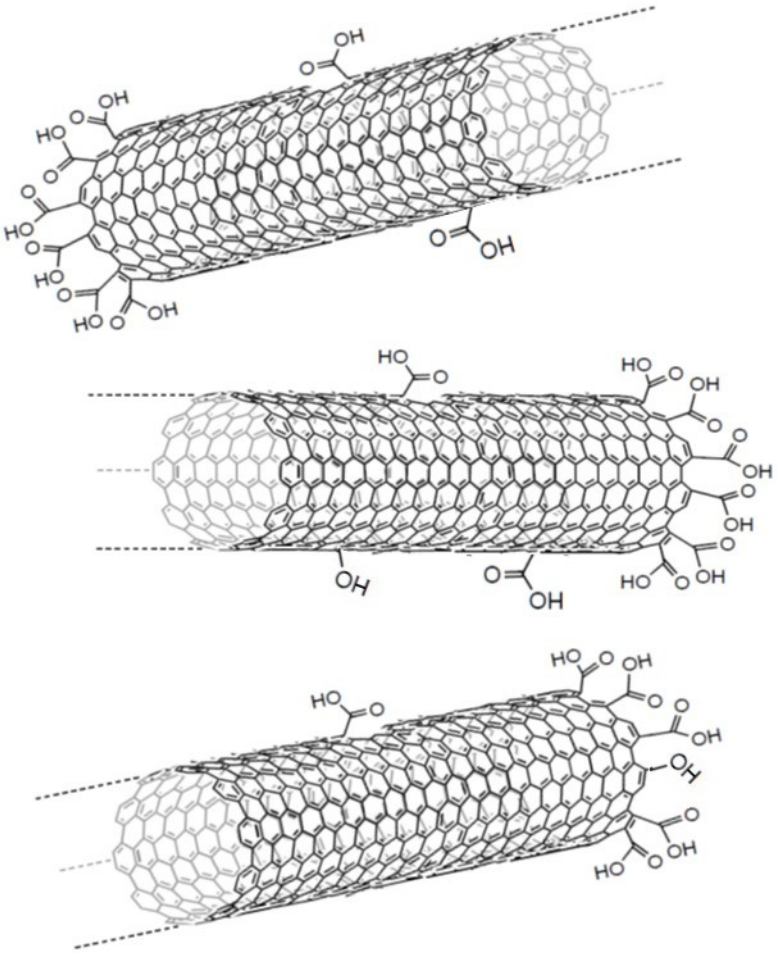


Figure 2. An idealized bundle of (10,10) nanotubes. In such bundles individual tubes are held together with strong $\pi-\pi$-stacking interactions. Almost all the tubes are organized in bundles in a typical sample.

48. Surface functionalization (*e.g.*, oxidation) of nanotubes can inhibit the π-π stacking mechanism, thus preventing the nanotubes from aggregating into clumps. Additionally, a POSITA would have known that the presence of oxidation species on the surface of the nanotubes can physically hinder the stacking of nanotubes, *e.g.*, the moieties on the nanotube walls act as physical barriers that prevent the nanotubes from getting close enough to one another to stack. That is, oxidation of nanotubes (as is the case in Sun's aqueous dispersion) can prevent the nanotubes, both chemically and

physically, from stacking into clumps, as illustrated in the figure below (which I have generated based on Scheme 1 of Hirsch (LGES-1041)).



49. A POSITA would have recognized that nanotubes treated according to the surface functionalization process described by Sun (*see* LGES-1004 (Sun), [0039], [0054]) can be functionalized with species such as carboxylic acid or hydroxyl groups, which can prevent the nanotubes from aggregating, and

instead the nanotubes would be present as individually separated, *e.g.*, discrete, nanotubes. The following paragraphs provide examples in the art demonstrating that surface functionalization under the conditions identified by Sun resulted in surface functionalization with species such as carboxylic acid or hydroxyl groups, and thus resulted in individually separated nanotubes.

50. In Gojny, multiwall carbon nanotubes "were heated with an excess of a mixture of sulphuric and nitric acid" to produce "oxidised MWCNTs." LGES-1014 (Gojny), 821. This treatment resulted in "[t]he presence of carboxylic acid groups on the surface," which "enables a better dispersion of CNTs." *Id.* Indeed, Fig. 1 shows "oxidised MWCNTs … [that] are completely dispersed in the [epoxy resin] matrix due to the appearance of carboxylic groups on the surface." *Id.*, 822.

51. Xing provides another example of functionalization: "multiwalled CNTs" were oxidized in "a mixture solution of HNO_3 and H_2SO_4." LGES-1015 (Xing), 4186. Xing demonstrated that oxidation for longer times resulted in "the CNTs becom[ing] more functionalized," as demonstrated by the results shown in Table 1 and Figure 2, and concluded that "the treated [oxidized] CNTs had better dispersion." *Id.*, 4186-4187.

52. Farrugia describes that carbon nanotube functionalization "results in one or more chemical moieties attached to the carbon nanotubes," which "results in better dispersion of the nanotubes throughout the matrix." LGES-1007 (Farrugia), [0027]. Specifically, Farrugia describes that "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." *Id.*, [0028].

53. Forohar describes that "extensive treatment of a nanotube with concentrated nitric and sulfuric acids … produces a nanotube decorated with carboxyl groups, which can be further modified through the creation of amide and ester bonds to generate a vast variety of functional groups." LGES-1016 (Forohar), 7:22-28; *see also* 13:40-45 ("Suitable oxidation reagents for introducing carboxylic acid groups to a carbon nanotube include, but are not limited to, … a mixture of nitric acid (NHO_3) and sulfuric acid (H_2SO_4)").

54. The following additional references further corroborate that oxidation of carbon nanotubes in the presence of sulfuric acid and nitric acid can result in functionalization of the nanotube surfaces with moieties including carboxylic acid groups and hydroxyl groups: LGES-1019 (Hatton), [0062] ("the MWCNT are firstly oxidised in a 3:1 mixture of concentrated sulphuric

acid and nitric acid to produce surface carboxylic acid functionality."), [0080] ("MWCNT oxidized in a 3:1 mixture of concentrated sulphuric and nitric acid to produce carboxylic acid and hydroxyl functionality on the nanotube surface"); LGES-1020 (Sundara), 8:54-58 ("functional groups such as carboxylic acid and hydroxyl groups were introduced on the surface of CNT upon treatment with concentrated nitric acid and concentrated sulfuric acid in the ratio of 1:3"); LGES-1021 (Gan), [0023] ("CNTs can be functionalized with carboxyl groups using known techniques such as by sonicating pristine CNTs in an acid bath of sulfuric acid and nitric acid in the ratio of 3:1 for an extended period of time, such as 6 hours. … Hydroxyl groups can be attached to the sidewalls of CNTs using similar known techniques.").

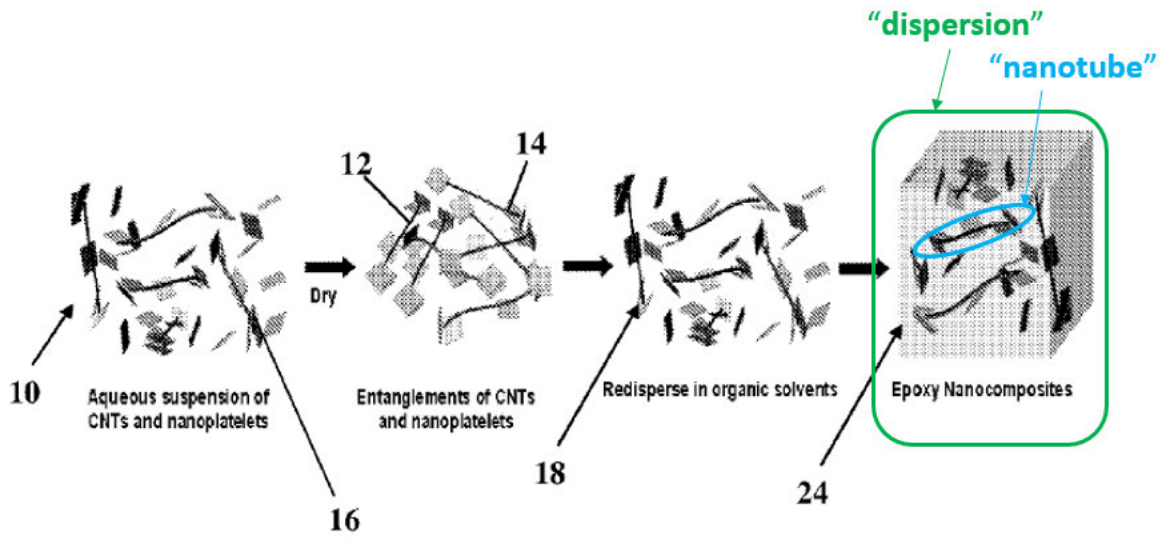
55. In light of this behavior of oxidized nanotubes, and given that Sun's oxidation process can lead to oxidized nanotubes, it is my opinion that a POSITA would have understood or found obvious that Sun's oxidized nanotubes can be present in the dispersion as generally individually distinct nanotubes, *e.g.*, that Sun's nanotubes are **discrete**.

Sun's nanocomposite includes oxidized, discrete carbon nanotubes

a. The nanotubes are oxidized carbon nanotubes

56. Sun's nanocomposite, which corresponds to the dispersion of claim 1, as I discussed above, includes **a plurality of oxidized carbon nanotubes**. This is the case at least because Sun forms its nanocomposite using the same nanotubes that were present in the aqueous dispersion: "[t]he nanotube-nanoplatelet association" of the dispersion 10 "comprises a nanofiller 16," which "may be dispersed in a medium … to form a nanocomposite 24." LGES-1004 (Sun), [0045]. These nanotubes, as I discussed above, are oxidized carbon nanotubes. That is, Sun's nanocomposite (outlined in green in FIG. 1, reproduced below) includes **oxidized carbon nanotubes** (one of which I have circled in blue). That the nanocomposite contains the same nanotubes—*i.e.*, oxidized carbon nanotubes—as the aqueous dispersion is further confirmed by the following citations: [0036] ("Oxidized, suspended carbon nanotubes (CNTs) 12 mixed [with] the aqueous solutions of nanoplatelets 14 are capable of being mixed to produce a …dispersible nanofiller 16, for implementation in nanocomposites 24"), [0046] ("The nanofiller and epoxy mixture comprises a nanocomposite 24"), [0044] (describing the nanotube-nanoplatelet association in the dispersion 10), [0068] ("The concept for re-dispersing exfoliated nanoplatelets and carbon nanotubes in polymers is summarized in FIG. 1"), claim 13; *see also* [0047]-

[0052] (describing that nanotubes from the aqueous dispersion "are dispersed in liquid precursors directly"), claim 20.



Except from Sun, FIG. 1 (annotated)

57. Sun's experimental procedure also confirms that the nanotubes of the nanocomposite are the same as those in the aqueous dispersion—that is, that the nanotubes of the nanocomposite are **oxidized carbon nanotubes**. Specifically, Sun describes its experimental procedure as an "aqueous dispersion, organic redispersion approach." LGES-1004 (Sun), [0069]; *see id.* ("epoxy nanocomposites containing MWNTs … prepared through the drying-redispersion process"). This approach involves preparation of an aqueous dispersion of nanotubes, redispersion of the nanotubes and nanoplatelets in acetone and mixing with epoxy monomer, and curing of the

epoxy mixture to form a nanocomposite. *See id.*, [0054]-[0058]. That is, in Sun's experimental procedure, the oxidized carbon nanotubes of the aqueous dispersion (*see* [0054]-[0056]) are the same nanotubes of the nanocomposite.

b. The nanotubes are discrete

58. Claim element [1a] also recites that the nanotubes in the dispersion are **discrete**. It is my opinion that a POSITA would have understood, or at least found obvious, that the oxidized nanotubes in Sun's nanocomposite are **discrete** nanotubes, *e.g.*, that they are generally individually distinct nanotubes, such as nanotubes that lack direct association with other nanotubes along the length of the nanotubes. For instance, a POSITA would have understood this or found it to be obvious based on Sun's description that its nanocomposites "contain[] ***individually dispersed CNTs***." LGES-1004 (Sun), [0014]. The following citations also support that the nanotubes in Sun's nanocomposite are discrete: Abstract ("the nanocomposite comprises at least one nanofiller, wherein said nanofiller comprises at least one nanotube… the nanotube comprises at least one ***exfoliated*** nanotube"), [0033] ("'exfoliated' as used herein refers to ***individually dispersed*** … nanotubes"), [0010]; claims 1, 5, 9, 11, 30-31. I note that Sun's nanocomposites contain multiple nanotubes, as described throughout, and

thus I interpret Sun's statement that its nanocomposite includes "at least one exfoliated nanotube" (*see id.*, Abstract) as referring to multiple exfoliated nanotubes.

59. Additionally, the dispersion of discrete nanotubes in the nanocomposite is depicted schematically in FIG. 1 (reproduced above), which shows discrete (*e.g.*, individually distinct) nanotubes in the nanocomposite.

60. Sun's experimental results (Example 1) also would have led a POSITA to understand or find obvious that the nanotubes in Sun's nanocomposite are **discrete**. For instance, Sun's TEM images "clearly suggest that … both MWNTs and nanoplatelets ***can be fully dispersed down to individual level*** in epoxy matrices," *e.g.*, that the nanotubes in the nanocomposite are discrete, *e.g.*, generally individually distinct nanotubes. LGES-1004 (Sun), [0069], FIG. 6.

61. As I discussed above, a POSITA would have understood, or at least found obvious, that nanotubes in Sun's aqueous dispersion are **discrete** because they are the same oxidized nanotubes as those of the aqueous dispersion, and because the nanotube oxidation process described by Sun can promote non-agglomeration of the nanotubes. *See* LGES-1004 (Sun), [0039], [0054].

[1b] at least one additive

62. Sun's aqueous suspension and Sun's epoxy nanocomposite (each of which is a dispersion, as I discussed above) both include at least one additive, as I discuss in the following paragraphs.

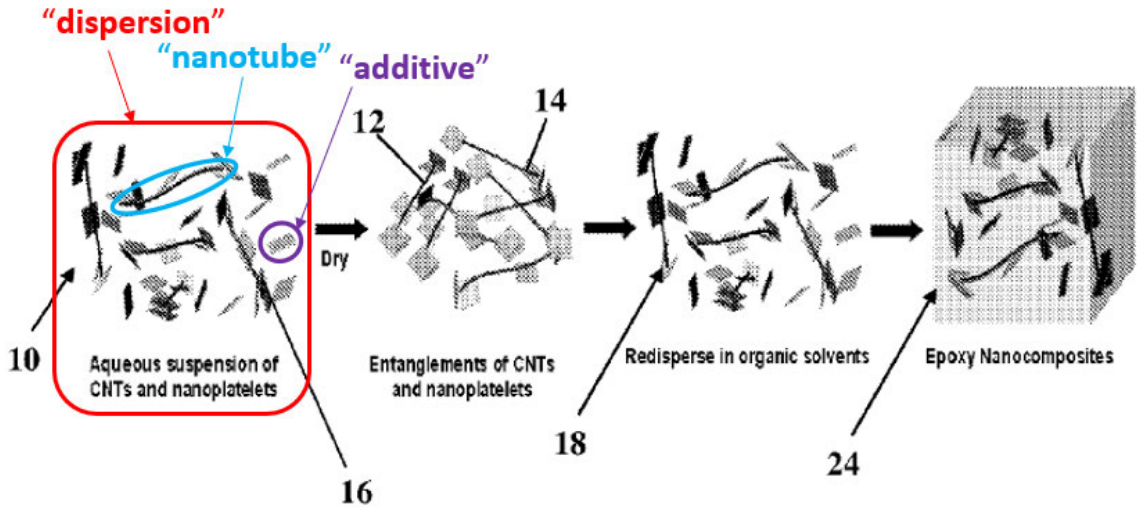
<u>*Sun's aqueous dispersion includes an additive*</u>

63. Sun describes that "nanotubes 12 and nanoplatelets 14 ***in solutions or solvents*** 18 forms a dispersion 10." LGES-1004 (Sun), [0036]; *see* Fig. 1. The solvent of the dispersion is one example of **an additive** in the dispersion. (I note that the '649 Patent identifies solvents as examples of additives to the dispersion. *See* LGES-1001, 7:46-53, claims 11-12.) Specifically, Sun's dispersion is an "aqueous solution," *e.g.*, a solution in water, and thus water (the solvent) is **an additive**. *Id.*, [0036]. *See also* the following cites confirming that Sun's dispersion is an aqueous solution: [0042] ("the ***aqueous-oxidized nanotube solution***, and aqueous-exfoliated nanoplatelet solution are admixed … to form a dispersion 10"), [0043] ("the ***aqueous*** nanotube-nanoplatelet dispersion 10"), [0056] ("Two ***Aqueous solutions*** containing oxidized MWNTs and fully exfoliated ZrP nanoplatelets were directly mixed"), [0068] ("the ***aqueous suspension*** 10, containing dispersed CNTs 12"), FIG. 1 ("***Aqueous suspension*** of CNTs and nanoplatelets") claim 13 (elements (a)-(d)), claim 20 (elements (b)-(f)).

I note that the ’649 Patent itself identifies solvents as an example of an additive: “In other embodiments such additives may include, for example, a solvent.” LGES-1001, 7:46-47.

64. Sun also describes that “***at least one surfactant*** is added to the nanotube-nanoplatelet mixture,” *i.e.*, to the aqueous dispersion, “to separate the nanotubes and nanoplatelets.” LGES-1004 (Sun), [0047]. The surfactant is a second example of **an additive** in the dispersion. *See also* the following cites describing inclusion of surfactant in the dispersion: *id.*, [0047] (“In certain instances, the surfactant comprises an ionic surfactant. The surfactant may preferably comprise sodium dodecyl sulfate (SDS)”), [0057] (“After dispersion, ***ionic surfactant*** such as sodium dodecyl sulfate (SDS) or polystyrene sulfonate (PSS) was added”). I note that the ’649 Patent itself identifies surfactants as an example of an additive: “Such additives may include, for example, various surfactants or dispersing aids and compounds such as sodium dodecyl sulfate,” among others. LGES-1001, 6:66-7:1.

65. The nanoplatelets are another example of **an additive** in Sun’s aqueous dispersion: “nanotubes 12 and ***nanoplatelets*** 14 in solutions or solvents 18 forms a dispersion 10.” LGES-1004 (Sun), [0036]. This is illustrated in Sun’s FIG. 1, which is reproduced and annotated below, with the nanoplatelets (**additive**) circled in purple.



Except from Sun, FIG. 1 (annotated)

66. Sun's nanoplatelets (an **additive**) can be composed of "any suitable material … such as clay, nanoclay, graphite, inorganic crystal, organic crystal, and combinations thereof," and in a specific example are composed of "alpha-zirconium phosphate (ZrP)." *Id.*, [0041]. The following citations provide further description of Sun's nanoplatelets in the aqueous dispersion: LGES-1004 (Sun), [0040] ("The nanoplatelets 14 are nanoparticles … having a thin, planar geometry"), [0042] ("the aqueous-oxidized nanotube solution, and ***aqueous-exfoliated nanoplatelet solution*** are admixed … to form a dispersion 10"), [0043] ("the aqueous nanotube-nanoplatelet dispersion 10 is a substantially homogeneous solution of dispersed nanotubes 12 and associated ***nanoplatelets*** 14"), [0055]-[0056] ("Two Aqueous solutions containing oxidized MWNTs and fully exfoliated ***ZrP nanoplatelets*** were

directly mixed" to form a dispersion), [0068] ("the aqueous suspension, containing dispersed CNTs 12 and ***exfoliated nanoplatelets 14***"), claim 13 ("admixing the functionalized nanotube solution and exfoliated ***nanoplatelet*** solution to form a nanofiller solution"), claim 16 ("wherein the nanofiller solution comprises, at least one exfoliated ***nanoplatelet*** associated with at least one oxidized nanotube"), [0044], [0047], claims 17-18.

Sun's nanocomposite includes an additive

67. Sun describes that the "nanofiller may be ***dispersed in a medium*** such as, but not limited to epoxy, plastic, or polymer, to form" the nanocomposite. LGES-1004 (Sun), [0045]. The medium (*e.g.*, epoxy) in which the nanofiller is dispersed to form the nanocomposite is one example of **an additive** in the nanocomposite—in either its uncured or cured form. *See also* the following cites describing the matrix medium of Sun's nanocomposite: LGES-1004 (Sun), Abstract ("the nanocomposite comprises at least one nanofiller, wherein said nanofiller comprises at least one nanotube, and a medium comprising a polymeric matrix"), [0014] ("epoxy nanocomposites containing individually dispersed CNTs"), [0058].

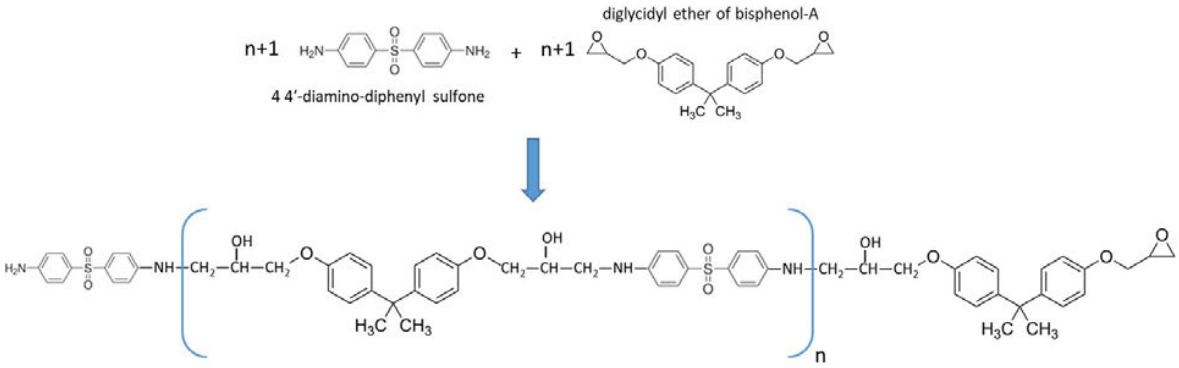
68. Specifically, the matrix medium of the nanocomposite can be considered an additive to the nanocomposite in its precursor (*e.g.*, uncured) form. Sun describes that "the epoxy" that is mixed with the nanofiller "comprises ***an***

epoxy monomer," such as "diglycidyl ether of bisphenol-A." LGES-1004 (Sun), [0045]. In this example, the epoxy monomer is the **additive**. The following cites provide further description of the uncured precursor to the matrix material of Sun's nanocomposite (*i.e.*, an example of an additive to the nanocomposite): LGES-1004 (Sun), [0058] ("The redispersed MWNTs:ZrP … were mixed with ***epoxy monomer***, diglycidyl ether of bisphenol-A"), Abstract ("redispersing at least one nanotube in ***a matrix precursor*** solution"), claims 30-31 ("A nanocomposite comprising: exfoliated carbon nanotubes … and an epoxy matrix having a first liquid state, and a second solid state," "wherein the exfoliated carbon nanotubes are dispersed in the first state of the epoxy matrix"), [0062], [0069], claims 1 and 13; *see also* [0052] (describing an embodiment in which "the nanotubes are dispersed in liquid precursors"), claims 20 and 28 ("admixing the isolated nanotube dispersion in a matrix precursor solution to form a carbon nanotube composite"). I note that the '649 Patent itself mentions uncured precursor as an example of an additive: "In other embodiments such additives may include, for example, at least one reactive species capable of creating a thermoset polymer such as, for example, epoxy, polyurethane, silicone, and mixtures thereof." LGES-1001, 7:53-57.

69. The medium (*e.g.*, the matrix of the nanocomposite) also can be considered **an additive** to the nanocomposite dispersion in its cured form. For instance, Sun describes the epoxy-nanofiller mixture is heated "to polymerize, crosslink, vulcanize, or otherwise cure … the epoxy nanocomposite." LGES-1004 (Sun), [0046]; *see* [0058] ("The epoxies were cured"), [0010] ("a nanotube reinforced nanocomposite" that includes "a medium, wherein said medium comprises a polymeric matrix; and wherein at least one nanotube is exfoliated within the medium", [0062] ("redispersing MWNTs and exfoliated ZrP into epoxy to form exfoliated nanocomposites"), [0069] ("epoxy nanocomposites containing MWNTs and exfoliated ZrP nanoplatelets"), Abstract, claims 1 and 13; *see also* [0052], claims 20 and 29 ("further compris[ing] curing the precursor solution"). The cured matrix material (*e.g.*, epoxy) is **an additive**.

70. A second example of **an additive** to Sun's nanocomposite is the curing agent, which is an additive to the uncured nanocomposite. Sun describes that "***a curing agent*** is added to the epoxy and nanotubes," where the curing agent is, *e.g.*, "4,4′-diamino-diphenyl sulfone." LGES-1004 (Sun), [0046]; *see* [0058]; *see also* [0052]. An example of the chemistry of curing diglycidyl ether of bisphenol-A (*see* LGES-1004 (Sun), [0045], [0058]) in

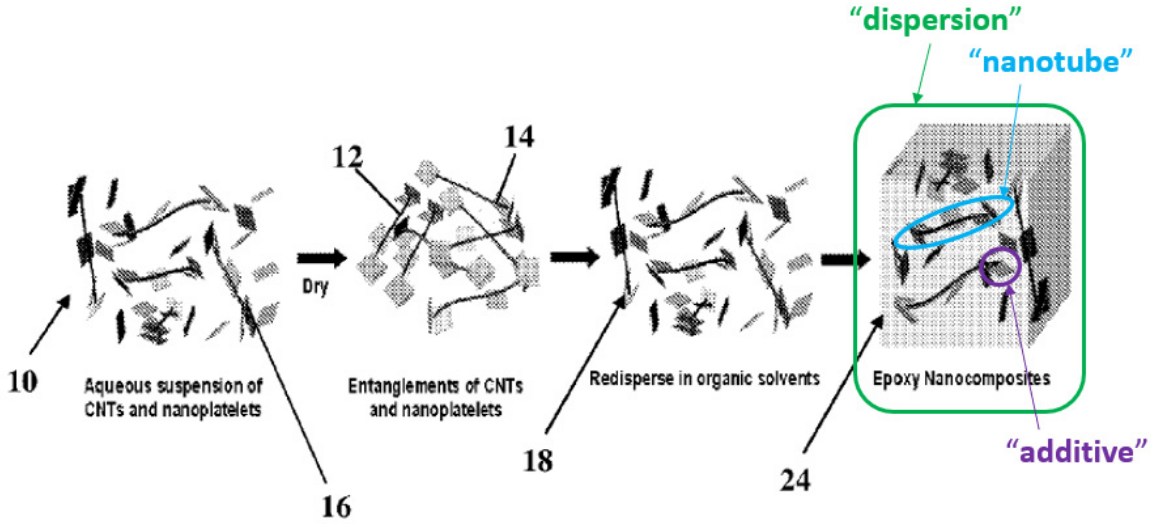
the presence of 4,4′-diamino-diphenyl sulfone as a curing agent to produce an epoxy polymer matrix is illustrated below:



71. A third example of **an additive** to Sun's nanocomposite is acetone. Sun describes that "the nanofiller is re-suspended in ***acetone*** prior to mixing in an epoxy." LGES-1004 (Sun), [0045]; *see* [0058] ("The redispersed MWNTs:ZrP with a weight ratio of 1:5 ***in acetone*** were mixed with epoxy monomer"). Acetone is an additive to the uncured nanocomposite dispersion, at the stage of processing after addition of the epoxy and prior to "the solvent [being] removed from the epoxy nanocomposite." *Id.*, [0046].

72. Sun's nanocomposite includes the same nanoplatelets that are included in the aqueous dispersion; these nanoplatelets are another example of **an additive** in both the uncured nanocomposite and the cured nanocomposite. *See* LGES-1004 (Sun), [0045] ("The nanotube-nanoplatelet association comprises a nanofiller 16" which "may be dispersed in a medium such as, but not limited to epoxy, plastic, or polymer, to form a nanocomposite 24").

I have outlined Sun's cured nanocomposite in blue in FIG. 1, reproduced below, and circled one nanoplatelet in purple and one nanotube in blue. The following citations provide further description of Sun's nanoplatelets in the nanocomposite: [0046] ("The nanofiller and epoxy mixture comprises a nanocomposite 24"), [0058] ("The redispersed MWNTs:ZrP [nanoplatelets] … were mixed with epoxy" to form a nanocomposite"), [0062] ("Here, we show the improved steps for redispersing MWNTs and exfoliated ZrP into epoxy to form exfoliated nanocomposites"), [0069] ("FIG. 6 shows the TEM images of epoxy nanocomposites containing MWNTs and exfoliated ZrP nanoplatelets"), FIG. 6; claims 3-4, 7-8, 13, 15-18, 20.



Except from Sun, FIG. 1 (annotated)

[1c] wherein the oxidized, discrete carbon nanotubes have an aspect ratio of 25 to 500,

73. Sun describes that the oxidized, discrete carbon nanotubes in its aqueous dispersion "have ***an aspect ratio***, or length to diameter ratio, of ***at least about 10***" or "***at least about 20***." LGES-1004 (Sun), [0038]. Sun also states that "[a]s used herein, the term 'nanotube(s)' or NT(s) refers to any cylindrical atomic allotrope or polyatomic molecule with … ***an aspect ratio (length to diameter ratio) of at least about 10***." *Id.*, [0029]. As I have previously discussed, the same nanotubes are present both in Sun's aqueous dispersion and in Sun's nanocomposite. *See id.*, [0044]-[0046]. Thus, the nanotubes in the nanocomposite also have an aspect ratio of "at least about 10" or "at least about 20." *Id.*, [0038]. These ranges of aspect ratios for Sun's nanotubes (at least about 10 or at least about 20) overlap with the range of aspect ratios recited in claim element [1c] (25 to 500).

74. Sun also provides specific dimensions, stating that "[t]he carbon nanotubes have a diameter of between about 1 nm and about 20 nm," and that "[t]he carbon nanotube length is at least about 100 nm." LGES-1004, [0038]. The aspect ratio of a nanotube is calculated by dividing length by diameter. At least some of the aspect ratios indicated by the dimensions given by Sun fall within the claimed 25-500 range of aspect ratios. For instance, taking the smallest length (100 nm) and the smallest diameter (1 nm) gives an aspect

ratio of **100**. As another example, another diameter within Sun's 1-20 nm range, such as 2 nm, and again the smallest length (100 nm), gives an aspect ratio of **50**.

75. I note that the '649 Patent does not describe particular unexpected results associated with nanotubes having an aspect ratio of 25 to 500 (as claimed) as opposed to those in the range of at least about 10 or at least about 20 (as described by Sun). The '649 Patent also does not provide any discussion or evidence that these ranges of aspect ratios are in any way critical.

[1d] are multiwall, and

76. The nanotubes in Sun's aqueous dispersion are **multiwall**. For instance, Sun describes that its aqueous dispersion includes "[o]xidized, suspended carbon nanotubes (CNTs) 12" such as "***multi-walled carbon nanotubes (MWNTs)***")." LGES-1004 (Sun), [0036]-[0038]. The following additional citations further confirm that Sun's aqueous dispersion includes multiwall nanotubes: [0054]-[0056] ("Two Aqueous solutions containing oxidized ***MWNTs*** and fully exfoliated ZrP nanoplatelets were directly mixed" to form a dispersion), claims 13-14 ("admixing the functionalized nanotube solution and exfoliated nanoplatelet solution to form a nanofiller solution," "wherein the carbon nanotube is selected from the group consisting of ***multi-walled***

carbon nanotubes, single walled carbon nanotubes, and combinations thereof"), claims 20 and 27, [0029]-[0030], [0068], [0006].

77. As I have previously discussed, the same nanotubes are present both in Sun's aqueous dispersion and in Sun's nanocomposite. *See id.*, [0044]-[0046]. Thus, the nanotubes in the nanocomposite are also multiwall nanotubes.

78. Sun's experimental procedure also confirms that the nanotubes in both its aqueous dispersion and its nanocomposite are **multiwall**. In Sun's Example 1, "[t]wo Aqueous solutions containing oxidized ***MWNTs*** and fully exfoliated ZrP nanoplatelets were directly mixed" to form the aqueous dispersion. LGES-1004 (Sun), [0056]. Additionally, "redispersed ***MWNTs***:ZrP" were mixed with epoxy precursors "to achieve a finial [sic] ***MWNT*** concentration … in epoxy nanocomposites." LGES-1004 (Sun), [0058]; *see* [0062]. Furthermore, Sun describes that its TEM images in FIGS. 6A-6B, which are images of experimental produced materials, show "epoxy nanocomposites ***containing MWNTs*** and exfoliated ZrP nanoplatelets." *Id.*, [0069]; *see* [0022]-[0023].

[1e] are present in the range of greater than zero to about 30% by weight based on the total weight of the dispersion.

Sun's aqueous dispersion has the claimed weight percentage of nanotubes

79. Sun describes that its aqueous dispersion has a "concentration of nanotubes" of "***at least 1 wt%***." LGES-1004 (Sun), [0043].

80. Sun also describes the concentration of nanotubes as follows: "The concentration of nanotubes 12 in the dispersion 10 is between about 0 ppm and about 1000 ppm; alternately between about 100 ppm and about 500 ppm; and in certain instances, the concentration of nanotubes is about 200 ppm." LGES-1004 (Sun), [0043]; *see* [0056] (In the aqueous dispersion of Example 1, "The final concentrations of CNTs and ZrP are 200, and 1000 ppm, respectively"). Although Sun does not explicitly state the basis on which its ppm (parts per million) values are calculated, it is my opinion that a POSITA would have understood these to refer to a mass or weight basis. On this basis, a concentration of nanotubes of 1% by weight is 1 part per hundred (pph), and a concentration of nanotubes of 0.0001% by weight is 1 ppm. Thus, Sun's disclosure of "between about 0 ppm and about 1000 ppm," for instance, is a disclosure of a weight percentage in the range of 0 to 0.1 wt%. *Id.*, [0043].

81. These ranges of weight percentages described by Sun (*e.g.*, "at least 1 wt%," LGES-1004 (Sun), [0043]) overlap with the range of weight percentages recited in claim element [1e] ("greater than zero to about 30% by weight").

82. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes at weight percentages of greater than zero to about 30%. The '649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

83. I also note that fewer than all of the nanotubes in Sun's dispersion may be oxidized, discrete carbon nanotubes. However, even if this is the case, those oxidized, discrete carbon nanotubes still constitute a portion of the "at least 1 wt %" of the entire population of nanotubes in Sun's dispersion, meaning that the population of oxidized nanotubes is still **greater than zero by weight**. LGES-1004 (Sun), [0043].

Sun's nanocomposite has the claimed weight percentage of nanotubes

84. Sun describes that its nanocomposite has a "concentration of nanotubes in the epoxy [of] ***between about 0.01 wt% and about 50 wt%***." LGES-1004 (Sun), [0045]. Sun also provides the following additional descriptions of the weight percentages of nanotubes in its nanocomposite: *id.* ("Alternatively, the nanotube concentration is ***between about 0.05 wt% and about 0.6 wt%***;

alternatively ***between about 0.1 wt% and about 0.4 wt%***"); [0058] ("The redispersed MWNTs:ZrP … were mixed with epoxy monomer … to achieve a finial [sic] ***MWNT concentration of 0.2 and 0.4 wt%*** in epoxy nanocomposites"), [0069] ("FIG. 6 shows the TEM images of epoxy nanocomposites with ***0.2 and 0.4 wt.% of MWNTs***"), [0022]-[0023], FIG. 6.

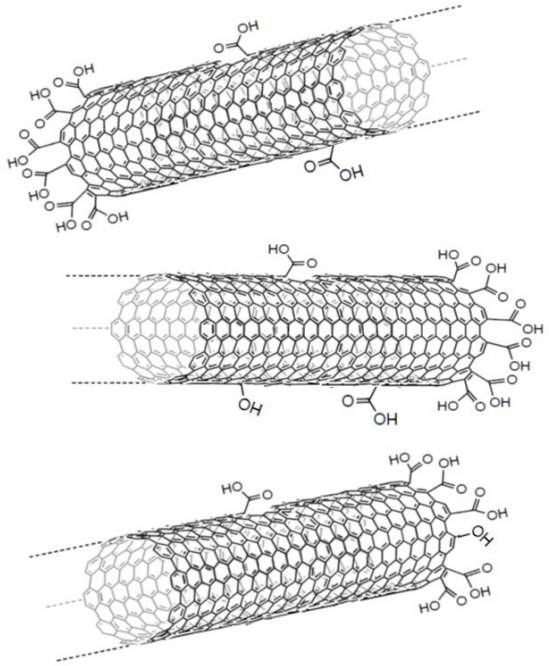
85. These ranges of weight percentages described by Sun (*e.g.*, between about 0.01 wt% and about 50 wt%; *see* LGES-1004 (Sun), [0045]) overlaps with the range of weight percentages recited in claim element [1e] ("greater than zero to about 30% by weight"). I note that the '649 Patent does not describe particular unexpected results associated with nanocomposites including nanotubes at weight percentages of greater than zero to about 30%. The '649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

86. Again, even if only a subset of Sun's nanotubes are oxidized, discrete carbon nanotubes, those oxidized, discrete carbon nanotubes still constitute a portion of the 0.01-50 wt% concentration of the entire population of nanotubes in Sun's nanocomposite, meaning that the population of oxidized nanotubes is still **greater than zero by weight**.

(2) Claim 2

[2] The dispersion of claim 1 wherein at least 70 percent by weight of the nanotubes are discrete.

87. As I previously discussed when addressing claim element [1a], a POSITA would have understood or found obvious that the nanotubes in Sun's aqueous dispersion and/or in Sun's nanocomposite can be **discrete**. For instance, this conclusion was based (at least in part) on Sun's description of the oxidation of the nanotubes in "a mixture of sulfuric acid and nitric acid," *e.g.*, where "the nitric acid to sulfuric acid ratio is approximately 3:1." LGES-1004 (Sun), [0039]; *see* [0054]. Oxidation of carbon nanotubes under these conditions (*i.e.*, with a 3:1 ratio of nitric to sulfuric acid) was well-known to result in surface functionalization with species such as carboxylic acid or hydroxyl groups, which causes the nanotubes to be individually separated, *e.g.*, discrete, as I discussed above, and as illustrated in a figure above (generated based on Scheme 1 of Hirsch (LGES-1041)), which is reproduced again here. *See* LGES-1014 (Gojny), 821-822; LGES-1015 (Xing), 4186-4187; LGES-1007 (Farrugia), [0027]-[0028]; LGES-1016 (Forohar), 7:22-28, 13:40-45.



88. It is my opinion that given that the oxidation treatment described by Sun was well-known to result in surface functionalization to a degree that the nanotubes are generally individually distinct, a POSITA would have understood or found obvious that substantially all of Sun's nanotubes can be surface functionalized such that they are present in the dispersion as discrete, *e.g.*, individually distinct, nanotubes. Specifically, a POSITA would have understood or found obvious that substantially all of the nanotubes being so functionalized (and thus discrete) encompasses **at least 70% by weight of the nanotubes** being so functionalized, and thus **discrete** nanotubes in the dispersion. Otherwise, it is alternatively/additionally my opinion that this

"at least 70% by weight" limitation is just a design choice. A POSITA developing a dispersion including discrete nanotubes would have considered various weight percentages of discrete nanotubes, of which 70% is an example, which is not described as corresponding to any unexpected results or unique solutions.

(3) Claim 3

[3] The dispersion of claim 1 in the form of free flowing particles.

89. As I previously discussed when addressing claim element [1a] and claim [2], a POSITA would have understood or found obvious that substantially all of the nanotubes in Sun's aqueous dispersion and/or in Sun's nanocomposite are discrete, *e.g.*, that they are generally individual, separated nanotubes. Individual, separated nanotubes are not aggregated into a clump, but instead are free flowing particles. For instance, individual, separated nanotubes, such as those in Sun's aqueous dispersion and/or nanocomposite (*e.g.*, at least Sun's uncured nanocomposite) can flow freely relative to other nanotubes in the dispersion. Thus, when substantially all of the nanotubes in the dispersion are discrete, the dispersion is **in the form of free flowing particles** at least because substantially all of the particles (*e.g.*, nanotubes) in the dispersion can flow freely relative to one another.

(4) Claim 4

[4] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes comprise an oxidation species selected from carboxylic acid or a derivative carbonyl containing species wherein the derivative carbonyl species is selected from ketones, quaternary amines, amides, esters, acyl halogens, and monovalent metal salts.

90. As I discussed above for claim element [1a], Sun's nanotubes are oxidized in "a mixture of sulfuric acid and nitric acid," *e.g.*, where "the nitric acid to sulfuric acid ratio is approximately 3:1." LGES-1004 (Sun), [0039]; *see* [0054] ("The CNTs were first oxidized in a mixture of concentrated sulfuric acid and concentrated nitric acid with a volume ratio of 3:1 by ultrasonication in a sonication bath for 2 hours"). I also previously discussed that it was well-known that oxidation of carbon nanotubes under these conditions described by Sun results in functionalization of the nanotube surfaces with moieties including carboxylic acid groups and hydroxyl groups, as I discuss further in the following paragraphs. Thus, it is my opinion that a POSITA would have understood or found obvious that Sun's nanotubes include at least **carboxylic acid oxidation species**.

91. The following citations provide examples of functionalization of carbon nanotubes in a mixture of sulfuric acid and nitric acid (*e.g.*, consistent with Sun's oxidation process), demonstrating that it was well-known that such an oxidation process results in carboxylic acid groups and hydroxyl groups on

the surface of the nanotubes: *See* LGES-1019 (Hatton), [0062] ("the MWCNT are firstly oxidized[sic] in a 3:1 mixture of concentrated sulphuric acid and nitric acid to produce carboxylic acid functionality"), [0080] ("MWCNT oxidized in a 3:1 mixture of concentrated sulphuric and nitric acid to produce carboxylic acid and hydroxyl functionality on the nanotube surface"); LGES-1020 (Sundara), 8:54-58 ("functional groups such as carboxylic acid and hydroxyl groups were introduced on the surface of CNT upon treatment with concentrated nitric acid and concentrated sulfuric acid in the ratio of 1:3"); LGES-1016 (Forohar), 7:22-26 ("the extensive treatment of a nanotube with concentrated nitric and sulfuric acids … produces a nanotube decorated with carboxyl groups"); LGES-1021 (Gan), [0023] ("CNTs can be functionalized with carboxyl groups … by sonicating pristine CNTs in an acid bath of sulfuric acid and nitric acid in the ratio of 3:1 for an extended period of time, such as 6 hours. … Hydroxyl groups can be attached to the sidewalls of CNTs using similar known techniques."); LGES-1014 (Gojny), 821-822.

92. These moieties—*e.g.*, carboxylic acid and hydroxyl groups—are oxidation species because they are functionalized onto the nanotubes as a result of an oxidation process. Not all oxidations necessarily involve oxygen. But when oxygen bearing functionality is added to a chemical system, it is generally an

oxidation process. Carboxylic acid and hydroxyl groups are oxygen bearing functionalities, and their addition to a carbon based system, like carbon nanotubes, represents oxidation.

93. It is my opinion that a POSITA would have understood or found obvious that Sun's nanotubes include a carboxylic acid oxidation species, given that, as I just discussed, it was well-known in the art that oxidation processes consistent with those described by Sun (*see* LGES-1004 (Sun), [0039]; [0054]) result in functionalization of carbon nanotubes with carboxylic acid groups. *See* LGES-1019 (Hatton), [0062], [0080]; LGES-1020 (Sundara), 8:54-58; LGES-1016 (Forohar), 7:22-26; LGES-1021 (Gan), [0023]; LGES-1014 (Gojny), 821-822.

94. I also note that a carboxylic acid functionality on the surface of an oxidized carbon nanotube is a reactive site to which other chemical species can react to produce derivative carbonyl species, such as ketones, esters, acyl halogens, or monovalent metal salts.

(5) Claim 5

[5] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes comprise an oxidation species selected from hydroxyl or derived from hydroxyl containing species.

95. Referring to my discussion of claim 4, it is also my opinion that a POSITA would have understood or found obvious that Sun's nanotubes include a

hydroxyl oxidation species, given that it was well-known in the art that oxidation processes consistent with those described by Sun (*see* LGES-1004 (Sun), [0039]; [0054]) result in functionalization of carbon nanotubes with hydroxyl groups. *See* LGES-1019 (Hatton), [0080] ("MWCNT oxidized[sic] in a 3:1 mixture of concentrated sulphuric and nitric acid to produce carboxylic acid and hydroxyl functionality on the nanotube surface"); LGES-1020 (Sundara), 8:54-58 ("functional groups such as carboxylic acid and hydroxyl groups were introduced on the surface of CNT upon treatment with concentrated nitric acid and concentrated sulfuric acid in the ratio of 1:3"); LGES-1021 (Gan), [0023] ("CNTs can be functionalized with carboxyl groups … by sonicating pristine CNTs in an acid bath of sulfuric acid and nitric acid in the ratio of 3:1 for an extended period of time, such as 6 hours. … Hydroxyl groups can be attached to the sidewalls of CNTs using similar known techniques.").

(6) Claim 6

[6] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes are present in the range of 15% to about 25% by weight based on the total weight of the dispersion.

96. As I discussed above for claim element [1e], Sun's aqueous dispersion has a "concentration of nanotubes" of "***at least 1 wt%***." LGES-1004 (Sun), [0043]. A POSITA would have found it obvious based on this disclosure

that the population of oxidized, discrete carbon nanotubes is likewise present in an amount of at least 1 wt.%. A concentration of oxidized, discrete nanotubes that is "at least 1 wt%" (LGES-1004 (Sun), [0043]) overlaps with and encompasses the range of weight percentages recited in claim [6] ("15% to about 25% by weight").

97. As I also discussed above for claim element [1e], Sun's nanocomposite has a "concentration of nanotubes in the epoxy [of] ***between about 0.01 wt% and about 50 wt%***." LGES-1004 (Sun), [0045]; *see* [0058]. A nanotube concentration of between about 0.01 wt% and about 50 wt% overlaps with and encompasses the range of weight percentages recited in claim [6] ("15% to about 25% by weight").

98. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes (either aqueous dispersions or nanocomposites) at weight percentages in the range of 15% to about 25%. The '649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

(7) Claim 7

[7] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes are present in the range of 18% to about 22% by weight based on the total weight of the dispersion.

99. As I discussed above for claim element [1e], Sun's aqueous dispersion has a "concentration of nanotubes" of "***at least 1 wt%***." LGES-1004 (Sun), [0043]. A POSITA would have found it obvious based on this disclosure that the population of oxidized, discrete carbon nanotubes is likewise present in an amount of at least 1 wt.%. A concentration of oxidized, discrete carbon nanotubes that is "at least 1 wt%" (LGES-1004 (Sun), [0043]) overlaps with and encompasses the range of weight percentages recited in claim [7] ("18% to about 22% by weight").

100. As I also discussed above for claim element [1e], Sun's nanocomposite has a "concentration of nanotubes in the epoxy [of] ***between about 0.01 wt% and about 50 wt%***." LGES-1004 (Sun), [0045]; *see* [0058]. A nanotube concentration of between about 0.01 wt% and about 50 wt% overlaps with and encompasses the range of weight percentages recited in claim [7] ("18% to about 22% by weight").

101. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes (either aqueous dispersions or nanocomposites) at weight percentages in the range of 18% to about 22%.

The'649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

(8) Claim 11

[11] The dispersion of claim 1, wherein the additive is a solvent.

102. As I discussed for claim element [1b], an example of an additive in Sun's aqueous dispersion is the solvent (*e.g.*, water): Sun describes that its dispersion is composed of "nanotubes 12 and nanoplatelets 14 ***in solutions or solvents***." LGES-1004 (Sun), [0036]; *see* [0042]-[0043] ("the aqueous-oxidized nanotube solution, and aqueous-exfoliated nanoplatelet solution are admixed, directly, and agitated to form a dispersion" that "is a substantially homogeneous solution of the dispersed nanotubes 12 and associated nanoplatelets 14"), [0056] ("Two Aqueous solutions containing oxidized MWNTs and fully exfoliated ZrP nanoplatelets were directly mixed"), [0068], claim 13 (elements (a)-(d)), claim 20 (elements (b)-(f)), Fig. 1. That is, an **additive** in Sun's aqueous dispersion **is a solvent**.

103. I also discussed for claim element [1b] that an example of an additive in Sun's nanocomposite (specifically, the uncured nanocomposite) is acetone: Sun describes that "the nanofiller is re-suspended in ***acetone*** prior to mixing in an epoxy." LGES-1004 (Sun), [0045]; *see* [0058] ("The redispersed MWNTs:ZrP … in acetone were mixed with epoxy monomer"), [0046].

After the nanofiller is suspended in acetone, the epoxy is added, generating the uncured nanocomposite, which thus still includes the acetone. That is, an **additive** in Sun's nanocomposite is acetone, which **is a solvent**.

(9) Claim 13

[13] The dispersion of claim 1, wherein the additive comprises at least one reactive species capable of creating a thermoset polymer.

104. As I discussed for claim element [1b], one example of an additive in Sun's uncured nanocomposite is the uncured matrix precursor. Sun describes that this uncured matrix precursor can be "***an epoxy monomer***" such as "diglycidyl ether of bisphenol-A," and describes that this epoxy monomer is heated in the presence of a curing agent to form an "epoxy nanocomposite." LGES-1004 (Sun), [0045]-[0046]; *see* [0058], claims 19 ("wherein the precursor solution is selected from the group consisting of epoxies…") and 30; *see also* [0052]. This epoxy monomer additive includes **a reactive species capable of creating a thermoset polymer**: the reactive groups of the epoxy precursor, which are capable of creating an epoxy when combined with a thermosetting hardener or curing agent and heated. Specifically, Sun's epoxy monomers include **reactive species** (namely, epoxides, of which diglycidyl ether of bisphenol-A is one example) that are **capable of creating epoxy polymers** through reaction with the curing agent. *See* LGES-1004 (Sun), [0045]-[0046] ("In a potential exemplary application, the

epoxy comprises an epoxy monomer. In certain instances, the epoxy monomer comprises diglycidyl ether of bisphenol-A. … A curing agent is added to the epoxy and nanotubes, and heated to polymerize, crosslink, vulcanize, or otherwise cure, without limitation, the epoxy nanocomposite. In certain instances, the curing agent comprises 4-4′-diamino-diphenyl sulfone, hereinafter, DDS"); *see* [0058], claims 19, 28 and 30. And these epoxy polymers that are created from this reaction are **thermoset polymers**. *See* LGES-1004 (Sun), [0045] ("an epoxy may comprise ***any thermosetting polymer*** that cures, crosslinks, and/or hardens when mixed with a catalyzing agent"); *see also* claims 2 and 10, which note that the cured matrix can be a "thermoset[]."

105. Bergmark corroborates that this epoxy-based chemistry is capable of creating thermoset epoxy resins: "Thermoset resins exemplified by epoxies may be resins wherein cross-linking occurs between the epoxy resin species comprising epoxide groups and curing agents composing corresponding functional grounds for crosslinking. The process of crosslinking is termed 'curing.'" LGES-1024 (Bergmark), [0057]. Moreover, Bergmark identifies "diglycidyl ether of bisfenol A" as an example epoxy. *Id.*, [0059]. Bergmark also teaches: "curing agents comprise… ***amines***." *Id.*, [0060] The curing

agent of Sun, 4-4′-di***amino***-diphenyl sulfone (DDS) is such an ***amine*** curing agent.

106. I note that Sun also describes additional precursors that also contain reactive species capable of creating a thermoset polymer, as listed in Sun's claim 19: "wherein the precursor solution is selected from the group consisting of epoxies, plastics, polymers, thermosets, thermoplastics, and combinations thereof." LGES-1004 (Sun), claim 19; *see* [0045] ("a nanofiller may be dispersed in a medium such as, but not limited to, epoxy, plastic, or polymer, to form a nanocomposite"), claim 28.

(10) Claim 14

[14] The dispersion of claim 13, wherein the thermoset polymer is selected from epoxy, polyurethane and silicone.

107. As I discussed for claim 13, Sun describes curing an epoxy monomer to create an epoxy, which Sun identifies as a **thermoset polymer**: "an epoxy may comprise ***any thermosetting polymer*** that cures, crosslinks, and/or hardens when mixed with a catalyzing agent." LGES-1004 (Sun), [0045]. That is, Sun's epoxy monomer additive includes a reactive species capable of creating a thermoset polymer (*see* my discussion in claim 13), and the resulting **thermoset polymer is an epoxy**. *See* LGES-1004 (Sun), [0014] ("epoxy nanocomposites containing individually dispersed CNTs and exfoliated nanoplatelets"), [0022]-[0023] and [0027] ("epoxy

nanocomposites containing … MWNTs"), [0045]-[0046] ("a nanofiller may be dispersed in a medium such as, but not limited to epoxy, plastic, or polymer, to form a nanocomposites"), [0058], [0062], [0069], [0071]-[0072], claim 2 ("the medium [in which the nanotubes are disposed] is selected from the group consisting of thermosets, thermoplastics, and combinations thereof"), claims 10, 19, 28, and 30-31; *see also* [0052].

(11) Claim 17

[17] The dispersion of claim 1 which further comprises at least one filler.

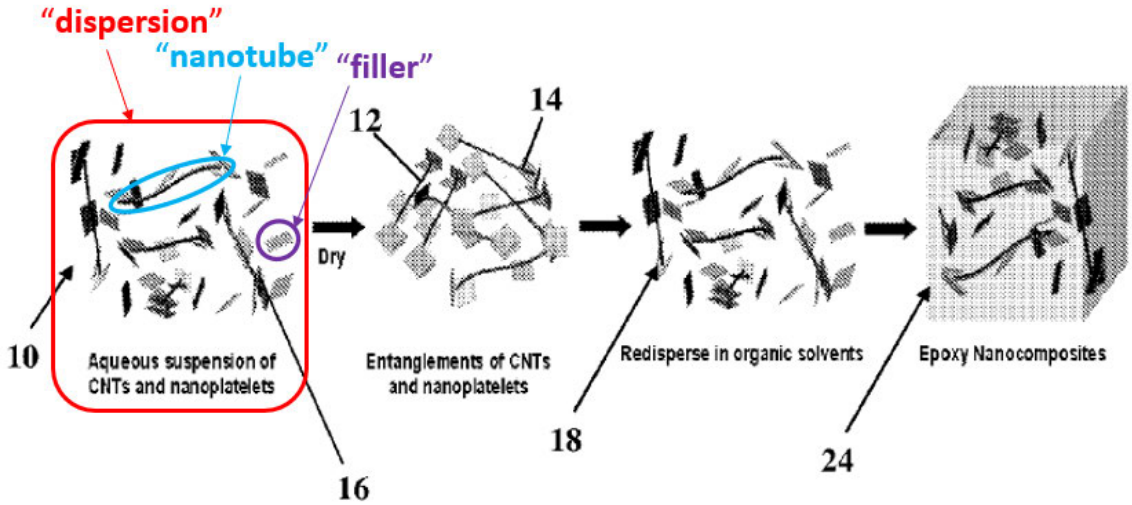
Sun's aqueous dispersion includes a filler

108. I have outlined Sun's aqueous dispersion in red in FIG. 1, which is reproduced below. Sun describes this aqueous dispersion: "nanotubes 12 and nanoplatelets 14 in solutions or solvents 18 forms a dispersion 10." LGES-1004 (Sun), [0036]. This aqueous dispersion includes nanoplatelets 14 one of which I have circled in purple. *Id.*. The nanoplatelets 14 and nanotubes 12 (one of which is circled in blue) form a dispersion 10 in solutions or solvents. *Id.*, [0036]. Sun's nanoplatelets can be considered **a filler** in this aqueous dispersion. Specifically, Sun's nanoplatelets can be composed of "any suitable material … such as clay, nanoclay, graphite, inorganic crystal, organic crystal, and combinations thereof," and in a specific example are composed of "alpha-zirconium phosphate (ZrP)." *Id.*,

[0041].[1] The following citations provide further description of Sun's nanoplatelets in the aqueous dispersion: LGES-1004 (Sun), [0040] ("The nanoplatelets 14 are nanoparticles … having a thin, planar geometry"), [0042] ("the aqueous-oxidized nanotube solution, and ***aqueous-exfoliated nanoplatelet solution*** are admixed … to form a dispersion 10"), [0043] ("the aqueous nanotube-nanoplatelet dispersion 10 is a substantially homogeneous solution of dispersed nanotubes 12 and associated ***nanoplatelets*** 14"), [0055]-[0056] ("Two Aqueous solutions containing oxidized MWNTs and fully exfoliated ***ZrP nanoplatelets*** were directly mixed" to form a dispersion), [0068] ("the aqueous suspension, containing dispersed CNTs 12 and ***exfoliated nanoplatelets 14***"), claim 13 ("admixing the functionalized nanotube solution and exfoliated ***nanoplatelet*** solution to form a nanofiller solution"), claim 16 ("wherein the nanofiller solution comprises, at least one

[1] The composition of Sun's platelets is consistent with the compositions identified by the '649 Patent for its fillers: "Such fillers may include, for example, a filler selected from silicon, lead, lead derivatives, carbon black, graphite, graphene, graphene oxides, paramagnetic particles and mixtures thereof." LGES-1001, 7:64-67.

exfoliated ***nanoplatelet*** associated with at least one oxidized nanotube"), [0044], [0047], claims 17-18.

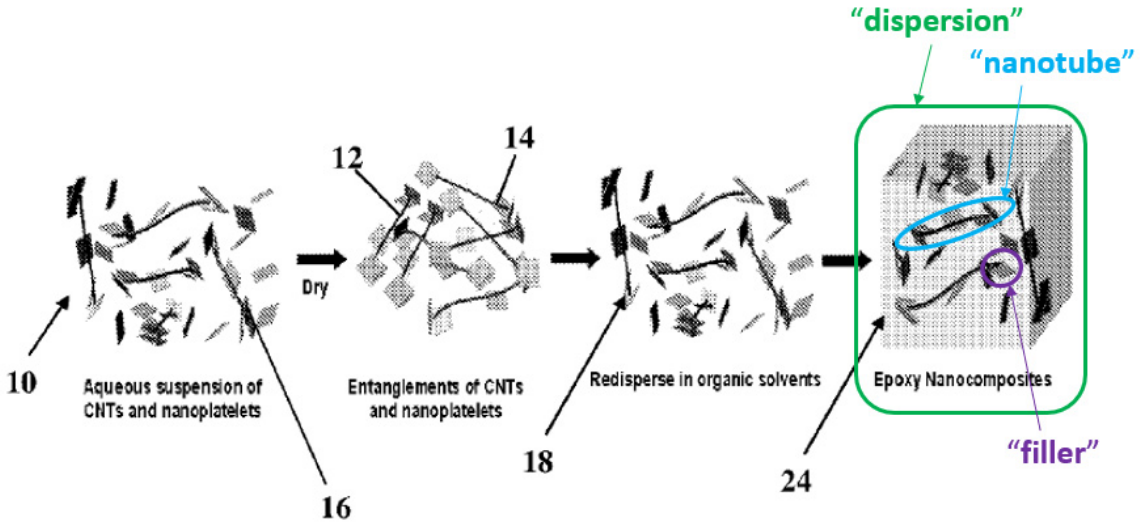


Excerpt from Sun, FIG. 1 (annotated)

Sun's nanocomposite includes a filler

109. Sun's nanocomposite includes the same nanoplatelets that are included in the aqueous dispersion; these nanoplatelets also can be considered **a filler** in the (uncured or cured) nanocomposite. *See* LGES-1004 (Sun), [0045] ("The nanotube-nanoplatelet association comprises a nanofiller 16" which "may be dispersed in a medium such as, but not limited to epoxy, plastic, or polymer, to form a nanocomposite 24"). I have outlined Sun's cured nanocomposite in blue in FIG. 1, reproduced below, and circled one nanoplatelet in purple and one nanotube in blue. The following citations provide further description of Sun's nanoplatelets in the nanocomposite: [0046] ("The

nanofiller and epoxy mixture comprises a nanocomposite 24"), [0058] ("The redispersed MWNTs:ZrP [nanoplatelets] … were mixed with epoxy" to form a nanocomposite"), [0062] ("Here, we show the improved steps for redispersing MWNTs and exfoliated ZrP into epoxy to form exfoliated nanocomposites"), [0069] ("FIG. 6 shows the TEM images of epoxy nanocomposites containing MWNTs and exfoliated ZrP nanoplatelets"), FIG. 6; claims 3-4, 7-8, 13, 15-18, 20.



Excerpt from Sun, FIG. 1 (annotated)

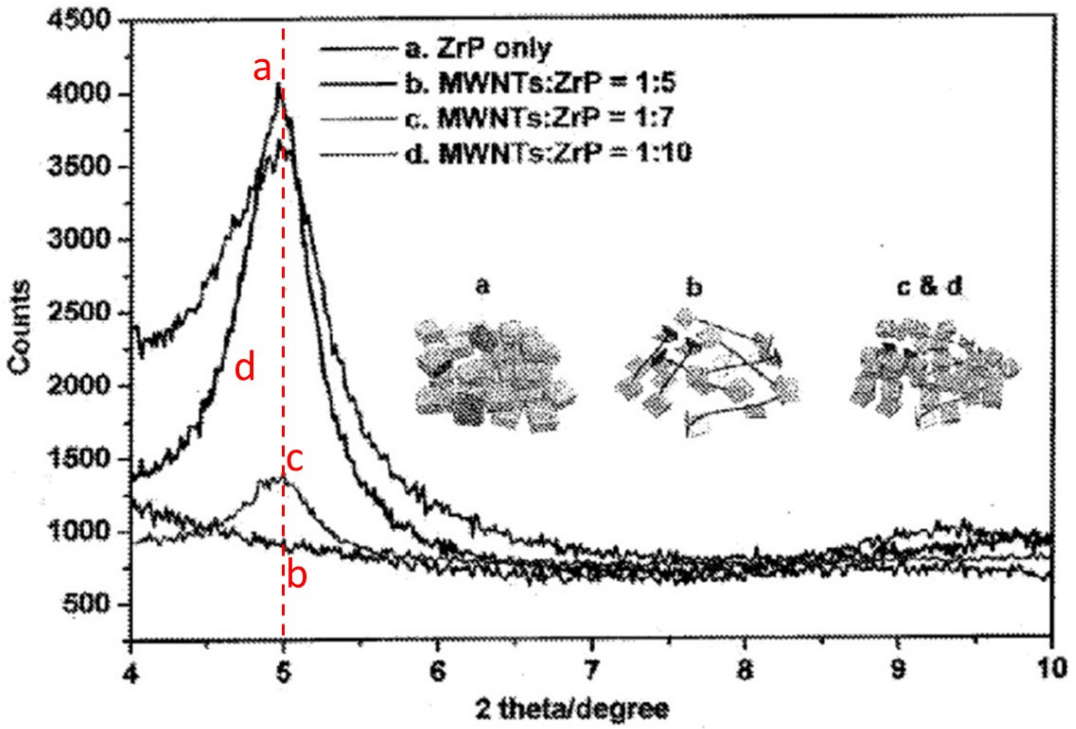
(12) Claim 18

[18] The dispersion of claim 17, wherein the filler has at least one dimension less than about 20 microns.

110. As I discussed with respect to claim 17, Sun's nanoplatelets are **a filler** in both the aqueous dispersion and the nanocomposite. Sun describes the size

range of the nanoplatelets: they "***have a diameter range from about 10 nm to about 20,000 nm***, and preferably between about 100 nm and 1000 nm." LGES-1004 (Sun), [0040]. This diameter range (from about 10 nm to about 20,000 nm, or between about 100 nm and 1000 nm) is **less than about 20 microns**. This size of Sun's nanoplatelets is corroborated by the TEM images of FIG. 2B. *See id.*, [0016]-[0017], [0055]-[0056].

111. Moreover, I note that Sun's X-ray diffraction (XRD) data presented in FIG. 5 (which I have reproduced below) shows how exfoliation changes the thickness of the ZrP platelets of Sun's experimental example. Generally speaking, exfoliation processes can separate platelets into their fundamental layers, thus reducing their thickness.



Excerpt from Sun, Fig. 5 (annotated)

112. I have labeled the XRD curves in Sun's Fig. 5 as best understood, based on my interpretation of the data in light of the description provided in [0068] of Sun. Curves a, c, and d in Fig. 5 all have peaks resulting from the repeat of the inter-sheet layer spacing of ZrP. This peak occurs at a scattering angle, 2θ, of about 5 degrees. Bragg's Law, $\lambda = 2d \sin\theta$, relates the wavelength, λ, of the X-rays to the layer spacing, d, and the scattering angle θ. *See* LGES-1045 (Cullity), 87. Note that the scattering angle used in Bragg's Law is half that recorded on the instrument and displayed in Sun Fig. 5. Although Sun does not specify the type of X-rays used, it is my opinion that a

POSITA would have understood the X-rays to be CuKα X-rays (λ = 0.154 nm), which are the only kind of X-rays available for this type of work in a laboratory setting not associated with a particle accelerator called a synchrotron. There are only a few synchrotron X-ray scatting facilities in the U.S.; they are associated with National Laboratories, and they are primarily used for academic and government research. It is a near certainty that Sun did their X-ray scattering in a laboratory using CuKα X-rays, and not at a synchrotron.

113. Braggs law can be rearranged to give the layer spacing: $d = \lambda/(2 \sin\theta)$. Based on the peak position in Sun's Fig 5, I calculate the layer spacing to be 1.77 nm. Given that this result was obtained by "eyeballing" the peak position off of a poor quality reproduction of the XRD data, I conclude that this approximate value is the same as the known 1.7 nm layer spacing of ZrP. *See* LGES-1031 (Kim), 1416-1417.

114. Curve b of Sun's Fig. 5 shows the disappearance of this peak when the carbon nanotubes and ZrP platelets are in an optimal ratio of 1:5, which allows them to complex with one another and exfoliate one another. In this case, the ZrP platelets are no longer stacked tightly with a layer spacing of 1.7 nm. Instead they are individualized and separated from one another by much larger distances. It is important to understand that the XRD peak does

not result from the layer thickness directly, but rather from the repetitive arrangement of multiple layers at a repeating distance equal to the layer thickness. When the layers are separated, that repetition disappears, and the peak at the scattering angle equivalent to that distance also disappears.

115. In the exfoliated state, the ZrP platelets still have their thickness of 1.7 nm; this is just no longer visible by XRD. XRD does potentially allow one to observe the average separation distance of the ZrP platelets in the exfoliated state, but any peak or other scattering feature representing that will occur at much lower scattering angles than displayed in Sun's Fig. 5. With specialized equipment, techniques and analysis, it is possible to observe scattering at these smaller scattering angles, but Sun has not done that.

116. This analysis demonstrates that the XRD results of Sun indicate that the ZrP platelets have at the very least a thickness dimension less than 20 μm, that dimension being as small as 1.7 nm (0.0017 μm) in the case where the ZnP filler is exfoliated. The in-plane platelet dimension, is also potentially less than 20 μm, *e.g.*, based on Sun's characterization of the diameter of the platelets, as I discussed above.

(13) Claim 19

[19] The dispersion of claim 18, wherein the filler is selected from silicon, lead, lead derivatives, carbon black, graphite, graphene, graphene oxides, paramagnetic particles and mixtures thereof.

117. As I discussed with respect to claim 17, Sun's nanoplatelets are **a filler** in both the aqueous dispersion and the nanocomposite. Sun describes the composition of the nanoplatelets: "The nanoplatelets comprise any suitable material … such as clay, nanoclay, ***graphite***, inorganic crystal, organic crystal, and combinations thereof." LGES-1004 (Sun), [0041]; *see* claims 4 and 15. That is, the filler in Sun's dispersions is **graphite**.

B. [GROUND 1B] – CLAIM 8 IS OBVIOUS OVER SUN AND BURETEA

118. It is my opinion that a POSITA would have found claim 8 to be obvious over the combination of Sun and Buretea, as I discuss in the following sections.

1. Buretea describes nanotube-resin composites including plasticizers

119. Buretea describes "a nanowire-material composite," such as "a freestanding nanowire material composite." LGES-1005 (Buretea), Abstract. The nanowires in Buretea's composite can be "any elongated conductive or semiconductive material" having dimensions in a specified range, and specifically can be "carbon nanotubes." *Id.*, 7:16-60. The nanowires (*e.g.*,

nanotubes) are embedded in a matrix material such as "polymers and resins," *e.g.*, "an elastomer, thermoplastic or thermosetting resin" such as epoxy resins. *Id.*, 15:28-62.

120. Buretea's composite "optionally comprises additives to modify the properties of the material." *Id.*, 15:64-66. The additive can be "a plasticizer." *Id.*, 15:66. Buretea defines a plasticizer as being "any material that can decrease the flexural modulus of a polymer," and which "may influence the morphology of the polymer and may affect the melting temperature and glass transition temperature." *Id.*, 15:67-16:20. Buretea identifies a number of types of plasticizers: "small organic and inorganic molecules, oligomers and small molecular weight polymers …, highly-branched polymers and dendrimers," and identifies many "[s]pecific examples." *Id.*, 16:4-20. Buretea also notes that the material can include other additives such as "fillers, antioxidants, colorants, initiators, crosslinking and curing agents, impact strength modifiers, heat and ultraviolet stabilizers, flame retardants, antistatic agents, electrical and thermal conductivity modifiers, drugs and biologically active compounds and molecules." *Id.*, 16:21-27.

2. The Sun-Buretea combination

121. The Sun-Buretea combination uses Sun's nanocomposite as a starting point. *See* LGES-1004 (Sun), [0045]-[0046]. In the combination, the nanocomposite also includes a plasticizer as an additive, as taught by Buretea. *See* LGES-1005 (Buretea), 15:64-66 ("The nanowire-material composite … comprises additives to modify the properties of the material 608. One example of an additive is a plasticizer."). The plasticizer is added into the uncured nanocomposite—*e.g.*, Sun's mixture of uncured matrix precursor and nanofiller—and remains through the curing process, and thus both the uncured and cured nanocomposites of the Sun-Buretea combination include the plasticizer. *See* LGES-1004 (Sun), [0045]-[0046].

122. As I mentioned in the previous section, Buretea describes numerous examples of plasticizers: "small organic and inorganic molecules, oligomers and small molecular weight polymers …, highly-branched polymers and dendrimers." *Id.*, 16:4-20. In the Sun-Buretea combination, one or more of these specific plasticizers can be used as the additive in the nanocomposite.

3. Reasons for the combination

123. It is my opinion that a POSITA would have been motivated to incorporate a plasticizer (as taught by Buretea) into Sun's nanocomposite for at least the three reasons I discuss in the following paragraphs.

124. The first reason why a POSITA would have been motivated to incorporate a plasticizer into the nanocomposite of the Sun-Buretea combination is in order to "influence the morphology" of the combination nanocomposite or to "decrease the flexural modulus" of the nanocomposite, which Buretea describes as capabilities provided by the inclusion of a plasticizer in a nanocomposite. LGES-1005 (Buretea), 16:1-2. The morphology of a material is the physical structure of the material, *e.g.*, structure on the scale of nanometers to micrometers. For instance, whether or not nanotubes in a nanotube-based nanocomposite are clumped together or exfoliated is a morphological question. The flexural modulus is a mechanical property. In some cases, although not always, changing the morphology of a material affects the mechanical properties, such as the flexural modulus, of the material. It is my opinion that a POSITA would have recognized that the ability to tailor the morphology or the flexural modulus of the nanocomposite has advantages, *e.g.*, in making the nanocomposites of the Sun-Buretea combination able to be used in a broad range of applications. For instance, with the ability to tailor morphology or flexural modulus of the nanocomposite, the nanocomposites would be able to be used in applications having a range of requirements for the morphology and mechanical properties of the nanocomposites. For instance, if the POSITA were

interested in using the nanocomposite in a component of a battery, such as a separator or electrode, that was to be wound into a cylindrical "jellyroll" battery cell, the flexural modulus of the composite would need to be adjusted to allow bending into this cylindrical shape.

125. The second reason why a POSITA would have been motivated to incorporate a plasticizer into the nanocomposite of the Sun-Buretea combination is to "affect the melting temperature and glass transition temperature" of the nanocomposite. LGES-1004 (Sun), 16:1:4. It is my opinion that a POSITA would have recognized that the ability to control the melting temperature and glass transition temperature of the nanocomposites would, again, enable the nanocomposites of the Sun-Buretea combination to be used in a variety of applications having a range of temperature requirements. For instance, a polymer material or a nanocomposite containing a polymer material may be stiff and rigid at room temperature, *e.g.*, in a rigid-glassy state, if its glass transition temperature is higher than room temperature. Addition of a plasticizer to that material can lower the glass transition temperature. If the glass transition temperature is lowered to below room temperature, the material is rendered soft and supple at room temperature, thus opening up a new range of applications for the material. Again, if the nanocomposite was to be used as a component of a battery cell

wound into a cylindrical geometry, it would be advantageous to plasticize the composite to render it supple enough to undergo winding.

126. Artificial leather, known as "vinyl," is a classic example of a material to which a plasticizer is added to tailor the glass transition temperature of the material. Vinyl is short for the polymer polyvinylchloride, which has a glass transition temperature of about 80°C (176°F). In its un-plasticized state (*i.e.*, without addition of a plasticizer), it is rigid enough at room temperature to serve as the material out of which "vinyl" records are made. However, the addition of a plasticizer decreases the "flexural modulus" of the "vinyl" and renders it suitable as an artificial leather substitute for clothing and upholstery. Thus addition of plasticizer reduces the flexural modulus and opens up a new market for the base material. A POSITA would have been well aware of this example from popular and common experience and would be motivated to try and replicate the success of "vinyl" by adding a plasticizer to his nanocomposite.

127. <u>The third reason</u> why a POSITA would have been motivated to incorporate a plasticizer into nanocomposite of the Sun-Buretea combination is because this combination is just the implementation of known techniques (specifically, incorporation of a plasticizer as an additive in a nanotube-based polymer matrix nanocomposite) to an already-known structure

(specifically, Sun's nanocomposite), and it produces predictable results (specifically, an polymer matrix nanocomposite containing carbon nanotubes and including plasticizer).

128. It is my opinion that a POSITA would have expected success when looking to incorporate a plasticizer (as taught by Buretea) into Sun's nanocomposite for at least the three reasons I discuss in the following paragraphs.

129. The first reason why a POSITA would have expected success is that Buretea's materials system is similar to the materials system of Sun's nanocomposite. For instance, both Buretea and Sun describe materials systems that include carbon nanotubes in a polymer matrix. *See* LGES-1004 (Sun), [0044]-[0046] ("The nanofiller," which includes nanotubes and nanoplatelets, "and epoxy mixture comprises a nanocomposite"); LGES-1005 (Buretea), 15:28-62 (describing a "nanowire-material composite"), 7:16-60. Indeed, in a specific example, both Buretea and Sun describe a nanocomposite that includes a thermoset polymer matrix such as an epoxy. *See* LGES-1004 (Sun), [0044]-[0046]; LGES-1005 (Buretea), 15:28-62 ("suitable polymers for material 608 include, but are not limited to an elastomer, thermoplastic or thermosetting resin" including "prepolymers such as phenoxy and epoxy prepolymers"). It is my opinion that this overlap in materials systems is an indication that a POSITA would have expected to

be successful in introducing a plasticizer, as taught by Buretea, into Sun's nanocomposite. That is, Buretea demonstrates that plasticizers can be used in nanocomposites having compositions similar to those described by Sun, and this would have led a POSITA to expect success in making this combination.

130. The second reason why a POSITA would have expected success, is that the nanotubes in Buretea's and Sun's nanocomposites are also similar. Buretea's nanocomposite contains nanowires, which can be "carbon nanotubes," and that have "at least one cross sectional dimension that is less than 500 nm, and preferably, less than 100 nm" and "an aspect ratio (length:width)" of, for instance, "greater than 10" or "greater than 50." LGES-1005 (Buretea), 7:17-28. Sun's nanocomposite also contains carbon nanotubes, and Sun's carbon nanotubes have similar dimensions to Buretea's nanotubes: a diameter of between 1 nm and 20 nm, and an aspect ratio of, for instance, "at least about 10" or "at least about 20." LGES-1004 (Sun), [0037]-[0038]. It is my opinion that these similarities are an indication that a POSITA would have expected to be successful in introducing a plasticizer, as taught by Buretea, into Sun's nanocomposite. That is, Buretea demonstrates that plasticizers can be used in nanocomposites containing nanotubes that have dimensions (*e.g.*, diameter

and aspect ratio) similar to the dimensions of Sun's nanotubes, and this would have led a POSITA to expect success in making this combination.

131. The third reason why a POSITA would have expected success is that the use of plasticizers as additives in resin composites, and specifically nanotube-containing resin composites, was well-known. *See* LGES-1005 (Buretea), 15:66-16:20 ("One example of an additive is a plasticizer"); LGES-1010 (Patel), [0039] ("the multifunctional nanomaterial-containing composites, may include additives such as a plasticizer"); LGES-1022 (Tour), [0041] ("other additives are added to the mixture to refine or enhance the composite/blend properties, or to impart them with new ones. Such other additives can include … plasticizers"). A POSITA pursuing a formulation that is well-known and used in the art would expect to be successful in doing so.

4. Claim-by-Claim Analysis

(1) Claim 8

[8] The dispersion of claim 1, wherein the additive is selected from the group consisting of dicarboxylic/tricarboxylic esters, timellitates, adipates, sebacates,

maleates, glycols and polyethers, polymeric plasticizers, bio-based plasticizers, and mixtures thereof.

The Sun-Buretea combination nanocomposite includes a polymeric plasticizer as an additive

132. As I discussed above, in the Sun-Buretea combination, Sun's nanocomposite is modified to include a plasticizer based on the teachings of Buretea. *See* LGES-1005 (Buretea), 15:64-66 ("The nanowire-material composite … comprises additives to modify the properties of the material 608. One example of an additive is ***a plasticizer***"). This plasticizer can be considered **an additive**. Additionally, it is my opinion that a POSITA would have understood that this additive—that is, Buretea's "plasticizer"—is **a polymeric plasticizer**, at least because it is an additive to a polymer matrix (that is, to the polymer matrix material, such as epoxy resin, of Sun's nanocomposite). *See* LGES-1004 (Sun), [0045]-[0046]). Notably, the use of Buretea's plasticizer as an additive to a polymer matrix is also consistent with its use in Buretea's composite. *See* LGES-1005 (Buretea), 15:67-16:1 ("Plasticizer is used herein to mean any material that can decrease the flexural modulus of a polymer"), 15:20-66 ("The nanowire-material composite … comprises additives to modify the properties of the material 608," where the material 608 can be "polymers and resins").

133. Alternatively or additionally, Buretea's "plasticizer" additive in the Sun-Buretea combination nanocomposite is **a polymeric plasticizer** because the plasticizer itself is a polymer. Buretea identifies polymers, such as "oligomers and small molecular weight ***polymers*** …, highly-branched ***polymers*** and ***dendrimers***," as "[e]xamples of plasticizers." LGES-1005 (Buretea), 16:4-20. When any of these materials are provided as the plasticizer additive to the nanocomposite in the Sun-Buretea combination, the combination nanocomposite thus includes **a polymeric plasticizer** in that it includes a plasticizer that is a polymer.

The Sun-Buretea combination nanocomposite includes glycols or polyethers as additives

134. Buretea identifies "alcohols such as glycerol, ***ethylene glycol, diethylene glycol, triethylene glycol***, [and] oligomers of ***ethylene glycol***" and "ethers such as oligomers of ***polyethylene glycol***, including PEG-500, PEG 1000 and PEG-2000" as "[e]xamples of plasticizers." LGES-1005 (Buretea), 16:4-20. At least some of these example plasticizers are **glycols**: ethylene glycol, diethylene glycol, triethylene glycol, oligomers of ethylene glycol, and oligomers of polyethylene glycol. Additionally, polyethylene glycol (also known as polyethylene oxide (PEO)) is a **polyether**. Specifically, PEG-500, PEG-1000, and PEG-2000 are polyethers.

135. In the Sun-Buretea combination, Sun's nanocomposite is modified to include a plasticizer based on the teachings of Buretea, as I have previously discussed. When any of these glycol plasticizers identified by Buretea are provided as the plasticizer additive to the nanocomposite in the Sun-Buretea combination, the combination nanocomposite thus includes **glycol** as **an additive**. Any of these glycol plasticizers that have sufficient number of repeating units to be an oligomer or a polymer is a polyethylene glycol, also known as polyethylene oxide (a polyether). Thus, when one of these glycol plasticizers (*e.g.*, PEG-500, PEG-1000, or PEG-2000) is used as the plasticizer additive in the Sun-Buretea combination, the combination nanocomposite includes a **polyether** as **an additive**.

C. [GROUND 1C] – CLAIMS 9-10, 15, AND 21-23 ARE OBVIOUS OVER SUN AND KAJI

136. It is my opinion that a POSITA would have found claims 9-10, 15, and 21-23 to be obvious over the combination of Sun and Kaji, as I discuss in the following sections.

1. Kaji describes nanotube-resin composites including carnauba wax and silicone oil additives

137. Kaji describes a "crystalline resin cured product which shows high thermal conductivity, low thermal expansion, high heat resistance, low moisture absorption, and good gas barrier properties." LGES-1006 (Kaji), [0001].

Kaji's resin product is a "crystalline resin composite material produced by combining a resin with a filler or base material." *Id.*, [0024]. Kaji provides numerous examples of the "filler or base material," including "carbonaceous materials" such as "carbon fibers, graphitized carbon fibers, natural graphite, artificial graphite, spherical graphite particles, mesocarbon microbeads, carbon whiskers, carbon microcoils, carbon nanocoils, carbon nanotubes, and carbon nanohones." *Id.*, [0068].

138. The resin matrix of Kaji's composite material is a "crystalline resin cured product." *Id.*, [0024]. Kaji notes that although "[t]he crystalline resin cured product … is not necessarily required to have a crosslinked structure," a "three-dimensional structure," *e.g.*, a crosslinked structure, "is desirable from the viewpoint of improving heat stability and mechanical properties." *Id.*, [0059]. Kaji also provides a specific example of "[a] procedure useful for forming a three-dimensional structure," in which "an aromatic dihydroxy compound together with an epoxy resin curing agent" is reacted "with an aromatic diglycidyl compound." *Id.*, [0059]. Kaji identifies suitable epoxy resin curing agents: dicyandiamide, imidazoles, amines, acid anhydrides, phenols, polymercaptains, polyaminoamides, isocyanates, and blocked isocyanates." *Id.*, [0059]. Kaji also explains that because "[t]hese curing agents contain three or more functional groups or have an ability to expedite

the reaction of the glycidyl group with the hydroxyl group of a secondary alcohol formed by the reaction of a glycidyl group with a hydroxyl group and they contribute to the formation of a three-dimensional structure." *Id.*, [0059]. In other words, Kaji's curing agents have three or more reactive sites on each molecule, which enables formation of chemical crosslinks in the cured resin.

139. Kaji's resin composite material can include a variety of additives, including "flame retardants such as brominated epoxy compounds and phosphorus-containing epoxy compounds, mold release agents such as carnauba wax and ester wax, coupling agents such as epoxysilanes, aminosilanes, ureidosilanes, vinylsilanes, alkylsilanes, organic titanates, and aluminum alcoholates, colorants such as carbon black, auxiliary flame retardants such as antimony trioxide, stress-lowering agents such as silicone oils, and lubricants such as higher fatty acids and metal salts of higher fatty acids." LGES-1006 (Kaji), [0067].

2. The Sun-Kaji combination

140. The Sun-Kaji combination uses Sun's nanocomposite as a starting point. *See* LGES-1004 (Sun), [0045]-[0046]. In the combination, the nanocomposite also includes silicone oil and/or a wax such as carnauba wax as an additive, as taught by Kaji. *See* LGES-1006 (Kaji), [0067] ("If

necessary, it is allowable to use … mold release agents such as carnauba wax and ester wax, … stress-lowering agents such as silicone oils"). The silicone oil and/or wax additives are added into the uncured nanocomposite—*e.g.*, Sun's mixture of polymer matrix material and nanofiller—and remain through the curing process, and thus both the uncured and cured nanocomposites of the Sun-Kaji combination include silicone oil and/or wax. *See* LGES-1004 (Sun), [0045]-[0046].

3. Reasons for the combination

Reasons for incorporation of wax, such as carnauba wax

141. It is my opinion that a POSITA would have been motivated to incorporate a wax, such as carnauba wax (as taught by Kaji), into Sun's nanocomposite for at least the three reasons I discuss in the following paragraphs.

142. The first reason why a POSITA would have been motivated to incorporate wax, such as carnauba wax, into the nanocomposite of the Sun-Kaji combination is because doing so would provide the nanocomposite of the combination with the "mold release" capabilities of carnauba wax. LGES-1006 (Kaji), [0067]. Mold release refers to the ability of the cured nanocomposite to be released easily from its mold, *e.g.*, without sticking, stretching, tearing, or other damage to the nanocomposite. *See* LGES-1025 (Ahsan), 5:46-52 ("Mold release agents are chemical agents commonly used

to assist in the release of the cured epoxy molding compounds from the curing mold"). It is my opinion that a POSITA would have recognized that a nanocomposite that releases easily from its mold is advantageous for processing, *e.g.*, in that processing time and complexity can be reduced when little effort is required to remove the nanocomposite from its mold. Additionally, it is my opinion that a POSITA would have recognized that a nanocomposite that releases easily from its mold is advantageous for structural integrity, *e.g.*, in that the nanocomposite is less likely to be damaged upon release.

143. The second reason why a POSITA would have been motivated to incorporate wax, such as carnauba wax, into the nanocomposite of the Sun-Kaji combination is to obtain the desirable anti-moisture properties that are provided by waxes, including carnauba wax. For instance, Rasmussen describes carnauba wax as "a plant-based antimoisture agent" which "inhibits moisture absorption by the composite." LGES-1011 (Rasmussen), [0050]-[0051]. It is my opinion that a POSITA would have recognized that including carnauba wax in the nanocomposite advantageously would provide the nanocomposite with improved resistance to the negative effects of moisture, which can include structural decay, degradation of mechanical

or electrical properties of the composite, growth of mold or mildew, and development of odors.

144. The third reason why a POSITA would have been motivated to incorporate wax, such as carnauba wax, into the nanocomposite of the Sun-Kaji combination is because this combination is just the implementation of known techniques (specifically, incorporation of carnauba wax as an additive in a nanotube-polymer matrix nanocomposite) to an already-known structure (specifically, Sun's nanocomposite), and it produces predictable results (specifically, an polymer matrix nanocomposite containing carbon nanotubes and including carnauba wax).

Reasons for incorporation of silicone oil

145. It is my opinion that a POSITA would have been motivated to incorporate silicone oil (as taught by Kaji) into Sun's nanocomposite for at least the three reasons I discuss in the following paragraphs.

146. The first reason why a POSITA would have been motivated to incorporate silicone oil into the nanocomposite of the Sun-Kaji combination is because doing so would provide the nanocomposite of the combination with the "stress-lowering" properties provided by silicone oil. LGES-1006 (Kaji), [0067]; *see* LGES-1026 (Akagi), 41:18-21 ("a stress-relaxing agent such as silicone oil"); LGES-1027 (Ikezawa), [0161] ("stress relaxation agent such

as silicone oil"). For instance, it is my opinion that a POSITA would have recognized that a nanocomposite containing silicone oil would advantageously have lower residual stress than a nanocomposite that does not contain silicone oil, and also would have recognized that this nanocomposite with lower residual stress would be more mechanically robust, for instance, resistant against applied strain or impact.

147. The second reason why a POSITA would have been motivated to incorporate silicone oil into the nanocomposite of the Sun-Kaji combination is because silicone oils are commonly used as mold release agents, and including silicone oil in the nanocomposite would provide the nanocomposite of the combination with the mold release capabilities of silicone oils. *See* LGES-1042 (Canter), 32.

148. In some manufacturing processes, a mold release agent is applied to the surface of the mold. Indeed, this is the approach followed by Sun. *See* LGES-1004 (Sun), [0046] ("The mixture is poured into a mold with mold release agent on the surfaces"). An alternative to this approach is to add the mold release agent (*e.g.*, silicone oil) directly into the resin of the composite, and the composite is then poured into the mold for curing. In this approach, the silicone oil is referred to as an internal release agent. During the curing process, a small amount of the silicone oil additive will coat the interface

between the resin composite and the mold surface, preventing the resin composite from sticking to the mold and thus acting as a mold release agent. This approach to mold release has advantages over approaches in which a mold release agent is applied to the mold after every process cycle. For instance, use of an internal release agent reduces process complexity and cycle time as compared to approaches in which a mold release agent is applied directly to the mold between process cycles. *See* LGES-1043 (Plevyak), 364.

149. A POSITA implementing the process described in Sun would apply a mold release agent onto the mold in which the nanocomposites are cured, following the teachings of Sun. LGES-1004 (Sun), [0046]. It is my opinion that POSITA, recognizing the advantages of using an internal mold release agent, would have been motivated to add silicone oil directly into the uncured nanocomposite to achieve these advantages, *e.g.*, to reduce process complexity and cycle time.

150. A POSITA would have recognized these advantageous effects of including silicone oil in the nanocomposite and been motivated to incorporate silicone oil into the resin of the Sun-Kaji composite to obtain these advantages.

151. <u>The third reason</u> why a POSITA would have been motivated to incorporate silicone oil into the nanocomposite of the Sun-Kaji combination is because

this combination is just the implementation of known techniques (specifically silicone oil as an internal mold release additive in a nanotube-polymer matrix nanocomposite) to an already-known structure (specifically, Sun's nanocomposite), and it produces predictable results (specifically, an polymer matrix nanocomposite containing carbon nanotubes and silicone oil, that does not stick to its mold).

Expectation of success in incorporating silicone oil or wax into the nanocomposite of the combination

152. It is my opinion that a POSITA would have expected success when looking to incorporate silicone oil and/or wax (*e.g.*, carnauba wax), as taught by Kaji, into Sun's nanocomposite for at least the three reasons I discuss in the following paragraphs.

153. The first reason why a POSITA would have expected success is that Kaji's materials system is similar to the materials system of Sun's nanocomposite. For instance, both Kaji and Sun describe materials systems that include carbon nanotubes in a polymer matrix. *See* LGES-1004 (Sun), [0044]-[0046], [0068]; LGES-1006 (Kaji), [0024] ("a crystalline resin composite material produced by combining a resin with a filler or base material"), [0059], [0068]. It is my opinion that this overlap in materials systems is an indication that a POSITA would have expected to be successful in introducing silicone oil and/or wax such as carnauba wax, as taught by Kaji,

into Sun's nanocomposite. That is, Kaji demonstrates that these additives can be used in nanocomposites similar to those described by Sun, and this would have led a POSITA to expect success in making this combination.

154. The second reason why a POSITA would have expected success is that Kaji and Sun describe nanocomposites containing similar amounts of carbon nanotubes. In Kaji's nanocomposite, the weight percentage of filler (*e.g.*, carbon nanotubes) is "10 to 90 wt% of the total weight." LGES-1006 (Kaji), [0024]. This is consistent with the weight percentage of carbon nanotubes in Sun's nanocomposite: "between about 0.01 wt% and about 50 wt%." LGES-1004 (Sun), [0045]. It is my opinion that these similarities are an indication that a POSITA would have expected to be successful in introducing silicone oil and/or wax such as carnauba wax, as taught by Kaji, into Sun's nanocomposite. That is, Kaji demonstrates that silicone oil and wax can be used in nanocomposites containing an amount of nanotubes that is similar to the amount of nanotubes contained in Sun's nanocomposite, and this would have led a POSITA to expect success in making this combination.

155. The third reason why a POSITA would have expected success is that the use of silicone oil and wax as additives in resin composites, and specifically nanotube-containing resin composites, was well-known. *See* LGES-1006 (Kaji), [0067]; LGES-1012 (Molitor), [0091] ("Other ingredients can be …

melt mixed with a formed nanocomposite composition … Examples of such materials include … waxes … silicone oil").

4. Claim-by Claim Analysis

(1) Claim 9

[9] The dispersion of claim 1, wherein the additives [sic] is a process oil.

156. As I discussed above, in the Sun-Kaji combination, Sun's nanocomposite is modified to include "stress-lowering agents such as ***silicone oils***" as an additive, based on the teachings of Kaji. LGES-1006 (Kaji), [0067]. Silicone oil is **a process oil**. Indeed, I note that the '649 Patent itself identifies silicone as an example of a process oil. *See* LGES-1001, 7:41-46, claim 10. Thus, when the Sun-Kaji nanocomposite includes silicone oil, the additive in the nanocomposite is a process oil.

(2) Claim 10

[10] The dispersion of claim 9, where in the process oil is selected from the group consisting of naphthenic oils, paraffin oils, paraben oils, aromatic oils, vegetable oils, seed oils, silicones, and mixtures thereof.

157. As I discussed above for claim 9, in the Sun-Kaji combination, the nanocomposite includes silicone oil, which is a process oil. *See* LGES-1006 (Kaji), [0067] ("stress-lowering agents such as ***silicone oils***"). LGES-1006 (Kaji), [0067]. That is, in the nanocomposite of the combination, the process oil is a silicone oil, namely, a **silicone**.

(3) Claim 15

[15] The dispersion of claim 1, wherein the additive comprises natural wax, synthetic wax, or mixtures thereof.

158. As I discussed above, in the Sun-Kaji combination, Sun's nanocomposite is modified to include "mold release agents such as ***carnauba wax and ester wax***" as an additive, based on the teachings of Kaji. LGES-1006 (Kaji), [0067]. Carnauba wax is **a natural wax**. *See* LGES-1011 (Rasmussen), [0051] (identifying "carnauba wax" as "a ***plant-based*** antimoisture agent"); LGES-1028 (Aga), [0107] ("natural waxes such as carnauba wax"). Thus, at least when the Sun-Kaji nanocomposite includes carnauba wax, the additive in the nanocomposite is a natural wax.

(4) Claim 21

[21] The dispersion of claim 15 which further comprises a crosslinking agent.

159. Sun describes that the nanocomposite includes a curing agent, which is a crosslinking agent. Specifically, to form Sun's nanocomposite, the nanofiller (which includes nanotubes and nanoplatelets) is "re-suspended in acetone prior to mixing in an epoxy," such as "an epoxy monomer." LGES-1004 (Sun), [0045]. Then, "the solvent [is] removed from the epoxy nanocomposite" and "***a curing agent*** is added to the epoxy and nanotubes, and heated to polymerize, crosslink, vulcanize, or otherwise cure … the epoxy nanocomposite." LGES-1004 (Sun), [0046]; *see* [0058]; *see also*

[0052]. Sun's curing agent is **a crosslinking agent** at least because it enables crosslinking in the epoxy to thus form a crosslinked epoxy polymer matrix.

(5) Claim 22

[22] The dispersion of claim 15 wherein the additive comprises natural wax wherein the natural wax is plant derived.

160. As I discussed above, in the Sun-Kaji combination, Sun's nanocomposite is modified to include "mold release agents such as ***carnauba wax***" as an additive, based on the teachings of Kaji. LGES-1006 (Kaji), [0067]. Carnauba wax is **a natural wax** that is **plant derived**. *See* LGES-1011 (Rasmussen), [0051] (identifying "carnauba wax" as "a ***plant-based*** antimoisture agent"); LGES-1028 (Aga), [0107] ("natural waxes such as carnauba wax"). Thus, at least when the Sun-Kaji nanocomposite includes carnauba wax, the additive in the nanocomposite is a natural wax that is plant derived.

(6) Claim 23

[23] The dispersion of claim 22 which further comprises a crosslinking agent.

161. I refer to my discussion of claim 21.

D. [GROUND 1D] – CLAIMS 16 AND 20 ARE OBVIOUS OVER SUN, KAJI, AND MATSUSHITA

162. It is my opinion that a POSITA would have found claims 16 and 20 to be obvious over the combination of Sun, Kaji, and Matsushita, as I discuss in the following sections.

1. Matsushita describes nanocomposites including fluororubber

163. Matsushita describes a "resin composite material" that is "obtained by molding a resin composition comprising the fibrous carbon-based material insulator … and a resin." LGES-1013 (Matsushita), [0072]. The "fibrous carbon-based material" can be, *e.g.*, "carbon nanotubes," such as "multi-walled carbon nanotubes." *Id.*, [0009], [0021]. Matsushita describes a variety of compositions for the resin (specifically, stating that "the resin is not particularly limited"), and identifies "thermosetting resins such as epoxy resin" as suitable examples. *Id.*, [0073]. Matsushita also describes that "[c]rosslinked rubbers such as … fluororubber… and composites of these and resins can also be used." *Id.*, [0073].

2. The Sun-Kaji-Matsushita combination provides a nanocomposite including a fluororubber

164. The Sun-Kaji-Matsushita combination uses the Sun-Kaji nanocomposite I have previously discussed as a starting point. *See* LGES-1004 (Sun), [0045]-[0046]. In the combination, the nanocomposite also includes a

fluororubber, as taught by Matsushita. *See* LGES-1013 (Matsushita), [0073]. Specifically, in one example implementation, a fluororubber is combined with the resin matrix of the Sun-Kaji nanocomposite based on the teachings of Matsushita, resulting in the matrix including "fluororubber," and specifically, being a "composite[] of these [*i.e.*, fluororubber]" with the resin matrix of the combination nanocomposite. LGES-1013 (Matsushita), [0073].

3. Reasons for the combination

165. It is my opinion that a POSITA would have been motivated to incorporate a fluororubber (as taught by Matsushita) into the Sun-Kaji nanocomposite for at least the two reasons I discuss in the following paragraphs.

166. <u>The first reason</u> why a POSITA would have been motivated to incorporate a fluororubber into the nanocomposite of the combination is so that the resulting nanocomposite would have good chemical and corrosion resistance, which are properties provided by fluoroelastomers (*e.g.*, fluororubbers). These properties are mentioned by Close (*see* LGES-1049, 4:54-57 ("An important aspect of the present invention is that the inherent resistance of the fluorocarbon elastomers to many fuels, hydrocarbons and solvents is not sacrificed"); Sumner (*see* LGES-1029 (Sumner), 5:39-41 ("corrosion resistant material such as a fluoroelastomer")), and Uchida (*see*

LGES-1030 (Uchida), 14:64-15:2 ("synthetic rubbers having good acid resistance or alkali resistance such as fluororubber")). For instance, a nanocomposite that has an polymer resin-fluororubber composite matrix would be useful for applications that require chemical- and corrosion-resistant materials, for instance, gaskets or seals. *See* LGES-1029 (Sumner), 2:4-9 (describing "a heat shrinkable chemically resistant moisture barrier of a fluorocarbon resin for sealing the connection between a metallic anode and the insulated conductor and for sealing the connection between two mechanically joined anode sections"); LGES-1030 (Uchida), 4:9-13 ("In order to further render the coupling assembly corrosion-resistant, it is preferred that the valve members are made of fluoroplastics having good chemical resistance"). It is my opinion that a POSITA would have recognized the utility of such a nanocomposite, and for that reason would have been motivated to use fluororubber in the nanocomposite of the combination.

167. For instance, a matrix including a fluoroelastomer and a polymer resin achieves the chemical resistance advantages of the fluoroelastomer while obtaining, through the presence of the polymer resin, an "improved adhesion" than what is generally possible with fluoroelastomers alone. LGES-1049, 4:54-66; *see* 6:63-65 ("the epoxy resin does significantly

improve adhesion in fluoroelastomer compositions containing metallic oxides and fillers").

168. The second reason why a POSITA would have been prompted to incorporate a fluororubber into the nanocomposite of the combination is because this combination is just the implementation of known techniques (specifically, incorporation of fluororubber into a resin matrix nanocomposite) to an already-known structure (specifically, the Sun-Kaji nanocomposite), and it produces predictable results (specifically, an polymer resin-based nanocomposite containing carbon nanotubes, and including fluororubber as part of the matrix).

169. It is my opinion that a POSITA would have expected success when looking to incorporate a fluororubber (as taught by Matsushita) into the Sun-Kaji nanocomposite for at least the four reasons I discuss in the following paragraphs.

170. The first reason why a POSITA would have expected success is that Matsushita's materials system is similar to the materials systems of Sun and Kaji. For instance, Matsushita's nanocomposite includes carbon nanotubes in a polymer matrix. *See* LGES-1013 (Matsushita), [0009], [0021], [0072]-[0073]. This composition is consistent with the composition of the Sun-Kaji nanocomposite, which also includes carbon nanotubes in a polymer matrix.

See LGES-1004 (Sun), [0044]-[0046], [0068]; LGES-1006 (Kaji), [0024], [0059], [0068]. It is my opinion that this overlap in materials systems is an indication that a POSITA would have expected to be successful in introducing a fluororubber, as taught by Matsushita, into the Sun-Kaji nanocomposite. That is, Matsushita demonstrates that fluororubber can be used in nanocomposites having compositions similar to those of the Sun-Kaji combination, and this would have led a POSITA to expect success in making this combination.

171. The second reason why a POSITA would have expected success is that Matsushita's nanotubes are similar to the nanotubes in the Sun-Kaji nanocomposite. The carbon nanotubes in Matsushita's nanocomposite have a diameter of "500 nm or less," "250 nm or less," or "150 nm or less," and an aspect ratio of, *e.g.*, "5 or more," "10 or more," or "20 or more." LGES-1013 (Matsushita), [0022]-[0023]. The carbon nanotubes in the Sun-Kaji nanocomposite fall within this size range: they have "a diameter of between about 1 nm and about 20 nm," and "an aspect ratio" of, *e.g.*, "at least about 10" or "at least about 20." LGES-1004 (Sun), [0037]-[0038]. It is my opinion that these similarities are an indication that a POSITA would have expected to be successful in introducing a fluororubber, as taught by Matsushita, into the Sun-Kaji nanocomposite. That is, Matsushita

demonstrates that fluororubbers can be used in nanocomposites containing nanotubes that have dimensions (*e.g.*, diameter and aspect ratio) similar to the dimensions of the nanotubes of the Sun-Kaji nanocomposite, and this would have led a POSITA to expect success in making this combination

172. The third reason why a POSITA would have expected success is that Matsushita describes that its resin nanocomposite including fluororubber can be used with additives including "mold release agents." LGES-1013 (Matsushita), [0074]. A mold release agent—carnauba wax—is already present in the Sun-Kaji nanocomposite, as I discussed above. *See* LGES-1006 (Kaji), [0067]; *see also* my discussion of claim 15. It is my opinion that a POSITA would have expected success in introducing a fluororubber into the Sun-Kaji nanocomposite in light of Matsushita's teaching that fluororubbers can be used in conjunction with mold release agents in nanocomposites.

173. The fourth reason why a POSITA would have expected success is that resin composites in which the resin includes fluororubber were well-known. See LGES-1013 (Matsushita), [0073] ("Crosslinked rubbers such as silicone rubber, ethylene-propylene-diene rubber, fluororubber, acrylonitrile rubber, and NBR, and composites of these and resins can also be used"); LGES-1049 (Close), 6:63-65 (describing materials containing "epoxy resin" with

"fluoroelastomer compositions containing metallic oxides and fillers"), 7:56-60 (identifying "fillers, such as carbon black" as example fillers), 4:41-44; LGES-1022 (Tour), [0035] ("Suitable elastomeric precursors (systems) include …tetrafluoroethylene based, copolymers of hexafluoropropylene and vinylidene fluoride,"), claim 16; LGES-1048 (Mijovic), 305-306.

4. Claim-by-Claim Analysis

(1) Claim 16

[16] The dispersion of claim 15 further comprising a fluoroelastomer.

174. As I discussed above, in the Sun-Kaji-Matsuhita combination, the Sun-Kaji nanocomposite is modified to include "[c]rosslinked rubbers such as … fluororubber," based on the teachings of Matsushita. LGES-1013 (Matsushita), [0073]. For instance, fluororubber is incorporated into the matrix of the nanocomposite.

175. Rubber is an elastomer. *See* LGES-1047 (Hiemenz), 381 ("An *elastomer*… [t]he quintessential everyday example is a rubber band."). A fluororubber is a rubber that is fluorinated. Thus, fluororubber is **a fluoroelastomer**. That is, the nanocomposite of the Sun-Kaji-Matsushita combination, which contains a fluororubber, includes **a fluoroelastomer**.

(2) Claim 20

[20pre] A dispersion comprising

[20a] a plurality of oxidized, discrete carbon nanotubes and

176. I refer to my discussion of claim elements [1pre]-[1a].

[20b] at least one wax additive and

177. I refer to my discussion of claim 15.

[20c] at least one fluoroelastomer,

178. I refer to my discussion of claim 16.

[20d] wherein the discrete carbon nanotubes have an aspect ratio of 25 to 500, are multiwall, and are present in the range of greater than zero to about 30% by weight based on the total weight of the dispersion.

179. I refer to my discussion of claim elements [1c]-[1e].

E. [GROUND 2A] – CLAIMS 1-8, 11-15, 17-19, AND 21 ARE OBVIOUS OVER FARRUGIA AND NARAYAN

180. It is my opinion that a POSITA would have found claims 1-8, 11-15, 17-19, and 21 to be obvious over the combination of Farrugia and Narayan, as I discuss in the following sections.

1. Farrugia describes toners including electrically conductive nanotube/polymer composites

181. Farrugia describes "a toner comprising a resin comprising a polymerized mixture," where "the polymerized mixture is a composite comprising carbon nanotubes and a polymer." LGES-1007 (Farrugia), [0019]. Farrugia explains that these toners "exhibit enhanced mechanical, chemical, thermal

and electrical properties." *Id.*, [0018]. For instance, Farrugia notes that "when the carbon nanotube/polymer composite comprises carbon nanotubes from about 2% to about 20% by weight of the composite, the toner exhibits increased conductivity, and thus is suitable for conductive developing methods." *Id.* Farrugia attributes this enhanced electrical conductivity to the aspect ratio of nanotubes: "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." *Id.*

182. Farrugia describes a process of making the nanotube/polymer composite that is incorporated into the toner. Specifically, Farrugia's nanotubes are "functionalized" by any of a variety of processes. LGES-1007 (Farrugia), [0028]; *see* [0020]. One approach to functionalization involves "oxidation … 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." *Id.* The resulting functionalized nanotubes have the general formula $[C_nH_L -]R_m$, where R encompasses a variety of functional groups. *See id.*, [0029]-[0037]. Farrugia also notes that in certain examples, "the carbon nanotubes are functionalized with carboxylic acid moieties," and that

"[c]arboxylic acid-functionalized carbon nanotubes are particularly useful because they can serve as the starting point for preparing other types of functionalized carbon nanotubes." *Id.*, [0039].

183. Farrugia's functionalized carbon nanotubes are "combined with a molar excess of a first monomer," and the nanotubes are "reacted with the first monomer to covalently attach the functionality moieties on the carbon nanotubes to the first monomer." LGES-1007 (Farrugia), [0040]-[0042]. That is, the first monomer, because it is present in "molar excess," reacts with substantially all of the available functional groups on the nanotubes (*e.g.*, the functional groups that are introduced in the prior functionalization step). The end result of this step is carbon nanotubes with the first monomer covalently bonded to their sidewalls. The first monomer is present in excess during this reaction step, which means that excess, unreacted first monomer remains even after the nanotubes are functionalized. *See id.*, [0045].

184. The nanotubes (now having the first monomer covalently attached) and the excess first monomer are mixed with a second monomer, and this mixture is then polymerized to form the composite: "The functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer, *e.g.*, through the formation of ester or amide bonds, to form a polymer matrix in which the

carbon nanotubes are dispersed." LGES-1007 (Farrugia), [0045]; *see* [0020]. During this polymerization reaction, the first monomers (including the first monomer attached to the nanotubes and the excess first monomer) and the second monomers react to form a polymer, thus covalently bonding the carbon nanotubes to the polymer matrix. Specific examples of this polymerization process are provided in Farrugia's Examples 2 and 3.

185. Farrugia provides various examples of "[t]he polymers formed from the polymerization of the second monomers"; examples include "polycarbonates, polyamides, polyesters and polyurethanes." LGES-1007 (Farrugia), [0048]; *see* [0049]-[0052]. To be clear, a POSITA would understand Farrugia to be teaching that the second monomer is polymerizing with the excess first monomer, which is also in the system, to produce the polymer resins listed. Due to the nature of the chemistry involved, the second monomers, by themselves, cannot produce these polymers. They can only do so in combination with the first monomers.

186. The carbon nanotube/polymer composite is "incorporated into a toner," which also may include any of a variety of additives to provide desired properties or characteristics to the toner. *Id.*, [0054]; *see* [0068]-[0084]. For instance, the process of making toner particles includes "aggregating an emulsion containing polymer binder and one or more optional waxes, one or

more optional colorants, one or more surfactants, an optional coagulant, and one or more additional optional additives to form aggregates," and further processing the aggregates into toner particles. *Id.*, [0055]. The "polymer binder" used in the aggregation is the "carbon nanotube/polymer composite resins" discussed in the preceding paragraphs. *Id.*, [0055]; *see* [0057] ("the toner process comprises forming a toner particle by mixing the carbon nanotube/polymer composite, an optional wax, an optional colorant dispersion, and an optional coagulant while blending at high speeds," and then further processing to obtain toner particles).

2. Narayan describes electrically conductive carbon nanotube/polymer compositions

187. Narayan describes "a polymeric foam and carbon nanotubes," and an "elastomeric composition compris[ing] an elastomer and carbon nanotubes." LGES-1008 (Narayan), Abstract. The Narayan's foams and elastomers are "electrically conductive … for electromagnetic shielding and electrostatic dissipation." *Id.*, [0002]. Narayan describes a wide variety of "polymer[s] for use in the polymeric electrically conductive polymer foams," including "thermoplastic resins, blends of thermoplastic resins, or thermosetting resins." *Id.*, [0009]; *see* [0010]-[0012] for a listing of specific compositions, including epoxies ([0011]).

188. The amount and characteristics of the carbon nanotubes in Narayan's polymeric foams are "selected so as to provide electrical conductivity" to the composite composition "while generally retaining the advantageous intrinsic physical properties of the polymeric foams or elastomers." *Id.*, [0008]. For instance, the carbon nanotubes have "diameters of about 0.5 to about 2000 nm, with aspect ratios greater than or equal to about 5 … 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." *Id.*, [0014]. These dimensional ranges are characteristics that Narayan describes as "suitable … for imparting electric conductivity to the polymeric foams and elastomers." *Id.*, [0014]. Additionally, Narayan notes that "[s]ingle and/or multiwall carbon nanotubes are used in amounts effective to provide the desired conductivity, generally in an amount of about 0.0001 to about 50 wt % of the total weight of the polymeric foam or elastomer composition." *Id.*, [0019].

3. The Farrugia-Narayan combination

189. The Farrugia-Narayan combination uses Farrugia's nanotube/polymer composite as the starting point. *See*, *e.g.*, LGES-1007 (Farrugia), [0019]. In the composite of the Farrugia-Narayan combination, the specific nanotubes used in the composite are dimensioned based on the teachings of Narayan.

Specifically, the nanotubes in the composite of the combination 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," as taught by Narayan. LGES-1008 (Narayan), [0014]. Notably, this is consistent with Farrugia's generic indication that the nanotubes of the composite have a "high aspect ratio." LGES-1007 (Farrugia), [0018].

190. Following the teachings of Farrugia, this combination composite is "incorporated into a toner," along with one or more additives such as those described by Farrugia. LGES-1007 (Farrugia), [0054]. Because the composite of the combination includes nanotubes that are dimensioned according to the teachings of Narayan, so, too, does the toner of the combination.

191. The claims of the '649 Patent relate to dispersions. It is my opinion that a POSITA would have found the claimed dispersions of the '649 Patent to be obvious over the nanotube/polymer composite of the Farrugia-Narayn combination—in both its unpolymerized and polymerized form. It is also my opinion that a POSITA, alternatively or additionally, would have found the claimed dispersions of the '649 Patent to be obvious over the toner of the Farrugia-Narayan combination. I discuss both of these alternatives in the following analysis, and provide specific citations to Farrugia's and

Narayan's descriptions to demonstrate why the Farrugia-Narayan combination renders obvious the claimed dispersion. I also note, and discuss further below, that certain of the dependent claims are obvious over only one of these alternatives—*e.g.*, over only the nanotube/polymer composite of the combination, or over only the toner of the combination.

4. Reasons for the combination

192. It is my opinion that a POSITA would have been motivated to use nanotubes having aspect ratios such as those described by Narayan in the nanotube/polymer composites and toners of the combination for at least the three reasons I discuss in the following paragraphs.

193. The first reason why a POSITA would have been motivated to use nanotubes having aspect ratios as described by Narayan in the Farrugia-Narayan combination is to obtain a "toner [that] exhibits increased conductivity." LGES-1007 (Farrugia), [0018] This is a design objective for Farrugia's toner, and Farrugia explains various advantages of using nanotubes to achieve high conductivity in toners. First, Farrugia states that by including carbon nanotubes in the composite, "a composite resin can be made more electrically conductive, and thus can be used as a component in a toner made by an emulsion/aggregation process for conductive developing methods." *Id.*, [0009]. Second, Farrugia also describes that "adding only a

small amount of carbon nanotubes to the resin composite can increase the nucleating size density of a crystalline polyester polymer," which in turns "improves the charge transfer in the toner particle," and notes that toners with improved charge transfer "are suitable for electrographic printing applications." *Id.*, [0009], [0018]. Third, Farrugia describes that "[a]dding carbon nanotube/polyester resin composites to a toner improves the resistivity, conductivity, and dielectric loss, of the toner," which "is particularly useful because low toner resistivity is known to be an issue in the development of black emulsion/aggregation toner, especially in the development of ultra-low melt toners, where the crystalline polyester is responsible for depressing the toner's resistivity." *Id.*, [0010]. Fourth, "adding carbon nanotube/polyester resin composites to a toner reduces dielectric loss in the toner," where "[d]ielectric loss poses problems in image formation by way of fogging, toner scattering, or transfer skip." *Id.*, [0011]. Fifth, Farrugia describes that "adding carbon nanotube/polyester resin composites to a toner improves conductivity of toners," which "makes the toners more suitable for conductive applications." *Id.*, [0012]. Finally, Farrugia notes that use of carbon nanotubes to achieve these advantages is economically desirable: "[a]s the price of carbon nanotubes has dramatically

decreased," toners including carbon nanotubes "offer the advantages of being both cost-effective and exhibiting improved properties." *Id.* [0018].

194. Farrugia notes that nanotubes, which have "a high aspect ratio," are suitable for achieving the desired "increased conductivity" that can lead to these many performance advantages in its toners. *Id.*, [0018]. Indeed, Farrugia specifically indicates that high aspect ratio nanotubes are desirable because "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." *Id.*, [0018]. But Farrugia provides no specific guidance as to what range of aspect ratios would enable a high electrical conductivity nanotube/polymer composite, and thus a high electrical conductivity toner, to be achieved.

195. Starting from this point—the desire to generate an electrically conductive nanotube/polymer composite (and toner), and the indication that the high aspect ratio of nanotubes are suitable for this purpose—it is my opinion that a POSITA would have looked to Narayan for specification as to the aspect ratio that is appropriate for achieving this objective. For instance, Narayan states that carbon nanotubes with "aspect ratios greater than or equal to about 5," greater than or equal to about 10," or "greater than or equal to about 100" are "suitable for imparting electrical conductivity" to

nanotube/polymer compositions. LGES-1008 (Narayan), [0014]. Narayan's specific guidance about a property (aspect ratio) already identified by Farrugia as being relevant to a shared objective (electrically conductive nanotube/polymer compositions) would have motivated a POSITA to implement the nanotubes in Farrugia's nanotube/polymer composite according to the specific teachings of Narayan.

196. The second reason why a POSITA would have been motivated to use nanotubes having aspect ratios as described by Narayan in the Farrugia-Narayan combination is because this combination is just the implementation of known techniques (specifically, nanotubes having aspect ratios greater than or equal to about 5, 10, or 100) to an already-known structure (Farrugia's nanotube/polymer composite and toner), and it produces predictable results (specifically, a nanotube/polymer composite including nanotubes sized as taught by Narayan, and a toner incorporating that composite).

197. It is my opinion that a POSITA would have expected success when looking to use nanotubes having aspect ratios such as those described by Narayan in the nanotube/polymer composites and toners of the combination for at least the four reasons I discuss in the following paragraphs.

198. The first reason why a POSITA would have expected success is that Narayan's materials system is similar to the materials system of Farrugia's nanotube/polymer nanocomposite. Narayan's polymer foam can be "a wide variety of thermoplastic resins, blends of thermoplastic resins, or thermosetting resins" such as "polyurethanes" or "polyesters," among others. LGES-1008 (Narayan), [0009]-[0011]. This composition of the polymer matrix of Narayan's material is consistent with the composition of the polymer matrix of Farrugia's nanotube/polymer nanocomposite: "polyesters and polyurethanes," among others. LGES-1007 (Farrugia), [0048]-[0052]. It is my opinion that this overlap in materials systems is an indication that a POSITA would have expected to be successful in using nanotubes dimensioned as taught by Narayan in a nanotube/polymer matrix such as Farrugia's. That is, Narayan demonstrates that nanotubes of these dimensions can be used with polymer compositions similar to those described by Farrugia.

199. The second reason why a POSITA would have expected success is that Narayan and Farrugia describe compositions containing similar amounts of carbon nanotubes. In Narayan's composition, the weight percentage of carbon nanotubes in Narayan's nanotube/polymer composition is "about 0.0001 to about 50 weight percent." LGES-1008 (Narayan), [0016]. This is

consistent with the weight percentage of carbon nanotubes in Farrugia's nanocomposite: "from about 2% to about 20% by weight." LGES-1007 (Farrugia), [0018]. It is my opinion that these similarities are an indication that a POSITA would have expected to be successful in introducing nanotubes dimensioned as taught by Narayan into in a nanotube/polymer matrix such as Farrugia's. That is, Narayan demonstrates that nanotubes of these dimensions can be used in polymer-based nanocomposites containing an amount of nanotubes that is similar to the amount of nanotubes contained in Farrugia's nanocomposite, and this would have led a POSITA to expect success in making this combination.

200. The third reason why a POSITA would have expected success is that both Narayan and Farrugia describe that carbon nanotubes are introduced into a polymer matrix to result in a composite having increased electrical conductivity. *See* LGES-1007 (Farrugia), [0009]-[0012] ("toners containing carbon nanotube/polyester composite resins exhibit enhanced properties. Depending on the concentration of nanotubes used, a composite resin can be made more electrically conductive"), [0018] ("Toners comprising carbon nanotube/polymer resins exhibit enhanced mechanical, chemical, thermal and electrical properties"); LGES-1008 (Narayan), [0008] ("The amount of carbon nanotubes (and other optional fillers) is preferably selected so as to

provide electrical conductivity … while generally retaining the advantageous intrinsic physical properties of the polymeric foams or elastomers"). Farrugia describes the concentration of nanotubes useful for achieving this objective, and indicates that "high aspect ratio" nanotubes are suitable for increasing electrical conductivity, but Farrugia does not identify what specific values of the aspect ratio can achieve this objective. *See* LGES-1007 (Farrugia), [0018] ("For example, when the carbon nanotube/polymer composite comprises carbon nanotubes from about 2% to about 20% by weight of the composite, the toner exhibits increased conductivity … 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"). Narayan, though, does identify the aspect ratios of nanotubes that can impart good electrical conductivity to nanotube/polymer composites: "Thus, mixing nanotubes with the polymer… may achieve excellent conductivity at low loading levels, because of the preservation of nanotube aspect ratio," and identifies "[s]uitable carbon nanotubes for imparting electrical conductivity" as having "diameters of about 0.5 to about 2000 nm, with aspect ratios greater than or equal to about 5." LGES-1008 (Narayan), [0014], [0025]. It is my opinion that a POSITA would have

expected success in relying on Narayan's teachings regarding aspect ratio given the shared objective (*e.g.*, increasing electrical conductivity), and given that Narayan provides a specific implementation of the high aspect ratio nanotubes already suggested by Farrugia. *See* LGES-1008 (Narayan), [0014], [0025]; LGES-1007 (Farrugia), [0018].

201. The third reason why a POSITA would have expected success is that the use of nanotubes having aspect ratios such as those described by Narayan in nanotube/polymer composites was well-known. *See* LGES-1008, [0014], [0025]; LGES-1046 (El Bounia), [0230] ("The carbon nanotubes used below are multi wall carbon nanotube… They have a diameter in between 5 and 70 nm and an aspect ratio greater than 5. Preferably, the aspect ratio is greater than 50 and more preferably greater than 100.").

5. Analysis

(i) Claim 1

[1pre] A dispersion comprising

202. To the extent the preamble is deemed to be limiting, it is my opinion that the Farrugia-Narayan combination renders it obvious.

Farrugia's nanotube/polymer composite is a dispersion

203. The nanotube/polymer composite of the Farrugia-Narayan combination is "a polymer matrix comprising ***carbon nanotubes dispersed in a polymer***

matrix." LGES-1007 (Farrugia), Abstract. This nanotube/polymer composite is **a dispersion**. For instance, the composite is a dispersion at least because the nanotubes are dispersed in the polymer matrix. The following citations from Farrugia further confirm that the nanotube/polymer composite is a dispersion: *id.*, [0020] ("The chemical moiety on the carbon nanotubes generally covalently attach to a suitable monomer. The monomers then polymerize … thereby forming ***carbon nanotubes dispersed in a polymer matrix***."), [0027]-[0028] ("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."), [0045] ("The functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer … to form ***a polymer matrix in which the carbon nanotubes are dispersed***"), [0001] ("The present disclosure relates to toners comprising carbon nanotubes dispersed in a polymer matrix"), claim 1 ("a polymer matrix comprising carbon nanotubes dispersed in a polymer"), claim 16 ("a crystalline polyester matrix comprising multi-walled carbon nanotubes dispersed in crystalline polyester").

204. The unpolymerized mixture of nanotubes, excess first monomer, and second monomer that is cured to form the nanotube/polymer composite is also **a dispersion**. Specifically, as I discussed above, nanotubes having first monomer covalently attached to their side walls, excess first monomer, and second monomer are mixed together and heated to generate the polymerized composite. *See* LGES-1007 (Farrugia), [0045] ("functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer … to form a polymer matrix in which the carbon nanotubes are dispersed"), [0041] ("The functionalized carbon nanotubes are dispersed in the first monomer").

205. This unpolymerized mixture is described in Farrugia's examples, in which "1,9-nonanediol" (*e.g.*, a first monomer) is melted and mixed with functionalized carbon nanotubes until "the MWNTs [multi-wall carbon nanotubes] were ***well-dispersed in the diol***." A second monomer, "1,12-dodecanedioic acid," was then added to the mixture, and the mixture was heated to melt the mixture, resulting in the MWNTs dispersed in molten first and second monomer. LGES-1007 (Farrugia), [0093]; *see* [0096].

206. The heated, unpolymerized[2] mixture of carbon nanotubes and monomers is **a dispersion** at least because the nanotubes are dispersed in a molten mixture of monomer (*e.g.*, molten excess first monomer and molten second monomer).

Farrugia's toner is a dispersion

207. In the Farrugia-Narayan combination, the "carbon nanotube/polymer composite" (*e.g.*, the composite including nanotubes having dimensions as described by Narayan) is "incorporated into a toner." LGES-1007 (Farrugia), [0054]. The toner of the combination is **a dispersion**. For instance, the toner is a dispersion at least because the toner includes the nanotube/polymer composite, which is a dispersion, as I discussed above.

[2] It is possible that the monomers start to react before the second monomer is fully melted. By the time the dispersion is fully fluid, some first and second monomers may have reacted to form pre-polymeric oligomers. There may then exist a state in which the carbon nanotubes are dispersed in a mixture of unreacted monomers and a smaller amount of oligomeric pre-polymers. For convenience, I will refer to this mixture as an unpolymerized mixture of nanotubes and monomers, keeping in mind that some limited reaction of monomers may have occurred.

208. The following citations further confirm that the Farrugia's toner includes the carbon nanotube/polymer composite, and thus that the toner is a dispersion: LGES-1007 (Farrugia), [0018] ("toners comprising carbon nanotube/polymer composite resins"), Abstract ("A toner comprising: a polymer matrix comprising carbon nanotubes dispersed in a polymer"), [0001] ("The present disclosure relates to toners comprising carbon nanotubes dispersed in a polymer matrix"), [0019] ("The present disclosure provides a toner comprising a resin comprising a polymerized mixture, … wherein the polymerized mixture is a composite comprising carbon nanotubes and a polymer"), [0020] ("The carbon nanotube/polymer composite …" includes "carbon nanotubes dispersed in a polymer matrix. This carbon nanotube/polymer composite resin can generally be incorporated into a toner"), claim 1 ("A toner comprising: a polymer matrix comprising carbon nanotubes dispersed in a polymer"), claim 11 ("A method of making a toner, comprising: … polymerizing the first monomer with a second monomer to form a polymer matrix comprising carbon nanotubes; and adding one or more colorants and/or one or more waxes to the polymer matrix to form a toner"), claim 16 ("An emulsion/aggregation toner comprising: a crystalline polyester matrix comprising multi-walled carbon nanotubes dispersed in crystalline polyester").

[1a] a plurality of oxidized, discrete carbon nanotubes and

209. First, Farrugia describes that the nanotubes are **carbon nanotubes**. *See* LGES-1007 (Farrugia), Abstract ("A toner comprising: a polymer matrix comprising ***carbon nanotubes*** dispersed in a polymer"). That the nanotubes are carbon nanotubes does not change with the combination with Narayan; Narayan impacts the dimension of the nanotubes, but not their composition.

210. Further confirmation that the nanotubes are **carbon nanotubes** is provided at the following citations: LGES-1007 (Farrugia), [0018] ("The present disclosure is directed to toners comprising ***carbon nanotube/polymer composite resins***"), [0045] ("The functionalized ***carbon nanotubes*** covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer … to form a polymer matrix in which the carbon nanotubes are dispersed."), [0001] ("The present disclosure relates to toners comprising carbon nanotubes dispersed in a polymer matrix"), [0019]-[0020] ("The present disclosure provides a toner comprising a resin comprising a polymerized mixture, … wherein the polymerized mixture is a composite comprising carbon nanotubes and a polymer"), [0053] ("The carbon nanotube content of the carbon nanotube/polymer composite is generally from about 0.05% to about 20% by weight of the composite"), [0054] ("The carbon nanotube/polymer

composite may be incorporated into a toner"), [0024]-[0028], [0039]-[0042], [0056]-[0057], [0088]-[0097], [0104]-[0107]; claims 1, 4-6, 8, 11, 16-18.

211. Second, Farrugia describes that the carbon nanotubes are **oxidized**. For instance, Farrugia's nanotubes are "functionalized" carbon nanotubes, where the "functionalized nanotubes can be directly prepared by … ***oxidation***." LGES-1007 (Farrugia), [0028]; *see* [0020]. That is, for instance, Farrugia's functionalized carbon nanotubes, in which the functionalization is carried out by oxidation, are **oxidized** carbon nanotubes.

212. Further confirmation that the nanotubes are **oxidized** is provided at the following citations, which indicate an oxidation process that can result in oxygen-containing species being covalently bound to the nanotubes: LGES-1007 (Farrugia), [0026] ("the carbon nanotubes are purified by reacting with one or more suitable reagents, such as oxidation agents"), [0045] ("The ***functionalized carbon nanotubes*** covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer … to form a polymer matrix in which the carbon nanotubes are dispersed."), [0028] ("Generally, functionalized nanotubes can be directly prepared by … oxidation, or other suitable means. In some embodiments, 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. Oxidation of nanotubes provides both improved stability and the ability to form electrostatically stabilized colloidal dispersions in water and alcohols."), [0029]-[0039], [0041]-[0042], [0045], [0089]; claims 8, 11.

213. Notably, the same nanotubes are present in the unpolymerized mixture of functionalized nanotubes and monomer, the nanotube/polymer composite, and the toner. For instance, neither the polymerization nor the incorporation of the composite into the toner affects the composition of the nanotubes. That is, the nanotubes in all three of the dispersions of the combination are **oxidized carbon nanotubes**.

214. Farrugia's experimental examples also confirm that the nanotubes in the unpolymerized mixture of functionalized nanotubes and monomer as well as in the nanotube/polymer composite are **oxidized** (*e.g.*, functionalized) **carbon nanotubes**. *See* LGES-1007 (Farrugia), [0088]-[0097] ("Example 1 describes the functionalization of carbon nanotubes. Examples 2 and 3 describe the synthesis of carbon nanotubes/semi-crystalline polyester resin compositions using the process described in Example 1."). Example 1 [0089] describes a process in which MWNTs oxidized (i.e. functionalized) using a treatment with nitric acid, which is known to oxidize carbon

nanotubes. Examples 2 and 3 describe the preparation first of the unpolymerized mixture of functionalized nanotubes and monomer and second of the polymerized nanotube/polymer composite using the "MWNTs produced in Example 1," which are oxidized carbon nanotubes. *Id.*, [0093].

215. Third, Farrugia describes that the nanotubes are **discrete**, *e.g.*, that the nanotubes in the dispersion are generally individually distinct, *e.g.*, lacking direct association with other nanotubes along substantially the length of the nanotubes. Indeed, Farrugia specifically states that "***[d]iscrete nanotubes***, aggregates of nanotubes, or both discrete and aggregate nanotubes may be suitable for use according to the present disclosure." LGES-1007 (Farrugia), [0024] (emphasis added). I note that although Farrugia indicates through this quote that aggregates of nanotubes can be used, it also indicates that solely discrete nanotubes can be used.

216. It is my opinion that a POSITA would have understood that Farrugia's discrete nanotubes are present in the unpolymerized mixture of functionalized nanotubes and monomer, the nanotube/polymer composite, and the toner, at least because of Farrugia's statement that "[d]iscrete nanotubes" are generally applicable in the context of the disclosure. LGES-1007 (Farrugia), [0024]. Additionally, a POSITA would understand that the oxidation, functionalization, and mixing ("stirring") procedures taught by

Farrugia will favor discrete nanotubes over aggregated nanotubes. *See* LGES-1007 (Farrugia), Examples 1-3.

[1b] at least one additive

217. The unpolymerized mixture of functionalized nanotubes and monomer, the nanotube/polymer composite, and the toner (each of which is a dispersion, as I discussed above) all include at least one additive, as I discuss in the following paragraphs.

The unpolymerized mixture of functionalized nanotubes and monomer includes an additive

218. The unpolymerized mixture of functionalized nanotubes and monomer includes nanotubes having first monomer covalently bonded to their side walls, excess first monomer, and second monomer. *See* LGES-1007 (Farrugia), [0040]-[0041].

219. In this mixture, the "second monomer" can be considered **an additive**. LGES-1007 (Farrugia), [0045]; *see* [0093] (identifying 1,12-dodecanedioic acid as the second monomer, *e.g.*, an additive), [0096]. More specifically, Farrugia provides numerous examples of second monomers, listed in [0046]-[0047]. Each of these compounds is a specific example of **an additive**.

220. The "excess unreacted first monomer" in this mixture can also be considered **an additive**. LGES-1007 (Farrugia), [0045]; *see* [0093] (identifying 1,9-nonanediol as the first monomer, *e.g.*, an additive), [0096]. More

specifically, Farrugia provides numerous examples of first monomers, listed in [0043]-[0044]. Each of these compounds is a specific example of **an additive**.

The nanotube/polymer composite includes an additive

221. Farrugia describes the nanotube/polymer composite as "a polymer matrix comprising carbon nanotubes dispersed in ***a polymer***." LGES-1007 (Farrugia), Abstract. The polymer in which the nanotubes are dispersed is **an additive**.

222. Specifically, Farrugia describes that "[t]he functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer … to form a polymer matrix in which the carbon nanotubes are dispersed," and notes that "[t]he resultant polymer," *e.g.*, the additive, "may be crystalline, semi-crystalline, amorphous, or a mixture thereof." LGES-1007 (Farrugia), [0045]. Farrugia provides numerous examples of these "polymers formed from the polymerization of the second monomers."[3] *Id.*, [0048]. Each of these

[3] A POSITA would understand that this statement does not mean that the second monomer polymerizes by itself. The chemistry taught by Farrugia is of a type (condensation polymerization) in which first monomer and second monomer react

polymers, which are listed in [0048]-[0052], is a specific example of **an additive**.

223. *See also* the following cites describing the polymer of the composite: [0001] ("A toner comprising: a polymer matrix comprising carbon nanotubes dispersed in a polymer"), [0018] ("The present disclosure is directed to toners comprising carbon nanotube/polymer composite resins"), [0019] ("The present disclosure provides a toner comprising a resin comprising a polymerized mixture … wherein the polymerized mixture is a composite comprising carbon nanotubes and a polymer"), [0020] ("The carbon nanotube/polymer composite according to the present disclosure is formed by known means in the art. The carbon nanotubes may be functionalized with one or more chemical moieties. … The chemical moiety on the carbon nanotubes generally covalently attach to a suitable monomer. The monomers then polymerize … thereby forming carbon nanotubes dispersed in a polymer matrix"), [0045] ("The functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer through the

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with one another to form polymers. Neither first monomer, nor second monomer is capable of homopolymerization.

formation of ester or amide bonds to form a polymer matrix in which the carbon nanotubes are dispersed. … The resultant polymer may be crystalline, semi-crystalline, amorphous, or a mixture thereof"), [0088] ("Examples 2 and 3 describe the synthesis of carbon nanotubes/semi-crystalline polyester resin compositions"), [0093]-[0097]; claims 1-6, 8, 10, 14, 16-18.

The toner includes an additive

224. Farrugia's toner contains the nanotube/polymer composite, as I discussed above. *See* LGES-1007 (Farrugia), [0054]. Thus, the polymer matrix, which is an additive to the nanotube/polymer composite (as I discussed above), is also **an additive** to the toner.

225. Farrugia also describes additional examples of **additives** in the toner of the Farrugia-Narayan combination, which I list in the following paragraphs. *See* LGES-1007 (Farrugia), [0054] ("The toner may optionally include one or more of the additives described later in the specification").

226. A first example of **an additive** in the toner is a ***wax***. Indeed, Farrugia explains that the toner includes "a polymer matrix comprising carbon nanotubes dispersed in a polymer" and "one or more waxes." LGES-1007 (Farrugia), Abstract. The following citations provide further examples of the inclusion of a wax additive in the toner: *id.*, [0071] ("One or more waxes

may be added to the toner in order to raise the image density and to effectively prevent the offset to a reading head and the image smearing. … Examples of suitable waxes include … polyolefin waxes, such as low molecular weight polyethylene, polypropylene, a fluorocarbon-based wax (Teflon), or Fischer-Tropsch wax, copolymers thereof, mixtures thereof, and the like"), [0019], [0055] (describing that the toner production process includes "aggregating an emulsion containing polymer binder and one or more optional waxes"), [0057], claims 1 and 11.

227. A second example of **an additive** in the toner is a ***surfactant***. For instance, Farrugia states that the toner production process includes "aggregating an emulsion containing polymer binder and … one or more surfactants." LGES-1007 (Farrugia), [0055]. Paragraphs [0072]-[0073] of Farrugia provide specific examples of "nonionic surfactants that may be used in the toner" and "suitable cationic surfactants"). *See also* [0074]-[0075] (describing "[a] suitable amount of surfactant" for inclusion in the toner).

228. Additional examples: Farrugia also lists a number of additional **additives** to the toner, including clarifiers, tackifiers, plasticizers, adhesives, antioxidants, and charge-controlling agents. *See* LGES-1007 (Farrugia), [0076]-[0083]. For instance, "[a]ntioxidants protect the images from oxidation and also protect the toner components from oxidation during the heating portion of

the toner preparation process." *Id.*, [0076]. "[A] charge-controlling agent may be added to the toner in order to help improve the electrification level and an electrification rate… and to obtain excellent fluidity." and can be "a charge-controlling agent (CCA) having a function to control charge … within a specific range and a charge-controlling resin (CRR) having a function to reinforce charge." *Id.*, [0078]-[0079]. Other examples of additives that can be incorporated in the toner include "a fluidization agent …; lubricants …; silica; a spacing agent; a dryer; a dispersant; a humectant; a stabilizer; a thickening agent; a gelatinizing agent; a defoaming agent and an initiator for photopolymerization." *Id.*, [0084].

[1c] wherein the oxidized, discrete carbon nanotubes have an aspect ratio of 25 to 500,

229. As I discussed above, the Farrugia-Narayan combination uses Farrugia's nanotube/polymer composite as the starting point, and sizes the nanotubes to have an aspect ratio based on the teachings of Narayan. Specifically, the nanotubes (which are oxidized, discrete nanotubes, as I discussed above for claim element [1a]) have an aspect ratio of "greater than or equal to about 5," "greater than or equal to about 10," or "greater than or equal to about 100," which are aspect ratios that Narayan teaches are suitable for achieving the desired high electrical conductivity. *See* LGES-1008 (Narayan), [0014].

230. The ranges of aspect ratios described in Narayan (*e.g.*, "greater than or equal to about 5," "greater than or equal to about 10," or "greater than or equal to about 100," LGES-1008 (Narayan), [0014]) overlap with the range of aspect ratios recited in element [1c] (25 to 500).

231. As I noted above when discussing the Sun ground, the '649 Patent does not describe particular unexpected results associated with nanotubes having an aspect ratio of 25 to 500 (as claimed) as opposed to those in the range of greater than or equal to 5, 10, or 100 (as in the Farrugia-Narayan combination). The '649 Patent also does not provide any discussion or evidence that these ranges of aspect ratios are in any way critical.

232. I also note that Narayan does disclose one range of aspect ratios ("greater than or equal to about 1000") that does not overlap with the claimed range of aspect ratios (25 to 100). I have been informed that I can rely on Narayan for all of the ranges it describes, even if the ranges I rely on are not the ranges that Narayan identifies as its preferred ranges.

[1d] are multiwall, and

233. Farrugia describes the use of **multiwall** nanotubes. *See* LGES-1007 (Farrugia), claim 16 ("An emulsion/aggregation toner comprising: a crystalline polymer matrix comprising ***multi-walled carbon nanotubes***");

see [0028] ("oxidation chemistry may be used to open the end caps of both single and ***multi-walled nanotubes***").

234. Notably, the same nanotubes are present in the unpolymerized mixture of functionalized nanotubes and monomer, the nanotube/polymer composite, and the toner. For instance, neither the polymerization nor the incorporation of the composite into the toner affects the composition of the nanotubes. That is, the nanotubes in all three of the dispersions of the combination are **multiwall** nanotubes.

235. Farrugia's experimental examples further confirm that the nanotubes in the unpolymerized mixture of functionalized nanotubes and monomer and in the nanotube/polymer composite are **multiwall** nanotubes (MWNTs). Specifically, Examples 2 and 3 describe preparation of "***MWNTs***/Polyester Composite[s]" using the MWNTs prepared in Example 1. LGES-1007 (Farrugia), [0089]-[0097]. Farrugia also describes its experimental results that characterize "The Effect of ***MWNTs*** on Recrystallization" in the composite and "The Effect of ***MWNTs*** on Resistivity and Conductivity" in the composite. *See* LGES-1007 (Farrugia), [0104]-[0107].

[1e] are present in the range of greater than zero to about 30% by weight based on the total weight of the dispersion.

The nanotube/polymer composite has the claimed weight percentage of nanotubes

236. Farrugia describes that its nanotube/polymer composite "comprises carbon nanotubes ***from about 2% to about 20% by weight*** of the composite," or "***from about 0.05% to about 10% by weight*** of the polymer composite." LGES-1007 (Farrugia), [0018]. That is, **nanotubes are present** in the nanotube/polymer composite **in the range of greater than zero to about 30% by weight**.

237. Farrugia provides the following additional cites confirming the weight percentage of nanotubes in its nanotube/polymer composite: [0053] ("The carbon nanotube content of the carbon nanotube/polymer composite is generally from about 0.05% to about 20% by weight of the composite, such as from about 0.05% to about 5%, or from about 5% to about 15%, or from about 7% to about 10% by weight of the composite. In one embodiment, the carbon nanotube content is from about 0.5% to 8% by weight of the composite."), claim 4 ("wherein the polymer matrix comprises carbon nanotubes from about 0.05% to about 20% by weight of the polymer matrix"), claim 5 ("wherein the polymer matrix comprises carbon nanotubes from about 1% to about 5% by weight of the polymer matrix"), claims 17-18.

238. These ranges of weight percentages described by Farrugia (*e.g.*, "from about 2% to about 20% by weight" or "from about 0.05% to about 10% by weight," LGES-1007 (Farrugia), [0018]) overlap with and fall within the range of weight percentages recited in claim element [1e] ("greater than zero to about 30% by weight").

239. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes at weight percentages of greater than zero to about 30%. The '649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

240. I also note that providing nanotubes having an aspect ratio in the range described by Narayan would not impact the weight percentages of nanotubes in the composite as specified by Farrugia. In particular, Farrugia prescribes suitable ranges of weight percentages for the nanotubes, but does not specify their aspect ratio. A POSITA would understand how to achieve a composite having a certain weight percentage of nanotubes regardless of the aspect ratio of the nanotubes.

241. Farrugia's examples also confirm that nanotubes are present in the nanotube/polymer composite at weight percentages that fall within the claimed range. Farrugia specifies the nanotube concentration in its

examples: Example 2: ("Preparation of **0.075% MWNTs**/ Polyester Composite.") Example 3: ("Preparation of **0.185% MWNTs**/Polyester Composite."). LGES-1007 (Farrugia), [0093], [0096]. As I discuss and calculate below, these are theoretical values, but the nanotube concentration in the unpolymerized mixture, as well as the actual experimental value for the nanotube concentration in the composite, are generally similar to these theoretical values.

242. Looking at Farrugia's examples, a POSITA would understand that the concentration of carbon nanotubes, determined at different times in the production of the composite, and calculated on different bases, will be slightly different from one another. All of these values are valid in their own ways, and a POSITA would understand how to utilize them in preparing a composite. These considerations are as follows: (1) The concentration of nanotubes in the unpolymerized monomer mixture is slightly lower than in the polymerized composite due to a loss of mass inherent in the polymerization mechanism of the monomers during polymerization. (2) Farrugia reports a carbon nanotube concentration in the polymerized composite, based on a **theoretical yield** of the polymerization reaction. (3) Farrugia provides all the necessary information for a POSITA to do their own calculation of the carbon nanotubes in the polymerized composite based

on actual experimental results, and this is slightly different from the concentration based on Farrugia's assumed theoretical yield. Below I will illustrate how the concentrations of the carbon nanotubes in Farrugia's Examples 2 and 3 are calculated. The results of these calculations are summarized in the following table:

	MWNT wt.% Farrugia Example 2	MWNT wt.% Farrugia Example 3
Un-polymerized mixture	0.066%	0.164%
Composite: Theoretical	0.075%	0.185%
Composite Experimental Results	0.072%	0.171%

243. Farrugia Example 3 [0096] states: "About 2.0 g of MWNTs (0.185% ***relative to 1080g polymer mass***) was added." This indicates that Farrugia is working on a mass basis, and therefore the percentage composition is a weight percentage.

244. The concentration of carbon nanotubes in the unpolymerized monomer mixture is the weight of carbon nanotubes divided by the total weight of all components in the mixture. For Example 2, the weight of carbon nanotubes is 0.81 g and the weight of all the components in the mixture is 500g of 1,9-nonanediol, plus 719 g of 1,12-dodecanedioic acid, plus 1.30 g of Fascat 4100 catalyst, plus the 0.81 g of carbon nanotubes. The concentration of carbon nanotubes in the unreacted Example 2 mixture is 0.81g / (500g +

719g + 1.30g + 0.81g), which is 0.066%. A similar calculation for Example 3 gives 0.164%.

245. The concentrations of nanotubes in the unpolymerized mixtures of Examples 2 and 3 are lower than either the theoretical or experimental values for the polymerized composites. This is because the polymerization reaction of 1,9-nonanediol and 1,12-dodecanedioic acid is a specific type of polymerization known as a **condensation polymerization**. In condensation polymerization, one molecule of water is lost by the molecular structure for each polymer backbone bond created between an alcohol of 1,9-nonanediol and a carboxylic acid of 1,12-dodecanedioic acid. This water of condensation is removed from the system and its mass is lost on conversion from the liquid monomer mixture to the solid polymer matrix.

246. As a result of loss of water of condensation, the nanotube concentrations in the unpolymerized monomer are slightly different than what Farrugia reports as **theoretical** concentrations of nanotubes in the nanotube/polymer composites in the titles of Examples 2 and 3. Example 2: ("Preparation of **0.075% MWNTs**/ Polyester Composite.") Example 3: ("Preparation of **0.185% MWNTs**/Polyester Composite."). The fact that these nanotube concentrations reported by Farrugia are theoretical can be found for Example 2 ("0.075% relative to 1080 g polymer **theoretical yield**," LGES-

1007 (Farrugia), [0093]) and for Example 3 ("0.185% relative to 1080g polymer mass," *id.*, [0096]), keeping in mind that Example 2 has already specified 1080g polymer mass, as the **theoretical yield** for the polymerization reaction which uses the same components in the same amounts (with the exception of the amount of nanotubes, which are not part of the polymerization and do not figure into the polymerization yield, theoretical or experimental) as for Example 2.

247. Finally, a POSITA can calculate the actual weight percentages of nanotubes in the Example 2 and Example 3 composites based on the experimental information, and in particular the amount of water of condensation lost. For Example 2 [0094]: ("The total water yield increased to about 95 ml"). With this information, a POSITA can calculate the experimental nanotube concentration in the polymerized composite of Farrugia's Example 2 [0093]: 0.81g of MWNTs are added to a mixture which polymerizes to form the polyester polymer. The total weight of polymer can be calculated by adding the 500g of 1.9-nonanediol, plus the 719 g of 1,12-dodecanedioic acid, plus the 1.30 g of Fascat 4100 catalyst, and subtracting the 95 ml of water (which weighs about 95 g) that is a byproduct of the reaction and is removed. *See* LGES-1007 (Farrugia), [0093], [0094] ("condensed water was collected in a graduated cylinder."). Dividing the weight of MWNTs by the net weight of

the polymer plus nanotubes and subtracting the weight of water lost, a POSITA will get an actual experimental nanotube concentration of 0.81g/(500g + 719g + 1.3g + 0.81g - 95g) = 0.00072 which is 0.072 weight %. This actual weight percent value is slightly different from the theoretical value (0.075%) given by Farrugia in the title of Example 2. A POSITA can do the same calculation for Farrugia Example 3 and will get a nanotube concentration of 0.171 weight %, which is again a little different from the theoretical value (0.185%) given by Farrugia.

248. A POSITA has a choice among numbers for weight percentage of nanotubes in the composites of Examples 2 and 3. Farrugia provides a direct statement of a theoretical number in the title of each example. A POSITA is very likely to simply use this as a guide. If, instead, a POSITA calculates actual experimental carbon nanotube weight percentages based on the experimental numbers given in these examples, they will get slightly lower weight percentages for these two examples. The differences among the carbon nanotube weight percentages in the unpolymerized mixture, the theoretical value for the polymerized composite, and the experimental value for the polymerized composite are sufficiently small that they could be used interchangeably by a POSITA in guiding the production of this type of composite.

249. In particular, all possible values of the MWNT concentration in Examples 2 and 3—whether for the unpolymerized mixture, the theoretical value for the polymerized composite, or the actual value for the polymerized composite—place the weight percentage of carbon nanotubes within the range of greater than zero to 30 wt. %, as claimed. Note also that Farrugia's Examples 2 and 3, discussed above, in both the polymerized and unpolymerized state also fall in the 0.05% to 10% by weight range, which is consistent with Farrugia's statements earlier in the description. *See* LGES-1007 (Farrugia), [0018].

250. I also note that fewer than all of the nanotubes in the nanotube/polymer composite may be oxidized, discrete carbon nanotubes. However, even if this is the case, those oxidized, discrete carbon nanotubes still constitute a portion of the entire population of nanotubes in the Farrugia-Narayan composite, meaning that the population of oxidized nanotubes is still **greater than zero by weight**.

The unpolymerized mixture of nanotubes and monomer has the claimed weight percentage of nanotubes

251. It is my opinion that a POSITA would have understood or found obvious that **nanotubes are present** in the unpolymerized mixture of functionalized nanotubes and monomer **in the range of greater than zero to about 30% by weight**. This opinion is based on Farrugia's description of the process of

producing its composite. In this process, the nanotubes and monomer are mixed, and then the mixture is polymerized to form the nanotube/polymer composite. *See* LGES-1007 (Farrugia), [0045], [0093], [0096]. Once the nanotubes and monomers are mixed, nothing is added to or removed from the mixture before the polymerization process. As I discussed above, though, during the polymerization process an amount of water is generated by the **condensation** polymerization process. This water it is removed from the system and quantified by its weight. This water comes from a slight loss of mass of the reacting monomers which takes place as they are connected together into polymer chains. As a result, the weight percentage of nanotubes in the unpolymerized mixture will be slightly less than the weight percentage of nanotubes in the polymerized nanotube/polymer composite.

252. The removal of this **water of condensation** is taken into account by Farrugia, on a theoretical basis, in the concentrations of the nanotubes in the final cured composites as reported by Farrugia, for instance, in the titles of Examples 1 and 2. And a POSITA can easily calculate the real experimental nanotube concentration in the final cured composite as I have shown above. Based on the table, which I reproduce again below and which I explained above, I conclude that the weight percentage of nanotubes in the unpolymerized mixture is similar enough to the weight percentage of

nanotubes in the nanotube/polymer composite, to be considered substantially equivalent from the point of view of a POSITA preparing such a composite.

	MWNT wt.% Farrugia Example 2	MWNT wt.% Farrugia Example 3
Un-polymerized mixture	0.066%	0.164%
Composite: Theoretical	0.075%	0.185%
Composite Experimental Results	0.072%	0.171%

253. That is, as demonstrated by Farrugia's examples, **nanotubes are present** in the unpolymerized mixture **in the range of greater than zero to about 30% by weight**, as claimed.

254. More generally, as I demonstrated above, the difference between the weight percentage of carbon nanotubes in the unpolymerized mixture and the stated (*e.g.*, theoretical) weight percentage of carbon nanotubes in the nanotube/polymer composite is sufficiently small as to be generally interchangeable. Farrugia states that the weight percentage of carbon nanotubes in the nanotube/polymer composite is "***from about 2% to about 20% by weight***" of the mixture, or "***from about 0.05% to about 10% by weight***" of the mixture. LGES-1007 (Farrugia), [0018]; *see* [0053], [0093], [0096], [0107], claims 4-5, claims 17-18. Based on this description, a POSITA would understand the weight percentage of carbon nanotubes in the unpolymerized mixture to be also in substantially the same range. That is, a

POSITA would understand that **nanotubes are present** in Farrugia's unpolymerized mixture in weight percentages that fall **in the range of greater than zero to about 30% by weight**, as claimed.

255. I also note that fewer than all of the nanotubes in the unpolymerized mixture may be oxidized, discrete carbon nanotubes. However, even if this is the case, those oxidized, discrete carbon nanotubes still constitute a portion of the entire population of nanotubes in the Farrugia-Narayan unpolymerized mixture, meaning that the population of oxidized nanotubes is still **greater than zero by weight**.

The toner has the claimed weight percentage of nanotubes

256. Farrugia describes preparation of toners with various weight percentages of multiwall carbon nanotubes; these preparations are listed in Table 2, which I have reproduced below.

TABLE 2

Resistivity of Carbon Black Toner Containing Crystalline Polyester Resins as a Function of MWNT Loading

MWNT (wt % of total toner weight)	Resistivity (Ohm cm) (Expected Values)
0%	7.1×10^{12}
0.075%	1.0×10^{13}
0.185%	5.0×10^{13}
1.0%	1.0×10^{14}
1.5%	1.0×10^{15}
2.0%	1.0×10^{16}
3.0%	1.0×10^{15}
5.0%	1.0×10^{13}

Excerpt from Farrugia, Table 2

257. This table shows that Farrugia's toners include nanotubes in the range of ***greater than 0 to 5% by weight*** of the "total toner weight." That is, **nanotubes are present** in the toner **in the range of greater than zero to about 30% by weight**. This range of weight percentages described for Farrugia's toner (up to 5% by weight; *see* Table 2) overlaps and falls within the range of weight percentages recited in claim element [1e] ("greater than zero to about 30% by weight"). And as I noted above, the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes at weight percentages of greater than zero to about 30, or provide any discussion or evidence that these ranges of weight percentage are in any way critical.

258. I also note that fewer than all of the nanotubes in the toner may be oxidized, discrete carbon nanotubes. However, even if this is the case, those oxidized, discrete carbon nanotubes still constitute a portion of the entire population of nanotubes in the Farrugia-Narayan toner, meaning that the population of oxidized nanotubes is still **greater than zero by weight**.

(ii) Claim 2

[2] The dispersion of claim 1 wherein at least 70 percent by weight of the nanotubes are discrete.

259. Farrugia states that "[d]iscrete nanotubes, aggregates of nanotubes, or both discrete and aggregate nanotubes may be suitable for use according to the present disclosure." LGES-1007 (Farrugia), [0024]. I interpret this statement to mean that Farrugia indicates that one option for the nanotubes in its composites is "[d]iscrete nanotubes," and a distinct option is a combination of "both discrete and aggregate nanotubes." *Id.* That is, if the composite includes "discrete nanotubes," then it does not include "both discrete and aggregate nanotubes," and thus substantially all of the nanotubes are discrete. It is my opinion that a dispersion in which substantially all of the nanotubes are discrete is a dispersion in which **at least 70 percent by weight of the nanotubes are discrete**, or at the very least, a POSITA would have found this to be obvious. Indeed, I note that the literature supports this interpretation that a dispersion in which substantially

all of the nanotubes are discrete has at least 70% by weight of the nanotubes being discrete. For instance, Huang states that "The term "singly dispersed carbon nanotubes" refers to a population of carbon nanotubes that is substantially completely dispersed. Specifically, each nanotube fiber is substantially separated from the others and is not part of a "bundle". They are separate entities and are free of strong interaction from each other. The nanotubes are also substantially uniformly distributed throughout the medium." LGES-1051, [0042].

260. It is also my opinion that a POSITA would have understood or found obvious that **at least 70 percent by weight of the nanotubes are discrete** based on the nanotube functionalization (*e.g.*, oxidation) process described by Farrugia. Farrugia describes the preparation of "functionalized carbon nanotubes," which have any of a variety of R groups covalently bonded onto their side walls. *See* LGES-1007 (Farrugia), [0028]-[0039]. These functionalized nanotubes are the nanotubes used in Farrugia's nanotube/polymer composite, and thus also in the toner. *See id.*, [0054]. As I discussed above with respect to the Sun ground, a POSITA would have understood or found obvious that the presence of these moieties on the nanotube walls prevents the nanotubes from stacking into clumps, and instead results in substantially all of the nanotubes being discrete, *e.g.*,

individually distinct, nanotubes in the composite. *See* [1a] and claim 2 of Ground 1A. Specifically, a POSITA would have understood or found obvious that substantially all of the nanotubes being so functionalized (and thus discrete) encompasses **at least 70% by weight of the nanotubes** being so functionalized, and thus **discrete** nanotubes in the composite.

261. Otherwise, it is alternatively/additionally my opinion that this "at least 70% by weight" limitation is just a design choice. A POSITA developing a dispersion including discrete nanotubes would have considered various weight percentages of discrete nanotubes, of which 70% is an example, and which, as I noted above, does not appear to correspond to any unexpected results or unique solutions.

(iii) Claim 3

[3] The dispersion of claim 1 in the form of free flowing particles.

262. As I previously discussed when addressing claim element [1a] and claim 2, the nanotubes in Farrugia's composites are **discrete**, *e.g.*, they are generally individual, separated nanotubes. Individual, separated nanotubes are not aggregated into a clump, but instead are free flowing particles. For instance, individual, separated nanotubes, such as those in Farrugia's unpolymerized mixture of nanotubes and monomer, can flow freely relative to other nanotubes in the mixture. Thus, when substantially all of the nanotubes in

the mixture are discrete, the mixture is **in the form of free flowing particles** at least because substantially all of the particles (*e.g.*, nanotubes) in the mixture can flow freely relative to one another.

(iv) Claim 4

[4] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes comprise an oxidation species selected from carboxylic acid or a derivative carbonyl containing species wherein the derivative carbonyl species is selected from ketones, quaternary amines, amides, esters, acyl halogens, and monovalent metal salts.

263. Farrugia describes that "functionalized carbon nanotubes can be directly prepared by … oxidization," which "produce[s] ***carboxyl***, carbonyl and hydroxyl groups at the opened ends and defects on the side walls." LGES-1007 (Farrugia), [0028]. Farrugia provides the following general formula as an example of its functionalized nanotubes:

$$[C_nH_L—]R_m$$

.

The C_nH_L element in the formula represents the nanotube, and the R_m element represents the functional group attached to the opened ends and side walls of the nanotube.

264. The R group (*i.e.*, the oxidation species on the nanotubes) can be ***COOH***, which is a **carboxylic acid**. LGES-1007 (Farrugia), [0029]-[0031]. Farrugia also provides additional detail about "carbon nanotubes [that] are

functionalized with ***carboxylic acid moieties***," noting that "[c]arboxylic acid-functionalized carbon nanotubes are particularly useful because they can serve as the starting point for preparing other types of functionalized carbon nanotubes." *Id.*, [0039].

265. I note that when the R group is a carboxylic acid, the carboxylic acid will naturally convert into a monovalent metal salt by the loss of a proton under even very mildly basic conditions. For instance, in the presence of even a small amount of sodium hydroxide, NaOH, the surface functionality becomes COO^{-} Na^{+}, which is a **monovalent metal salt**.

266. The R group (*i.e.*, the oxidation species on the nanotubes) can be COOR', where R' can be "hydrogen, alkyl, aryl, cycloalkyl, or aralkyl, cycloaryl, or poly(alkylether)." LGES-1007 (Farrugia), [0029]-[0033]. When R' is alkyl, aryl, cycloalkyl, aralkyl, cycloaryl, or poly(alkylether), the R group is **an ester**. *See also* [0039] ("alcohols or amides[4] can be easily linked to the acid [on the nanotube] to give stable ***esters or amides***").

[4] A POSITA would have understood that the term "amides" in [0039] should have been "amines," which react with acids to produce "amides."

267. The R group (*i.e.*, the oxidation species on the nanotubes) can be COCl. LGES-1007 (Farrugia), [0029]-[0031]. COCl is acid chloride, which is an **acyl halogen**.

268. These functional groups (that is, the R groups I discuss in the previous paragraphs) are **oxidation species** because they are functionalized onto the nanotubes in an oxidation process or because they are enabled by the oxidation process, as described by Farrugia: "***oxidation chemistry*** may be used … to produce carboxyl, carbonyl and hydroxyl groups at the opened ends and defects on the side walls." LGES-1007 (Farrugia), [0028]. *See also id.*, [0039] ("Functionalization may be carried out, for example, via chlorate, nitric acid, or ammonium persulfate ***oxidation***" to functionalize the nanotubes with carboxylic acid moieties). For instance, carboxylic acid functional groups are functionalized onto the nanotubes as a direct result of the oxidation process. Other of the functional groups I discussed above, such as amides, acyl halogens, and esters, are enabled by the oxidation process.

(v) Claim 5

[5] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes comprise an oxidation species selected from hydroxyl or derived from hydroxyl containing species.

269. Continuing my discussion from claim [4], the R group (*i.e.*, the oxidation species on the nanotubes) can be OH, which is a **hydroxyl** species. LGES-1007 (Farrugia), [0029]-[0031]. Farrugia also specifically notes that "oxidation chemistry may be used … to produce carboxyl, carbonyl and ***hydroxyl groups*** at the opened ends and defects on the side walls." *Id.*, [0028]. The hydroxyl groups are **oxidation species** for the same reasons I discussed above for claim [4].

270. Like the carboxylic acid group I discussed for claim [4], the hydroxyl group is a versatile chemical functionality, and once it exists on the surface of a carbon nanotube, it opens up numerous chemical routes to attach other chemical species. For instance, a carboxylic acid and a hydroxyl can react to form an ester. As previously discussed, a carboxylic acid on the nanotube surface can react with an alcohol, which is a compound containing the hydroxyl (OH) group, to form an ester bond. In Farrugia's Examples 2 and 3, 1,9-nonanediol (a diol is a molecule with two alcohol functionalities, one at each end) is reacted with carboxylic acids on nanotube surface to produce ester bonds. In these examples, subsequent reaction with a second

monomer, 1,12-dodecanedioic acid (this is molecule with two carboxylic acid functionalities, one on each end), results in a polyester polymer.

271. Farrugia's original oxidation process, which produces the carboxylic acids on the CNT surface (*see* Farrugia's description of Example 1; LGES-1007 (Farrugia), [0089]-[0092]) also produces some hydroxyl groups on the surface of the nanotubes. This hydroxyl functionality on the nanotubes does not react with the first monomer in Examples 2 and 3, because the first monomer is another alcohol (1,9-nonanediol). Instead, it remains unreacted until the second monomer, the diacid (1,12-dodecanedioic acid) is added. The diacid then reacts with the hydroxyl surface functionality and produces additional ester bonds to the nanotube surface, initiating the growth of additional polyester polymer chains from the nanotube surface.

(vi) Claim 6

[6] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes are present in the range of 15% to about 25% by weight based on the total weight of the dispersion.

272. As I discussed above for claim element [1e], the nanotube/polymer composite includes "carbon nanotubes ***from about 2% to about 20% by weight*** of the composite." LGES-1007 (Farrugia), [0018]; *see*[0053]: ("The carbon nanotube content of the carbon nanotube/polymer composite is generally from about ***0.05% to about 20% by weig*****ht** of the composite, such

as from… about ***5% to about 15%***"), claim 4, claim 17. These range of weight percentages overlap with the range of weight percentages recited in claim [6].

273. The ranges quoted in Farrugia at these locations (LGES-1007 (Farrugia), [0018].[0053], claim 4, claim 17) are for the polymerized composite. As explained above for claim element [1e], the carbon nanotube concentrations in the unpolymerized mixtures are slightly lower. For instance, in Farrugia's Example 2, the carbon nanotube concentration in the unpolymerized mixture is 8.3% less than in the final cured composite (experimentally determined result). Likewise, in Farrugia's Example 3, the carbon nanotube concentration in the unpolymerized mixture is 4.1% less than in the final cured composite (experimentally determined result). Applying an adjustment in this approximate range of magnitude to the composition ranges given in LGES-1007 (Farrugia), [0018]; *see* [0053], claims 4, claims 17, results in nanotube concentrations in the unpolymerized mixture of functionalized nanotubes and monomer which still fall **in the range of 15% to about 25% by weight based on the total weight of the dispersion**. In particular, the range of ***from about 2% to about 20% by weight*** quoted by Farrugia (*see* [0018], [0053]) will still overlap with the '649 patent's claim 6 range when corrected for a unpolymerized mixture, which would be necessary to hit the

stated 2 to 20% range in the final cured polymer composite. Specifically, if the carbon nanotube concentration in the unpolymerized mixture is 4.1% less than in the final composite (as in Farrugia's Example 2) then the 20% upper end of the ranges taught by Farrugia becomes 19.2 %, which still falls within the 15 to 25% range of claim 6 of the '649 patent. If the carbon nanotube concentration in the unpolymerized mixture is 8.3% less than in the final composite (as in Farrugia's Example 3) then the 20% upper end of the ranges taught by Farrugia becomes 18.3%, which still falls within the 15 to 25% range of claim 6 of the '649 patent.

274. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes (either aqueous dispersions or nanocomposites) at weight percentages in the range of 15% to about 25%. The '649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

(vii) Claim 7

[7] The dispersion of claim 1 wherein the oxidized, discrete carbon nanotubes are present in the range of 18% to about 22% by weight based on the total weight of the dispersion.

275. As I discussed above for claim element [1e], the nanotube/polymer composite includes "carbon nanotubes ***from about 2% to about 20% by weight*** of the composite." LGES-1007 (Farrugia), [0018]; *see* [0053] ("The

carbon nanotube content of the carbon nanotube/polymer composite is generally from about ***0.05% to about 20% by weight*** of the composite"), claim 4, claim 17. This range of weight percentages overlap with the range of weight percentages recited in claim [7].

276. The range quoted in Farrugia at these locations (LGES-1007 (Farrugia), [0018].[0053], claim 4, claim 17) are for the polymerized composite. As explained above for claim element [1e], the carbon nanotube concentrations in the unpolymerized mixtures are slightly lower. For instance, in Farrugia's Example 2, the carbon nanotube concentration in the unpolymerized mixture is 8.3% less than in the final cured composite (experimentally determined result). Likewise, in Farrugia's Example 3, the carbon nanotube concentration in the unpolymerized mixture is 4.1% less than in the final cured composite (experimentally determined result). Applying an adjustment in this approximate range of magnitude to the composition ranges given in LGES-1007 (Farrugia), [0018]; *see* [0053], claims 4, claims 17, results in nanotube concentrations in the unpolymerized mixture of functionalized nanotubes and monomer which still fall **in the range of 18% to about 22% by weight based on the total weight of the dispersion**. In particular, if the carbon nanotube concentration in the unpolymerized mixture is 4.1% less than in the final composite (as in Farrugia's Example 2) then the 20% upper

end of the ranges taught by Farrugia becomes 19.2%, which still falls within the 18 to 22% range of claim 7 of the '649 patent. If the carbon nanotube concentration in the unpolymerized mixture is 8.3% less than in the final composite (as in Farrugia's Example 3) then the 20% upper end of the ranges taught by Farrugia becomes 18.3%, which still falls within the 18 to 22% range of claim 7 of the '649 patent.

277. I note that the '649 Patent does not describe particular unexpected results associated with dispersions of nanotubes (either aqueous dispersions or nanocomposites) at weight percentages in the range of 18% to about 22%. The'649 Patent also does not provide any discussion or evidence that these ranges of weight percentage are in any way critical.

(viii) Claim 8

[8] The dispersion of claim 1, wherein the additive is selected from the group consisting of dicarboxylic/tricarboxylic esters, timellitates, adipates, sebacates, maleates, glycols and polyethers, polymeric plasticizers, bio-based plasticizers, and mixtures thereof.

278. As I discussed above for claim element [1b], in the unpolymerized mixture of functionalized nanotubes and monomer, the excess first monomer is an **additive**. *See* LGES-1007 (Farrugia), [0045]. Farrugia identifies numerous example compounds for the first monomer, including "ethylene glycol, diethylene glycol, triethylene glycol, dipropylene glycol, tripropylene

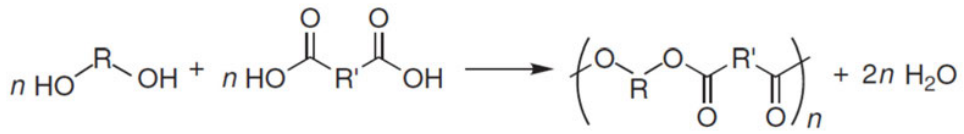
glycol." LGES-1007 (Farrugia), [0044]. These compounds are glycols; that is, the first monomer (*i.e.*, **the additive**) **is a glycol**.

279. As I also discussed above for claim element [1b], in the nanotube/polymer composite and in the toner, the polymer matrix is an **additive**. *See* LGES-1007 (Farrugia), [0020], [0027]-[0028], [0045]. Farrugia identifies numerous example compositions for the polymer matrix, including "poly(ethylene-sebacate), poly(propylene-sebacate), poly(butylene-sebacate), poly(pentylene-sebacate), poly(hexylene-sebacate), and poly(octylene-sebacate)," or "polyethylene-sebacate, polypropylene-sebacate, polybutylene-sebacate." LGES-1007 (Farrugia), [0049]-[0050]. These compositions are sebacates; that is, the polymer matrix (*i.e.*, **the additive**) can be **a sebacate**. Farrugia also identifies the following compositions as other examples of the polymer matrix: "poly(ethoxylated bisphenol co-maleate), poly(butyloxylated bisphenol co-maleate), poly(co-propoxylated bisphenol co-ethoxylated bisphenol co-maleate, poly(1,2-propylene maleate)." *Id.*, [0050]. These compositions are maleates; that is the polymer matrix (*i.e.*, **the additive**) can be **a maleate**.

280. Third, Farrugia's polymer matrix (*i.e.*, the additive) can be a polyester polymer that is produced by reaction of a diol and a dicarboxylic acid; this polyester polymer contains many repetitions of dicarboxylic esters in its

backbone. That is, the polymer matrix (*i.e.*, **the additive**) can include **dicarboxylic esters**. Specifically, Farrugia identifies "diols having from 2 to 36 carbons" as examples of compounds for the first monomer, and provides a list of example diols. LGES-1007 (Farrugia), [0043]-[0044]; *see* [0093], [0096] (describing the specific use of 1,9-nonanediol in its experimental examples). Farrugia also identifies "dicarboxylic acids" as examples of compounds for the second monomer. *Id.*, [0046]; *see* [0093], [0096] (describing the specific use of 1,12-dodecananedioic acid in its experimental examples). The reaction of a diol (the first monomer) with a dicarboxylic acid (the second monomer) produces a polyester polymer. *See* LGES-1047 (Hiemenz), 43, excerpted below:

1. Polyesters—successive reactions between diols and dicarboxylic acids:



See LGES-1007 (Farrugia), [0045] ("The functionalized carbon nanotubes covalently attached to a first monomer and any excess unreacted first monomer may be polymerized with a second monomer through the formation of ester or amide bonds"), [0048] ("The polymers formed from the polymerization of the second monomers may be … polyesters").

(ix) Claim 11

[11] The dispersion of claim 1, wherein the additive is a solvent.

281. As I discussed above for claim element [1b], in the unpolymerized mixture of functionalized nanotubes and monomer, the excess first monomer and the second monomer are examples of an **additive**. *See* LGES-1007 (Farrugia), [0045]. The excess first monomer and the second monomer (the **additive**) are molten in the mixture. For instance, Farrugia describes an experimental procedure in which nanotubes were "added to the ***molten 1,9-nonanedio*** [sic]" (a first monomer) and "well-dispersed in the diol." LGES-1007 (Farrugia), [0093]; *see* [0096]. Farrugia then describes that "***1,12-dodecanedioic acid***" (a second monomer) was added and "***melt[ed]***." *Id.*, [0093]; *see* [0096]. Thus, in the mixture, the functionalized nanotubes are dispersed in the liquid first monomer and the liquid second monomer. This means that each of the first monomer and the second monomer can be considered **a solvent** in this mixture, *i.e.*, the **additive** (the first or second monomer) **is a solvent**. There is nothing unusual about a solvent that can be a solid below its melting temperature. Water is a solid (ice) below its freezing point, but is a very common solvent at higher temperatures.

282. A state in the production of the composite exists in which the functionalized carbon nanotubes are dispersed in the excess first monomer and then the

second monomer is added, and at least in Farrugia's examples, this second monomer is a solid, prior to melting. In this case, the liquid first monomer is a solvent for the dispersion of nanotubes and the solid particles of second monomer. In this case, the first monomer is an additive, and that additive is a solvent.

283. Subsequently, in the production of Farrugia's composite as illustrated in the Examples, it is possible that reaction of the second monomer with the first monomer will begin to occur before the second monomer is completely melted. By the time the second monomer is completely melted, and the mixture of first and second monomers is completely fluid, some reaction of first and second monomer will have produced some oligomeric, prepolymer species. In this case the liquid, first and second monomers are solvents for the carbon nanotubes and any prepolymer that may exist. In this case, the first and second monomers are additives, and these additives are solvents.

(x) Claim 12

[12] The dispersion of claim 11, where in the solvent is selected from the group consisting of substituted or unsubstituted, halogenated or nonhalogenated hydrocarbons.

284. As I discussed above for claim [11], the excess first monomer is **a solvent**. Farrugia identifies "diols having from 2 to 36 carbons" as examples of compounds for the first monomer, and provides a list of example diols.

LGES-1007 (Farrugia), [0043]-[0044]. Farrugia also describes the specific use of 1,9-nonanediol in its experimental examples. *See id.*, [0093], [0096]. Diols are **substituted, nonhalogenated hydrocarbons**, as I discuss in the following paragraphs. Thus, when the first monomer is a diol, the solvent (*i.e.*, the molten first monomer) is a substituted, nonhalogenated hydrocarbon.

285. As I also discussed above for claim [11], the second monomer is also **a solvent**. Farrugia identifies "dicarboxylic acids" as examples of compounds for the second monomer, and provides a list of example dicarboxylic acids, including 1,4-cyclohexanedicarboxylic acid, 1,3-cyclohexanedicarboxylic acid, 1,12-dodecanedioic acid, among others. *See* LGES-1007 (Farrugia), [0046]. Farrugia also describes the specific use of 1,12-dodecanedioic acid in its experimental examples. *See id.*, [0093], [0096]. These dicarboxylic acids are **substituted, nonhalogenated hydrocarbons**, as I discuss in the following paragraphs. Thus, when the second monomer is a dicarboxylic acid, the solvent (*i.e.*, the molten second monomer) is a substituted, nonhalogenated hydrocarbon.

286. The International Union of Pure and Applied Chemistry (IUPAC) is an international body responsible for establishing standards in the naming of chemical compounds. According to these IUPAC standards, Farrugia's

"diols having from 2 to 36 carbons" are **substituted hydrocarbons**, and specifically are **substituted, nonhalogenated hydrocarbons**. *See* LGES-1032 (Skonieczny), 1633-1634. For instance, the particular first monomer used in Examples 2 and 3, 1,9-nonanediol, is a substituted hydrocarbon and a substituted, nonhalogenated hydrocarbon. Also according to these IUPAC standards, Farrugia's second monomers that are dicarboxylic acids [0046], such as 1,12-dodecananedioc acid used in Examples 2 and 3, are **substituted hydrocarbons**, and are **substituted, nonhalogenated hydrocarbons**.

287. As explained in Skonieczny, the naming process of organic compounds involves a number of steps. Step 1 is to "[d]etermine the principal functional group" in Table 1. LGES-1032 (Skonieczny), 1633. Table 1 includes hydroxyl (-OH), which is the functional group that makes an alcohol, and Table 1 also includes carboxylic acid (-COOH). *Id.*. Step 2 is to "[d]etemine the parent hydrocarbon." *Id.* In the case of first monomer 1,9-nonanediol, the parent hydrocarbon is *n-nonane*, which is a linear chain of nine carbon atoms completely saturated with hydrogens. It is a hydrocarbon. In the case of the second monomer, 1,12-dodecanedioc acid, the parent hydrocarbon is *n-dodecane*, which is a linear chain of twelve carbon atoms completely saturated with hydrogens. It is a hydrocarbon.

288. Subsequent steps in the naming process involve establishing a numbering scheme for the parent hydrocarbon, which is used to specify the locations on the parent hydrocarbon at which the functional groups, referred to as "substituents," are substituted for one or more of the hydrogen atoms of the parent hydrocarbon. LGES-1032 (Skonieczny), 1634. The numbers specifying the locations of the ***substituents*** are then used in the name to indicate where the substitutions have occurred. For instance, the name 1,9-nonanediol, tells a POSITA that it is a hydrocarbon chain of nine carbons that is substituted with two alcohols (diol means two alcohols) at positions 1 and 9, which are the two ends of the nine carbon chain. Likewise, the name 1,12-dodecananedioc acid, tells a POSITA that it is a hydrocarbon chain of 12 carbons that is substituted with two carboxylic acid functionalities at positions 1 and 12, which are the two ends of the 12 carbon chain. Similar observations apply to the other diols and diacids listed as first and second monomers by Farrugia. *See*, *e.g.*, LGES-1007 (Farrugia), [0043]-[0044], [0046]-[0047]

289. When the substituents are not halogens (F, Cl, Br, I), then the substituted hydrocarbon is ***a substituted, nonhalogenated hydrocarbon***. This is the case for 1,9-nonanediol, 1,12-dodecananedioc acid and the other diols and diacids listed as first and second monomers by Farrugia.

(xi) Claim 13

[13] The dispersion of claim 1, wherein the additive comprises at least one reactive species capable of creating a thermoset polymer.

The unpolymerized mixture of functionalized nanotubes and monomer includes trifunctional diacids or trifunctional diols

290. As I discussed above for claim element [1b], in the unpolymerized mixture of functionalized nanotubes and monomer, the second monomer is an example of an **additive**. *See* LGES-1007 (Farrugia), [0045]. As I discuss in the following paragraphs, many of the compounds Farrugia identifies as examples of the second monomer are **reactive species capable of creating a thermoset polymer**.

291. First, Farrugia identifies sulfonated diacids (or equivalents such as sulfonated phthalic acids, sulfonated phthalates, or sulfonated acid anhydrides) as examples of second monomers. *See* LGES-1007 (Farrugia), [0046]-[0047]. Examples of these types of compounds include dimethyl-5-sulfo-isophthalate, dialkyl-5-sulfo-isophthalate, 4-sulfo-18-naphthalic anhydride, 4-sulfo-phthalic acid, dimethyl-4-sulfo-phthalate, dialkyl-4-sulfo-phthalate, sulfo-terephthalic acid, dimethyl-sulfo-terephthalate, 5-sulfo-isophthalic acid, dialkyl-sulfo-terephthalate. *See id.*. Second, Farrugia identifies sulfonated diols as examples of second monomers. *See id.*. Specific examples of sulfonated diols include "sulfoethanediol, 2-

sulfopropanediol, 2-sulfobutanediol, 3-sulfopentanediol, 2-sulfohexanediol, 3-sulfo-2-methyl-pentanediol, [and] 2-sulfo-3,3-dimethylpentanediol." *Id.*, [0047].

292. These types of second monomers (*i.e.*, diacids, diacid equivalents, or diols) are reactive species that are capable of reacting to form polymers. For instance, diacids (or diacid equivalents) can react with diols to form polyesters, as in Farrugia Examples 2 and 3. Additionally, diols can react with diisocyanates to form polyurethanes or with di-epoxides, such as diglycidyl ether of bisphenol A, to form epoxies.

293. Additionally, these types of second monomers are sulfonated compounds that contain a sulfonate group, which provides a third reactive site on each monomer (*i.e.*, the monomer is trifunctional). These sulfonate groups can be reacted to crosslink the polymer, thereby producing a crosslinked polymer such as a crosslinked polyester, a crosslinked polyurethane, or a crosslinked epoxy. A crosslinked polymer is a thermoset polymer. (Indeed, I note that the'649 Patent acknowledges that polyurethane is a thermoset polymer. *See* LGES-1001, 7:55-56, claim 14.) Thus, when Farrugia's second monomer (the additive in the unpolymerized mixture of functionalized nanotubes and monomer) is a trifunctional monomer such as a sulfonated diacid, sulfonated diacid equivalent, or sulfonated diol, (*see* LGES-1007 (Farrugia), [0046]-

[0047]), **the additive includes a reactive species capable of creating a thermoset polymer**.

294. Furthermore, I note that Farrugia specifies that the second monomer may be a mixture of monomers. *See* LGES-1007 (Farrugia), [0047]. It is my opinion that a POSITA would have understood that he could mix a certain amount of a sulfonated, trifunctional (*i.e.*, crosslink promoting) monomer with a difunctional monomer to tune the degree of crosslinking that would be achieved.

295. I also discussed above for claim element [1b] that the first monomer is an example of **an additive** in the unpolymerized mixture of functionalized nanotubes and monomer. *See* LGES-1007 (Farrugia), [0045]. As I discuss in the following paragraphs, many of the compounds Farrugia identifies as examples of the first monomer are **reactive species capable of creating a thermoset polymer**.

296. Farrugia's first monomer can be a trifunctional sulfonated diol, such as 2-sulfo-1,3-propanediol. *See* LGES-1007 (Farrugia), [0043]-[0044]. As I noted above, this is a trifunctional monomer that is capable of reacting to create a crosslinked, thermoset polymer.

297. Farrugia's first monomer also can be a non-sulfonated, difunctional diol. *See* LGES-1007 (Farrugia), [0043]-[0044] ("Suitable first monomers …

include, for example, diols having from 2 to 36 carbons"). These compounds are **a reactive species capable of creating a thermoset polymer** if the second monomer is suitable trifunctional monomer, such as the sulfonated diacids, sulfonated diacid equivalents, or sulfonated diols discussed above.

(xii) Claim 14

[14] The dispersion of claim 13, wherein the thermoset polymer is selected from epoxy, polyurethane and silicone.

298. When the second monomer is a suitable multifunctional isocyanate, the thermoset polymer that results from the polymerization reaction can be a thermoset **polyurethane**. *See* LGES-1007 (Farrugia), [0048]. For instance, a thermoset polyurethane is generated when the first monomer is sulfonated diol and the second monomer is a diisocyanate, or the first monomer is a unsulfonated diol and the second monomer is a trifunctional (or greater functionality) isocyanate. Farrugia teaches just this type of first monomer and second monomer reaction to form a polyurethane: "the polyurethane of isocyanate and alcohol, the polyurethane of phenyl isocyanate with ethanol, the polyurethane of toluene diisocyanate and ethylene glycol" *See* LGES-1007 (Farrugia), [0048].

(xiii) Claim 15

[15] The dispersion of claim 1, wherein the additive comprises natural wax, synthetic wax, or mixtures thereof.

299. As I discussed above for claim element [1b], **the additive** in the toner is a wax, such as "polyolefin waxes, such as low molecular weight polyethylene, polypropylene, a fluorocarbon-based wax (Teflon), or Fischer-Tropsch wax, copolymers thereof, mixtures thereof, and the like." LGES-1007 (Farrugia), [0071]. Each of these waxes is **a synthetic wax**.

(xiv) Claim 17

[17] The dispersion of claim 1 which further comprises at least one filler.

300. Farrugia describes that the toner can include "***colorants***," such as "pigment, dye, mixtures of pigment and dye, mixtures of pigments, mixtures of dyes, and the like." LGES-1007 (Farrugia), [0068]. Example colorants include "carbon black, lamp black, iron black, ultramarine," or a variety of other colorants listed by Farrugia. *Id.*, [0069]; *see* [0106] ("carbon black toner containing crystalline polyester resins and various MWNT loadings"). Any of these example colorants can be considered **a filler** in the toner.

301. Further discussion of colorants is provided at the following citations: *id.*, Abstract ("A toner comprising: a polymer matrix comprising carbon nanotubes dispersed in a polymer; optionally ***one or more colorants***"), [0057] ("In embodiments, the toner process comprises forming a toner

particle by mixing the carbon nanotube/polymer composite, an optional wax, an optional colorant dispersion, and an optional coagulant while blending at high speeds"), [0068]-[0070] ("The toner compositions may be produced as a colored toner by adding a colorant during toner production.", [0019], [0055] claims 1 and 11.

302. Farrugia also describes the use of another **filler** in its toner: silica (*i.e.*, glass particles): "suitable additives include a fluidization agent such as colloidal silica … [or] silica." LGES-1007 (Farrugia), [0084].

(xv) Claim 18

[18] The dispersion of claim 17, wherein the filler has at least one dimension less than about 20 microns.

303. As I discussed for claim [17], the filler can be carbon black. *See* LGES-1007 (Farrugia), [0069]. Carbon black particles used for pigment were well-known to have dimensions of less than about 20 microns. *See* LGES-1033 (Shimomura), 2:29-35 ("carbon black having a primary particle size of 5 nm or more and 40 nm or less"); LGES-1034 (Mathias), 2:3-6 ("The carbon black used may preferably be a pigment carbon black with an average particle diameter of 8 to 80 nm, preferably 10 to 35 nm"); LGES-1036 (Miyabe), 11:10-25 (identifying carbon black as a black pigment, which "is preferred to have an average particle diameter of no more than 20 μm, preferably not more than 5 μm"); LGES-1037 (Koike), 8:32-40 ("In the

example of the pigment available in the present invention, carbon black is suitable as the pigment used in the black ink. … The preferably used carbon black pigment has a primary particle diameter of 15 to 40 nm"). Given that the standard size range for carbon black was well-established, it is my opinion that a POSITA would have understood or found obvious that the carbon black (*i.e.*, the filler) in the toner **has at least one dimension less than about 20 microns**.

304. As I also discussed for claim [17], the filler can be lamp black. *See* LGES-1007 (Farrugia), [0069]. Lamp black particles used for pigment were well-known to have dimensions of less than about 20 microns. *See* LGES-1033 (Shimomura), 2:29-35 ("Examples of the black pigment include carbon black pigments such as … lamp black … Among the carbon black pigments described above, carbon black having a primary particle size of 5 nm or more and 40 nm or less"); LGES-1034 (Mathias), 1:63-2:6 ("The carbon black used may be … lamp black … The carbon black used may preferably be a pigment carbon black with an average particle diameter of 8 to 80 nm, preferably 10 to 35 nm"); LGES-1036 (Miyabe), 11:10-25 (identifying lamp black as a black pigment, which "is preferred to have an average particle diameter of no more than 20 μm, preferably not more than 5 μm"); LGES-1037 (Koike), 8:32-40 ("In the example of the pigment available in the

present invention, carbon black is suitable as the pigment used in the black ink. Specific examples of the carbon black pigment include … lamp black … The preferably used carbon black pigment has a primary particle diameter of 15 to 40 nm"). Given that the standard size range for lamp black pigment particles was well-established, it is my opinion that a POSITA would have understood or found obvious that lamp black (*i.e.*, the filler) in the toner **has at least one dimension less than about 20 microns**.

305. I also discussed for claim [17] that the filler in the toner can be ultramarine. *See* LGES-1007 (Farrugia), [0069]. Ultramarine particles used for pigment were well-known to have dimensions of less than about 20 microns. *See* LGES-1038 (Itou), 5:48-49 ("ultramarine colored pigments, 0.01 μm diameter"); LGES-1039 (Hasegawa), 3:33-48 ("The pigment or extender pigment in the present invention embraces inorganic pigments" such as ultramarine. "Preferred among these examples are fine particles or superfine particles (smaller than 1 μm in diameter) of titanium oxide and color pigments (such as … ultramarine…)"). Given that the standard size range of ultramarine particles for pigment was well-established, it is my opinion that a POSITA would have understood or found obvious that ultramarine (*i.e.*, the filler) in the toner **has at least one dimension less than about 20 microns**.

(xvi) Claim 19

[19] The dispersion of claim 18, wherein the filler is selected from silicon, lead, lead derivatives, carbon black, graphite, graphene, graphene oxides, paramagnetic particles and mixtures thereof.

306. As I discussed in claim [17], the filler in Farrugia's toner is a colorant such as **carbon black**. *See* LGES-1007 (Farrugia), [0069].

(xvii) Claim 21

[21] The dispersion of claim 15 which further comprises a crosslinking agent.

307. Farrugia identifies "diols" as examples of first monomers and diacids, such as "dicarboxylic acids" as examples of second monomers. LGES-1007 (Farrugia), [0044]-[0047]. A polymerization reaction between diols and dicarboxylic acids produces a polymer, such as a polyester polymer, as I discussed above for claim [8]. *See* LGES-1047 (Hiemenz), 43. When the diols, dicarboxylic acids, or both, contain a sulfonate group as a third functional site (*see* my discussion of claim [13]), these trifunctional monomers act as **crosslinking agents** during the polymerization reaction. That is, the sulfonate can react with and link to an alcohol, which is present on the diols. Some of the diols and dicarboxylic acids identified as Farrugia's first and second monomers, respectively, contain sulfonate groups and thus act as **cross linking agents**. For instance, the first monomer "2-sulfo-1,3-propanediol" and the second monomer "4-sulfo-phthalic acid"

each includes a sulfonate functionality, which is capable of acting as a crosslinking agent. LGES-1007 (Farrugia), [0044], [0047].

308. Farrugia also identifies "crosslinked poly(styrene-acrylate) resins, … crosslinked poly(styrene-methacrylate) resins, … crosslinked poly(styrene-butadiene) resins," and other crosslinked resins, as examples of the polymers in its nanotube/polymer composite. LGES-1007 (Farrugia), [0050]. A POSITA would have understood or found obvious that one way for these crosslinked resins to be produced is for the dispersion (*e.g.*, the unpolymerized mixture of functionalized nanotubes and monomer) to include a **crosslinking agent** in order to facilitate creation of these crosslinked resins.

F. [GROUND 2B] – CLAIMS 22-23 ARE OBVIOUS OVER FARRUGIA, NARAYAN, AND NANYA

309. It is my opinion that a POSITA would have found 22-23 to be obvious over the combination of Farrugia, Narayan, and Nanya, as I discuss in the following sections.

1. Nanya describes toners including lubricants

310. Nanya describes "a dry-type toner for use in electrophotography and electrostatic printing," and specifically a "toner which exhibits high fixing ability even at low image-fixing temperatures." LGES-1009 (Nanya), 1:8-12. Nanya's toner includes "a binder resin, a coloring agent, and a carnauba

wax serving as a lubricant or releasant, which is substantially free of free aliphatic acids." *Id.*, 2:30-34. The use of such a carnauba wax is advantageous in that it can "be much better dispersed in the binder resin than the conventional carnauba wax." *Id.*, 2:46-52. As a result, "a carrier does not adhere to the toner," and "the toner exhibits high resistance to both the off-set and the winding phenomena." *Id.*, 2:52-57.

311. The binder resin can be a "thermoplastic resin[]." LGES-1009 (Nanya), 2:66-67. Nanya provides various "examples of the thermoplastic resins usable in the present invention," including, *e.g.*, "a styrene-based resin" having specific characteristics, or "a polyester resin," because "they can impart to the toner high fixing ability and high resistance to the winding phenomenon." *Id.*, 2:66-3:37. The toner also can include various "auxiliary components," including "an electro-conductivity-imparting agent such as carbon black or tin oxide." *Id.*, 4:8-16.

2. The Farrugia-Narayan-Nanya combination

312. The Farrugia-Narayan-Nanya combination uses Farrugia's toner (with nanotubes dimensioned as described by Narayan, as I discussed above) as the starting point. Following the teachings of Nanya, Farrugia's toner is modified to include carnauba wax, such as "[a] conventionally employed carnauba wax" or "a carnauba wax … substantially free of free aliphatic

acids." LGES-1009 (Nanya), 2:38-45; *see* LGES-1007 (Farrugia), [0055], [0071]. Specifically, I rely on Nanya as teaching that either "conventionally employed" carnauba waxes or the specific carnauba waxes ("substantially free of aliphatic acids") discussed by Nanya can be advantageously incorporated into toners. LGES-1009 (Nanya), 2:38-45. I note that Farrugia already describes that waxes can be included in the toner particles. *See* LGES-1007 (Farrugia), [0071]. In the Farrugia-Narayan-Nanya combination, carnauba wax is included either in addition to or instead of the waxes listed by Farrugia.

3. Reasons for the combination

313. It is my opinion that a POSITA would have been motivated to use carnauba wax in the toner of the combination for at least the four reasons I discuss in the following paragraphs.

314. <u>The first reason</u> why a POSITA would have been motivated to use carnauba wax is so that the carnauba wax in the toner would "impart to [the] toner … high resistance to the off-set phenomenon." LGES-1009 (Nanya), 1:61-63. This advantage applies whether the toner is modified to include "conventionally employed" carnauba waxes or the specific carnauba waxes ("substantially free of aliphatic acids") discussed by Nanya. LGES-1009 (Nanya), 2:38-45.

315. The second reason why a POSITA would have been motivated to use carnauba wax is so that the toner of the combination would have "excellent fixing ability at low temperatures." LGES-1009 (Nanya), 1:61-64. This advantage applies whether the toner is modified to include "conventionally employed" carnauba waxes or the specific carnauba waxes ("substantially free of aliphatic acids") discussed by Nanya. LGES-1009 (Nanya), 2:38-45; *see* LGES-1028 (Aga), [0107] ("In the case where a polymerized toner particle … comprises a fixability improving agent, the fixability improving agent usable is natural waxes such as carnauba wax").

316. The third reason why a POSITA would have been motivated to use carnauba wax relates specifically to carnauba waxes that are free of aliphatic acids. It is my opinion that a POSITA would have been motivated to use these specific carnauba waxes to obtain their specific advantages in the toner of the combination: "high resistance to both the off-set and the winding phenomena," a toner that is "free from … filming problem[s]," and a toner to which "a carrier does not adhere." LGES-1009 (Nanya), 2:46-57.

317. The fourth reason why a POSITA would have been prompted to use carnauba wax is because this combination is just the implementation of known techniques (specifically, carnauba wax as an additive in a toner) to an already-known structure (the toner of the Farrugia-Narayan combination),

and it produces predictable results (specifically, toner including carnauba wax).

318. It is my opinion that a POSITA would have expected success when incorporating carnauba wax into the toner of the combination for at least the three reasons I discuss in the following paragraphs.

319. The first reason why a POSITA would have expected success is because Farrugia already indicates that its toners can include wax. *See* LGES-1007 (Farrugia), [0071]. Additionally, while Farrugia identifies certain example waxes that can be used in its toner, this list is open-ended, which indicates the possibility that other waxes not included on the list can also be incorporated successfully into the toner. *See id.*

320. The second reason why a POSITA would have expected success is that Nanya's materials system is similar to the materials system of Farrugia's toner. Nanya's toner includes a binder resin, which can be, for instance, thermoplastic resins such as styrene-based resins and polyester resins. LGES-1009 (Nanya), 3:1-38. This composition of the resin matrix is consistent with the polymer of the nanotube/polymer composite that is used in Farrugia's toner (which can be, *e.g.*, polycarbonates, polyamides, polyesters and polyurethanes). *See* LGES-1007 (Farrugia), [0048]-[0052]. Farugia's toner can also include styrene-based resins [0050]. I also note that

both Nanya's and Farrugia's toners include conductive components—*e.g.*, "an electro-conductivity-imparting agent such as carbon black or tin oxide" in Nanya's toner and nanotubes for providing "increased conductivity" in Farrugia's toner. LGES-1009 (Nanya), 4:14-15; LGES-1007 (Farrugia), [0018]. It is my opinion that this overlap in materials systems is an indication that a POSITA would have excepted to be successful in using carnauba wax as taught by Nanya in Farrugia's toner. That is, Nanya demonstrates that carnauba wax can be used in toners having compositions similar to those described by Farrugia.

321. The third reason why a POSITA would have expected success is that the use of carnauba wax in toners was well-known. *See* LGES-1009 (Nanya), 2:38-39 (describing "conventionally employed carnauba wax"); LGES-1028 (Aga), [0107].

322. The fourth reason why a POSITA would have expected success is that both Farrugia and Nanya relate generally to toners. *See* LGES-1007 (Farrugia), [0018]-[0019]; LGES-1009 (Nanya), 2:15-34.

4. Analysis

(i) Claim 22

[22] The dispersion of claim 15 wherein the additive comprises natural wax wherein the natural wax is plant derived.

323. As I discussed above, in the Farrugia-Narayan-Nanya combination, the toner includes carnauba wax, *e.g.*, either "[a] conventionally employed carnauba wax" or "a carnauba wax … substantially free of free aliphatic acids." LGES-1009 (Nanya), 2:38-45. The carnauba wax can be included along with one or more of the waxes identified by Farrugia, or as an alternative in place of those waxes. *See* LGES-1007 (Farrugia), [0071]. Carnauba wax is **a natural wax**. *See* LGES-1011 (Rasmussen), [0051] (identifying "carnauba wax" as "a ***plant-based*** antimoisture agent"); LGES-1028 (Aga), [0107] ("natural waxes such as carnauba wax").

(ii)Claim 23

[23] The dispersion of claim 22 which further comprises a crosslinking agent.

324. I refer to my discussion of claim [21].

VI. LEGAL PRINCIPLES

A. Anticipation

325. I have been informed that a patent claim is invalid as anticipated under 35 U.S.C. § 102 if each and every element of a claim, as properly construed, is found either explicitly or inherently in a single prior art reference. Under

the principles of inherency, if the prior art necessarily functions in accordance with, or includes the claimed limitations, it anticipates.

326. I have been informed that a claim is invalid under 35 U.S.C. § 102(a) if the claimed invention was known or used by others in the U.S., or was patented or published anywhere, before the applicant's invention. I further have been informed that a claim is invalid under 35 U.S.C. § 102(b) if the invention was patented or published anywhere, or was in public use, on sale, or offered for sale in this country, more than one year prior to the filing date of the patent application (critical date). And a claim is invalid, as I have been informed, under 35 U.S.C. § 102(e), if an invention described by that claim was described in a U.S. patent granted on an application for a patent by another that was filed in the U.S. before the date of invention for such a claim.

B. Obviousness

327. I have been informed that a patent claim is invalid as "obvious" under 35 U.S.C. § 103 in light of one or more prior art references if it would have been obvious to a POSITA, taking into account (1) the scope and content of the prior art, (2) the differences between the prior art and the claims, (3) the level of ordinary skill in the art, and (4) any so called "secondary considerations" of non-obviousness, which include: (i) "long felt need" for

the claimed invention, (ii) commercial success attributable to the claimed invention, (iii) unexpected results of the claimed invention, and (iv) "copying" of the claimed invention by others.

328. I have been informed that a claim can be obvious in light of a single prior art reference or multiple prior art references. To be obvious in light of a single prior art reference or multiple prior art references, there must be a reason to modify the single prior art reference, or combine two or more references, in order to achieve the claimed invention. This reason may come from a teaching, suggestion, or motivation to combine, or may come from the reference or references themselves, the knowledge or "common sense" of one skilled in the art, or from the nature of the problem to be solved, and may be explicit or implicit from the prior art as a whole. I have been informed that the combination of familiar elements according to known methods is likely to be obvious when it does no more than yield predictable results. I also understand it is improper to rely on hindsight in making the obviousness determination.

VII. ADDITIONAL REMARKS

329. I currently hold the opinions set expressed in this declaration. But my analysis may continue, and I may acquire additional information and/or attain supplemental insights that may result in added observations.

330. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code.

Dated: June 7, 2024 By: _____

APPENDIX A

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Professional Experience

1999-Present
Associate Professor of Polymer Science and Engineering – University of Massachusetts, Amherst, MA

2006-2014
Associate Director for Facilities, Materials Research Science and Engineering Center (MRSEC) on Polymers at the University of Massachusetts Amherst.

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2006-2010
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2000-2006
Vice President, Smart Surfaces, LLC. 600 Snow Goose Ln., Annapolis, Maryland.

2000-2005
Director and Principal Investigator: U.S. Army Research Laboratory-University of Massachusetts Collaborative Polymer Materials Center of Excellence.

1999-2002
Graduate Program Director, Polymer Science and Engineering Department – University of Massachusetts, Amherst, MA

1994-2010
Director of the W. M. Keck Electron Microscopy Laboratory – University of Massachusetts, Amherst, MA

1993-1998
Assistant Professor of Polymer Science and Engineering – University of Massachusetts, Amherst, MA

1989-1992
Graduate Research Assistant, Department of Materials Science and Engineering – Massachusetts Institute of Technology, Cambridge, MA

1987-1988
Research Fellow, Textile Research Institute – Princeton University, 601 Prospect Avenue, Princeton, NJ

1987 Research Intern – Separations & Purifications Research - Dow Chemical, U. S. A., Michigan Division, Midland MI

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1993 *Ph. D.* Massachusetts Institute of Technology, Cambridge, MA
Chemical Engineering and Polymer Science and Technology
Thesis Advisor: Professor Edwin L. Thomas – Materials Science & Engineering Department

1988 *B.S.E. – Magna Cum Laude,* Princeton University, Princeton, NJ, Chemical Engineering

Academic Honors

University of Massachusetts

- The Mettler Toledo Thermal Analysis Education Award in Honor of Prof. Edith A. Turi. (Shared with E. B. Coughlin), 2002.
- The OMNOVA Solutions Foundation, OMNOVA Solutions Signature University Award for Research, 2000
- NSF Faculty Early Career Development (CAREER) Award, 1996
- Army Young Investigator Award, 1995.
- W. M. Keck Foundation Award, 1994

Graduate School

- Materials Research Society (MRS) Graduate Student Award Winner, 1991
- Sigma Xi, National Scientific Research Honor Society, 1990
- Austin Endowed Fellowship, sponsored by Dow Chemical, 1988

Undergraduate

- Tau Beta Pi National Engineering Honor Society, Princeton University Chapter, 1986
- Gilbert Solar Scholarship of the American Society for Metals, 1984
- Carl J. Long Science Award & Scholarship of the Pennsylvania Air Force Association, 1984

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21. Ambrose, Stephen D. (Longmont CO, US); Gido, Samuel P. (Hadley MA, US) "AUDIO DEVICE, SYSTEM AND METHOD" WIPO Patent Application WO/2012/061594 A2 · Filed: 11/03/2011 · Published: 05/10/2012

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23. Ambrose, Stephen D. (Longmont CO, US); Gido, Samuel P. (Hadley MA, US) "AUDIO DEVICE, SYSTEM AND METHOD" European Patent Application EP2636225 A2 · Filed: 11/03/2011 · Published: 09/11/2013

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28. Mays, Jimmy W. (Knoxville, TN, US); Gido, Samuel P. (Hadley, MA, US); Weidisch, Roland (Halle, DE) "Multigraft Copolymers as Superelastomers" United States Patent Application 20140161858 A1 · Filed: 09/30/2011 · Published: 06/12/2014

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3. P. Jeanene Willcox and Samuel P. Gido, "Cholesteric Liquid Crystalline Intermediate in Natural Silk Spinning Processes Resulting from the Chirality of Protein Secondary Structure," *ACS Polymer Preprints* **1996**, *37(2)*, 484.

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5. Jennifer L. David, Samuel P. Gido and Bruce M. Novak, "Synthesis and Characterization of Rodcoil Diblocks Comprised of Thermally Labile Polyguanidine and Polystryene Segments" *Abstracts of Papers of the American Chemical Society* 216: U26-U26, 085-POLY Part 3, Aug. 23, 1998.

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10. R. L. McKiernan, A. M. Heintz, S. L. Hsu, S. P. Gido and J. Penelle, "From Aliphatic Polyurethanes to Linear Polyethylene: Influence of Hydrogen Bonding on the Morphology of Semi-Crystalline Polymers," *Polymeric Materials: Science & Engineering* **2001**, *84*, 416.

11. X. D. Wu, S. Hong, T. P. Russell, S. P. Gido, S. Pispas, N. Hadjichristidis. "Morphological Studies of a Semicrystalline Diblock Copolymer." *Abstracts of Papers of the American Chemical Society* 224: U453-U453 552-Poly Part 2, Aug. 18, 2002.

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13. Mays, Jimmy W.; Huang, Tianzi; Gido, Samuel P. "Synthesis and morphology of fluorinated and sulfonated block copolymers" *Abstracts of Papers of the American Chemical Society* 231: 109-PMSE MAR 26 2006

14. Cardoen, G.; Mays, J.W.; Gido, S.P.; Russell, T.P.; Hu, X.C.; Baskaran, D.; Coughlin, E.B. "POSS containing multiblock copolymers as model of well-defined organicinorganic hybrid materials" *Abstracts of Papers of the American Chemical Society* 230: U4013-U4014 31-POLY AUG 28 2005

15. Chen, S.J.; Gido, S.P.; Tsoukatos, T; Avgeropoulos, A; Hadjichristidis, N; Hong, K.L., Mays, J.W.. "Defects in a noncentrosymmetric lamellar block copolymer blend" *Abstracts of Papers of the American Chemical Society* 230: U4123-U4124 271-POLY AUG 28 2005.

16. Lin, Ying ; Daga, Vikram K.; Anderson, Eric A. ; Gido, Samuel P.; Watkins, James J. "High volume production of well-ordered hybrid materials for device applications" *Abstracts of Papers of the American Chemical Society* Volume: 242 Meeting Abstract: 37-PMSE Published: AUG 28 2011

17. Gido, Samuel P. "Control of block copolymer morphology and properties through molecular architecture variation" *Abstracts of Papers of the American Chemical Society* Volume: 242 Meeting Abstract: 487-POLY Published: AUG 28 2011.

18. Gido, Samuel P.; Schulein, Robert, B.; Ambrose, Stephen D. "Sound Reproduction within a Closed Ear Canal: Acoustical and Physiological Effects" *Journal of the Audio Engineering Society*, 8319, 2011. http://www.aes.org/e-lib/browse.cfm?elib=15786

19. Ambrose, Stephen D.; Schulein, Robert, B.; Gido, Samuel P. "Diaphonic Pump: A Sound Actuated Alternating to Static Pressure Converter" *Journal of the Audio Engineering Society*, 8361, 2011. http://www.aes.org/e-lib/browse.cfm?elib=15828

20. Gido, Samuel P. "Grain structure development and influence on block copolymer transport and mechanical properties" *Abstracts of Papers of the American Chemical Society* Volume: 243. Meeting Abstract: 50-PMSE Published: MAR 25 2012.

21. "Energy filtered TEM for element specific imaging of new poly(isoprene-b-benzofulvene) block copolymers" Samuel Gido. *Abstracts of Papers of the American Chemical Society*. Volume: 249 Meeting Abstract: 430. Published: MAR 22 2015

22. "Characterization of novel high temperature thermoplastic elastomers polybenzofulvene-block-polyisoprene-block-polybenzofulvene" Mays, J; Wang, WY; White, T; Kang, NG; Hong, KL; Schlegel, R; Beiner, M; Williams, K; Gido, S. *Abstracts of Papers of the American Chemical Society*. Volume: 249 Meeting Abstract: 37. Published: MAR 22 2015

Invited Lectures

1. Samuel P. Gido "Control of Morphology and Properties Through Control of Molecular Architecture in Block and Graft Copolymers" Department of Chemistry, University of Massachusetts Lowell. October 1, 2020.

2. "Control of Block Copolymer Morphology and Properties Through Molecular Architecture Variation", Samuel P. Gido and Jimmy Mays, Fraunhofer-Institut für Werkstoffmechanik, Halle, Germany, August 7, 2014.

3. "Effect of Macromolecular Architecture on Morphology and Mechanical Properties of Block Copolymers", J. W. Mays, S. Gido, and R. Weidisch, invited presentation at Goodyear Tire & Rubber Company, May 30, 2014, Akron, OH.

4. "Effect of Macromolecular Architecture on Morphology and Mechanical Properties of Block Copolymers", J. W. Mays, S. Gido, and R. Weidisch, invited presentation at Kraton Polymers, June 3, 2014, Houston, TX.

5. "Optimization of Macromolecular Architecture in Styrene/Diene Block Copolymer Thermoplastic Elastomers", J. W. Mays and Samuel P. Gido, Case Western Reserve University, March 8, 2013.

6. “Optimization of Macromolecular Architecture in Styrene/Diene Block Copolyme Thermoplastic Elastomers”, Samuel P. Gido and Jimmy Mays, Fraunhofer-Institut für Werkstoffmechanik, Halle, Germany, July 11, 2013.

7. “Optimization of Macromolecular Architecture in Styrene/Diene Block Copolymer Thermoplastic Elastomers”, J. W. Mays, S. Gido, R. Weidisch. DYNACOP Final Conference on Dynamics of Architecturally Complex Polymers: Current and Future Trends, Leeds, England, December 12-14, 2012.

8. Samuel P. Gido “Characterization of Bicontinuous Morphologies in Silicone Hydrogel Contact Lenses” CooperVision Inc., Pleasanton, California. March 23, 2012

9. Samuel P. Gido. “Grain structure development and influence on block copolymer transport and mechanical properties” ACS Polymer Award: Symposium in Honor of Edwin L. Thomas. 243rd ACS National Meeting, San Diego, California. PMSE: Division of Polymeric Materials Science and Engineering, Sunday, March 25, 2012

10. Samuel P. Gido “Control of block copolymer morphology and properties through molecular architecture variation.” American Chemical Society, National Meeting, Denver Colorado, August 31, 2011. POLY: Division of Polymer Chemistry. Mark Senior Scholar Award: Symposium in Honor of Jimmy Mays.

11. Ying Lin, Vikram K. Daga, Eric A. Anderson, Samuel P. Gido, James J. Watkins “High volume production of well-ordered hybrid materials for device applications” American Chemical Society, National Meeting, Denver Colorado, August 28, 2011. PMSE: Division of Polymeric Materials: Science and Engineering

12. Gido, Samuel P.; Schulein, Robert, B.; Ambrose, Stephen D. “Sound Reproduction within a Closed Ear Canal: Acoustical and Physiological Effects” *Audio Engineering Society 130th Convention, 2011 May 13–16, London, UK*

13. Ambrose, Stephen D.; Schulein, Robert, B.; Gido, Samuel P. “Diaphonic Pump: A Sound Actuated Alternating to Static Pressure Converter” *Audio Engineering Society 130th Convention, 2011 May 13–16, London, UK*.

14. “Defects, Grain Growth, and Long Range Order in Block Copolymer Nanostructures” Massachusetts Institute of Technology, Program in Polymer Science & Technology Seminar. March 10, 2010. Samuel P. Gido

15. "Morphology of Silicone Hydrogels Materials" Samuel P. Gido. March 26, 2009. CIBA Vision R&D, Duluth, Georgia

16. "Next Generation Foul Release Materials" Samuel P. Gido. October 10, 2008. FUJIFILM, Tokyo, Japan

17. “Branched Block Copolymers: Morphology, Mechanical Properties and Applications” Symposium in honor of Prof. T. Kajiyama, University of Massachusetts. April 2007. Samuel P. Gido

18. “Phase Behavior of Multicomponent Polymer Systems for Nanostructured Applications” National Starch, Bridgewater, NJ August 2, 2007, Samuel P. Gido.

19. American Physical Society National Meeting, Denver, Colorado, March 2007: Session L24: John H. Dillon Award Symposium in Honor of Darrin Pochan, 2:30 PM–5:30 PM, Tuesday, March 6, 2007, Colorado Convention Center – 201, Sponsoring Unit: DPOLY Abstract: L24.00002 : "Morphological Characterization of Silicone Hydrogels"

20. "Advanced Silicone Foul Release Coatings" International Workboat Show. New Orleans, LA November 30, 2006. Samuel P. Gido

21. International Symposium on Polymer Analysis and Characterization (ISPAC), Oak Ridge, Tennessee, on June 12-14, 2006 *Characterization of Bicontinuous Morphologies in Silicone Hydrogels*.

22. Center for Nanophase Materials Science (CNMS), Oak Ridge National Laboratory, Annual User Meeting, June 15, 2006. *Star and Graft Copolymers of Precisely Controlled Molecular Architecture, Morphology, and Properties.*

23. S. P. Gido and S. Chen, J. Mays, N. Hadjichristidis. Pacifichem 2005 Honolulu, Hawaii, Symposium 157 "Precise Characterization of Complex Polymers and Their Hierarchical Structures" December 15 - 20, 2005. *Influence of block copolymer molecular architecture and processing conditions on development of long range order during self-assembly of nanostructures.*

24. "Superelastic Properties of Multigraft Copolymers", R. Weidisch, U. Staudinger, S. P. Gido, D. Uhrig, J. Mays, and N. Hadjichristidis, presented at the International Chemical Congress of Pacific Basin Societies, "Pacifichem 2005", Honolulu, HI, December 15 - 20, 2005.

25. "Bulk and Solution Properties of Polymers Based on 1,3-Cyclohexadiene", J. W. Mays, K. Hong, G. Wignall, Y. Melnichenko, A. Yun, P. F. Britt, J. M. Simonson, T. Huang, M. Pyda, B. Farmer, S. Gido, X. Wu, K. Terao, and Y. Nakamura, presented at the International Chemical Congress of Pacific Basin Societies, "Pacifichem 2005", Honolulu, HI, December 15 - 20, 2005.

26. "Synthesis, Morphology, and Properties of Novel Branched Block Copolymers", J. W. Mays, S. Gido, and R. Weidisch, invited presentation at CIQA National Research Center, Saltillo, Mexico, April 4, 2006.

27. "Effect of Copolymer Architecture on the Morphology and Properties of Styrene/Isoprene Block Copolymers", J. W. Mays, S. Gido, and R. Weidisch, invited presentation at the American Chemical Society Rubber Division Thermoplastic Elastomers Conference, Akron, Ohio, May 11-12, 2006.

28. "Effect of Macromolecular Architecture and Chain Stiffness on Self-Assembly and Properties of Block Copolymers", J. W. Mays, D. Uhrig, K. Hong, S. Gido, and R. Weidisch, invited presentation at the Department of Chemical Engineering, University of South Florida, September 16, 2005.

29. Tripathy Memorial Symposium, University of Massachusetts Lowell, Dec. 2, 2005. *Noncentrosymmetric Block Copolymer Nanostructures.*

30. University of Tennessee at Knoxville, Chemistry Dept. Oct. 14, 2005. *Grain structure, defects and long range order in block copolymer nanostructures.*

31. Oak Ridge National Laboratory, Oakridge, TN, Oct. 13, 2005. *Grain structure, defects and long range order in block copolymer nanostructures.*

32. American Chemical Society, Symposium on Defects in Polymer Nanostructures, Washington DC, August 29, 2005. *Defects in noncentrosymmetric lamellar block copolymer blends.*

33. U. S. Army Research Laboratory, Aberdeen, MD. June 6, 2005. *Polymer Materials Center of Excellence, Research Highlights.*

34. University of Massachusetts Lowell. March 31, 2005. *Defects, Grain Growth, and Long Range Order in Block Copolymer Nanostructures.*

35. University of Massachusetts Dartmouth. March 10, 2005. *Influence of Block Copolymer Molecular Architecture and Annealing Conditions on Development of Long Range Order During Self-Assembly*.

36. Faraday Discussion 128. Self-Organizing Polymers. University of Leeds, UK. July 19-21, 2004. *The Effect of Molecular Architecture on the Grain Growth Kinetics of AnBn Star Block Copolymers.*

37. 12th Annual National Textile Center (NTC) Forum. "Four Points of Light Lecture" Hilton Head, SC. February 14, 2004. *Phase Behavior and Enhanced Spinnablity of Silk Under Osmotic Stress.*

38. Institut für Polymerforschung Dresden, Germany. September 12, 2003. *Effect of Controlled Molecular Architecture on Block and Graft Copolymer Morphology and Properties*

39. Elastomers Gordon Research Conference, June 29-July 4, 2003. Colby Sawyer College, New London, New Hampshire. Invited Speaker: *Multigraft Copolymers of Well Defined Molecular Architecture as Novel Thermoplastic Elastomers.*

40. Third International Symposium on Silk. June 16-19, 2003. Montreal, Canada. *Silk Under Osmotic Stress.*

41. University of Massachusetts Amherst, Polymer Science & Engineering Dept. Weekly Seminar. April 18, 2003. *Defect Structures in Block Copolymers and Blends*.

42. Seoul National University, Seoul, Korea. The 4th Symposium on Hyperstructured Organic Materials. Oct. 23, 2002. *Effect of Controlled Molecular Architecture on Block and Graft Copolymer Morphology and Properties.*

43. Symposium on "Polymer Interfaces and Thin Films" Materials Research Society National Meeting, November 26, 2001. Boston, Massachusetts, invited speaker, *Crystallographic Registry in Coil-Crystalline Block Copolymer Thin Films*.

44. IUPAC International Symposium on Ionic Polymerization, October 22-26, 2001. Crete, Greece, *Effect of controlled molecular architecture on block copolymer morphology.*

45. Polymers East Gordon Research Conference, July 8-14, 2001. Colby Sawyer College, New London, New Hampshire, invited speaker, *Crystallographic Registry in Coil-Crystalline Block Copolymers*.

46. Martin-Luther Universitat Halle Wittenberg, Institut fur Werkstoffwissenschaft, June 26, 2001. Halle, Germany, *Effect of molecular architecture on transport and mechanical properties of model graft copolymers.*

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47. Polytechnic University, Department of Chemical Engineering, November 10, 1999. Brooklyn, New York, *Defect Structures and Long Range Order in Block and Graft Copolymer Morphology.*

48. Massachusetts Institute of Technology Program in Polymer Science and Technology, weekly seminar, March 10, 1999. Cambridge, Massachusetts, *Controlling Morphology and Long Range Order with Graft Copolymer Molecular Architecture.*

49. West Coast Polymer Gordon Research Conference, January 14, 1999. Ventura, California, *Controlling Morphology and Long Range Order with Graft Copolymer Molecular Architecture.*

50. University of Massachusetts Lowell, Department of Chemistry, November 5, 1998. Lowell, Massachusetts, *Effect of Block and Graft Copolymer Molecular Architecture on Morphology.*

51. University of Alabama at Birmingham, Chemistry Department, May 10, 1998. Birmingham, Alabama, *Controlling Morphology and Long Range Order with Graft Copolymer Molecular Architecture.*

52. Kyoto University, September 11, 1998. Kyoto, Japan, *Effect of Block and Graft Copolymer Molecular Architecture on Morphology.*

53. Kyoto Institute of Technology, September 10, 1998. Kyoto, Japan, *Interfacial Control of Silk Protein Chain conformation and Orientation.*

54. Tokyo Institute of Technology, September 9, 1998. Tokyo, Japan, *Effect of Block and Graft Copolymer Molecular Architecture on Morphology.*

55. Tokyo University of Agriculture and Technology, September 7, 1998. Tokyo, Japan, *Electron Microscopy and the Silk I Structure.*

56. International Symposium on Silk and NMR, Tokyo University of Agriculture and Technology, August 29, 1998. Tokyo, Japan, *The Structure of Bombyx mori Silk Foams.*

57. Second International Silk Symposium, University of Virginia, June 15, 1998. Virginia, *Orientation of Silk III at the Air-Water Interface.*

58. American Physical Society National Meeting, March 19, 1998. Los Angeles, California, *Interfacial Control of Protein Chain Conformation and Orientation.*

59. Asilomar Conference on Polymers, February 24, 1998. Pacific Grove, California, *Hollow Cylinder Morphology in Blends of poly(styrene-b-cyclohexadiene) Diblock Copolymers and Homopolymer.*

60. Fourth Greek Symposium for Polymers, November 22, 1997. Petras, Greece, *Architectural Control of Graft Copolymer Morphology and Long Range Order.*

61. University of Athens, November 19, 1997. Athens, Greece, *Morphological Studies of Polymers Using Electron Microscopy and Scattering Techniques.*

62. Max Planck Institut fur Polymerforschung, November 18, 1997. Mainz, Germany, *Architectural Control of Interfacial Curvature, Morphology, and Long Range Order in Graft Copolymers.*

63. Fourth International Symposium on Polymers for Advanced Technologies, September 1, 1997. Leipzig, Germany, *Architectural Control of Graft Copolymer Morphology and Long Range Order.*

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64. Alza Corporation, July 18, 1997. Palo Alto, California, *Influence of Interfaces on the Crystalline Structure of Silk Protein.*

65. Bayer Corporation, March 5, 1997. Indian Orchard, Massachusetts, *Architectural Control of Graft Copolymer Morphology and Long Range Order.*

66. Asilomar Conference on Polymers, February 8, 1997. Pacific Grove, California, *Architectural Control of Graft Copolymer Morphology and Long Range Order.*

67. University of Wisconsin, Materials Science Program, November 14, 1996. Madison, Wisconsin, *Effect of Molecular Architecture on Morphology and Interfacial Curvature in Microphase Separated Polymer Materials.*

68. NIST, November 12, 1996. *Effect of Molecular Architecture on Morphology and Interfacial Curvature in Microphase Separated Polymer Materials.*

69. Army Research Laboratory, August 20, 1996. Wilmington, Delaware, *Synthesis, Morphology and Properties of Model Graft and Flexible Semi-Rigid Block Copolymers.*

70. American Chemical Society Meeting, Division of Polymer Chemistry "Materials Chirality" Symposium, August 25-30, 1996. Orlando, Florida, *Cholesteric Liquid Crystalline Intermediate in Natural Silk Spinning Processes Resulting from the Chirality of Protein Secondary Structure.*

71. University of Connecticut Weekly Polymer Science Seminar, February 23, 1996. Storrs, Connecticut, *Research on Block Copolymer Morphology and Natural Silk Spinning Processes.*

72. Hoechst-Celanese, March 12, 1995. Summit, New Jersey, *Research on Block Copolymer Morphology and Natural Silk Spinning Processes.*

73. United States Military Academy at West Point, Department of Chemistry, October 18, 1995. *Block and Graft Copolymer Morphologies and Interfacial Curvature Properties.*

74. Academia Sinica, Institute of Chemistry, August 24, 1995. Guangzhou, Peoples Republic of China, *Block and Graft Copolymer Morphologies and Interfacial Curvature Properties.*

75. The International Microsymposium on Polymer Physics, August 25-28, 1995. Xi'an, Peoples Republic of China, *Block and Graft Copolymer Morphologies and Interfacial Curvature Properties.*

76. Materials Research Society (MRS) Fall 1995 National Meeting, Symposium Q: Characterization and Properties of Defects in Polymeric Materials, November 28, 1994. Boston, Massachusetts, *Lamellar Diblock Copolymer Grain Boundary Morphology.*

77. University of Massachusetts Amherst, Department of Physics Condensed Matter Seminar, October 6, 1994. Amherst, Massachusetts, *Self-Assembling Grain Boundary Structures in Block Copolymer Mesophases.*

78. Seventeenth Asilomar Conference on Polymers, February 8, 1994. Pacific Grove, California, *Nano-Scale Structures of Self Ordering Polymers.*

79. University of Alabama at Birmingham, Department of Chemistry, November 18, 1993. Birmingham, Alabama, *Morphologies Produced by Aqueous Processing of Biopolymers.*

80. University of Southern Mississippi, Department of Polymer Science and Engineering, November 17, 1993. *Morphologies Produced by Aqueous Processing of Biopolymers.*

81. U. S. Army Watertown Arsenal, December 15, 1992. Modeling of Transport Behavior in Microphase Separated Block Copolymer Materials.

Contributed Presentations

1. W. P. Zhang, S. P. Gido, F. Ignatious, S. W. Kantor, and R. W. Lenz. "The Crystal Structure of Poly(tetramethylene terephthaloyl-bis-4-oxybenzoate)." Presented at the American Physical Society Meeting, Pittsburgh, Pennsylvania, March 21, 1994.
2. S. P. Gido, R. Valluzzi, D. J. Pochan, and B. Novak. "Double, Double Helical Morphology Observed in Crystals of a Rod-Coil-Rod Triblock Copolymer" Presented at the American Physical Society Meeting, Pittsburgh, Pennsylvania, March 21, 1994.
3. R. Valluzzi, W. P. Zhang, and S. P. Gido. "A Pseudo-Threefold Helical Structure in Silk Protein" Presented at the American Physical Society Meeting, Pittsburgh, Pennsylvania, March 21, 1994.
4. D. J. Pochan, S. P. Gido, J. Mays. "Microphase Separation Morphologies in Semiflexible/Flexible Diblock Copolymers." Presented at the American Physical Society Meeting, Pittsburgh, Pennsylvania, March 23, 1994.
5. J. Zhou, J. W. Mays, S. P. Gido, and D. Pochan. "Synthesis and Morphological Study of Flexible/Semi-Rigid Diblock Copolymer (Polyisprene-Poly(t-butyl methacrylate))." Presented at the Southeast Regional Meeting of the American Chemical Society, Birmingham, Alabama, October 16-19, 1994.
6. P. J. Willcox, W. P. Zhang, and S. P. Gido. "Morphological Investigation of Liquid Crystalline Phases in Natural Silk Processing." Presented at the American Physical Society National Meeting, San Jose, CA, March 1995.
7. D. Pochan, S. P. Gido and J. W. Mays. "Microphase Separation Morphology of a Semiflexible/Flexible Diblock Copolymer." Presented at the American Physical Society National Meeting, San Jose, CA, March 1995.
8. R. Valluzzi and S. P. Gido. "A New Crystal Structure Found in Ultrathin Films of *B. mori* Silk." Presented at the American Physical Society National Meeting, San Jose, CA, March 1995.
9. D. J. Pochan, S. P. Gido, J. Zhou and J. W. Mays. "Microphase Separated Morphologies in Conformationally Asymmetric Diblock Copolymers." Presented at the Polymers West Gordon Research Conference, Ventura, CA, Jan. 1995
10. S. Pispas, J. W. Mays, D. J. Pochan, and S. P. Gido. "Synthesis and Characterization of Model Graft Copolymers." Presented at the 47th Southeast / 51st Southwest American Chemical Society Joint Regional Meeting, Memphis, TN, November 29-December 1, 1995.
11. J. Zhou, J. W. Mays, D. J. Pochan, and S. P. Gido. "Morphological Study on Flexible/Semi-Rigid Polyisoprene/Poly(t-butyl methacrylate) Diblock Copolymers." Presented at the 47th Southeast / 51st Southwest American Chemical Society Joint Regional Meeting, Memphis, TN, November 29-December 1, 1995.
12. D. J. Pochan, S. P. Gido, S. Pispas, J. W. Mays, A. J. Ryan, J. P. Fairclough, I. W. Hamley, and N. Terrill. "Bulk Morphological Behavior of Simple Graft A_2B Block Copolymers." Presented at the American Physical Society National Meeting, St. Louis, MO, March 19 1996.

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13. P. J. Willcox, and S. P. Gido "Observations of Liquid Crystalline and Crystalline Structures in Natural Silk Spinning." Presented at the American Physical Society National Meeting, St. Louis, MO, March 20 1996.

14. C. Lee, D. J. Pochan, S. P. Gido, S. Pispas, and J. W. Mays "Bulk Morphological Behavior of Block Copolymers with Novel Architectures" Presented at the American Physical Society National Meeting, St. Louis, MO, March 21 1996.

15. J. L. David, B. M. Novak, and S. P. Gido "A Comparison of the Bulk Crystal Structure of Racemic and Enantiomerically Pure Polyisocyanide Homopolymers." Presented at the American Physical Society National Meeting, St. Louis, MO, March 21 1996.

16. R. Valluzzi, and S. P. Gido "Silk Protein Crystal Structure at the Air-Water Interface." Presented at the American Physical Society National Meeting, St. Louis, MO, March 21 1996.

17. S. P. Gido, D. J. Pochan, J. W. Mays, and S. Pispas "Morphology of Model Graft Block Copolymers." Materials Research Society (MRS) Spring 1996 National Meeting, Symposium Y: "Structure Controlled Macromolecules of Nanoscopic Dimensions", San Francisco, CA, April 10, 1996.

18. S. P. Gido, N. Hadjichristidis, C. Lee, J. W. Mays, S. Pispas, M. Pitsikalis, and D. Pochan. "Synthesis and Morphology of Model Poly(isoprene-graft-styrenes)." Presented at the 9th International Symposium on Polymer Analysis and Characterization, Oxford, England, July 1-3, 1996

19. Y. Poulos, S. Pispas, N. Hadjichristidis, J. Mays, and S. P. Gido. "Synthesis and Morphology of Model Non-Linear Block Copolymers of H and Pi Shape." Presented at the 6th European Polymer Federation Symposium on Polymeric Materials, Aghia Pelaghia, Crete, Greece, October 7-11, 1996.

20. S. P. Gido, C. Lee, D. J. Pochan, J. W. Mays, and N. Hadjichristidis "Architectural Control of Graft Copolymer Morphology." Materials Research Society (MRS) Spring 1997 National Meeting, Symposium W, San Francisco, CA, April 1, 1997.

21. S. P. Gido, D. J. Pochan, J. W. Mays. "Effect of Conformational and Architectural Asymmetry on Long Range Order in Microphase Separated Block Copolymers." Materials Research Society (MRS) Spring 1997 National Meeting, Symposium W, San Francisco, CA, April 1, 1997.

22. R. Valluzzi, and S. P. Gido. "Fibroin Conformation Selection at the Aqueous-Organic Interface." Presented at the American Physical Society National Meeting, Kansas City, MO, March 17 1997.

23. S. P. Gido, D. J. Pochan, C. Lee, and J. W. Mays. "Effect of Architecture and Conformational Asymmetry on Interfacial Curvature and Long Range Order in Microphase Separated Block and Graft Copolymers." Presented at the American Physical Society National Meeting, Kansas City, MO, March 18 1997.

24. S. P. Gido, and Z.-G. Wang "Interfacial Curvature and Morphology in Block and Graft Copolymers." Presented at the American Physical Society National Meeting, Kansas City, MO, March 18, 1997.

25. D. J. Pochan, C. Lee, and S. P. Gido. "Architecturally and Compositionally Induced Long-Range Order of Microphase Separated Block Copolymer Microstructure." Presented at the American Physical Society National Meeting, Kansas City, MO, March 18, 1997.

26. C. Lee, D. J. Pochan, S. P. Gido, S. Pispas, J. W. Mays, N. Beck-Tan, S. F. Trevino. "Morphologies of A_2B Simple Graft Copolymer Blends: Copolymer/Copolymer and Copolymer/Homopolymer Systems to Further Elucidate the Stability of Simple Graft Phase Behavior." Presented at the American Physical Society National Meeting, Kansas City, MO, March 18 1997.

27. F. Beyer, S. P. Gido, A. Avgereopoulos, Y. Poulos, N. Hadjichristidis, and J. Roovers. "Morphological Characterization of Miktoarm Vergina Star Block Copolymers." Presented at the American Physical Society National Meeting, Kansas City, MO, March 18 1997.

28. S.-J. He, R. Valluzzi, and S. P. Gido. "Crystal Structure of Bombyx mori Silk Foams" Presented at the American Physical Society National Meeting, Kansas City, MO, March 19 1997.

29. D. J. Pochan, S. P. Gido, J. Zhou, J. W. Mays, M. D. Whitmore, and A. J. Ryan. "Morphologies of Microphase Separated Conformationally Asymmetric Diblock Copolymers" Presented at the American Physical Society National Meeting, Kansas City, MO, March 19 1997.

30. C. Lee, D. J. Pochan, S. P. Gido, J. W. Mays, Y. Poulos, and N. Hadjichristidis. "Architectural Control of Graft Copolymer Morphology." Presented at the American Physical Society National Meeting, Kansas City, MO, March 19, 1997.

31. S. -J. He, R. Valluzzi, and S. P. Gido "Structure Formation in *Bombyx mori* Silk Foams." Second International Silk Symposium, University of Virgina, June 16, 1998

32. R. Valluzzi, S. Szela, S. -J. He, S. P. Gido, and D. L. Kaplan. "Structure Formation in Fibroin Films at Aqueous Organic Interfaces." Second International Silk Symposium, University of Virgina, June 15, 1998.

33. P. J. Willcox, K. Schmidt-Rohr, D. A. Hoagland, and S. P. Gido "Structural Changes in Poly(vinyl alcohol) Hydrogels with Freeze-Thaw Processing and Time: Electron Microscopy, X-ray Diffraction, and Solid State NMR." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 17 1998.

34. S. J. He, R. Valluzzi, and S. P. Gido. "Structure Formation in Bombyx mori Silk Foams" Presented at the American Physical Society National Meeting, Los Angeles, CA, March 17 1998.

35. F. Beyer, S. P. Gido, M. Xenidou, and N. Hadjichristidis "Morphology of Random Multigraft Block Copolymers." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 18 1998.

36. J. L. David, S. P. Gido, J. Zhou, and J. W. Mays "Filled Cylinder Morphology in Polystyrene-Polycyclohexadiene Diblock Copolymers." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 18 1998.

37. C. Lee, S. J. He, S. P. Gido, S. M. Yu, and D. A. Tirrell "Structural Characterization of Monodisperse Derivatives of Poly(□-benzyl □, L-glutamate) (PBLG)." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 18 1998.

38. P. J. Willcox, S. K. Siddiquee, J. W. Van Egmond, S. P. Gido "Silk Solution Properties and the Natural Silk Spinning Process." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 18 1998.

39. J. L. David, B. M. Novak, and S. P. Gido "Synthesis and Morphological Characterization of Diblocks Formed with Thermally Labile Polycarbodiimides." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 19 1998.

40. K. S. Laverdure, L. Yang, C. Lee, D. J. Pochan, S. P. Gido, S. Pispas, J. W. Mays, and N. Hadjichristidis "Morphological behavior of I_2S Simple Graft Copolymer and Homopolymer Blends." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 19 1998.

41. D. J. Yontz, Shaw L. Hsu, and S. P. Gido. "Interdependence of Morphology and Mechanical Properties of a Crosslinked, Phase Separated System." Presented at the American Physical Society National Meeting, Los Angeles, CA, March 20 1998.

42. F. L. Beyer, S. P. Gido, H. Iatrou, D. Uhrig, J. W. Mays, N. Beck Tan. "Morphology of Regular Multigraft Copolymers with Tetrafunctional Branch Points" American Physical Society National Meeting, March 23, 1999, Atlanta, Georgia.
43. S. J. He, R. Valluzzi, S. P. Gido. "Crystallization and Structure Formation of The Silk I Structure in Natural Silk and Synthetic Polypeptides American Physical Society National Meeting, March 25, 1999, Atlanta, Georgia.
44. S. P. Gido, S. J. He, C. Lee, S. M. Yu, D. A. Tirrell. "A TGB-like Twisted Smectic Phase in Monodisperse Poly(γ-benzyl α, L-glutamate) Produced by Recombinant DNA Techniques" American Physical Society National Meeting, March 26, 1999, Atlanta, Georgia.
45. L. Yang, S. P. Gido, D. Uhrig, S. Pispas, J. W. Mays. "Order-Order Transitions of Miktoarm Star Block Copolymer – Homopolymer Blends" American Physical Society National Meeting, March 20, 2000, Minneapolis, Minnesota.
46. L. Yang, S. P. Gido, S. Pispas, K. Hong, J. W. Mays. "Sporatic Nucleation and Growth in the Microphase Separation Process of an I2S Miktoarm Star Block Copolymer and its Blends with Homopolymer" American Physical Society National Meeting, March 20, 2000, Minneapolis, Minnesota.
47. S. Hong, W. J. MacKnight, T. P. Russell, S. P. Gido. "Morphology of Semicrystalline / Amorphous Diblock Copolymer in Thin Films" American Physical Society National Meeting, March 20, 2000, Minneapolis, Minnesota.
48. L. Yang, S. Hong, S. P. Gido. "Gryroid Single Crystal Diffraction" American Physical Society National Meeting, March 12, 2001, Seattle, Washington
49. S. P. Gido, and E. Burgaz. "T-Junction Grain Boundaries in Block Copolymer-Homopolymer Blends" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

50. S. Chen, S. P. Gido, R. Valluzzi, D. Kaplan. "Crystal Structure of a Model Spider Silk Peptide" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

51. Y. Zhu, S. P. Gido, M. Moshakou, H. Iatrou, N. Hadjichristidis. "Effect of central junction point of AnBn star block copolymers on chain conformation in the strong segregation limit" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

52. S. Hong, W. J. MacKnight T. P. Russell, S. P. Gido. "Crystallization of Multi-layered Crystalline Block Copolymer Thin Films" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

53. S. Hong, L. Yang, W. J. MacKnight, S. P. Gido. "Morphology of Crystalline Block Copolymers" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

54. K. S. Laverdure, S. P. Gido, B. Garetz, N. Balsara. "Mass Transport within Block Copolymers: The Relationship with Morphology and Grain Structure" American Physical Society National Meeting, March 12, 2001, Seattle, Washington

55. A.M. Heintz, R.L. McKiernan, S.P. Gido, J. Penelle, S.L. Hsu. "The Crystallization Behavior of Strongly Interacting Chains",.American Physical Society National Meeting, March 18, 2002, Indianapolis, Indiana

56. E. Burgaz, S.P. Gido. "Cylinder Nucleation at T-Junction Grain Boundaries in Lamellar Block Copolymer/Homopolymer Blends", American Physical Society National Meeting, March 18, 2002, Indianopolis, Indiana

57. Y. Zhu, S.P. Gido. "Morphologies and tensile properties of Block-Double-Graft Copolymers" American Physical Society National Meeting, March 18, 2002, Indianapolis, Indiana

58. T. Xu, Y. Zhu, S. P. Gido, and T. P. Russell. "Interfacial Energy Dependence of Microdomain Orientation in Symmetric Diblock Copolymer Thin Films" Materials Research Society Fall Meeting, December 3, 2002, Boston, Massachusetts

59. E. Burgaz, and S. P. Gido "Nucleation and Growth of Cylindrical Morphology at Tilt Grain Boundaries in Lamellar Block Copolymer / Homopolymer Blends." *Materials Research Society* Fall Meeting, December 3, 2002, Boston, Massachusetts

60. S. P. Gido, and Engin Burgaz. "Wulff Shape of Cylindrical Domains Growing in Lamellar Block Copolymer Homopolymer Blends, and Nucleation of a Second Phase at Grain Boundaries" American Physical Society National Meeting, March 5, 2003, Austin, Texas

61. B. Garetz, M. Newstein, N. Balsara, C. Marques, and S. Gido. "Anisotropy of lamellar block copolymer grains" American Physical Society National Meeting, March 5, 2003, Austin, Texas

62. R. Weidisch, Y. Zhu, E. Burgaz, and S. P. Gido. "Morphologies and tensile property study on multigraft copolymers with tri-, tetra-, and hexafunctional junction points" American Physical Society National Meeting, March 3, 2003, Austin, Texas

63. X. Wu, D. H. Kim, S. Gido, and T. Russell. "Evolution of Morphology in Thin Films of Poly(butadiene-b-ethylene oxide)" American Physical Society National Meeting, March 6, 2003, Austin, Texas

64. Y. Zhu, S. P. Gido, H. Iatrou, and N. Hadjichristidis. "Microphase-Separation of Cyclic Block Copolymers of Styrene and Butadiene and of Their Corresponding Linear Triblock Copolymers" American Physical Society National Meeting, March 3, 2003, Austin, Texas

65. S. Sohn, H. H. Strey, and S. P. Gido "Silk Fibroin under Osmotic Stress" American Physical Society National Meeting, March 4, 2003, Austin, Texas

66. S. Sohn, H. H. Strey, and S. P. Gido "New Routes to Fiber Spinning From Regenerated Silk Fibroin" Third International Symposium on Silk. June 16-19, 2003.

67. Engin Burgaz, Lei Zheng, Gregoire Cardoen, E. Bryan Coughlin, S.P. Gido *"Morphology and Phase Behavior of Polyhedral Oligomeric Silsesquioxane-Polybutadiene Random Copolymer Blends in Bulk and Thin Films"* American Physical Society National Meeting. Montreal, Canada. March 22, 2004.

68. Ashoutosh Panday, Samuel Gido "A Modeling study of azimuthal orientational correlations of anisotropic particles and its application to evolution of order in block copolymers" Physical Society National Meeting. Montreal, Canada. March 24, 2004.

69. Samuel Gido, Engin Burgaz "Comparison of Wulff Shapes of Cylindrical Domains Growing at Homogenous and Heterogenous Nucleation Sites in Lamellar Block Copolymer Homopolymer Blends" American Physical Society National Meeting. Montreal, Canada. March 24, 2004.

70. Roland Weidisch, Ralf Lach, Yuging Zhu, Engin Burgaz, Samuel Gido, David Uhrig, Jimmy Mays, Nikos Hadjichristidis. "Influence of Molecular Architecture on Tensile Properties of Multigraft Copolymers" American Physical Society National Meeting. Montreal, Canada. March 23, 2004.

71. Sungkyun Sohn, Helmut H. Strey, Samuel P. Gido "Silk-I is a Hydrate" American Physical Society National Meeting. Montreal, Canada. March 23, 2004.

72. Thomas P. Russell, Ting Xu, Yuqing Zhu, Samuel P. Gido, Oleg Gang, Ben Ocko "Electric field induced Sphere to cylinder transition in diblock copolymer thin films" American Physical Society National Meeting. Montreal, Canada. March 23, 2004.

73. Xiaochuan Hu, Samuel Gido, Thomas Russell, Ferass Abuzaina, Bruce Garetz "The Effect of Molecular Architecture on the Coarsening of AnBn Star Block Copolymers" American Physical Society National Meeting. Montreal, Canada. March 25, 2004.

74. Shujun Chen, Samuel P. Gido, Thodoris Tsoukatos, Apostolos Avgeropoulos, Nikos Hadjichristidis, Kunlun Hong, Jimmy W. Mays "Defect Study on Noncentrosymmetric Lamellar Block Copolymer Blends" American Physical Society National Meeting. Montreal, Canada. March 25, 2004.

75. E. Burgaz, L. Zheng, G. Cardoen, E. B. Coughlin, S. P. Gido. "Microphase Segregation in Organic-Inorganic Randomly Grafted Copolymers" American Physical Society National Meeting. Los Angeles, CA. March 22, 2005.

76. Shujun Chen, Samuel P. Gido, Souvik Nandi, H. Henning Winter. "Long-range Periodic Structure in Porous High Density Polyethylene Crystallized from the Gel State." American Physical Society National Meeting. Los Angeles, CA. March 25, 2005.

77. Ashoutosh Panday, Samuel Gido. "Shape Templating Effects Among Growing Anisotropic Particles." American Physical Society National Meeting. Los Angeles, CA. March 24, 2005.

78. Ashoutosh Panday, Samuel Gido, Kunlun Hong, Jimmy Mays. "Stability of Core-Shell-Cylinder Structure of Poly(styrene-b-1,3-cyclohexadiene) Diblock Copolymers." American Physical Society National Meeting. Los Angeles, CA. March 21, 2005.

79. Shujun Chen, Samuel P. Gido. "Morphological Transitions in a Triblock Copolymer and Its Sulfonated Ionomer: Thermal Annealing and Solvent Effects." American Physical Society National Meeting. Los Angeles, CA. March 21, 2005.

80. Akinbode Isaacs-Sodeye, Shujun Chen, Samuel Gido. "Electric Field induced alignment and morphological transitions of triblock copolymers." American Physical Society National Meeting. Los Angeles, CA. March 21, 2005.

81. Sungkyun Sohn, Tamako Hata, Helmut H. Strey, Samuel P. Gido. "Self-Assembly of Natural Silk Fibroin under Osmotic Stress." American Physical Society National Meeting. Los Angeles, CA. March 21, 2005.

82. Xiaochuan Hu, Samuel Gido, Thomas Russell, Hermis Iatrou, Nikos Hadjichristidis, Ferass Abuzaina, Bruce Garetz. "Grain Growth Kinetics of AnBn Star Block Copolymers in Supercritical Carbon Dioxide." American Physical Society National Meeting. Los Angeles, CA. March 23, 2005.

83. Gregoire Cardoen, Jimmy W. Mays, Samuel P. Gido, Thomas P. Russell, Xiaochuan Hu, D. Baskaran, and E. Bryan Coughlin. *POSS containing Multiblock Copolymers as Model of Well-defined Organic-Inorganic Hybrid Materials.* American Chemical Society, General Papers: Polymers in Nanotechnology, Washington DC, August 28, 2005

84. "Synthesis, Morphology, and Tensile Properties of Multigraft Copolymers with Regularly Spaced Tri-, Tetra-, and Hexa-functional Junction Points", D. Uhrig, J. W. Mays, Y. Zhu, E. Burgaz, S. P. Gido, U. Staudinger, and R. Weidisch, poster presentation at the 19th International Symposium on Polymer Analysis and Characterization (ISPAC), Oak Ridge, Tennessee, June 12 - 14, 2006.

85. "Synthesis and Morphology of Fluorinated and Sulfonated Block Copolymers", J. W. Mays, T. Huang, and S. P. Gido, presented at the National Meeting of the American Chemical Society, Atlanta, GA, March 26 - 30, 2006; see also: Polym. Mater. Sci. Eng., 94, 165-166 (2006).

86. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session K25: Polymer Electrolytes and Conduction, Tuesday, March 14, 2006, "Proton Conducting Membranes from Fluorinated Poly(Isoprene)-{block}-Sulphonated Poly(Styrene): A Structure Vs Property Study" Akinbode Isaacs-Sodeye, Samuel Gido, Tianzi Huang,Jimmy Mays

87. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session V30: Focus Session: Mechanical Properties: Microscale Deformation and Failure, March 16, 2006 "Tensile Properties and Hysteresis Behavior of Graft Copolymers with Complex Molecular Architecture" R. Weidisch, U. Staudinger, Y. Zhu, S.P. Gido, D. Uhrig, J.W. Mays, N. Hadjichristidis,H. Iatrou

88. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session C1: Poster Session I, March 13, 2006. "Oriented Lamellar Structure and Pore Formation Mechanism in CSX-Processed Porous High-Density Polyethylene" Shujun Chen, Samuel P. Gido, Souvik Nandi, H. Henning Winter.

89. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session H24: Polymers at Interfaces, March 14, 2006 "Defects in a Noncentrosymmetric Lamellar Block Copolymer Blends" Shujun Chen, Samuel P. Gido, Thodoris Tsoukatos, Apostolos Avgeropoulos, Nikos Hadjichristidis, Kunlun Hong, Jimmy W. Mays.

90. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session N30: Block Copolymer Phase Behavior, March 15, 2006, "Azimuthal Orientational Correlations due to Excluded Volume Epitaxy in Growing Anisotropic Grains" Ashoutosh Panday, Samuel Gido.

91. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session C1: Poster Session I, March 13, 2006 "Morphology of Fluorinated and Sulfonated diblock Copolymers" Tomonori Hosoda, Sam Gido, Tianzi Huang, Jimmy Mays.

92. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session C1: Poster Session I, March 13, 2006 "Osmotic Pressure induced by Poly(ethylene glycol) at High Salt Concentrations" Sungkyun Sohn, Helmut Strey, Sam Gido.

93. American Physical Society National Meeting, Baltimore, Maryland. March 2006: Session C1: Poster Session I, March 13, 2006 "Scaling of Avrami Kinetics of Growing Anisotropic Grains" Samuel Gido, Ashoutosh Panday.

94. American Physical Society National Meeting, Denver, Colorado March 2007: Session A17: Charged and Ion Containing Polymers, March 5, 2007. "Proton Conducting Membranes from Fluroinated Poly(Isoprene-b-sulfonated polystyrene): Structure and Transport Properties. Akinbode Isaacs-Sodeye, Samuel Gido.

95. American Physical Society National Meeting, Denver, Colorado March 2007: Session W24: Microphysical Properties of Block Copolymer Aggregates, Going Beyond Structure, March 8, 2007. "Superelastric materials based on multigraft copolymers" U. Staudinger, R. Weidisch, Y. Zhu, S. P. Gido, D. Uhrig, J. W. Mays, M. Klueppel, G. Heinrich.

96. Eric Anderson, Vikram Daga, James Watkins, Sam Gido. "Blends of Diblock Copolymers with Attempt to Achieve Triblock Morphologies." American Physical Society National Meeting, Portland, OR. March 15, 2010. Abstract C1.00030.

97. Lin, Ying ; Daga, Vikram K.; Anderson, Eric A. ; Gido, Samuel P.; Watkins, James J. "High volume production of well-ordered hybrid materials for device applications" Division: PMSE: Division of Polymeric Materials: Science and Engineering. Sunday, August 28, 2011. ACS Award for Team Innovation: Symposium in Honor of Patricia Burns.

98. Qingshou Wei, Ying Lin, Eric R. Anderson, Alejandro L. Briseno, Samuel P. Gido, James J. Watkins. "Additive-driven assembly of block copolymer-nanoparticle hybrid materials for solution processable floating gate memory" Nanofabricated Surfaces: Techniques, Materials and Applications. 243rd ACS National Meeting, San Diego, California. PMSE: Division of Polymeric Materials Science and Engineering. Monday, March 26, 2012

99. Eric Anderson, YuYing Tang, Craig Versek, Mark Tuominen, Samuel Gido, James Watkins. "Incorporation of Functionalized Metal Oxides into Poly(Ethylene Oxide) Based Solid Polymer Electrolytes for Lithium-Ion Batteries", APS March Meeting 2012, Session C1: Poster Session I, Monday, February 27, 2012; Boston, Massachusetts

100. Qingshuo Wei, Ying Lin, Eric Anderson, Alejandro Briseno, Samuel Gido, James Watkins. "Additive-Driven Assembly of Block Copolymer-Nanoparticle Hybrid Materials for Solution Processable Floating Gate Memory" APS March Meeting 2012, Session C1: Poster Session I, Monday, February 27, 2012; Boston, Massachusetts

Research Funding

$100,000 2013-2015 Bill & Melinda Gates Foundation "Ultra-Sensory Condoms Based on New Superelastomer™ Technology" Grand Challenges in Global Health: Development of the Next Generation Condom. Phase I. Co-I's: J. W. Mays (U. Tenn.), S. P. Gido (U. Mass.)

$1,500,000 2012-2015 National Science Foundation ($1,050,000 from NSF, $450,000 Univ. Match) Award Number 1229302 "MRI: Acquisition of a Field Emission Gun Energy Filtered Transmission Electron Microscope" PI: S. P. Gido. Co-I's: A. Ribbe; T. P. Russell; T. Emrick; M. Klingbeil.

$600,000 2012-2015 National Science Foundation "PFI-BIC: Superelastomers: New Thermoplastic Elastomers based on Multigraft Copolymers." PI: J. W. Mays (Univ. of Tennessee). UMass did not participate in this grant. Gido's involvement was through small businesses: BBB Elastomers LLC and Asius Technologies LLC.

$720,000 2009-2012 National Science Foundation (NSF): "MRI: Acquisition of a High Resolution Scanning Electron Microscope" Co-PIs: T. Baskin, S. P. Gido, J. Goldstein, D. "DV" Venkataraman.

$115,000 2012 Saint Gobain Inc. "Exploration of the morphology of C-flex tubing, development of next generation tubing with improved temperature stability, solvent bonding capacity, and clarity." Joint Project with E. B. Coughlin.

$50,000 2007 FUJIFILM Hunt Smart Surfaces, LLC. Unrestricted Support

$5,000 2005 Arkema Inc. Unrestricted Support.

$335,000 2004-2007 U.S. Army Research Office (ARO) W911NF-04-1-0329**.** New Fluorinated and Sulfonated Copolymers: Synthesis, Morphology, and Properties. S. P. Gido is PI. The project involves collaboration with Prof. J. W. Mays of U. Tenn. who receives a subcontract.

$40,000 2004-2006 Kureha Chemical Industry (Japan). Unrestricted Support.

$85,000 2003-2004 Boston Scientific Corp. Unrestricted Support.

$3,750,000 2001-2006 U. S. Army Research Laboratory (ARL) DAAD19-01-2-0002. *University-ARL Collaborative Center for Flexible Protective Materials*. S. P. Gido is PI. The Center involves 9 collaborating faculty at the Univ. of Massachusetts, the Univ. of Tennessee, and Amherst College.

$487,039 2001-2004 U. S. Army Research Office (ARO) DAAD19-01-1-0544 *Morphology and Properties of Model Block and Graft Copolymers.*

$9,000 2001 Metabolix Inc. *Transport Property Measurements on Bacterially Synthesized Polyesters.*

$456,750 2000-2003 U. S. Department of Education, Graduate Assistantships in Areas of National Need (GAANN) *Graduate Fellowships in Polymer/Plastics Engineering.*

$15,000 1999 U. S. Army Research Office (ARO) *Workshop on Flexible Barrier Materials for the Soldier System.*

$331,239 1998-2002 U. S. Army Research Office (ARO) DAAG55-98-1-0116 *Systematic Control of Morphology and Properties Through Graft Copolymer Molecular Architecture.*

$337,240 1998-2002 National Science Foundation (NSF) *Control of Protein Conformation and Mesophases via Interfacial Engineering.* Joint grant with Professor David L. Kaplan of Tufts University.

$506,400 1997-2000 U. S. Army Research Office (ARO) DAAG55-98-1-0005 *Synthesis, Properties and Applications of New Model Block and Graft Copolymers.* Joint grant with Professor Jimmy W. Mays of the University of Alabama at Birmingham.

$325,000 1996-2001 National Science Foundation (NSF), Faculty Early Career Development (CAREER) Program. DMR-9624306 *Morphological Study of Aqueous Processing of Biopolymers.*

$25,000 1997-1998 Loctite Corporation. *Unrestricted Support.*

$100,000 1996-1998 Center for UMass Industry Research on Polymers (CUMIRP) *Cluster M: Fracture Mechanics and Morphology of Semicrystalline Polymers.* (Funded by Exxon and Advanced Elastomer Systems) S. P. Gido and A. J. Lesser, Co-primary investigators.

$150,000 1995-1997 U. S. Army Research Office (ARO) Young Investigator Program. DAAH04-95-1-0305 *Morphology and Surface Structure of Model Conformationally Asymmetric Block Copolymers.*

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$130,664 1995-1997 U. S. Army Research Office (ARO) ASSERT DAAH04-95-1-0303 *Research Training in Block Copolymer Morphology and Surface Structure.*

$406,584 1995-1997 U. S. Army Research Office (ARO) DAAH04-95-1-0126 *Structure, Morphology, and Transport Relationships in Segmented Polyurethanes: A Molecular Simulation Approach.*

$268,144 1994-1997 U. S. Army Research Office (ARO) DAAH04-94-G-0245 *Synthesis and Properties of Model Graft and Flexible-Semirigid Block Copolymers.* Joint grant with Professor Jimmy W. Mays of the University of Alabama at Birmingham.

$50,000 1994-1995 Molecular Simulations, Inc. 16 New England Executive Park, Burlington, MA 01803. *Development of Computer Algorithms to Simulate Phase Separation Behavior and Associated Transport Properties of Polymers.*

$550,000 1994-1995 W. M. Keck Foundation. *The W. M. Keck Electron Microscopy Laboratory at the University of Massachusetts – Amherst.*

$245,660 1994-1997 NSF DMR-9413740 *Academic Research Infrastructure for the Acquisition of Scanning Electron Microscope Instrumentation.*

$150,000 1994-1995 U. S. Army Research Office (ARO) Defense University Research Instrumentation Program (DURIP) DAAH04-95-1-0005 *Instrumentation for Spatially Resolved Composition Mapping in the Electron Microscope.*

$35,000 1994-1995 U. S. Army Research Office (ARO) DAAL03-91-C-0034-TCN-94320 Through Battelle Columbus. *Analysis of Biopolymers for Novel Materials Applications.*

$49,263 1993-1994 U. S. Army Research Office (ARO) DAAL03-91-C-0034-TCN-93444 Through Battelle Columbus. *Bioengineered Proteins and Bioceramics.*

Doctoral Students Supervised

Advisor or co-advisor to 21 Ph.D. candidates.

Student	Year Graduated	Dept.	General Area	Co-Advisor	Employment
Darrin J. Pochan	1997	PS&E	Block Copolymers		Prof. U.Del.
Regina Valluzzi	1997	PS&E	Biopolymers, fibers		Res. Prof. Tufts U.
Chin Lee	1997	Chem. E.	Block Copolymers		Dupont
P. Jeanene Willcox	1998	PS&E	Biopolymers, fibers		U. Penn. Staff
Jennifer David	1999	PS&E	Block Copolymers	Novak	GE Plastics
Rich Beyer	1999	PS&E	Block Copolymers		U.S. Army Res. Lab.
Shi-Juang He	1999	Chem. E.	Biopolymers, fibers		Dow Chemical Co.
Ken Laverdure	2001	Chem. E.	Block Copolymers		Dow Chemical Co.
Lizhang Yang	2001	PS&E	Block Copolymers		3M Company

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Robin McKeirnan	2002	PS&E	Polyolefins, fibers	Pennelle	Proctor & Gamble
Sheng Hong	2002	PS&E	Polyolefins	MacKnight	Accenture
Yuqing Zhu	2003	PS&E	Block Copolymers		Eastman Chemical
Engin Burgaz	2005	PS&E	Block Copolymes		Prof. Turkey
Xiaochuan Hu	2005	PS&E	Block Copolymers	Russell	Ashland Chemical
Shujun Chen	2006	PS&E	Bio & Block Copolym.		Exponent Consulting
Ashoutosh Panday	2006	PS&E	Block Copolymers		Faculty UPES India
Sungkyun Sohn	2006	Chem.E.	Biopolymers, fibers		Eastman Chemical
Akinbode Isaacs-Sodeye	2007	PS&E	Fuel Cells & Batteries		Intel Corp.
Eric Anderson	2020	PS&E	Batteries	Watkins	Intel Corp.
Katherine Williams		PS&E	Block Copolymers	Coughlin	
Ned Burnett		PS&E	Photovoltaics	Briseno	