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(54) METHODS FOR PROCESSING MULTIFUNCTIONAL, RADIATION TOLERANT NANOTUBE-POLYMER STRUCTURE COMPOSITES

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ABSTRACT (57)

Embodiments provide a composite material with oriented nanotubes and a method for making the composite material. The composite material can be formed by distributing a plurality of nanotubes in a polymer matrix. The nanotubes can be further magnetically oriented during the formation of the polymeric matrix, while the polymer matrix is magnetically annealed. The composite material can provide enhanced mechanical and electrical properties, and effective radiation resistance against high-energy ionizing radiation particles and/or electromagnetic interferences. The composite material can be useful for lightweight armors incorporated into vehicles, aircrafts or personnel protection with high ballistic properties, and efficient dissipation of radiation energies, photovoltaic cells with improved polymer solar cell efficiency, improved light emitting diodes (LEDs) with controllable optical properties, or infrared screening devices with increased extinction coefficient.

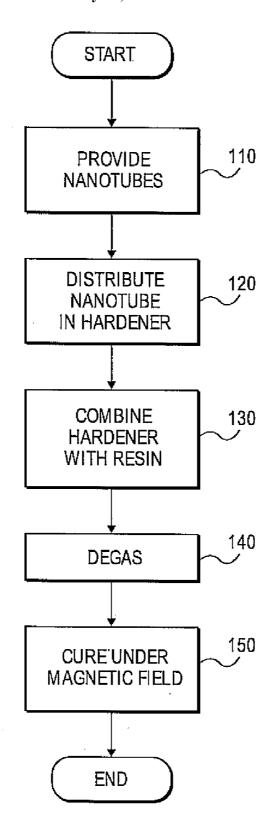


FIG. 1

<u>200</u>

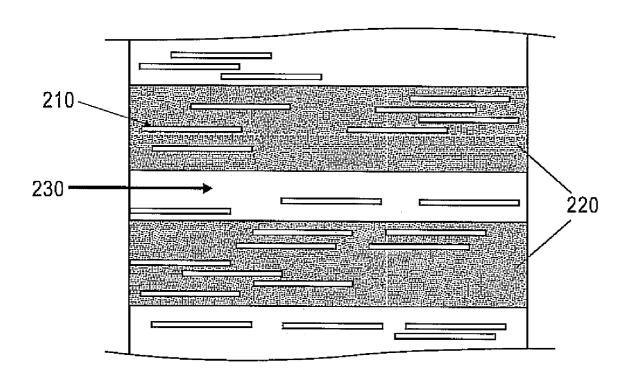


FIG. 2

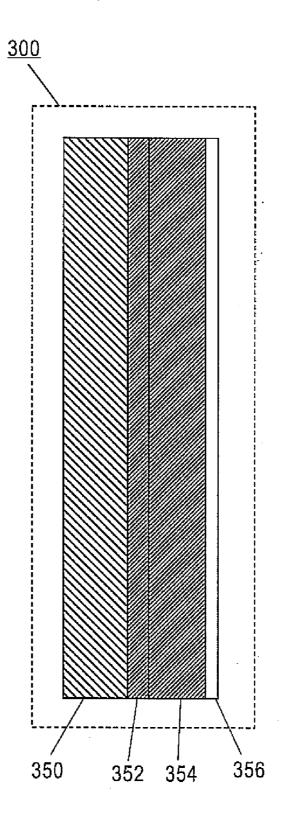


FIG. 3

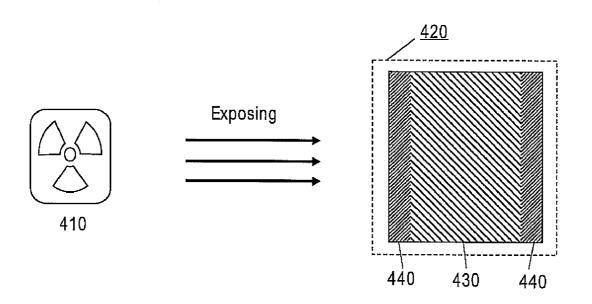


FIG. 4

METHODS FOR PROCESSING MULTIFUNCTIONAL, RADIATION TOLERANT NANOTUBE-POLYMER STRUCTURE COMPOSITES

RELATED APPLICATIONS

[0001] This application claims priority from U.S. Provisional Patent Applications Ser. No. 60/711,678, filed Aug. 29, 2005, and Ser. No. 60/726,652, filed Oct. 17, 2005, which are hereby incorporated by reference in their entirety.

FIELD OF THE INVENTION

[0002] This invention relates generally to composite materials and, more particularly, to composite materials with magnetically oriented nanotubes in a polymer matrix.

BACKGROUND OF THE INVENTIONS

[0003] Organic materials such as polymers offer an attractive route for a wide variety of applications, such as armor devices, photovoltaic devices, light emitting diodes (LEDs), or infrared screening devices, due to their advantages of lightweight (i.e. mass-effective), low cost, ease of fabrication, and flexibility. For example, polymers used as protective armor materials against low-level of threats (e.g., NIJ level III or lower) offer the distinctive advantage of lower density over materials such as metals or ceramics. However, because of their relatively low strength and hardness, polymers are commonly reinforced with either organic or ceramic fibers/whiskers, and are used in conjunction with harder metals and ceramics when they are used in protective systems against higher level of threats, such as NIJ Level IV or higher. An example is standard body armor where a ceramic armor plate is combined with KevlarTM (a type of polymeric synthetic fiber from DuPont Company (Wilmington, Del.)) and graphite fiber in a polyurethane and urea matrix to provide sufficient protection. In another example, polymers may be used as infrared screening films since conventional armor materials such as metal alloy brass are highly toxic. However, problems arise due to the low electrical conductivity for most polymers.

[0004] Carbon nanotubes possess exceptional mechanical properties and superior electric and thermal properties and can be used as reinforcement fibers for structural composites. For example, a cast composite film consisting of polystyrene and randomly oriented carbon nanotubes (5% volume fraction) has been shown to increase the specific modulus by 100% and the strength of the polystyrene by 25%. In addition, carbon nanotube reinforcement can increase the toughness of the composite by absorbing energy because of its high elastic behavior during loading. Furthermore, carbon nanotubes are environmental friendly compared with materials such as brass in conventional infrared screening devices.

[0005] Therefore, it is desirable to combine carbon nanotubes with polymers to provide distinctive properties. Limitations arise, however, because utilizing the unique properties of carbon nanotubes depends on the spatial control and dispersion of individual nanotubes in the polymer matrix, and on the interaction between the polymer and the nanotubes, such as, the ability to transfer load from the matrix to the nanotubes.

[0006] Thus, there is a need to overcome these and other problems of the prior art and to provide a controlled processing of nanotubes with polymer matrix forming a composite material with oriented nanotubes.

SUMMARY OF THE INVENTION

[0007] According to various embodiments, the present teachings include a composite material with magnetically oriented nanotubes. To form the composite material, a plurality of nanotubes are distributed in one of a resin and a hardener to form a first mixture. A second mixture is then formed by combining the other of the resin and the hardener with the first mixture. The second mixture is then degassed and cured under a magnetic field of about 15 Tesla or more to orient the nanatubes.

[0008] According to various embodiments, the present teachings further include a composite material including a polymer matrix with distributed nanotubes. The polymer matrix includes at least one of thermosetting polymers or thermoplastic polymers. The nanotubes are magnetically aligned during formation of the polymeric matrix.

[0009] Additional objects and advantages of the invention will be set forth in part in the description which follows, and in part will be obvious from the description, or may be learned by practice of the invention. The objects and advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims.

[0010] It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011] The accompanying drawings, which are incorporated in and constitute a part of this specification, illustrate several embodiments of the invention and together with the description, serve to explain the principles of the invention.

[0012] FIG. 1 is a block diagram of an exemplary method for making a composite material in accordance with the present teachings.

[0013] FIG. 2 is a schematic diagram for an exemplary magnetically aligned composite material 200 in accordance with the present teachings.

[0014] FIG. 3 depicts an exemplary armor device 300 providing protection from projectile threats in accordance with the present teachings.

[0015] FIG. 4 depicts an exemplary method for resisting ionizing radiations with composite armor material in accordance with the present teachings.

DESCRIPTION OF THE EMBODIMENTS

[0016] Embodiments provide a composite material including oriented nanotubes and a method for making the composite material. The composite material may be formed by distributing a plurality of nanotubes in a polymer matrix. The nanotubes may be further magnetically oriented during curing of the polymeric matrix. The composite material may provide enhanced mechanical and electrical properties, and

effective radiation resistance against high-energy ionizing radiation particles and/or electromagnetic interferences. The composite material can be useful for many applications including, but not limited to, armors for vehicles, aircrafts and personnel protection, with high ballistic properties, and efficient dissipation of radiation energies, photovoltaic devices with improved polymer solar cell efficiency, improved LEDs with controllable optical properties, and infrared screening devices with increased extinction coefficient.

[0017] Reference will now be made in detail to exemplary embodiments of the invention, an example of which is illustrated in the accompanying drawings. Wherever possible, the same reference numbers will be used throughout the drawings to refer to the same or like parts.

[0018] In the following description, reference is made to the accompanying drawings that form a part thereof, and in which is shown by way of illustration specific exemplary embodiments in which the invention may be practiced. These embodiments are described in sufficient detail to enable those skilled in the art to practice the invention and it is to be understood that other embodiments may be utilized and that changes may be made without departing from the scope of the invention. The following description is, therefore, merely exemplary.

[0019] Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the invention are approximations; the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contains certain errors necessarily resulting from the standard deviation found in their respective testing measurements. Moreover, all ranges disclosed herein are to be understood to encompass any and all sub-ranges subsumed therein. For example, a range of "less than 10" can include any and all sub-ranges between (and including) the minimum value of zero and the maximum value of 10, that is, any and all sub-ranges having a minimum value of equal to or greater than zero and a maximum value of equal to or less than 10, e.g., 1 to 5.

[0020] As used herein, the term "nanotube" refers to any cylindrical shaped material (including organic or inorganic material) with a diameter of about 100 nanometers or less. The term "nanotubes" also refers to single wall nanotubes, multiwall nanotubes, and their various functionalized and derivatized fibril forms, which include nanofibers. The nanofibers can be fibrils with diameters of 100 nm or less in at least one form of thread, yarn, fabrics, etc.

[0021] FIG. 1 shows a block diagram of an exemplary method for forming a composite material in accordance with the present teachings. It should be readily obvious to one of ordinary skill in the art that the method depicted in FIG. 1 represents a generalized schematic illustration and that other steps may be added or existing steps may be removed or modified.

[0022] As shown in FIG. 1, at 110, a plurality of nanotubes may be provided. In various embodiments, the provided nanotubes may be carbon nanotubes, which may include but are not limited to single wall carbon nanotubes (SWCNs) or multi-wall carbon nanotubes. In some embodiments, the SWCNs may be armchair type nanotubes (n,n).

[0023] In various embodiments, the nanotubes may be obtained in low and high purity dried paper forms or may be

purchased in various solutions. The nanotubes may also be available in the as-processed, unpurified condition, which may carry with them numerous unwanted impurities that may affect composite properties. Accordingly, at 110, the plurality of nanotubes may be provided from a purification process, which utilizes ultrasonically assisted filtrations. The ultrasonic energy source may be, for example, a highintensity ultrasonic processor. In the purification process, for example, the nanotubes can be ultrasonically suspended in a first solvent, such as, toluene, and then filtered to extract the soluble fullerenes leaving an insoluble fraction. The insoluble fraction may then be ultrasonically re-suspended in a second solvent, such as a methanol, and transferred in a filtration funnel configured with a filter membrane. A pressure differential of, for example, about 50 Torr, may be applied across the filter membrane. The filter membrane may be, for example, a polycarbonate track-etched filter membrane with a pore size of about 0.8 µm. The obtained nanotubes may then be washed with a third solvent, such as a sulfuric acid with an exemplary concentration of 6 M, to remove traces of metal such as titanium introduced into the sample from the ultrasonic horn.

[0024] At 120 of FIG. 1, a plurality of nanotubes can be distributed in a resin. Distributing nanotubes in a resin may further include first dispersing nanotubes in a solvent, such as an ethanol, and then ultrasonically mixing nanotubeethanol, for example, at about 10% amplitude for about 90 seconds using the high-intensity ultrasonic processor. Meanwhile, the resin may also be ultrasonically mixed with ethanol at about 10% amplitude for about 90 seconds. The nanotubes/ethanol mixture may then be combined with the resin/ethanol mixture and ultrasonically mixed at about 50% amplitude for about 90 seconds. This process may promote the distribution of nanotubes over the surface of the resin molecules and prevent nanotube clustering. The exemplary embodiment described herein utilizes a polymer matrix formed of DERAKANE 411-350 epoxy vinyl ester resin manufactured by Ashland Inc. (Covington, Ky.).

[0025] Still at 120 of FIG. 1, to form the composite material, a designated weight fraction of purified nanotubes, such as, for example, 35% or less by weight, may be dispersed in a hardener part of an epoxy by first dispersing the plurality of nanotubes in a solvent such as ethanol in an ultrasonic bath at room temperature for about one hour. Then the ethanol-based nanotube solution may be mixed with the hardener and the mixture may be stirred for at least one hour at about 2000 rpm or more. During this stirring process, the nanotube-hardener mixture may be kept at room temperature to maintain a low viscosity using a silicon oil bath for example. In various embodiments, the solvent ethanol may be removed by evaporation where the mixtures may be placed in a vacuum oven at about 60° C. or more for at least one hour.

[0026] At 130 of FIG. 1, a resin part of the epoxy may then be added to the nanotube-hardener mixture to form a composite mixture with a desired resin/hardener ratio, such as 4:1 by weight. The epoxy may include, but are not limited to, one or more of aeropoxy, thixotropic epoxy, Derakane-441, or other type of epoxy. The composite mixture may be stirred at about 2000 rpm or more for at least 5 minutes.

[0027] At 140 of FIG. 1, the composite mixture may be degassed moderately until no gas bubbles can be seen. The

degassed mixture may then be loaded into molds of a desired shape which may result in a variety of 3-D structures for the nanotube-polymer composite, such as, for example, a sheet, a fiber, a cylinder, a foam or other 3-D structure. The molds may be sealed for a subsequent magnetic process. In various embodiments, the degassed composite mixture may be formulated as a film or coated on various substrates and then be loaded for a subsequent magnetic process.

[0028] At 150 of FIG. 1, the composite mixture may be cured at room temperature with a low viscosity under a high magnetic field, such as 15 Tesla or more for at least 2 hours. Then, still under the high magnetic field, the curing temperature may be increased up to about 60° C. or more for also at least 2 hours. During the magnetic process, the polymer may be annealed and the nanotubes may be oriented in the polymer matrix (i.e. the curing mixture of the resin and the hardener of the epoxy in this example) due to the cooperative effect of the magnetic torque exerted by the magnetic field directly on the nanotubes and by hydrodynamic torque and viscous shear (i.e. drag forces) exerted on the nanotubes by the polymer chains. In addition, the magnetic field may be penetrable and its direction and strength may be controllable. Accordingly, the alignment of the nanotubes may be controlled for desired orientation(s) depending on specific applications. More specifically, the alignment profile may be specially designed for desired enhanced properties of the composite material, such as enhanced mechanical and electrical properties, or efficient radiation resistance.

[0029] Turning to 150 of FIG.1, the magnetic field may then be removed and the composite mixture may remain cured at about 60° C. or more for at least 2 hours to fully cure the polymer matrix, i.e., the resin and the hardener of the epoxy in this example. One of ordinary skill in the art will understand that other polymers may be also used for the polymer matrix including but not limited to thermosetting polymers and thermoplastic polymers. In various embodiments, boron carbide particles, silicon carbide particles, or other similar hard materials may be incorporated into the polymer matrix for the composite material.

[0030] FIG. 2 shows a schematic diagram of an exemplary magnetically oriented composite material 200 including a plurality of nanotubes 210 and polymer fibrils 220 formed in accordance with the present teachings. Arrow 230 indicates a direction of the applied magnetic field. It should be readily obvious to one of ordinary skill in the art that the exemplary magnetically oriented composite material 200 depicted in FIG. 2 represents a generalized schematic illustration and that more nanotubes or polymer fibrils may be added or existing nanotubes or polymer fibrils may be removed or modified.

[0031] As shown in FIG. 2, the plurality of nanotubes 210 can be oriented in a direction parallel to the magnetic field indicated by the arrow 230. In some embodiments, the nanotubes 210 may be magnetically oriented single wall carbon nanotubes (SWCNs). In other embodiments, the nanotubes 210 may be locally oriented, for example, through a mechanical stretching, or a pressing through a die or electric field. As a result, the composite material 200 with locally oriented nanotubes may provide specially-varying mechanical properties for specific applications, such as, for example, a composite tube with strong exterior and soft interior.

[0032] The polymer fibrils 220 may be uniform along the direction of the magnetic field indicated by the arrow 230. When a magnetic field is applied during the formation of the polymer, such as, for example, during the curing of a liquid epoxy, the polymer molecules may also be annealed (e.g., aligned) along the direction of the applied magnetic field and taking fibril shape. Accordingly, the polymer fibrils 210 may be magnetically aligned epoxy fibrils in accordance with various embodiments.

[0033] In various embodiments, the magnetically oriented composite material 200 may provide enhanced mechanical and electrical properties. The enhanced mechanical properties may be demonstrated by a specific strength and a specific modulus. The specific strength (or modulus) may be determined by a material strength (or modulus) divided by its density (e.g., weight per unit volume). For example, the magnetically oriented composite material 200 may provide a specific strength of such as about 20 GPa·cm²/g to about 50 GPa·cm²/g and a specific modulus of such as about 100 GPa·cm²/g to about 200 GPa·cm²/g.

[0034] The enhanced electrical properties for the magnetically oriented composite material 200 may be demonstrated by the electrical conductivity, such as, for example, an electrical conductivity of about 10⁶ s·cm⁻¹ or higher. Because of this enhanced electrical conductivity, the magnetically oriented nanotube-polymer composite material 200 may be used for, for example, improved polymer-based light emitting diodes (LEDs), especially when the polymer used is a photo-active polymer. Compared with conventional LEDs, the nonotube-polymer based composite material may be able to increase photoluminescence/electro-luminescence yield, which may provide a means to alter the optical properties of the polymer to tune the color or emission for organic light emitting devices. One more example for using the enhanced electrical conductivity of the magnetically oriented nanotube-polymer composite material 200 may be for infrared screening devices. The magnetically oriented composite material 200 may be used as an environmental friendly alternative to screen infrared radiations compared to highly toxic materials that is conventionally in use such as brass. More importantly, the enhanced electrical conductivity of the magnetically oriented composite material 200 may increase the extinction coefficient for infrared screening.

[0035] Accordingly, the magnetically oriented composite material 200 with enhanced mechanical and electrical properties may be used for ballistic resistant material such as armor devices, that may be as effective as steel against projectiles—at considerably lower weight. FIG. 3 shows an illustration for an armor device 300 that may provide protection from possible projectile threats in an application of such as a ground combat vehicle. It should be readily obvious to one of ordinary skill in the art that the armor device 300 depicted in FIG. 3 represents a generalized schematic illustration and that other layers may be added or existing layers may be removed or modified.

[0036] In the armor device 300, the magnetically oriented composite material 200 may be configured as armor interior 350. The armor interior 350 may be overlaid by a layer 352 of metal, such as aluminum, then a layer 354 of ceramic, such as alumina (i.e., aluminum oxide), and then a layer 356 of metal, such as steel. The layer 352, 354, and 356 may be configured as an armor cover over the armor interior 350.

[0037] To form the armor interior 350, the magnetically oriented composite material 200 may undergo a scale up fabrication process to meet specific applications. Accordingly, the resulting armor interior 350 may be at least one form of films, sheets, fibers, cylinders, foams, coatings or pastes.

[0038] As used for the armor interior 350, the nanotubes may be magnetically oriented in certain directions with a high anisotropy due to its one-dimensional structure. Such magnetic orientation of nanotubes (including nanofibers) may be controlled to confer specific properties to the armor interior 350. For example, by rearranging the orientations of nanotubes, superior mechanical and physical properties may be tailored to the armor interior 350. In some embodiments, the oriented nanotubes in the armor interior 350 may be aligned co-axially with the line of fire to provide shear load transfer in the frontal impact layer. In other embodiments, the oriented nanotubes in the armor interior 350 may be aligned orthogonally to the line of fire to provide tensile load support in the structural baking layer.

[0039] In various embodiments, the magnetically oriented composite material 200 may further be used as a shielding material and provide effective radiation resistance against high-energy ionizing radiation particles and/or electromagnetic interferences in applications such as armor devices.

[0040] FIG. 4 depicts a method for resisting radiation including a radiation source 410 and an armor device 420. The armor device 420 may include a composite armor material 430 enclosed in an armor enclosure 440 formed with materials, such as aluminum. The composite armor material 430 may include the magnetically oriented composite material 200.

[0041] The radiation source 410 may provide ionizing particles, electromagnetic interferences, or a combination of various radiations. The ionizing particles may include alpha particles, beta particles, gamma rays or x rays, cosmic ray or solar flares. To measure the radiation resistance, the radiation source 410 may provide radiations with high energy, such as, for example, an ionizing particle with a proton beam for a radiation energy of 15 MeV (i.e. megaelectron volts) or higher. Alternatively, the radiation source 410 may provide a proton beam with high intensities, such as, ranging from the direct level of 109 beam particles per second to 10 beam particles per second. Such high energy radiation may be far more intense than would be expected in a real environment, such as in space.

[0042] The armor device 420 may be exposed to the radiation source 410 to measure the radiation resistance of the composite armor material 430. The radiation resistance may be demonstrated by a shielding effectiveness (or attenuation fraction) that may be measured. For example, when using a high-energy proton beam as radiation source 410, the shielding effectiveness (or attenuation fraction) may be measured in terms of the number of high-energy particles in the beam before and after the proton beam hits the shielding material, i.e. the magnetically oriented composite armor material 430. The shielding effectiveness or attenuation fraction for the composite armor material 430 may be, for example, about 0.60 or higher. In some embodiments, the composite armor material 430 may include magnetically aligned nanotubes with high aspect ratio for providing enhanced transport properties for effective electromagnetic interference shielding for electronics, which in some cases may also need to be guarded against impact damages.

[0043] Generally, armor devices, such as, for example, the armor device 300 in FIG. 3 or the armor device 420 in FIG. 4, may be incorporated within vehicles, aircrafts or personnel armors to provide lightweight protection against ballistic threats, enhanced mechanical and electrical properties, and radiation protection against high-energy ionizing particles and/or electromagnetic interferences.

[0044] In various embodiments, the magnetically oriented composite material 200 may also provide electronic properties based on morphological modification or electronic interaction between the two components, such as, for example, π -conjugated polymers and carbon nanotubes. In particular, the combination of carbon nanotubes with π -conjugated polymers may form an electronic conjugation which may enable the polymers to be used as an active material for photovoltaic devices, such as a photovoltaic cell. The controlled magnetic processing of carbon nanotubes with π -conjugated polymers may improve the exciton dissociation and carrier transport of the system and thus resulting in an improved polymer solar cell efficiency.

[0045] Other embodiments of the invention will be apparent to those skilled in the art from consideration of the specification and practice of the invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with a true scope and spirit of the invention being indicated by the following claims.

What is claimed is:

1. A composite material formed by the steps comprising: providing a plurality of nanotubes;

distributing the plurality of nanotubes in one of a resin and a hardener to form a first mixture;

forming a second mixture by combining the other of the resin and the hardener with the first mixture;

degassing the second mixture; and

curing the second mixture under a magnetic field of about 15 Tesla or more to orient the plurality of nanotubes.

- 2. The composite material of claim 1, wherein the plurality of nanotubes comprise nanofibers.
- 3. The composite material of claim 1, wherein the plurality of nanotubes comprise carbon nanotubes, wherein the carbon nanotubes comprise one of single wall carbon nanotubes (SWCNs) or multi-wall carbon nanotubes.
- **4**. The composite material of claim 3, wherein the SWCNs comprise armchair type nanotubes having a chirality where n=m.
- 5. The composite material of claim 1, wherein a weight percentage of the plurality of is 35% or less of the composite material.
- **6**. The composite material of claim 1, wherein the resin and the hardener form an epoxy with low viscosity at room temperature.
- 7. The composite material of claim 1, wherein the plurality of nanotubes are oriented anisotropically.
- **8**. A ballistic resistant material comprising the composite material of claim 1, wherein the plurality of nanotubes are aligned co-axially to a line of impact.

- **9**. A ballistic resistant material comprising the composite material of claim 1, wherein the plurality of nanotubes are aligned orthogonally to a line of fire or a line of impact.
- 10. A ballistic resistant material comprising the composite material of claim 1 further comprising at least one of boron carbide and silicon carbide.
- 11. The composite material of claim 1 further comprising at least one form of a film, sheet, fiber, cylinder, foam, coating or paste.
- 12. A method for making a composite material comprising:

providing a plurality of carbon nanotubes;

distributing the plurality of carbon nanotubes in a hardener:

forming a mixture by combining a resin with the hardener and the plurality of carbon nanotubes;

degassing the mixture; and

curing the mixture under a magnetic field of about 15 Tesla or more to align the plurality of nanotubes.

13. The method of claim 12 further comprising:

adding a first solvent to the plurality of carbon nanotubes, wherein the first solvent comprises an ethanol;

adding a second solvent to the resin, wherein the second solvent comprises an ethanol; and

combining the first solvent and the plurality of carbon nanotubes with the second solvent and the resin.

14. The method of claim 12, wherein providing the plurality of carbon nanotubes comprises purifying the carbon nanotubes.

- 15. The method of claim 12 further comprising providing a solvent for steps of providing carbon nanotubes and distributing the carbon nanotubes in the hardener, wherein the solvent comprises an ethanol.
- **16**. The method of claim 12 further comprising placing the carbon nanotubes distributed hardener in a vacuum at about 60° C. or more for at least one hour.
- 17. The method of claim 12 further comprising stirring the mixture at about 2000 rpm or more for at least 5 minutes prior to the step of curing.
- **18**. The method of claim 12, wherein curing the mixture comprises:
 - curing the mixture at room temperature for at least two hours under the magnetic field;
 - curing the mixture at about 60° C. or more for at least two hours under the magnetic field; and
 - curing the mixture at about 60° C. or more for at least two hours under no magnetic field.
 - 19. A composite material comprising:
 - a polymer matrix, wherein the polymer comprises at least one of a thermosetting polymer and a thermoplastic polymer; and
 - a plurality of nanotubes distributed in the polymer matrix, wherein the plurality of nanotubes are magnetically aligned during formation of the polymeric matrix.
- **20**. A ballistic resistant material comprising the composite material of claim 19, wherein the plurality of nanotubes are aligned co-axially to a line of impact.

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