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**CHALLENGES AND OPPORTUNITIES
FOR NANOTECHNOLOGY IN MULTI-
FUNCTIONAL COMPOSITE
STRUCTURES (PREPRINT)**



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14. ABSTRACT Incorporation of nanoparticles into engineered composites represents one of the largest volume applications for nanocomposites currently envisioned. Of the wide variety of structural applications, fiber-reinforced composites for aerospace structures have some of the most demanding applications with extreme requirements in physicals, chemical, electrical, thermal, and mechanical properties. Nanocomposites offer tremendous potential to improve these properties for advanced engineered composites with modest additional weight and easy integration into current processing schemes. Significant progress has been made in fulfilling this vision. Within this paper, we review the relative status of nanocomposites incorporation into aerospace composite structures and the need for continued development. In particular, nanocomposites have been applied at specific and multiple locations within the hierarchical composites to improve specific properties and to offer yet another method and level to optimize multiple properties of the overall structural composite.					
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Challenges and Opportunities for Nanotechnology in Multi-functional Composite Structures

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Abstract

Incorporation of nanoparticles into engineered composites represents an important application for nanocomposites. Of the wide variety of structural applications, fiber-reinforced composites for aerospace structures have some of the most demanding applications with extreme requirements in physical, chemical, electrical, thermal, and mechanical properties. Nanocomposites offer tremendous potential to improve these properties for advanced engineered composites with modest additional weight and easy integration into current processing schemes. Significant progress has been made in fulfilling this vision. Within this paper, we review the relative status of nanocomposites incorporation into aerospace composite structures and the need for continued development. In particular, nanocomposites have been applied at numerous locations within the hierarchical composites to improve specific properties and to offer yet another method and level to optimize multiple properties of the overall structural composite.

Keywords: *nanocomposite, fiber-reinforced, polymer matrix composite. applications.*

Introduction

Current advanced engineered material systems, such as the organic matrix composites, have a myriad of applications including aerospace structures. Composite aerospace structures often have extreme demands in properties that make the adoption of higher performance materials systems, such as nanocomposites, inviting. However, there is also a strong need to balance the multiple demands of performance, weight, processability, risk and/or life cycle cost in selecting new structural materials. Within this paper, we will discuss the current and potential impact of nanotechnology on composites with particular attention on their relevance to aerospace applications.

As air travel continues to grow, light-weight, multi-functional and easily manufactured structural materials such as polymer matrix composites (PMC) are finding increased use to combat increased fuel and maintenance cost which each respectively account for roughly 50% and 20% of the operation cost beyond ownership of a commercial airplane.¹ Considering that the recent cost of launching a heavy lift system into Low Earth Orbit (LEO) is \$6000 - \$20,000/kg² and approximately \$36,000/kg for geosynchronous orbit (GEO)³, PMC's are also finding increase use for space structural applications. PMC's are generally preferred for moderate temperature applications (<600°F) over metals based on weight savings, fatigue resistance, corrosion suppression, and significantly decreased part-count including fasteners. Future aerospace systems and current developmental systems, seek to further enhance both the mechanical and multi-functional properties of PMC by incorporating nanoparticles.

Property Improvement of Composite Structures with Nanoparticles

A particular challenge for traditional composites is the integration, control and exploitation of nanoparticle-enabled properties within a hierarchical composite made with commercially

viable processing methods. Though efforts continue to pursue low-cost processing methods for composites fabrication, the current large capital investment of composite processing equipment (pre-preg machines, fiber tow placement, infusion, autoclave, etc.) make it initially preferable to use traditional composite processing schemes for integrating nanocomposites. Thus, nanoparticles can be incorporated in a number of different ways within the traditional composite material forms including within a fiber, as a thin coating on a fiber, in place of a fiber tow, as an inner layer, as a veil or coating, or as a part of the polymer resin system. Where the nanoparticles should be placed will depend on the property being sought and the ability to exploit the suite of properties imparted. An illustration of the potential multi-scaled incorporation is shown in Figure 1. A notional summary of the approaches and application of nanocomposites to aerospace structural composites is given in Table 1.

In comparison, many of the current non-nanocomposite approaches to property improvement incorporate larger-scale conducting materials (foils, grids, coatings, etc) which can often have increased weight, leading to manufacturing issues, or complicate repairs due to phase discontinuities and their larger sizes. Nanocomposites provide an opportunity to lessen these traditional trade-offs as properties are improved with small additions of nanoparticles, without significant changes in the manufacturing process, and without the incorporation of an additional bulky phase that often requires formal connections. Overall, such a system is expected to eliminate redundancy, improve fabrication efficiency, reduce weight and volume, and decrease operating or repair cost. Below are some specific examples of improvements currently being made.

Physical /Chemical Properties

The ability for a solid material to maintain dimensional and chemical stability in adverse environments is important for both air and space structures. For example, some large space-based optical structures require that the deviation in optical pathlength not exceed a few picometers as the asset is cycling in and out of extreme thermal environments. Ideally, the structural material would be chemically inert to the radiation environment and be resistant to dimensional or chemical changes as a function of temperature until the end of the material's service life.

One of the most promising nano-scaled fillers for enhancing the physical and chemical integrity of a polymer system without adding significant weight are layered silicates. Loadings of only a few percent exfoliated silicate nanoparticles in the appropriate polymer can result in significant enhancement of a number of physical properties, including resistance to atomic oxygen in Low Earth Orbit,⁴ gas barrier properties for cryogenic tanks,⁵ heat distortion temperatures, resistance to solvent swelling, and flammability resistance.⁶ For example, previous work demonstrated the reduction of oxygen permeability from a value near 100 cc mil./m² day atm for the pristine polymer to values of 1 to 0.1 cc mil./m² day atm for film nanocomposites.⁷ Figure 2 shows the decrease in helium permeability of a composite-overwrapped tank using layered silicates within the resin. There was also an additional 45-55% decrease in structural weight with the elimination of the inner liner.⁵

Current reusable launch concepts that would use such tanks are envisioned to thermally cycle hundreds of times and require the tank to experience thermal extremes of ~-240 °C (-400°F) at the inner cryogenic fluid wall and up to ~343 °C (~650°F) at the outer wall.

Such thermal extremes will require management of the internal stresses that can lead to microcracking. A major contributor to these stresses will be those induced by the coefficient of thermal expansion mismatch between the carbon fiber and the thermosetting resin. Preferential swelling of the polymer by diffusing species (moisture, condensate, etc.) can also contribute to internal stresses. Fortunately, in addition to decreasing permeability, organically-modified layered silicates have been shown to decrease the coefficient of thermal expansion (CTE) and the moisture expansion coefficient. For example, at modest layered silicate loadings, decreases in CTE of 20-50% for some epoxies⁸⁻⁹ and about 20% for some polyimides¹⁰ as well as decreases in moisture values of 40-80% for epoxy¹¹ and ~30-40% for polyimides¹⁰ have been observed. Impeding the transport of oxidative species also appears promising as indicated by the substantial increase in atomic oxygen resistance with a few percent of layered silicate dispersed in epoxy matrix.⁹ Other nanoparticles such as functionalized carbon nanotubes (CNT) and graphite flakes have also been investigated,¹²⁻¹⁴ and may be able to add multifunctional properties such as electrical and thermal conductivity.

Mechanical Properties

Mechanical improvements to traditional composite materials through the use of nanocomposites can be targeted towards improvement of resin-dominated properties or, eventually, towards fiber-dominated properties. Resin-dominated properties include interlaminar shear strength, compression strength, and fatigue properties. Fiber-dominated properties include modulus and ultimate tensile strength in which the theoretically predicted high modulus and strength of nanoparticles such as single-walled carbon nanotubes are of interest. For resin-dominated properties such as interlaminar shear strength (ILSS) and toughness, a detailed understanding of the fracture mechanics within the nanocomposite morphology is needed to fully exploit the ultimate properties of nanocomposites. Characterization of the deformation and failure mechanisms in a manner similar to that used for traditional composites (pull-out, bridging, interfacial cracking, matrix cracking, etc.) will be needed to more fully understand and exploit improvements made with nanocomposites. Though more work is needed, increases in ILSS of 20% for only 0.3wt% functionalized double-walled carbon nanotubes are reported in the literature for glass fiber epoxy composite¹⁵ and increases of 180% for a similar epoxy/glass fabric composite with carbon nanofibers have been reported by a commercial material provider.¹⁶

A key benefit of nanocomposites within traditional fiber reinforced composites is the ability to selectively tailor the deformation mechanism at different locations within the composite. Thus, one might need only use the nano enhancement within selected plies. A recently reported extension of this concept is to grow carbon nanotubes directly on the fiber ply to create a mechanically and multi-functionally enhanced composite.¹⁷ With these fiber-like, out-of-ply nano reinforcements, the final composite begins to more closely resemble a hierarchical 3D-reinforced fabric rather than a composite with nano-enhanced resin and highlights the great flexibility of using nanoparticles within a composite material. Table 2, which compares some of the properties of carbon nanotubes against more traditional aerospace materials, illustrates the basis of this potential. Though nanocomposite properties are discussed in more detail elsewhere,¹⁸ these properties raise the intriguing possibility of applying the nanotubes as super-strong reinforcing fibers with orders of magnitude higher strength and stiffness than any other known material.

Though major advances have been made on the fabrication of carbon nanotube sheets¹⁹⁻²⁷ and yarns,²⁸⁻³³ no one has yet assembled carbon nanotubes into yarns and sheets that retain the spectacular properties of the individual single-walled carbon nanotubes. Individual single-walled nanotubes properties including a ten fold higher strength than existing sheets and yarns, a higher thermal conductivity than diamond, a thousand-fold higher current carrying capability than copper, and many other fascinating and useful properties for active devices. Though some of the produced CNT yarns are nearly as tough as the Kevlar fibers used for antiballistic vests, efforts to increase this strength, yarn toughness, and production capability continue. Correspondingly, the actual performance of nanotube/polymer composites has also been below the theoretically predicted performance.

Thermal Properties

Thermal management of aerospace structures is important for many applications including space platforms, re-entry vehicles, propulsion systems, electronics, and high energy (laser, etc.) systems. For example, heat generated by spacecraft components often presents difficult thermal design problems because of local high peak heat flux, large total power needing dissipation, and wide temperature changes occurring over time. The increasing need to fly larger, higher-performance payloads with higher-density microprocessors for longer periods of time has escalated power dissipation and heat flux at the silicon level. Future military communication satellites will have higher power density microelectronics packaging design concepts that will increase by five to ten times the data processing throughput, resulting in up to a ten fold increase in the thermal density.³⁴

Next generation aerospace structures could potentially use more thermally conductive materials to spread-out and judiciously direct heat flow in satellites, thermal protection systems, near-propulsion structures, electronic boxes, chip packaging, directed energy systems, radiators, and their accompanying thermal interfaces. Carbon nanotubes offer some of the strongest promise since the measured thermal conductivity of individual MWNTs (3000 W/m·K) is higher than diamond³⁵ and predicted values are even higher³⁶. The theoretical thermal conductivity of single-walled carbon nanotubes is 6000 W/m·K³⁷ which, when combined with the exceptional tensile strength, could form the basis of a multitude of future high-performance materials.

Experimentally, un-optimized MWNT sheets have been measured to give a room temperature thermal conductivity of about 150 W/m·K, which is close to the thermal conductivity reported for magnetically oriented SWNT sheets,³⁸ but substantially higher than the <3 W/m·K reported for highly loaded (up to 20wt%) vapor grown carbon nanofibers (VGCNF) in epoxy.³⁹ Inefficient phonon transport between nanotubes is considered the major limitation to approaching the thermal conductivity of the individual nanotubes.⁴⁰ Since the phonon dispersion occurs at discontinuities such as tube ends, a simple argument would suggest that increasing nanotube length from 0.3 mm to 3 mm will increase nanotube yarn thermal conductivity ten-fold or from 150 W/m·K to 1500 W/m·K. However, creating nanotube junctions in which contacted tubes efficiently exchange thermal energy would also significantly increase the thermal conductivity of composite resins and sizings that incorporate nanotubes. One of the first aerospace applications using this type of nanofiller could be thermally conductive adhesives and reinforced interface gaskets.

Electrical Properties

Many aerospace applications require electrically conducting, polymer-based composites for static discharge, electrical bonding, radio frequency interference shielding, primary and secondary power, and current return through the structure. However, present carbon-reinforced polymer composites alone cannot provide robust solutions to satisfy these requirements. Traditionally, secondary conductive materials such as foils, wires/straps, and coatings would be incorporated into the structure with additional processing steps. However, integrated composite structures that utilize percolated networks of conductive particle with high aspect ratios appear to be a preferred method for obtaining low resistivity composites at low volume fractions and with little additional weight. For example, materials such as carbon nanofibers,^{39,41} SWNT buckypaper,⁴² nickel nanostrands,⁴³ or graphite flakes¹⁴ have potential application in near-term electrostatic dissipation and electromagnetic shielding systems as discussed in detail below .

Control of Electrostatic Dissipation (ESD)

Composite structures, including graphite/epoxy and Kevlar are known to locally charge to 4000 volts or 6000 volts respectively when exposed to electron fluxes despite the presence of a conductive path through carbon fibers.⁴⁴ To avoid discharging to a proximate ground and harming electronics or personnel, electrical shielding is required over the entire exposed surface. Materials that have resistivity values above 1×10^{13} ohm/square can develop a static charge that will not dissipate even when bonded. The measurement of ohms/square refers to the resistance (in ohms) of a surface film multiplied by the surface width and divided by the length. If one considers a square of equal length and width, the resistivity can be expressed as ohms per unitless square. A resistivity of 1×10^7 ohm/square is sufficient to dissipate charge for an electrically bonded structure and is generally the range for graphite reinforced composites when the conductive carbon filaments are adequately connected to the proximate ground. By adding high aspect ratio conductive nanoparticles into the resin rich regions of a composite, the electrical connection with the proximate ground and the conducting carbon fibers can be assured at modest additional weight. For example, coatings of vapor grown carbon nanofibers (VGNF) and hybrid VGNF/graphene platelet nanocomposites on conventional composite substrates reduces the surface resistivity from $>10^{12}$ ohm/square to that of $10^3 - 10^4$ ohm/square and successfully satisfies the ESD requirements for spacecraft applications.⁴⁵

Conducting Adhesives

A good electrical bonding of joint is needed to assist in controlling and dissipating the build-up of electrostatic charges. Typically, the requirement is less than 1000 ohms for bonds between composite materials and structure. For bonding across joints in composite materials an electrically conductive adhesive is filled with high amounts of powdered silver, nickel, or carbon black, up to 60% by volume. However, this makes the adhesive bond weaker, and hence, a structural adhesive is needed in addition to the electrically conductive adhesive. Recent industry and government research efforts are developing a multifunctional carbon nanofiber filled adhesive with high electrical conductivity, while meeting the requirements for lap shear strength and viscosity.⁴⁶

Lightning Strike Protection

Another related application involving much larger currents (up to 200 kA) is lightning strike protection. With 40 aircraft accidents and 290 fatalities attributed to lightning strike incidents between 1963 and 1989,⁴⁷ this unpredictable act of nature can lead to loss of pilot control as well as burning, eroding, and distortion of the structure. As more structures use composite materials, strategies that use bulky films and meshes are being employed for protection. The key challenge is to either fully dissipate the energy or direct it into an easily repairable failure modes without compromising the structure or the flight.^{48,49} New approaches which attempt to exploit lighter weight conductive nano-filled composites are actively being explored by several researchers.⁴³

Enclosures for Electromagnetic Shielding

Traditional enclosures of electronics and equipment for air and space structures have usually been made of aluminum and other electrically conductive metals that provide electrostatic discharge protection, electromagnetic shielding, fault current return, an antenna ground plane, and lightning protection. In recent years composite materials have been used because of their lighter weight, high strength, and ease of fabrication. But, extra steps must again be taken to achieve the desired electromagnetic properties. In this particular application, the electrical properties of the structure must meet the $> 60\text{dB}$ shielding effectiveness level in order to shield against electrical interference. One traditional solution is to use a 3 mil aluminum foil co-cured with the graphite composite prepregs. However, this has not proved to be the best solution due to cost, questionable adhesion and limitation in the ability to repair or rework areas despite the favorable shielding efficiency. An alternative approach is to use a lightweight veil of nickel nanostrands to achieve the desired level of shielding with good adhesion and the ability to repair. A composite with alternating layers of fibers oriented 0 and 90 degrees relative to each other and with the two top layers of nickel nanostrand veils showed shielding levels of greater than 60 dB over the useful frequency range, as shown in Figure 3.^{43,50} Hence, only a few mils of a nanostrand composite film were found to create a highly effective electromagnetic interference (EMI) shield across a wide bandwidth. While 60 dB is a respectable shielding level, it is anticipated that thicker or more concentrated nanostrands may provide even better broadband shielding with minimal weight increase.

Conclusion

Within this article we have attempted to give a general overview of the near-term promise for nanotechnology within aerospace polymer matrix structural composites. While these composites are often considered light-weight and integrated alternatives to traditional metals, they also have trade-offs in dimensional stability, temperature capability, electrical conductivity and thermal transport. Nanocomposites are addressing many of the near term needs of composites structures by providing mechanical and multi-functional improvements with modest additional weight. In the far term, one can envision integrated structures in which more advanced functions are embedded into the structure to actively manage mechanical, thermal, electrical, or optical loads, as well as actively select, sense, convert, store and transmit the various energies within an intelligent structures.

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Figures

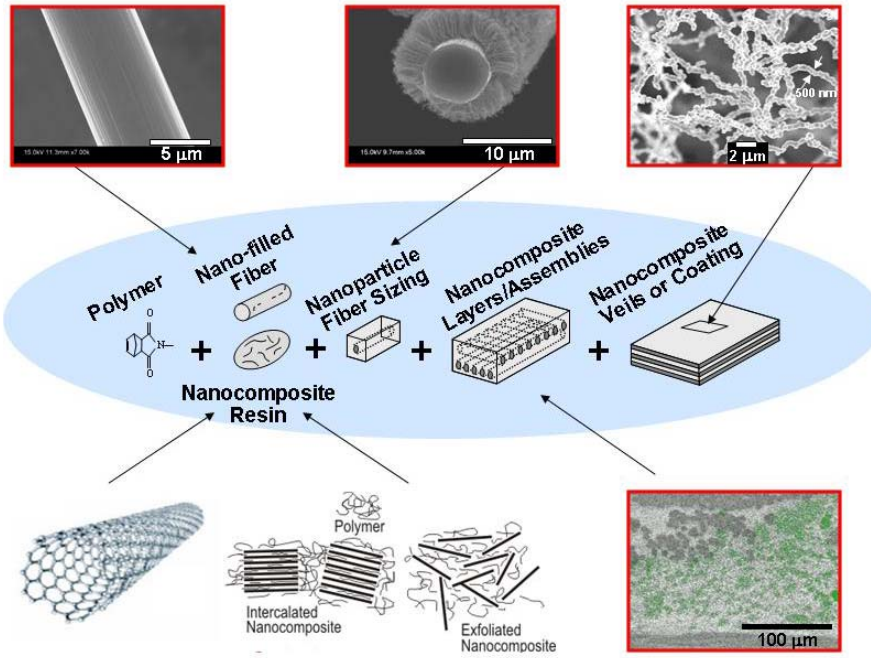


Figure 1

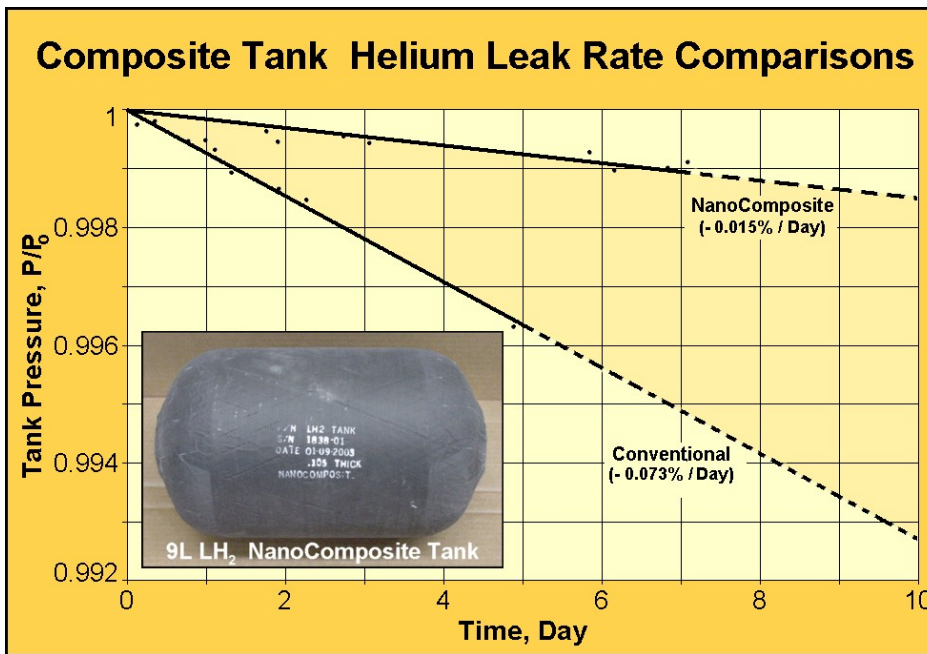


Figure 2

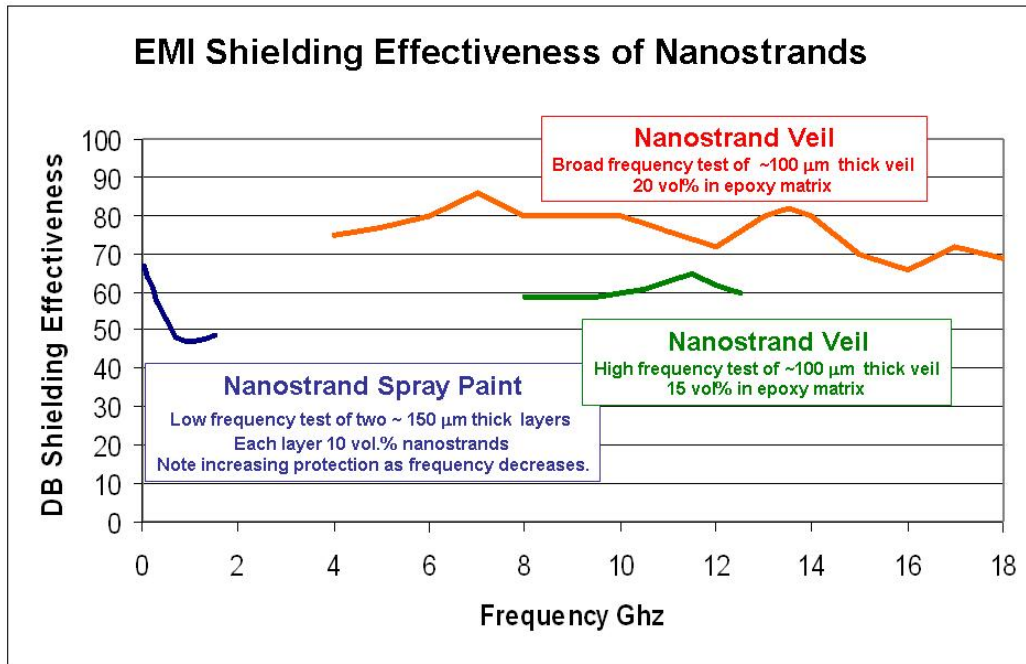


Figure 3.

Figure and Table Captions

Figure 1. Potential hierarchical integration of nanoparticles within a multi-scaled composite.

Figure 2. Nanoclays show reduced permeability to helium for tanks applications.

Figure 3. Summary of Shielding Effectiveness (SE) test results for composites with nickel nanostrand coating and epoxy infused nickel nanostrand veil of two different thickness values

Table 1. Approaches to and applications of nanocomposites within aerospace structural composites. Abbreviations include: CNT – carbon nanotube CNF – carbon nanofiber, POSS – polyhedral oligomeric sisesquioxane materials, ESD –electrostatic dissipation.

Table 2. Properties of single-walled carbon nanotubes in comparison to other aerospace materials.

Tables

Table 1

Property	Common Nanocomposite Approach	Potential Application
Physical/Chemical		
Permeability	Inclusion of impermeable, high aspect ratio silicate or graphite flake in resin	Cryogenic tanks, durability to diffusion species
Outgassing	Inclusion of impermeable, high aspect ratio silicate or grapheme in resin	Optical benches, interferometer, antenna truss structures
Oxidative Resistance	Incorporate high temperature, oxidative resistant fillers (silicate, CNT,POSS, etc.) that form passivating layers or slow oxidative erosion in resin or as coating	Thermal protective systems, atomic oxygen resistance
Electrical		
ESD	Incorporate high aspect ratio conductive particles such as CNT, graphite flake, metals, etc as percolated network in resin between conductive fibers	Adhesives, coatings, gap fillers
EMI	Create films of highly percolated network of conductive nanofillers (nickel nanostrand veil, SWNT buckypaper, etc.) that can both absorb and dissipate broadband frequencies	Bus compartment enclosure, electronic enclosures
Lightning Strike	Incorporate conductive nanofillers (nickel nanostrands, CNT, etc.) as highly percolated coatings, appliquéés, resins, or veils that can carry large currents and have controlled failure modes	Composite aircraft exterior
Thermal		
Thermal Conductivity	Incorporate highly thermal conductive particles (CNT's, metals, etc.) into resin and optimize structure for heat transfer along continuous path to heat sink	Adhesives, gaskets, radiators, doublers, electronics board, solid state laser heat removal
Thermal Protection Systems	Use thermally conductive and insulating nanofillers within resin to assist larger structure components to direct heat away from protected systems	Aircraft brakes, re-entry vehicles, missiles
Coefficient of Thermal Expansion	Incorporate nanofillers with low expansion coefficients and good matrix bonding such as (functionalized CNT, CNF, silicates, etc.) into resin or as fiber sizing to reduce CTE mismatch with fiber by composite effect and restriction of polymer motion	Adhesives, space apertures
Mechanical		
Toughness	Incorporate nanofillers like CNT into resin to increase energy dissipation on failure through deformation, pull-out, crack bridging, etc. at needed plies	Membrane structures, damage tolerant structures
Modulus	Incorporate high modulus nanoparticles like continuous CNT yarns/sheets as reinforcement or grow reinforcements between plies to increase out of plane modulus	Precision stable structures
Compression Strength	Incorporate high strength nanoparticles such as functionalized carbon nanotubes into the resin	Propulsion tanks, fittings
Interfacial Shear Stress	Grow high strength nanoparticles such as CNT from fiber to tailor the interfacial properties as a smart sizing	High temperature composites, vehicle health monitoring
Interlaminar Shear Strength	Incorporate nanofillers like CNT that can increase energy dissipation on failure through deformation, pull-out, crack bridging, etc. into resin at mid plies via coating or prepregging	Tubular structures

Table 2

Material	Specific Gravity (g/cm³)	Yield Strength (GPa)	Elastic Modulus (GPa)	Thermal Cond. (W/m-K)	Elect. Resistivity (μOhm-cm)	Normalized Strength-to-Mass Ratio
Carbon Nanotube (SWNT Theory)	1.4	65	1000	~6000	<1	225
Measured SWNT Yarns and Sheets	1.4	1.8	80	150	3	7
Conventional Carbon Fiber, M55J	2.2	4	550	70	220	9
IM7 Carbon Composites	1.6	2.1	152	30	2000	7
Titanium	4.5	0.9	103	12	127	1
Aluminum	2.7	0.5	69	180	4.3	1

Author Biographies

Jeff Baur is a Senior Research Engineer for the Advanced Composites Branch within the Air Force Research Laboratory's Materials and Manufacturing Directorate. He received his Ph.D. from the Massachusetts Institute of Technology's (MIT's) Program in Polymer Science and Technology in 1997. He has held research and research management positions within the Air Force Research Lab, Borden Chemical UV Coating Division, and at MIT's Institute for Soldier Nanotechnologies with publications in advanced electrical, optical and mechanical properties of polymer composites. His current interests are in nanocomposite for improvement of fiber reinforced composite structures and materials for morphing structures.

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