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A COMPARISON OF ELECTRICAL PROPERTIES OF CARBON NANOTUBE-LOADED RESINS

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ABSTRACT

Increasing attention toward electromagnetic interference (EMI) in defense applications has resulted in initiatives to develop multifunctional materials that can satisfy structural performance requirements while effectively shielding electronic components from EMI. The goal of this article is to characterize the electrical properties of carbon nanotube (CNT) loaded resins with an emphasis on those properties that directly influence EMI shielding effectiveness; particularly conductivity. Limiting the measurements to conductivity allowed studying a wide range of candidate materials to identify the most promising combinations of overall cost, manufacturing process and materials. Various parameters affecting the conductivity of CNT-loaded resins were considered in this study from CNT characteristics (CNT loading as weight percentage and functionalization) and dispersion processes (sonication or microfluidization) used during fabrication. Electrical testing of specimens was conducted using a low-frequency impedance analyzer in order to measure the conductivity of manufactured CNT-loaded materials for a wide range of frequencies depending on the experiment. For the materials and conditions tested, the percolation threshold (CNT loading that produces a conductive material) was established to be approximately 0.3% by weight. Given the low percolation threshold, these results can be considered as a positive indication that CNT-loaded resins can be incorporated into conventional composites intended for load bearing applications and provide EMI shielding as well. An even more promising approach is to incorporate CNTs into composites using nanocomp-non-woven-fabric, which results in conductivities of 10^2 S/cm.

Keywords: Carbon nanotube, electrical properties, electromagnetic interference shielding, nanocomposites.

1 INTRODUCTION

There is significant interest in the design and manufacture of multi-functional structural materials that can integrate electrical, thermal, healing, and sensing functions with the basic properties of strength and stiffness in load-bearing applications [1]. Of particular interest is the development of materials that can satisfy structural performance requirements as well as provide shielding from electromagnetic interference (EMI) in demanding electromagnetic environments. Protecting electrical components is critical where many high electric powered systems are in close proximity, and is increasingly becoming a concern with the growth and dominance of commercial electronics equipment [2].

The discovery of carbon nanotubes (CNTs) by Iijima [3] and their subsequent integration with fiber-reinforced polymer (FRP) composites has allowed the development of multifunctional composite materials that are capable of providing EMI shielding and still satisfy performance requirements associated with impact resistance. Due primarily to the advantageous properties of CNTs (high electrical conductivity, high tensile strength, high flexibility, high stiffness, and high thermal conductivity) traditional FRP composites can experience significant improvement in mechanical, electrical, and thermal properties when they are loaded with CNTs [4]. Nonetheless, obstacles remain toward a complete implementation of CNTs as reinforcing materials because of:

- lack of standardized manufacturing processes associated with integrating CNTs within composite materials (particularly dispersion),

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- variation in physical and chemical properties of CNT-infused resins, which are greatly affected by the quality of CNT dispersion,
- variation in purity of commercially available CNTs, which depends on source and fabrication technique, and
- high cost of CNT materials.

In this article, we highlight a specific aspect of multi-functional materials – EMI shielding, which strongly depends on the electrical properties of materials. For this reason, we concentrate on characterizing the electrical properties of CNT loaded resins; particularly their dependence on dispersion processes and CNT characteristics, such as CNT percent content by weight (CNT wt.%). In addition, we examine the electrical properties of an innovative approach to distribute CNTs uniformly in E-glass/epoxy composite panels using CNTs assembled into a non-woven textile form.

1.1 Overview of EMI shielding

EMI is caused by rapidly changing voltages and currents in various electrical and electronic devices. Ambient EMI can be considered electromagnetic pollution, most of which consists of spurious, conducted or radiated signals of electrical origin, such as radiation emitted from telecommunication equipments. This electromagnetic pollution can adversely alter the operation of sensitive circuits in solid state electronic components found in many defense and civilian instruments. A means of properly protecting this type of equipment is to utilize materials capable of EMI shielding as containment or housing.

There are three mechanisms involved in EMI shielding: reflection, absorption, and multiple reflections of EMI radiation.

- The reflection mechanism requires materials to possess mobile charge carriers (electrons or holes) to interact with incoming electromagnetic radiation waves; i.e. electrically conductive materials, which are only required to have a low level of conductivity (or its inverse, resistivity) to provide effective shielding. A volume resistivity on the order of 1.0 ohm-cm is typically sufficient for effective shielding [5].
- The absorption mechanism requires the material to have electric dipoles (materials with high-dielectric constant values) and/or magnetic dipoles (materials with high-magnetic permeability values) to interact with incoming electromagnetic radiation waves, and is highly dependent on the thickness of the shield.
- The multiple reflections mechanism requires the material to have large surface or interface areas where multiple reflections of radiation waves can occur.

In all three mechanisms, shielding is achieved by electromagnetic radiation losses that are controlled by a material's electrical conductivity and/or magnetic permeability.

Polymers and fibers (glass or carbon) generally contain negligible concentrations of mobile charge carriers or electric dipoles. This lack of mobile charge carriers makes polymer-based composites non-conductive (electrically insulating) and thus transparent to electromagnetic radiation. In order for FRP composites to achieve any level of EMI shielding capacity, they must be made electrically conductive by incorporating (loading) intrinsically conductive fillers, such as carbon black, metal particulates, or nickel-coated short carbon fibers [6]. A very promising polymer filler to achieve EMI shielding is multiwall CNTs (MWCNTs). Previous studies have shown that MWCNT polymer nanocomposites provide EMI shielding through the absorption mechanism [5]. Also, the multiple-reflection mechanism's shielding effectiveness is controlled by the shield thickness; if the shield is thicker than the so called 'skin depth' the contribution from this mechanism can be ignored [7], which we assume in this study. The sum of electromagnetic radiation losses due to reflection, absorption, and multiple reflections (which is negligible relative to the other two) constitutes EMI shielding effectiveness. Losses from reflection and absorption are difficult to characterize for CNT nanocomposites since they depend on many factors that are difficult to control; this has been reported in a number of studies where shielding effectiveness values display large variability in similar materials [8]. Some of the more critical factors include processing synthesis, purity of CNTs, and dispersion of CNTs. There are two primary approaches used to predict EMI shielding effectiveness of CNT nanocomposites; one is experimental and the other is semi-empirical. In both cases, material conductivity plays an important role; other parameters that are important include the dissipation factor and the dielectric constant [9], neither of which was studied.

Intuitively, the conductivity of polymer-based composites should increase as conductive filler loading (filler concentration) is increased. However, full conductivity does not occur instantaneously, a critical filler concentration is needed for the material to display a dramatic increase in conductivity – essentially the point at which the material is converted from an insulator to a conductor. This critical concentration is known as the electrical percolation threshold concentration, or the percolation threshold. This is the point at which the filler particles form a continuous 3-D conductive network (percolating networks) throughout the resin matrix. These percolating networks allow electrons to tunnel from one filler particle to another in order to overcome the inherent high resistance of the resin matrix. The formation of percolating networks depends primarily on the filler's intrinsic conductivity, the particles' geometric aspect-ratio and the distribution of the particles.

Since CNTs have high conductivity and high aspect-ratio, the resulting nanocomposites can be made conductive at a low percolation threshold provided that the CNTs are uniformly dispersed throughout the resin. The conductivity of nanocomposites is also affected by the type of polymer, CNT type, CNT surface functionalization, and synthesis method [8]. The polymer type is typically chosen to address a function other than EMI shielding (e.g. structural performance or durability); or it can be selected based on availability and cost. CNTs tend to agglomerate because of inherent electrical charge, which adversely affects the uniform dispersion of CNTs throughout the resin material. One approach used to provide uniform CNT spatial distribution is to chemically functionalize (surface chemical treatment) CNTs. However, this technique disrupts the extended p-conjugation of CNTs and reduces electrical conductivity, as will be shown in the results section. In this study, various parameters affecting the electrical properties of CNT-loaded resins were considered, including CNT characteristics (loading by wt.% and functionalization) and dispersion processes (microfluidization or sonication). Additionally, the use of an innovative approach to assemble CNTs into a non-woven textile form to uniformly distribute CNTs in polymer composites is investigated.

The microfluidizing process uses a proprietary apparatus (Microfluidics Inc.), which entails a pump that forces the mixture of resin and CNTs through a Z-shape chamber with a very small rectangular cross-section (as small as 50 mm across); the sharp turns and small orifice of which create a shear force, impact, and cavitation to deagglomerate and disperse CNTs into the resin. The main parameters affecting the effectiveness of this dispersion process are the number of passes through the pump, the pump pressure, and the chamber pressure. The sonication (or acoustic cavitation) process uses alternating acoustic pressures above the cavitation threshold to create large numbers of small cavities in the liquid resin to deagglomerate and disperse CNTs into the resin. This however is limited to a small region close to the source of the ultrasound waves, usually a probe. The main parameter affecting the effectiveness of this dispersion process is the frequency of the applied sound (pressure) waves (approximately 20 kHz in our study). Therefore, the microfluidizing process is the most effective approach to uniformly disperse CNTs because it is continuous, repeatable, and scalable.

CNT-based nanocomposites have a number of advantages over conventional metal-based and other EMI shielding materials; including light weight, corrosion resistance, flexibility, and ease of processing. In fact, the use of CNTs can lead to a significant reduction of filler loading required to achieve a desired level of EMI shielding. For instance, percolation thresholds in the range of 5–15% volume concentration are typical for carbon black filler, and are even higher for dispersed metal particle fillers, 10–30% [9]. By comparison, 0.3% weight percolation thresholds for CNTs were found in this study, which demonstrates the potential to reduce weight and overall costs if incorporated in FRP composites.

2 MATERIALS AND TESTING

In this article nanocomposites were manufactured using the vacuum assisted resin transfer molding (VARTM) process. Multi-wall CNTs produced by catalytic chemical vapor deposition (CCVD) were obtained from www.cheaptubes.com. In general, the CNT/epoxy manufacturing process first involved dispersion of CNTs in an epoxy resin via microfluidization or sonication. Next, the mixture was poured into room temperature vulcanizing (RTV) silicone molds and bagged. The samples were then subjected to vacuum pressure for 24 hours followed by an elevated post-curing temperature of 100°C for 1 hour. Twenty four hours after the elevated temperature post-cure, the samples were removed from the molds and cut into 1-mm thick specimens with dimensions of 63.5 mm (2.5 inches) long and 12.7 mm (0.5 inches) wide. For further analysis, the two dimensions were measured at three different points and averaged.

The non-woven CNT fabric was sandwiched in an E-glass FRP composite, which was fabricated using the VARTM process. The non-woven CNT textile is proprietary and was obtained from Nanocomp Technologies, Inc (www.nanocomptech.com). E-glass fiber reinforcement in woven rovings having a unit weight of 800 g/m^2 and width of 1.27 m was used. SC-15 epoxy resin was used due to its high strength, low viscosity, and extensive use in structural composite applications. A composite panel was manufactured using the aforementioned CCVD and post-cure process, and cut into samples of 63.5 mm long and 12.7 mm wide. The only difference being that the glass FRP composite samples were 4.6 mm thick. Details of the manufacturing process are provided in Estrada *et al.* [10].

An HP 4192 low frequency impedance analyzer was used to measure conductivity of CNT-loaded resin samples and glass FRP nanocomposite samples. The ends of each specimen were coated with silver paint to ensure connectivity with the impedance analyzer during measurements, see Fig. 1. Conductivity of specimen was measured for a wide range of frequencies depending on the experiments.

2.1 Test plan

There are two primary approaches used to predict EMI shielding effectiveness of CNT-based nanocomposites, one is entirely experimental and one semi-empirical. Both are discussed in Estrada *et al.* [10]. In this article, we focus on conductivity (or the inverse of resistivity) measurements of CNT-loaded resins as part of the semi-empirical approach to evaluating EMI shielding effectiveness. Establishing conductivity is considered critical in order for a

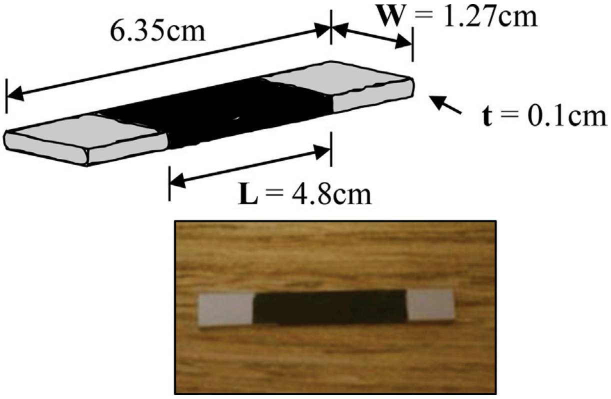

Figure 1: Sample dimensions and preparation procedure for impedance testing.

material to provide EMI shielding; therefore, this was the primary material property characterized from fabricated materials in this experimental program. The manufacturing of samples and test plan was developed in order to evaluate the composite conductivity as a function of CNT properties (CNT loading as weight percentage and functionalization) and dispersion process (sonication or microfluidizing). The effects of these different factors on conductivity were examined according to the test plan detailed in Table 1. All experiments were performed using CNTs with diameter of 8–15 nm and length of 10–50 µm loaded in SC-15 epoxy resin.

Experiments 1–3 examined samples that were fabricated using the microfluidizing dispersion process with CNT loadings between 0.2 and 0.8 weight percent (wt.%). These first three experiments were used to determine the minimum number of passes needed during the manufacturing process in order to achieve conductivity. Twenty passes through the microfluidizer were performed for each experiment, with five samples manufactured every two passes.

With optimal dispersion identified and conductivity established, experiments 4–7 were performed in order to understand the effect of functionalizations (OH and COOH) on material conductivity. Experiments 8 and 9 were performed to investigate the combined effect of functionalized and non-functionalized CNTs. These experiments were intended to determine the influence of partial functionalization. Functionalization is intended to improve dispersion of CNTs; however, as shown in Table 1, it prevents CNT-loaded resins from developing conductivity and thereby prevents its EMI shielding effectiveness. Experiment 10 was performed to compare the effects of dispersion via sonication versus microfluidization.

3 RESULTS

3.1 Microfluidized samples

As listed in Table 1 most of the experiments were conducted with samples fabricated using the microfluidizing process (except experiment 10). The dispersion process is noted in Table 1 under the ‘Process’ column, which describes the chamber pressure and shear pressure used in each experiment. During microfluidizing, the temperature was maintained at 25°C. The conductivity was measured at three different frequencies, 0.1, 1, and 10 kHz; all provided similar conductivities for the same conditions – as will be shown later.

Table 1: Test plan for conductivity of MWCNT-loaded resin materials.

Exp. No.	Functionalization	CNT wt.%	Process[1,2]	Notes	Conductive
1	—	0.2	M/100/15	20 passes	Very low
2	—	0.4	M/100/14	20 passes	Yes
3	—	0.8	M/100/15	20 passes	Yes
4	OH	0.8	M/70/11	20 passes	No
5	OH	1.6	M/70/11.5	20 passes	No
6	COOH	1.6	M/70/11.5	4 passes	No
7	COOH	4	M/70/13	3 passes	No
8	OH	1	M/70/13	4 passes	Yes
	—	0.4	M/75/13		
9	COOH	1	M/70/12	4 passes	Yes
	—	0.4	M/80/14		
10	—	0.6	S	—	Yes

[1] Process = M/100/15 (microfluidizing at 100-psi chamber pressure and 15-ksi shear pressure).

[2] Process = S (sonication).

The following factors were found to have the greatest effect on conductivity of microfluidized samples: CNT loading, chamber pressure, shear pressure, and number of passes through the microfluidizer. Experiments 1–3 were an attempt to isolate the effect of each of these variables with respect to CNT loading and the number of passes necessary to achieve conductivity. CNT loadings of 0.2, 0.4, and 0.8 wt.% were used in order to identify the percolation threshold (loading when the material achieves conductivity). The results of 0.2 wt.% CNT loading specimens (experiment 1) are excluded because their conductivity was negligible; and since the 0.4 wt.% CNT-loaded specimens were conductive, it was concluded that the percolation threshold is between the 0.2 wt.% and 0.4 wt.% CNT loadings.

The results of the conductivity for 0.4 wt.% and 0.8 wt.% CNT loadings (experiments 2 and 3) can be obtained using the results depicted in Fig. 2 – conductivity is the inverse of resistivity. The samples revealed no statistically significant conductivity improvement after *two passes*; thus this was considered the adequate number of passes to achieve proper dispersion. Two passes are ideal, particularly for higher CNT loadings for which mix viscosity increases.

Figure 2 also provides a comparison of the resistivity for CNT loadings of 0.4 wt.% and 0.8 wt.% at different frequencies. From these results it appears that the resistivity increases slightly, which suggests that the conductivity decreases, as the number of passes through the microfluidizer increases. This change in conductivity is speculated to be the result of a gradual decrease in the CNT aspect ratio with every pass because of CNTs being broken up into smaller lengths. Because of the smaller aspect ratio, particles tend to attract resulting in faster agglomeration and a more drastic increase in resistivity.

In general, the results of conductivity measurements for CNT-loaded resins at 0.4% and 0.8% indicate little potential for EMI shielding applications. The CNT-loaded epoxy specimens display a conductivity of about 10^{-6} S/cm, which others have correlated to a shielding effectiveness of less than 5 dB. A greater load of CNTs would need to be loaded in the resin in order to attain a meaningful level of shielding effectiveness. For instance, Lin *et al.* have observed that resins containing 7.0 wt.% CNT loading have conductivity of 0.7 S/cm and a corresponding shielding effectiveness of 33 dB sufficient for commercial electronics [11].

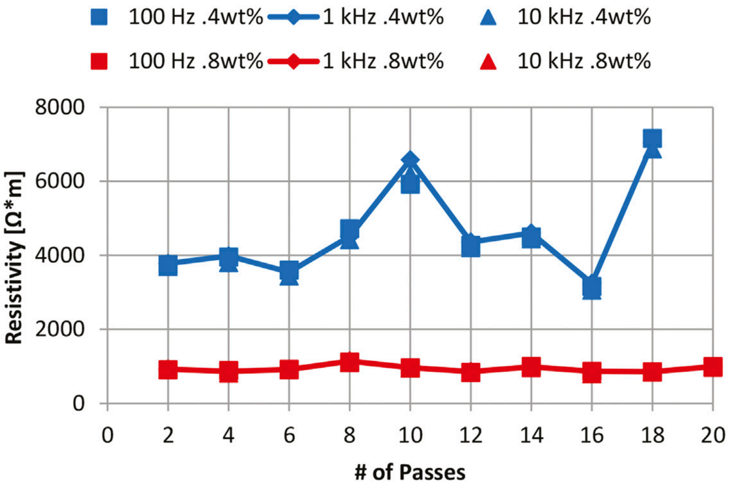

Figure 2: Comparison of 0.4 wt.% and 0.8 wt.% resistivity versus number of passes through the microfluidizer – experiments 2 and 3.

3.2 Sonication and microfluidization

Experiment 10 was conducted primarily to compare the electrical properties of sonication to microfluidizing dispersion methods. The sonication samples showed conductivity comparable to that of microfluidized samples. The electrical properties results for 0.4 wt.% CNT loading sonicated (experiment 10) and microfluidized (experiment 2 with two passes) are listed in Table 2; and the resistivity is compared in Fig. 3. Though the results of the sonicated samples are comparable to those using the microfluidizing process, the later process is a better choice for dispersing CNTs because it is more efficient. Not only is the sonication process more time-intensive, the process also induces a large heat buildup during dispersion, which can adversely influence resin properties. Also, the size-distribution of sonicated CNT samples must be further analyzed to determine any damage done during sonication.

After the samples cured, microscopy was used to examine the dispersion of CNTs. The microscopy was conducted using a scanning electron microscope (Jeol Model JSM-6390 SEM) to view the CNTs in the specimens at the nanoscale. The results of the morphological investigation are used to characterize the distribution of CNTs in the hardened resin matrix. In order to view the CNTs in the resin material, the samples were first fractured, which

Table 2: Results of 0.4 wt.% CNT loading in SC-15 using sonication (experiment 10) and microfluidizing (experiment 2 with two passes).

	Dispersion process	0.1 kHz	1 kHz	10 kHz
Resistivity (Ω m)	Sonication	3387	3044	3401
	Microfluidizing	3744	3775	3697
Conductivity (μS/m)	Sonication	295	328	294
	Microfluidizing	267	265	272

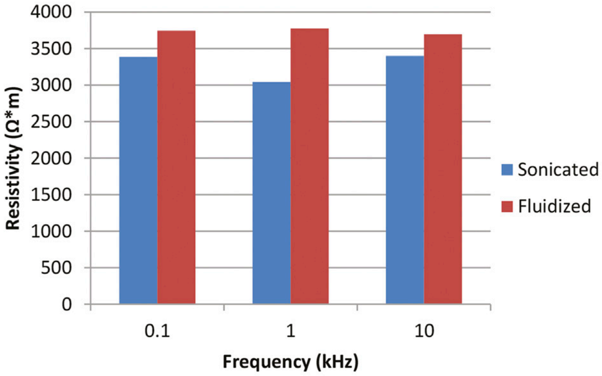

Figure 3: Sonication (experiment 10) versus microfluidizing (experiment 2 with two passes) resistivity for 0.4 wt.% CNT loading.

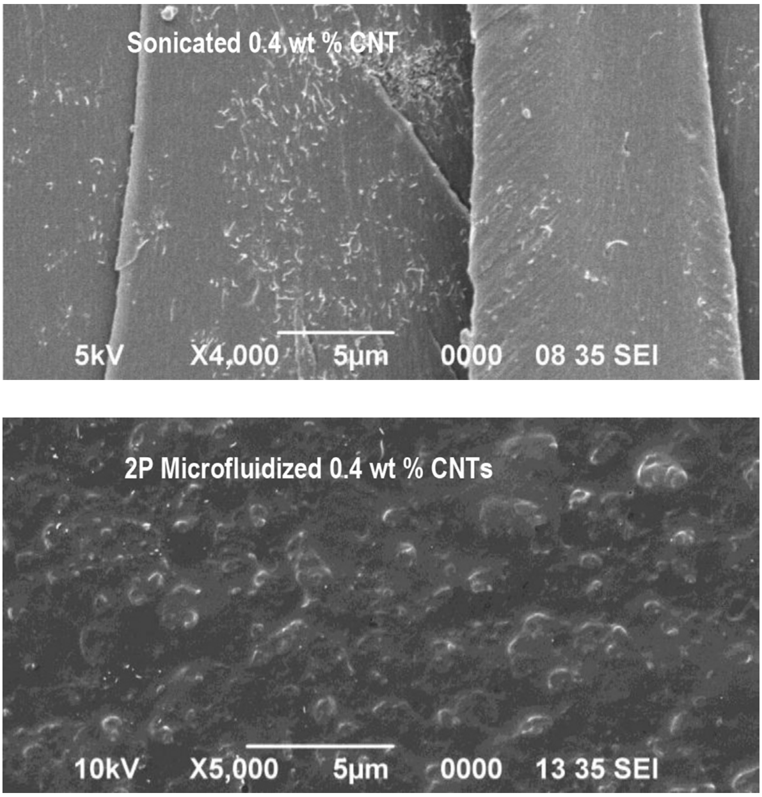

Figure 4: Morphological comparisons of sonicated and microfluidized samples.

allowed any CNTs bridging the fracture surfaces to pull out from the resin material. One side of the fractured sample was then prepared for imaging. Figure 4 shows a comparison of samples containing CNTs dispersed using the sonication and microfluidizer (two passes) dispersion methods. Notice that the microfluidizer method produces a more uniform CNT dispersion – the white dotted structures are CNTs.

3.3 Effect of functionalization

Experiments 4–7 focused on studying the effect of functionalized CNTs on resin conductivity. There are several types of functionalized CNTs available; the one used in these experiments has carboxyl functional groups (COOH) and hydroxyl functional groups (OH). The main advantage of the functionalized CNTs is a smaller increase in resin viscosity compared to regular CNTs. CNT loadings of up to 0.4 wt.% were fluidized without any viscosity-related problems. Although the functional groups occupied between 0.2% and 0.4% of the CNTs by surface area, they likely increased the tunneling distance and obstructed the network from achieving electrical percolation. This resulted in insignificant conductivity of the material. Although the conductivity of the material was compromised by the functionalized CNTs, mechanical properties were noticeably improved.

While samples loaded with functionalized CNTs were not conductive; it is hypothesized that this occurrence is a result of functional groups preventing CNTs from forming the networks needed to initiate percolation. In experiments 8 and 9, functionalized and regular CNTs were combined, with regular CNTs intended to bridge the gaps that functional groups cause. The combination of functionalized and regular CNTs resulted in conductivity comparable to that of the non-functionalized CNT-loaded resins in experiments 2 and 3, as shown in Fig. 5. These results indicate that the addition of non-functionalized CNTs did not bridge the gaps caused by the functional groups between CNTs, but instead created their own percolating network. However, the addition of regular CNTs substantially increased the viscosity of the mix and made it very difficult to process the material as compared to non-functionalized samples. It is also interesting to note that a negative conductivity was recorded for the 1 wt.% COOH material as shown in Fig. 5. Although negative resistivity is possible [12], this may actually be an error in the readings.

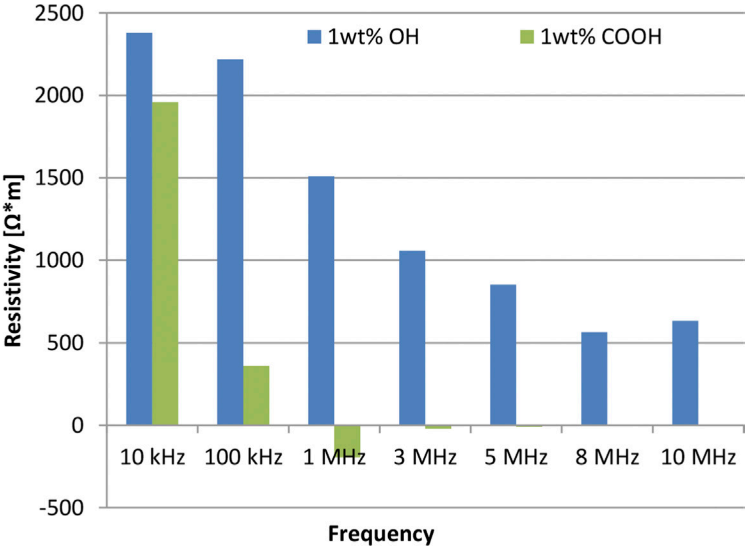

Figure 5: Resistivity of 0.4 wt.% non-functionalized CNTs with varying combinations of functionalized CNTs in SC-15 – experiments 8 and 9.

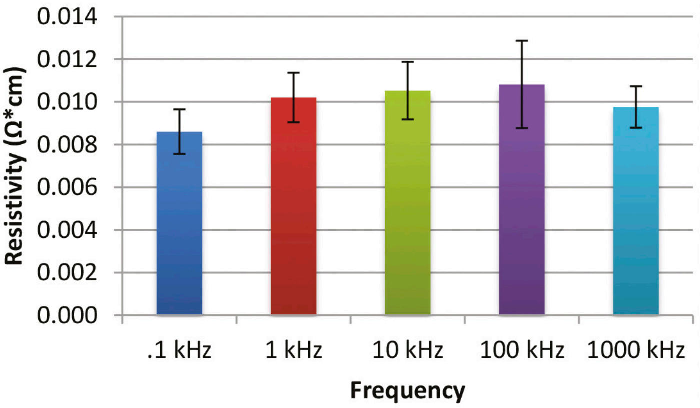

Figure 6: Resistivity of nanocomp composite panel.

3.4 GFRP composite with non-woven CNT fabric layer

This section summarizes the results of glass FRP (GFRP) composites manufactured with a non-woven CNT fabric layer. The non-woven CNT fabric or nanocomp material was used because embedding CNTs in sheet form, as opposed to mixing with the resin, facilitates the manufacturing process and production needed in infrastructure applications. The results for resistivity for nanocomp composite samples are shown in Fig. 6. The figure includes the average of a series of three tests and corresponding standard deviations, which are shown as error bars. The resistivity varied with the orientation of the samples in the impedance analyzer test rig (up to 62% as shown by the error bars). However, when averaged, the resistivity values converged to results close to those of more repeatable samples.

The nanocomp fabric exhibits a much higher conductivity than the nanocomposites prepared using powder CNTs. The results indicate that at all frequencies the average resistivity is comparable; the low resistivity indicates that the material is highly conductive (10^2 S/cm). The high value of conductivity implies that the nanocomposite is a viable candidate for shielding EMI. And based on a semi-empirical model discussed in Estrada *et al.* [10], this material can provide a calculated 99% EMI shielding effectiveness.

4 CONCLUSIONS

This article presents an overview of EMI shielding provided by fiber reinforced CNT-loaded epoxy composite (nanocomposite) materials. The main focus of this article was the characterization of electrical properties of CNT loaded resins with an emphasis on those properties that directly influence EMI shielding effectiveness; particularly conductivity. This was deliberate so as to study a wide range of candidate materials to identify the most promising ones based on combinations of overall cost, manufacturing process, and materials. That is, the experimental work centers on the development of a material system with electrical properties that can shield EMI waves, which a minimum requires a material to be electrically conductive. Since most epoxy resins are not electrically conductive, a separate conductive material must be integrated in order to develop conductivity. Electrically conductive particulate materials (such as CNTs) can make epoxy conductive even at low loadings (concentrations) since electron tunneling occurs even when particles do not come in contact.

Several conclusions regarding the conductivity of CNT-loaded resins can be drawn from the results of this experimental program.

1. The microfluidizing dispersion process for CNT loadings is more effective than sonication. Also, microfluidization displays the most potential for scaling to large quantities; particularly considering that it only requires two passes of the resin through the system for optimal conductivity. Several loadings were tested and the percolation threshold was estimated at approximately 0.3 wt.%.
2. Though OH and COOH functionalizations lead to a smaller increase in resin viscosity compared to regular CNT loadings (and thus better dispersion), functionalization prevents the CNT-loaded material from developing conductivity. However, in cases where a large amount of CNTs may be needed to improve mechanical or thermal properties, non-functionalized CNTs can be added to the functionalized CNT-loaded resin to develop a conductive material system.
3. While conductivity was developed in CNT-loaded resin at 0.4% and 0.8%, the low values of conductivity (~10^{-6} S/cm) indicates that these materials have very low potential as effective EMI shielding. Their properties may potentially be improved at higher CNT concentrations; however, additional studies would be required related to manufacturing processes. Although the fabrication process has challenges, one sheet of nanocomp non-woven fabric can produce high conductivities that can provide up to 99% EMI shielding effectiveness as estimated in Estrada *et al*. [10].

While these results show that CNT loaded materials have great promise for use in multi-functional applications, additional work needs to be completed in order to validate the behavior of practical infrastructure components and systems manufactured using these innovative materials. Specifically, more detailed investigations of combining CNT-loaded epoxy and glass fibers into a composite material need to be performed in order to take full advantage of the multi-functionality of these materials. Furthermore, there are a number of issues that remain with respect to experimental validation of EMI shielding effectiveness. Therefore, direct testing of EMI shielding effectiveness using these materials is necessary in order to further validate conductivity results.

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