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Detonation Nanodiamonds and Carbon Nanotubes as Reinforcements in Epoxy Composites—A Comparative Study

A comparative study between detonation nanodiamonds (DNDs) and carbon nanotubes (CNTs) as low concentration additives to epoxy composites is presented. The dispersibility of the different nanocarbons in resin solutions leading to uniform composite formation is also discussed. Significant increase in glass transition temperature was observed, which were 37 °C and 17 °C for DNDs and CNTs, respectively. Unlike the pure epoxy, the fractured surface of both composites showed resistance to crack propagation. Tensile properties of DNDs and CNTs composites showed an increase in microhardness by 41% for DNDs and 12% for CNTs, and a decrease in electrical resistivity by 2 orders of magnitude, with the CNTs showing lower resistivity. In general, the DNDs were found to be quite effective and at the reported concentrations between 0.1% and 0.5% and showed superior enhancement compared to the CNTs. [DOI: 10.1115/1.4024663]

Keywords: nanocomposites, detonation nanodiamond, carbon nanotube, thermal properties, microhardness, scanning electron microscopy (SEM)

Highlights

- Enhanced properties of low concentration detonation nanodiamond and carbon nanotube epoxy composites are presented.
- A significant increase in glass transition temperature (by 37 °C) for detonation nanodiamonds has been observed.
- Microhardness of the composites increased by 41% for the detonation nanodiamonds and 12% for the nanotubes and electrical resistance decreased by 2 orders of magnitude.

Introduction

Epoxy resins are an important class of structural polymers having excellent mechanical, thermal, and electrical properties, and are extensively used in fiber reinforced composite, laminates, structural adhesives, and protective coatings. In spite of some highly desirable attributes, the cross-linked epoxy polymers tend to be brittle and rigid. The crack propagates quickly and the low impact strength reduces its end applications. The poor conductivity also enhances its degradation rate as it accumulates electrical charges.

Epoxy nanocomposites have been synthesized using a range of nanocarbons such as carbon nanotubes (CNTs), fullerenes, and carbon fibers [1-5]. These nanocarbons have shown enhanced mechanical, thermal, electrical properties and offer higher corrosion resistance, fracture toughness and fatigue resistance [4,6-8].

A more recent development is epoxy-nanodiamond composites [9–12]. The relatively inexpensive large scale production of nanodiamonds by a detonation synthesis process (DNDs) has made it commercially viable for a broad range of applications [13–17]. The DNDs have tetrahedral network structures, and they comprise of a diamond core (sp³), a middle core (sp^{2+x}) and a graphitized outer core (sp²) that is often partially oxidized. The graphitized

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Fig. 1 (a) Photograph of dispersion of 0.05 wt. % MWCNTs (left) and DNDs (right) in mixed solvent (1:1 xylene-butanol) and (b) stability of in presence of 5 wt. % epoxy resin with time

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Table 1Particle size of 0.05 wt. % DNDs and CNTs in xylene-
butanol and in presence of 5% epoxy resin and TETA

Nanomaterials	Particle size with the addition of epoxy		Particle size with addition of TETA	
	0 wt. % epoxy	5 wt. % epoxy	0 wt. % TETA	5 wt. % TETA
DNDs CNTs	326.5 81.6	334 87.8	326.5 81.6	887.7 187.2

core provides chemical functionalizability as well as electrical conductivity. They also have large grain boundary density, and low to negative electron affinity which makes them suitable for electronic applications [18]. In addition, their high mechanical strength, stiffness, light weight, and low coefficient of friction are attractive for the fabrication of structural composites [19–22] and lubricants [23]. For the most part, studies on polymernanodiamond composites showed moderate improvement in mechanical properties of the composites [10–12,23–26]. A decrease in storage modulus of epoxy-nanodiamond composite has also been reported by Spitalsky et al. [27].



Fig. 2 Schematic representation of epoxy-nanocomposite with CNTs (left) and DNDs (right)





Fig. 3 Photographs of (*a*) epoxy-DND composite, (*b*) epoxy-CNT composite and 3D images of (*c*) epoxy-CNT composite surface and (*d*) epoxy-DND composite surface from digital microscope (Keyence). The concentration of DNDs and CNTs were 0.1% (scale bar: red 67.7, yellow 38.7, green 29.06, and dark blue 0.0μ m). For black and white print; the circle and black spot corresponds to 67.7 and 0 micron, respectively.

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 Image = 128 40 X r
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CNT composite, and (e) epoxy-DND composite. The percentage of DNDs and CNTs in the composites were 0.1% (scale bars—Figs. 4(a) and 4(c) are one micron, (b) and (d) are 200 nm, Fig. 4(e) is 300 nm).

As the epoxy-nanodiamond composites evolve, it is important to develop an understanding of their advantages especially with respect to CNTs and it is anticipated that they may have some distinct features. For example, it has been reported that the overall load does not transfer to another shell in nanotubes, thus the inner shells of multiwalled tubes do not contribute toward the load bearing cross-sectional area of the system. However, this problem is absent in nanodiamonds because of its crystalline structure [28]. Moreover, the long length of CNTs (few microns) can hinder the curing reaction of a polymer, and the mechanical properties of final composites are known to decrease when CNTs are present at higher concentration (>1.0%) [29]. On the other hand, the nanodiamonds have been reported to provide hard, water resistant coating for metallic and polymeric biomaterials [30].

In general, it is important to compare these two interesting and versatile materials as applied to epoxy composites. The objective of this study is the synthesis of epoxy-DNDs and epoxy-CNTs nanocomposites and compares their thermal, electrical and mechanical properties. The dispersibility of the nanocarbons is considered to be an important parameter and is to be taken into consideration.

Experimental

Chemicals and Materials. A bisphenol A diglycidylether based epoxy resin (D.E.R.TM 332, epoxy equivalent weight 176) and an amine hardener Triethylenetetramine (TETA) were used in this study. Xylenes (ACS Reagent, 98.5%) and 1-butanol (anhydrous, 99.8%) were used as solvents. All the materials and DNDs were purchased from Sigma Aldrich Inc., Saint Louis, MO. The raw multiwalled carbon nanotubes (MWCNTs) were purchased from Cheap Tubes Inc., Brattleboro, VT. For this study, both the CNTs and DNDs were purified by washing with dilute nitric acid. The average diameters of the CNTs were ~30 nm and a length of up to 15 μ m. The average particle size of DNDs was less than 10 nm.

Colloidal Behavior of Nanocarbons in Epoxy Resin and TETA. Effective dispersal of nanomaterials within the polymer matrices is an important step during the fabrication process. The nanocarbons were dispersed in a xylene, butanol (1:1) mixture. The mixed solvent provided compatibility for both nanotubes and the epoxy resin. The mixture was stirred mechanically for 30 min and then put in an ultrasonic bath for 3 h. The nanomaterials were

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Fig. 5 Tensile properties of pure epoxy and 0.1% epoxynanocomposites (*a*) tensile strength and (*b*) elongation at break (%).

found to be more stable for longer period of time in epoxy solution than in TETA. Hence, epoxy resin was added to the dispersion first and then mechanically stirred for an hour. The mixture was kept overnight in a vacuum oven at 70 °C for complete solvent removal. The dispersion was cooled under intense stirring condition and hardener was added for 10 min, cured for 24 h at room temperature and finally postcured at 60 deg for 6 h.

The colloidal behavior of CNTs and DNDs dispersed in dilute solution of epoxy resin and TETA was studied using dynamic light scattering (Beckman Coulter N4 Plus submicron particle size analyzer, operated at 90 deg detector angle) at 25 °C. The dispersions were prepared using 50 mg l^{-1} in xylene-butanol solution using sonication for 3 h. 5 wt. % of epoxy resin or TETA was added to the dispersion and stirred for 10 min. The stability of the nanomaterials for a period of time has been also studied.

Characterization of Nanocomposites. The nanocarbons and the composites were characterized by scanning electron microscope (SEM). SEM data were collected on a LEO 1530 VP scanning electron microscope equipped with an energy-dispersive X-ray analyzer. The composites were further characterized with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The glass transition temperature (T_g) was measured using a DSC analyzer (model DSC822e, Mettler Toledo, Switzerland). The temperature range for these experiments was 0–180 °C at a scanning rate of 10 °C/min. TGA (Perkin-Elmer Pyris 7 TGA system with a heating rate of 10 °C per minute under air atmosphere) was used to investigate the degradation of composite materials during thermal stress condition. The 3D image of the surface was taken by digital microscope (Keyence) and it provided information regarding planarity of the composite surface and the surface distribution of nanocarbons.

Mechanical properties of epoxy nanocomposites were measured by Instron 8516. Tensile tests were performed at ambient temperature and at the constant cross-head speed of 3 mm/min. Specimens were made in the dumbbell shape by compression molding. Vickers microhardness tests were performed using Leco Microindentation Hardness Testing Systems under a constant load of 25 gf. Volume resistivity of different composites was measured by using Keithley 6715B electrometer. Surface resistance was also measured using two silver paint electrodes leaving an effective area of 1×1 cm². The data represented in the figures and tables are the average of at least three experiments with relative standard deviations less than 1%.

Results and Discussion

The colloidal behavior of nanocarbons plays an important role in the composite formation. Due to strong electrostatic and van der Walls interactions, nanomaterials tend to form agglomerates, which behave as defects in polymer matrices, and consequently nano particles seldom reach their full potential [12]. The stability of the particles in a resin forming environment is important because it relates to the dispersibility into the polymer matrix. The change in particle size after sonication showed the agglomeration behavior of nanomaterials. Figure 1(a) shows the photographs of DNDs and CNTs dispersions in the mixed solvent system, where the DNDs suspension was found be optically transparent. It is noted that the particle size of CNTs and DNDs were not directly comparable because CNTs are cylindrical in shape. However, the relative growth of particles provides insight into the rate of agglomeration. As evidenced by the growth of the agglomerates shown in Table 1, it was found that both nanocarbons were quite stable in dilute epoxy solution for a longer period of time than in TETA. Figure 1(b) shows particle size as a function of time after the addition of epoxy. It is quite evident that the agglomeration was minimum during this period. Therfore the procedure involved first disperison in epoxy followed by the additon of TETA.

Characteristics of DNDs and CNTs Composites. Figure 2 shows the schematic representation of the composite formation where the DNDs and CNTs are incorporated into the structure. The photographs of the composites are shown in Figs. 3(a) and 3(b). The CNTs composite was significantly darker. Figures 3(c) and 3(d) shows three dimensional images of the nanocomposite surfaces. It is clear from the images that epoxy-DND composite



Fig. 6 Optical images of vickers indents produced with a load of 1 kgf for (*a*) pure epoxy, (*b*) epoxy-CNT composite and (*c*) epoxy-DND composite. Concentration of nanocarbons in the composites were 0.1%.

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Fig. 7 (a) TGA thermogram of pure epoxy and 0.1% epoxy-nanocomposites and (b) DSC curve of pure epoxy and epoxy-nanocomposites.

had more planer surface in comparison to epoxy-CNT composite, indicating less agglomeration of nanoparticles and better leveling effect of polymer.

Figures 4(a) and 4(b) shows the SEM images of the original CNTs and DNDs, and Figs. 4(c)-4(e) shows the SEM images of cross-sectional view of pure and epoxy-nanocomposites. Good dispersion of both CNTs and DNDs within the matrix was observed. Due to the brittle nature of epoxy, the pure polymer is known to show interface cracking under quasi static and cyclic loading [31]. With incorporation of DNDs into the polymer matrix the strength and stiffness are expected to improve. The cross-sectional SEM images of both composites showed resistance to crack propagation and the nanocarbons are clearly visible. The fracture surface of pure epoxy was smoother and flatter than that of nanocomposites, which is known to indicate lower impact resistance and fracture toughness [32]. The distribution of DNDs was more uniform than the CNTs, which was attributed to their size as well as dispersibility.

Figure 5(*a*) shows the increase in tensile strength of the epoxy with the addition of DNDs and CNTs into the polymer matrix. It is interesting to observe that epoxy-DND composite exhibited better mechanical properties compared to the with epoxy-CNT composite at low nanocarbon content, up to 0.3 wt. %. This may be due to the fact that small DNDs particles reduce the free volume fraction of the polymer matrix (as also observed in T_{o}) thus offer

resistance to propagation of cracks. The well dispersed DNDs are more efficient than the CNTs in transferring applied load at lower loadings. However, higher nanocarbon loading increases the tendency to agglomerate, which reduces the enhancement in tensile properties. The improvement in the tensile strength of epoxy nanodiamond composites reinforced with 3 and 0.3 wt. % of ND has been reported to be 4.6 [33] and 4.8% [34], respectively. In our study, the enhancement in tensile strength was found to be 6.4% for epoxy-DND composite and 2.9% for epoxy-CNT composite at 0.1% loading. Figure 5(b) shows the effect of nanocarbon content on elongation at break (EAB). It is clear from the figure that with increase in nanocarbon content EAB of both composite is reduced. However, CNTs showed less reduction in EAB in comparison with DNDs. Under tensile load, CNT showed a tendency to be pulled out and this was observed in the SEM. Slippage between individual graphene shells of MWCNTs is also a possibility as only weak van der Walls forces exist between the layers.

The surface hardness properties were measured using Vicekr Microhardness. The hardness value for pure epoxy was found 17 HV. A significant enhancement in hardness values of 41% was obtained for DND-composite, whereas the CNT-composite showed only 12% improvement. Therefore, even a small concentration of DNDs as well as CNTs led to significant improvement in microhardness. Additionally, a significant reduction in cracks can be seen for epoxy-DND composite at the junction of the

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Table 2Volume resistivity and surface resistance of pure ep-oxy and 0.5% epoxy nanocomposites

Sample	Volume resistivity $(\Omega \text{ cm})$	Surface resistance of 1 cm^2 area (Ω)
Epoxy-pure Epoxy-CNT composite	$\begin{array}{c} 1.31 \times 10^{11} \\ 0.81 \times 10^{9} \end{array}$	$7.1 imes 10^9 \\ 0.89 imes 10^9$
Epoxy-DND composite	6.15×10^9	$1.8 imes 10^9$

diagonals at large loads (as shown in Fig. 6) which imply that it is comparably less brittle and has higher fracture toughness.

The thermal degradation behavior and thermal stability of epoxy-nanocomposites were studied with TGA. The TGA curves are shown in Fig. 7(a). At this low concentration, the CNT did not appear to enhance thermal stability, but the DNDs were quite effective in lowering the degradation rate. However, the largest enhancement was observed in the DSC analysis. Figure 7(b) shows the DSC curves of the pure epoxy and epoxynanocomposites prepared in the current study. It was observed that with incorporation of 0.1 wt. % nanocarbon into the polymer matrix enhanced the $T_{\rm g}$ from 78 °C to 95 °C for the CNTcomposite and 115 °C for the DND-composite. The increase in T_{g} suggests a well dispersion of nanofiller for the polymer matrix, thereby decreasing the chain mobility. CNTs are dimensionally similar to that of polymer chain (30 nm in diameter and $15 \,\mu m$ length) and the presence of CNTs influences the alignment of polymer and thus T_{g} . The high enhancement in T_{g} for DNDcomposite was due to the fact that small spherical equiaxial DNDs (particle size is ~ 10 nm) were free of entanglement and could be better dispersed into the polymer matrices which helped the epoxy chain alignment with maximum packing density [35] as shown in Fig. 2. Moreover, spherical, untangled DNDs represent a higher direct surface interaction with the polymer compared to the CNTs [36]. DNDs with its small particle size also did not interfere with the epoxy curing, which has been reported for CNTs [4].

Cured epoxy is known to be a poor conductor of electricity. The inability to dissipate built up static charge under strong electrical field is known to degrade the polymer. However, incorporation of conducting nanomaterials into the polymer matrix can reduce the resistivity, thus releasing the build up static charge. Table 2 presents the volume resistivity and surface resistance of pure epoxy and the epoxy-nanocomposites. Table 2 shows that with incorporation of 0.5 wt. % nanocarbons into the polymer matrix decreased volume resistivity by 2 orders of magnitude. High aspect ratio and percolation network of CNTs favors the reduction in resistivity of the composite [37]. However, the surface resistance values did not differ significantly because of the low nanocarbon concentration and surface leveling effect of low viscous liquid epoxy resin.

Conclusions

Both CNTs and DNDs showed excellent colloidal stability in resin solution that led to homogeneous composite formation. SEM images of composites showed uniform distribution of nanomaterials into the polymer matrices. Rough fracture surface of nanocomposites indicate higher impact resistance and fracture toughness. DNDs were particularly effective in increasing the glass transition temperature, which rose by 37 °C. The DNDs also showed better mechanical properties while the CNTs showed lower resistivity.

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