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Electrical properties of carbon nanofiber reinforced multiscale polymer composites

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ABSTRACT

Carbon nanofiber (CNF) reinforced epoxy matrix nanocomposites and CNF reinforced glass hollow particle filled syntactic foams are studied for electrical properties. The effect of CNF weight fraction, hollow particle volume fraction, and hollow particle wall thickness on impedance and dielectric constant are characterized. The results show that the impedance decreases and the dielectric constant increases with increasing CNF content in the composites. Nanocomposites containing 10 wt.% CNFs showed significantly higher dielectric constant because of the presence of a continuous network of CNFs in the composite. CNF reinforced syntactic foams showed higher dielectric constant than the neat resin. The CNF content had a more prominent effect on the dielectric constant than the glass hollow particle volume fraction and wall thickness. The Maxwell–Garnett and the Jayasundere–Smith models are modified to include the effect of hollow particle wall thickness and obtain predictions of dielectric constants of syntactic foams. The semi-empirical predictions obtained from Maxwell–Garnett models are closer to the experimental values. Lightweight syntactic foams, tailored for electrical properties, can be useful in electronic packaging applications.

1. Introduction

Carbon nanofibers (CNFs) are promising in developing multifunctional nanocomposites. The low cost of CNFs is attractive in developing bulk composite materials for structural applications. Individual CNFs have been tested and their modulus is measured to be in the range 180–245 GPa [1], which can provide composites with high modulus. In addition, crack bridging and branching promoted by CNFs can result in increased strength and failure strain in composites. The electrical and thermal conductivity of CNFs can be used in conjunction with the mechanical properties to develop multifunctionality in composite materials. High mechanical properties [2], low coefficient of thermal expansion [3–4] and high electrical conductivity [5–9] are achieved in CNF reinforced composites.

Hollow particle filled composites called syntactic foams are used in modern aerospace and marine applications due to their tailorability and excellent compressive strength-to-weight ratio [10–13]. The applications of syntactic foams are discussed in a recent article [14]. A high volume fraction of hollow particles in syntactic foams is important in order to obtain the benefit of weight reduction. Presence of additional nanoscale reinforcement in the matrix resin in the interparticle region can further improve the properties of syntactic foams [15]. Nanoclay, nanotubes, and nanofibers are examples of nanomaterials that have been used for reinforcing syntactic foams [16–18]. In addition, use of CNFs can result in the development of electrical and thermal conductivity in syntactic foams. Preliminary studies on the effect of CNF reinforcement on syntactic foams have shown enhancement in the strength, modulus, and thermal stability of the overall composite [4,18–19].

The current study is focused on studying the electrical properties of CNF-reinforced epoxy matrix syntactic foams. In order to understand the trends observed in the properties of CNF reinforced syntactic foams, neat matrix resin and CNF reinforced epoxy matrix nanocomposites are also characterized under the same conditions. The CNF content is varied in the range 1–10 wt.% in the composites and the experiments are conducted using the AC impedance method to obtain the electrical impedance and dielectric constant. The experimental results are compared with the predictions obtained from the Maxwell–Garnett (M–G) and the Jayasundere–Smith (J–S) models, which are modified to include the effect of hollow particle wall thickness.

2. Materials and methods

2.1. Constituent materials

Vapor grown PR-24 XT-PS CNFs (Pyrograf Products, Inc., OH) are used in the study, where "XT" denotes that the CNFs have been de-

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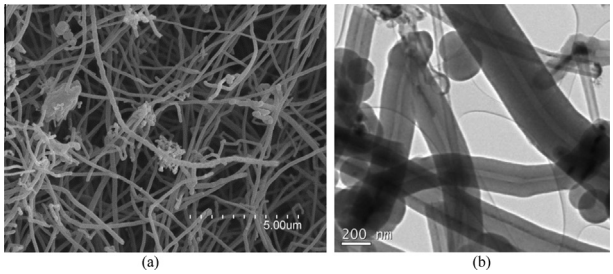

Fig. 1. (a) Scanning electron micrograph of as-received carbon nanofibers and (b) transmission electron micrograph of carbon nanofibers showing hollow core structure.

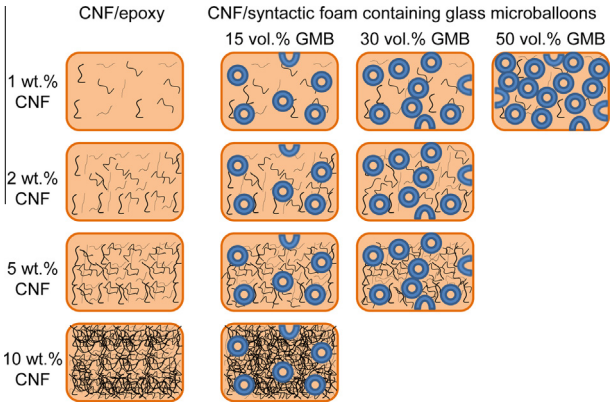

Fig. 2. Illustration of CNF nanocomposites and CNF reinforced syntactic foams fabricated in the present work. Sixteen compositions of CNF reinforced syntactic foams are synthesized with two types of glass hollow particles.

bulked to allow for uniform bulk density, and "PS" denotes that the fibers have been pyrolytically stripped of aromatic hydrocarbons. A scanning electron micrograph of CNFs is shown in Fig. 1a. The transmission electron micrograph in Fig. 1b shows that CNFs have a hollow core structure.

DER 332 epoxy resin with DEH 24 hardener manufactured by the DOW Chemical Co. is used as the matrix. Two types of nanocomposites are fabricated in the present work as illustrated in Fig. 2. In the first set, 1, 2, 5 and 10 wt.% CNFs are dispersed in epoxy resin. In the second set, glass hollow particles are dispersed along with CNFs to create multiscale lightweight composites. Glass microballoons (3M, MN), referred to as GMB, of 220 and 460 kg/m^3 nominal true particle densities are used in 15, 30 and 50 vol.% to fabricate multiscale syntactic foams, referred to as CNF/syntactic foams. Sixteen compositions of CNF/syntactic foams are fabricated as illustrated in Fig. 2. The CNF density is 1950 kg/m^3 and the epoxy resin density is 1160 kg/m^3, as obtained from the respective manufacturer's datasheets. The composite nomenclature follows the trend NXX-YYY-ZZ where N represents CNFs, XX corresponds to CNF weight fraction, YYY denotes GMB density and ZZ refers to GMB vol.%.

2.2. Specimen preparation

A mechanical mixer with a high shear impeller is used to obtain uniform dispersion of CNFs in the epoxy resin [20]. CNFs are initially mixed in the epoxy for 30 min at 650 rpm to form a composite slurry. Hardener is added and slowly mixed using wooden dowels and then cast in aluminum molds to obtain CNF/epoxy nanocomposites. To fabricate CNF/syntactic foams, GMBs are added to the slurry and mixed slowly with a wooden dowel for an additional 15 min [21]. The hardener is added in the end, mixed, and then cast in aluminum molds. The molds are placed on top of a shaker to degas the mixture and remove air voids entrapped in the resin during mixing (such voids are called matrix porosity). The mixture is cured for at least 24 h and then post-cured in a convection oven for 2 h at 100 °C. Specimens of nominal dimensions 18 × 14 × 1 mm^3 were cut from the slab using a precision Isomet diamond blade saw (Buehler Ltd., Lake Placid, N.Y.) for electrical testing.

2.3. Electrical impedance testing

The electrical impedance was measured using a CH Instruments 700D (Austin, TX) electrochemical potentiostat. Specimens were placed between gold electrodes in order to measure the impedance and dielectric constant with respect to frequency in the range 10^{-2} to 10^6 Hz with applied AC wave amplitude of 500 mV. The impedance, denoted by Z , was obtained using the measurement of the electrical resistance R and the capacitive reactance X_c as [22]

$$|Z| = \sqrt{R^2 + X_c^2} \tag{1}$$

The resistance and the reactance, which represent the real and imaginary parts of the impedance, respectively, can also be utilized to calculate the phase angle, ϕ

$$\phi = \tan^{-1}\left(\frac{X_c}{R}\right) \tag{2}$$

The capacitance C is calculated by

$$C = \frac{1}{2\pi f X_c} \tag{3}$$

where f is the frequency. Finally, the dielectric constant is calculated by

$$\varepsilon = \frac{Ct}{\varepsilon_0 A} \tag{4}$$

where t is the specimen thickness, A is the area, and ε_0 is the permittivity of free space. The value of ε_0 is taken as 8.854 × 10^{-12} F/m.

3. Results

Fig. 3 shows a micrograph of a representative N10 specimen. The CNFs are observed to be dispersed uniformly in the material microstructure. Extensive microscopic observations are taken on several specimens of different composites to confirm the wetting

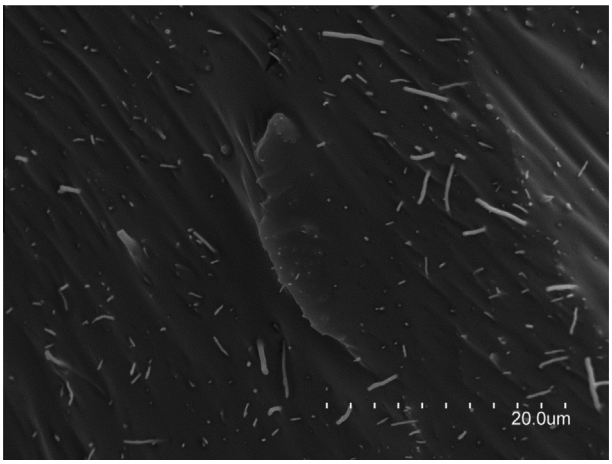

Fig. 3. Microstructure of CNF/epoxy composite containing 10 wt.% CNFs are uniformly dispersed in epoxy matrix.