Electrical and Thermal Conductivity and Tensile and Flexural Properties of Carbon Nanotube/ Polycarbonate Resins

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ABSTRACT: Adding conductive carbon fillers to insulating thermoplastic polymers increases the resulting composite's electrical conductivity. Carbon nanotubes (CNTs) are very effective at increasing composite electrical conductivity at low loading levels without compromising composite tensile and flexural properties. In this study, varying amounts (2–8 wt %) of CNTs were added to polycarbonate (PC) by melt compounding, and the resulting composites were tested for electrical conductivity (1/electrical resistivity), thermal conductivity, and tensile and flexural properties. The percolation threshold was less than 1.4 vol % CNT, likely because of CNTs high aspect ratio (1000). The addition of CNT to PC increased the composite electrical and thermal conductivity and tensile and flexural modulus. The 6 wt % (4.2 vol %) CNT in PC resin had a good combination of properties for electrical conductivity applications. The electrical resistivity and thermal conductivity were 18 Ω-cm and 0.28 W/m · K, respectively. The tensile modulus, ultimate tensile strength (UTS), and strain at UTS were 2.7 GPa, 56 MPa, and 2.8%, respectively. The flexural modulus, ultimate flexural strength, and strain at ultimate flexural strength were 3.6 GPa, 125 MPa, and 5.5%, respectively. Ductile tensile behavior is noted in pure PC and in samples containing up to 6 wt % CNT. J Appl Polym Sci 118: 2512–2520, 2010

Key words: composites; fillers; injection molding; nanocomposites

INTRODUCTION

Most polymer resins are electrically insulating. Increasing the electrical conductivity of these resins allows them to be used in other applications, such as static dissipative (e.g., handling trays used in electronic equipment assembly, etc.) and moderate electrical conductive (e.g., fuel gauges, etc.) applications. One approach to improve the electrical conductivity of polymer is through the addition of a conductive filler material, such as carbon and metal.[1–14] Recently, carbon nanotubes (CNTs) have been used to increase the electrical conductivity of a resin.[15–26] CNTs have many unique characteristics. For example, only a small amount of CNTs need to be added to a polymer to increase the composite's electrical conductivity without decreasing the material's mechanical properties and without significantly increasing the melt viscosity.

In this work, researchers performed compounding runs followed by injection molding of CNT filled polycarbonate (PC) resins. Composites containing varying amounts of CNTs were fabricated and tested for electrical and thermal conductivity, along with tensile and flexural properties. The goal of this project was to determine the effects of the CNTs on the composite thermal conductivity, electrical conductivity (1/electrical resistivity), and tensile and flexural properties.

MATERIALS AND EXPERIMENTAL METHODS

Materials

The matrix used for this project was Sabic Innovative Plastics's (Pittsfield, MA) Lexan HF1130-111 PC resin. The properties of this polymer are shown in Table I.[27]

Hyperion Catalysis International's (Cambridge, MA) FIBRIL™ nanotubes were used in this study.

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TABLE I
Properties of Sabic's Polycarbonate Lexan HF 1130[27]

Melt flow rate (300 C/1.2 kg)	25 g/10 min
Density	1.2 g/cc
Electrical resisitiviy	1×10^{17} Ω-cm
Thermal conductivity	0.19 W/m · K

TABLE II
Properties of FIBRIL™ Carbon Nanotubes[18,22,28]

Composition	Pure carbon
Diameter	0.01 μm
Length	10 μm
Morphology	Graphitic sheets wrapped around a hollow 0.005 μm core
BET (N_2) surface area	250 m^2/g
Density	2.0 g/cc of nanotube wall, 1.75 g/cc for the hollow nanotube

This is a conductive, vapor grown, multiwalled CNT. They are produced from a high purity, low-molecular weight hydrocarbons in a proprietary, continuous, gas phase catalyzed reaction. The outside diameter of the tube is 10 nm and the length is 10 μm, which gives an aspect ratio (length/diameter) of 1000. Because of this high aspect ratio, very low concentrations of nanotubes are needed to produce an electrically conductive composite. This material was provided by Hyperion Catalysis International in a 15 wt % FIBRIL™ masterbatch MB6015-00. Table II shows the properties of this CNT filler.[18,22,28]

The concentrations (shown in wt % and the corresponding vol % using 1.75 g/cm^3 for the CNT density) for all of the single-filler composites tested in this research are shown in Table III.[18,22] We note that increasing filler amount increases composite melt viscosity. Table III also shows the electrical resistivity and thermal conductivity results that will be described later in this article.

Test specimen fabrication

Before extrusion and injection molding, the Lexan HF1130-111 was dried in an indirect heated dehumidifying drying oven at 121°C for 12 h. The Hyperion fibrils MB6015-00 was dried in an indirect heated dehumidifying drying oven at 121°C for 6 h. The extruder used for melting the compound was an American Leistritz extruder (Somerville, NJ) Model ZSE 27. This extruder has a 27 mm corotating intermeshing twin screw with 10 zones and length/diameter ratio of 40. The screw design, which is shown in Figure 1, was chosen to obtain a minimum amount of filler degradation still dispersing the fillers well in the polymers. The pure PC pellets and the Hyperion FIBRIL™ masterbatch MB6015-00 (containing 15 wt % CNTs) pellets were mixed at the appropriate weight ratio to yield the desired CNT concentration and introduced into Zone 1. The two types of pellets were similar in size; therefore, there was no segration of pellet type in the feed hopper.

After passing through the extruder, the polymer strands (3 mm in diameter) entered a water bath and then a pelletizer that produced nominally 3 mm long pellets. After extrusion, the PC-based composites were dried in an indirect heated dehumidifying drying oven at 121°C for 12 h and then stored in moisture barrier bags before injection molding.

A Niigata (Tokyo, Japan) injection molding machine, model NE85UA$_4$, was used to produce test specimens. This machine has a 40 mm diameter single screw with a length/diameter ratio of 18. The lengths of the feed, compression, and metering sections of the single screw are 396, 180, and 144 mm, respectively. A four cavity mold was used to produce 3.2 mm thick ASTM Type I tensile bars (end gated), 127 mm long by 12.7 mm wide flexural bars (end gated), and 6.4 cm diameter disks (end gated).

Field emission scanning electron microscope and transmission electron microscopy test methods

A Hitachi *S*-4700 (Pleasanton, CA) cold field emission scanning electron microscope (FESEM) was used to view the cryrofractured surface of the CNT/PC composite (3.2 mm thick by 12.7 mm wide cross

TABLE III
Single Filler Loading Levels in Polycarbonate and Electrical Resistivity and Thermal Conductivity Results

Formulation	Filler (wt %)	Filler (vol %)	Electrical resistivity (Ω-cm)	Thermal conductivity (W/ m · K)
PC	0	0.0	$1.06 \times 10^{17} \pm 7.96 \times 10^{16}$, $n = 7$	0.218 ± 0.002, $n = 5$
2CNT	2	1.38	4610 ± 1120, $n = 6$	0.232 ± 0.002, $n = 5$
3CNT	3	2.08	216 ± 44, $n = 6$	0.241 ± 0.003, n = 5
4CNT	4	2.78	73 ± 10, $n = 6$	0.255 ± 0.001, $n = 5$
5CNT	5	3.48	43 ± 7, $n = 6$	0.266 ± 0.003, $n = 5$
6CNT	6	4.19	18 ± 2, $n = 6$	0.275 ± 0.003, $n = 5$
8CNT	8	5.63	7.8 ± 0.4, $n = 6$	0.306 ± 0.003, $n = 5$

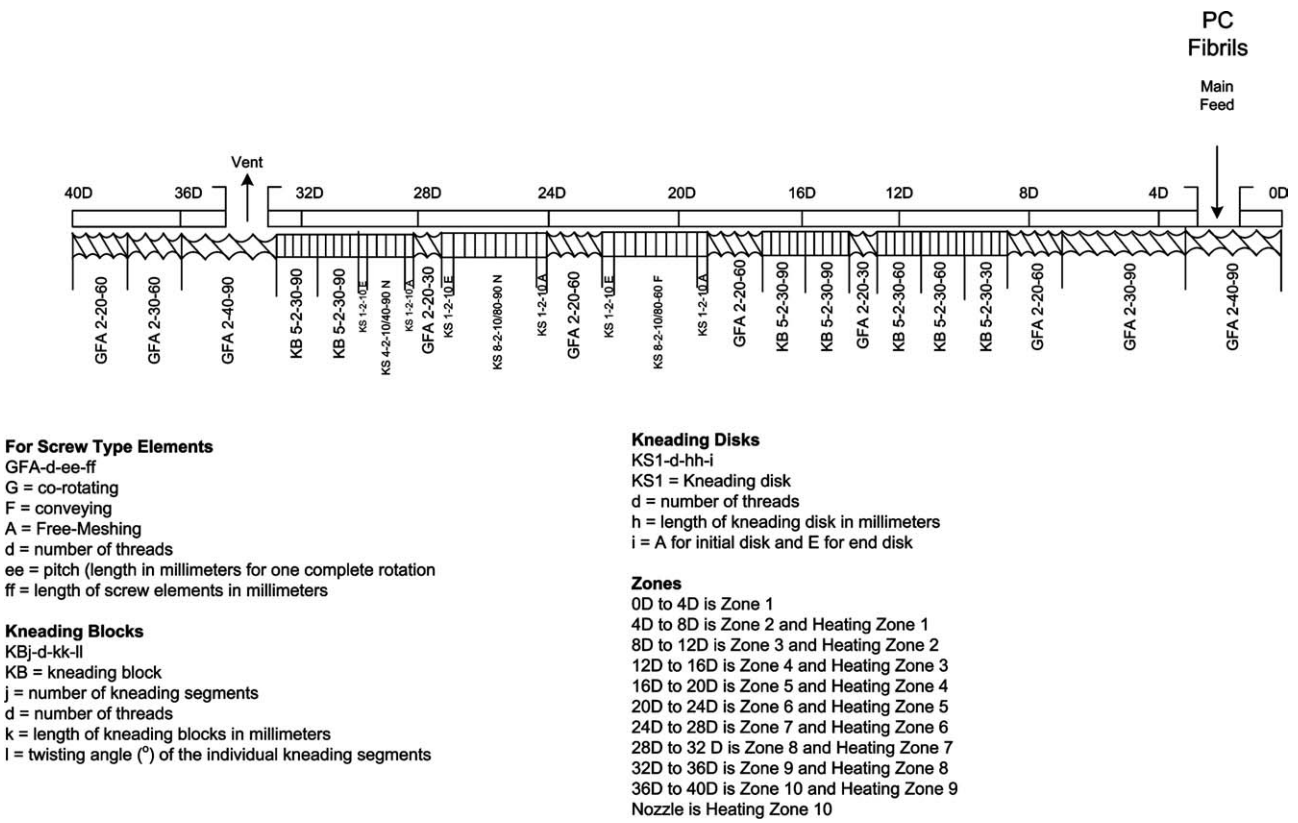

Figure 1 Twin screw extruder design.

section from an injection molded tensile bar). The samples were prepared for observation by cyro-fracture, where the composite was submerged in liquid nitrogen until frozen, and then quickly retracted and fractured. Afterward, the samples were attached to aluminum mounting disks and were observed in the FESEM at 2 kV accelerating voltage, and 2 mm working distance using the upper secondary electron detector. This method was used to view the CNT.

Ultra-thin ($\sim$ 50 nm) transmission electron microscopy (TEM) sections of the composite were prepared by Leica UCT ultramicrotome (Bannockburn, IL). The sections were supported on a copper 300 mesh lacey formvar carbon-coated grid and then examined using a JEOL JEM-4000FX (Peabody, MA) operated at 200 kV accelerating voltage. The CNT distributions were imaged at a magnification of 12,000×, and digital images were acquired using a Gatan Orius camera (Pleasanton, CA). This technique was used to view the dispersion of the CNT in PC.

Electrical resistivity test method

For samples with an electrical resistivity $>10^4$ Ω-cm, the volumetric through-plane electrical conductivity test was conducted. In this method, a constant voltage (typically 100 V) was applied to the as-molded test specimen, and the resistivity was measured according to ASTM D257 using a Keithley 6517A electrometer/high-resistance meter (Cleveland, OH) and a 8009 resistivity test fixture.[29] The Keithley 6524 high resistance measurement software was used to automate the conductivity measurement. Each test specimen was an injection molded disk that was 6.4 cm in diameter and 3.2 mm thick. This test method was used on the pure PC samples and seven samples were tested. Before testing, the samples were conditioned at 23°C and 50% relative humidity for 2 days.

The in-plane volumetric electrical resistivity of the center 60 mm long, 3.2 mm thick, 12.7 mm wide tensile bars (rectangular necked area) injection molded tensile bars was determined according to ASTM D 4496 at 23°C.[30] Before testing, the samples were conditioned at 23°C and 50% relative humidity for 2 days. Six samples were tested for each formulation. This test was conducted with two probes. In the two probe method, the tensile bar was scratched with a razor blade, placed in liquid nitrogen, and then broken manually at the desired location. Hence, a fracture surface was created on both ends of the in-plane sample. Then, the 3.2 mm thick by 12.7 mm wide ends were coated with silver paint and allowed to dry for 1 h. One probe was placed on each of silver-painted fracture surface and a constant voltage was placed across the sample using a

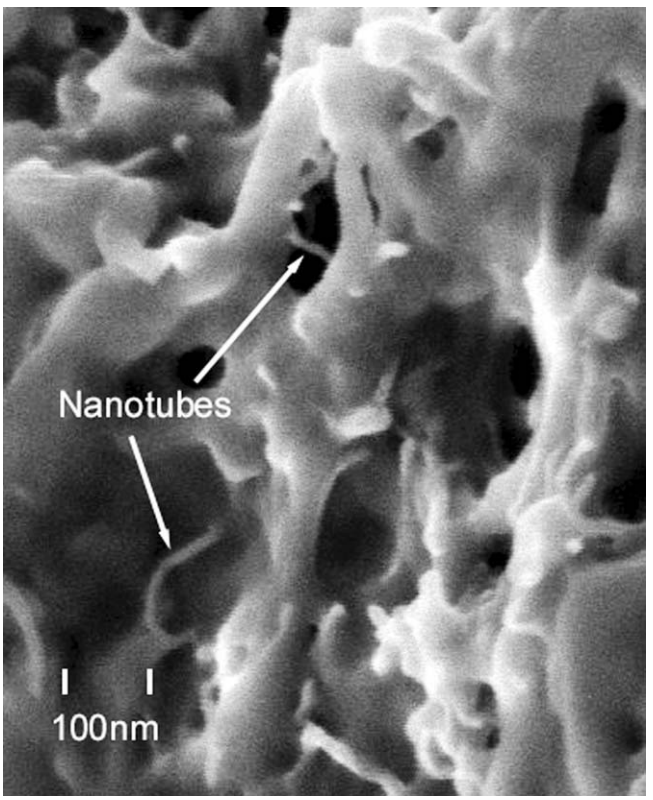

Figure 2 Field emission scanning electron microscope photomicrograph of 6 wt % CNT in polycarbonate composite.

Keithley 2400 Source Meter. The resulting current was also measured on this same Keithley 2400. The volume electrical resistivity is calculated from eq. (1).

$$\mathrm{ER} = \frac{(\Delta V)(w)(t)}{(i)(L)} \quad (1)$$

where:

ER = volume electrical resistivity, Ω-cm.

ΔV = voltage drop over length of sample, volts

w = sample width, 1.27 cm.

t = sample thickness, 0.32 cm.

i = current, amps.

L = length over which ΔV is measured (6 cm).

Thermal conductivity test method

The through-plane thermal conductivity of a 3.2 mm thick, 5 cm diameter disk shaped test specimen was measured at 55°C using a Holometrix (Burlington, MA) Model TCA-300 thermal conductivity analyzer, which uses the ASTM F433 guarded heat flow meter method.[31] For each formulation, five samples were tested. Before testing, the samples were conditioned at 23°C and 50% relative humidity for 2 days.

Tensile test method

The tensile properties (at ambient conditions, 16.5 cm long, 3.2 mm thick ASTM D638 Type I sample geometry) from all formulations were determined using ASTM D638 at a crosshead rate of 5 mm/min for reinforced plastics.[32] An Instru-Met Sintech (Union, NJ) screw driven mechanical testing machine was used. Tensile modulus was calculated from the initial linear portion of the stress–strain curve. For each formulation, at least five samples were tested. Before testing, the samples were conditioned at 23°C and 50% relative humidity for 2 days.

Flexural test method

The flexural properties were determined using three-point loading at ambient conditions from all formulations according to ASTM D790[33] at a crosshead rate of 5.3 mm/min. A 16 : 1 span to thickness ratio was used in an Instru-Met Sintech screw driven mechanical testing machine. Deflection was measured using a linear variable displacement transducer. Flexural modulus was calculated from the initial linear portion of the stress–strain curve. For each formulation, at least five samples were tested. Before testing, the samples were conditioned at 23°C and 50% relative humidity for 2 days.

RESULTS

FESEM and TEM results

Figure 2 shows the CNT (white fibers) in the sample containing 6 wt % CNT in PC. Clearly, networks of CNT are seen in this figure. Figure 3 shows the dispersion of the CNT in the 3 wt % and 6 wt % CNT composites.

Electrical resistivity results

The mean, standard deviation, and number of sample test for each formulation containing varying

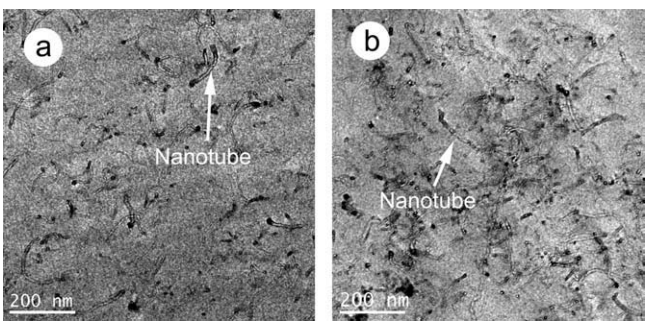

Figure 3 TEM photomicrographs of carbon nanotube (CNT)/polycarbonate composites: (a) 3 wt % CNT and (b) 6 wt % CNT.

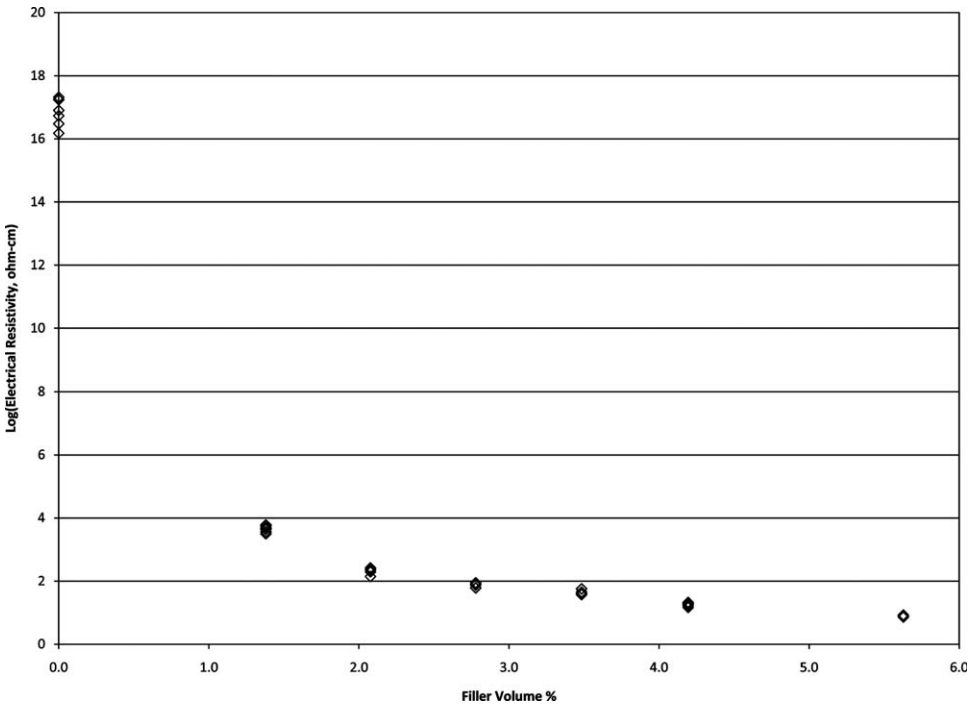

Figure 4 Electrical resistivity results for carbon nanotube/polycarbonate composites.

amounts of single fillers are shown in Table III. Figure 4 shows the log (electrical resistivity in Ω-cm) for composites containing varying amounts of CNT as a function of filler volume fraction. In these figures, all the data points have been plotted. Figure 4 follows the typical electrical resistivity curve. At low filler loadings, the electrical resistivity remains similar to that of the pure polymer. Then at a point called the percolation threshold, the resistivity decreases dramatically over a very narrow range of filler concentrations. At higher filler loadings, the electrical resistivity begins to level out again at a value many orders of magnitude lower than that of the pure polymer.[5,34]

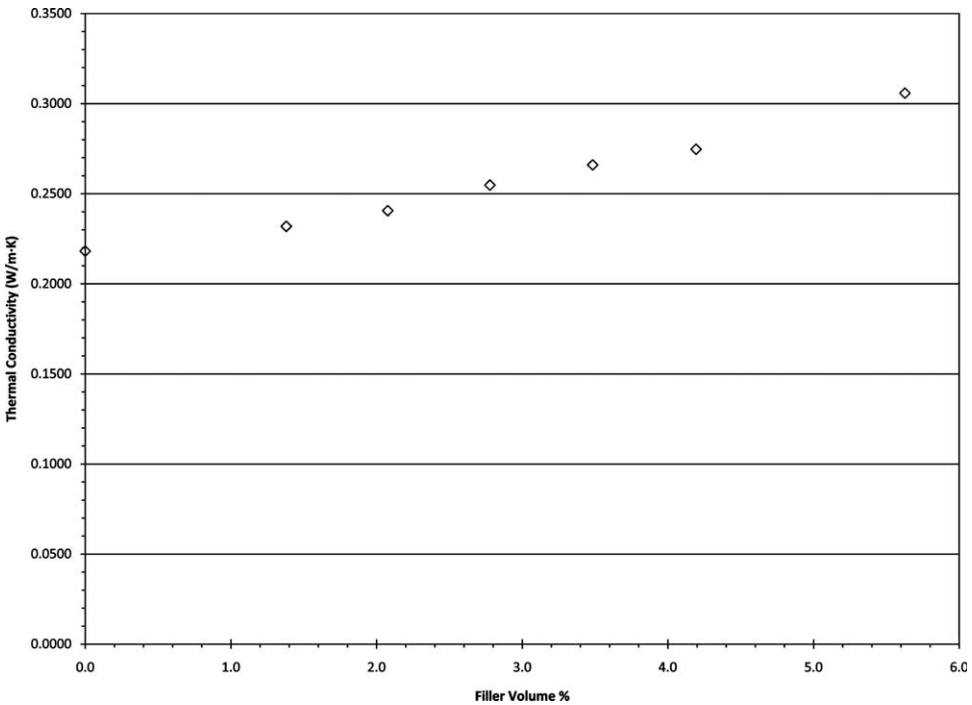

Figure 5 Thermal conductivity results for carbon nanotube/polycarbonate composites.

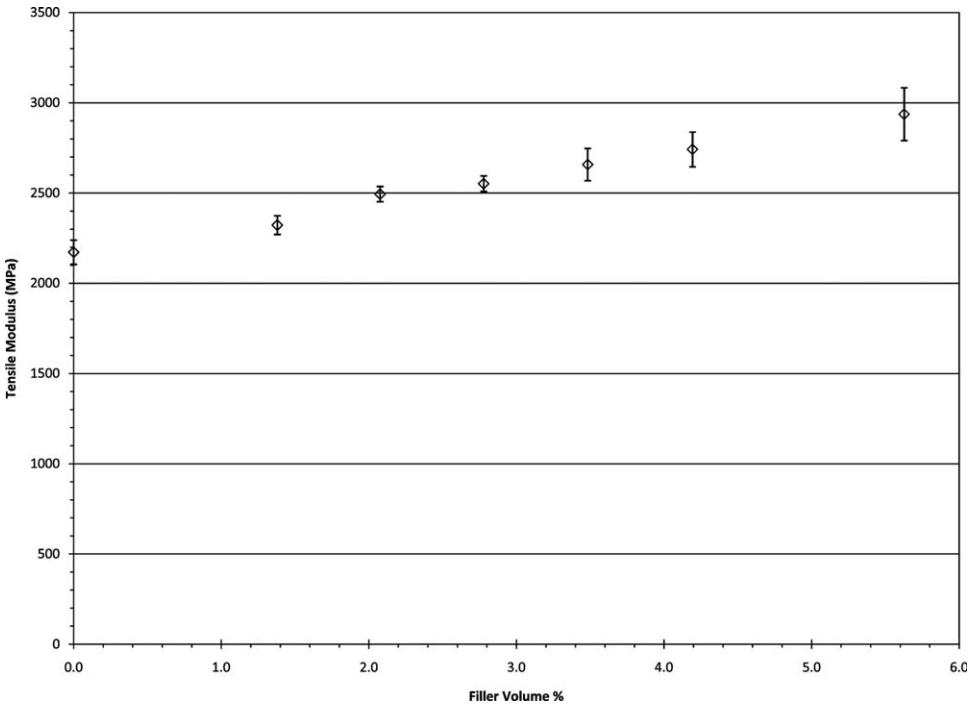

Figure 6 Tensile modulus for carbon nanotube/polycarbonate composites.

Figure 4 illustrates that CNTs are effective at decreasing the electrical resistivity (1/electrical conductivity) at low filler loadings. The pure PC has a mean electrical resistivity of 1.1×10^{17} Ω-cm, which agrees with the vendor literature value (Table I). The percolation threshold occurs below 1.4 vol % (2 wt %) for CNTs. At the highest filler concentration, the CNTs produced a mean composite resistivity of 8 Ω-cm (8 wt % = 5.6 vol %). The percolation threshold is low for the CNT composites because of the extremely high aspect ratio (length/diameter) of 1000. These results are similar to those obtained by Hornbostel et al. for single-walled CNT in PC,[15] and Potschke et al.[23,25] and Pegel et al.[26] for multiwalled

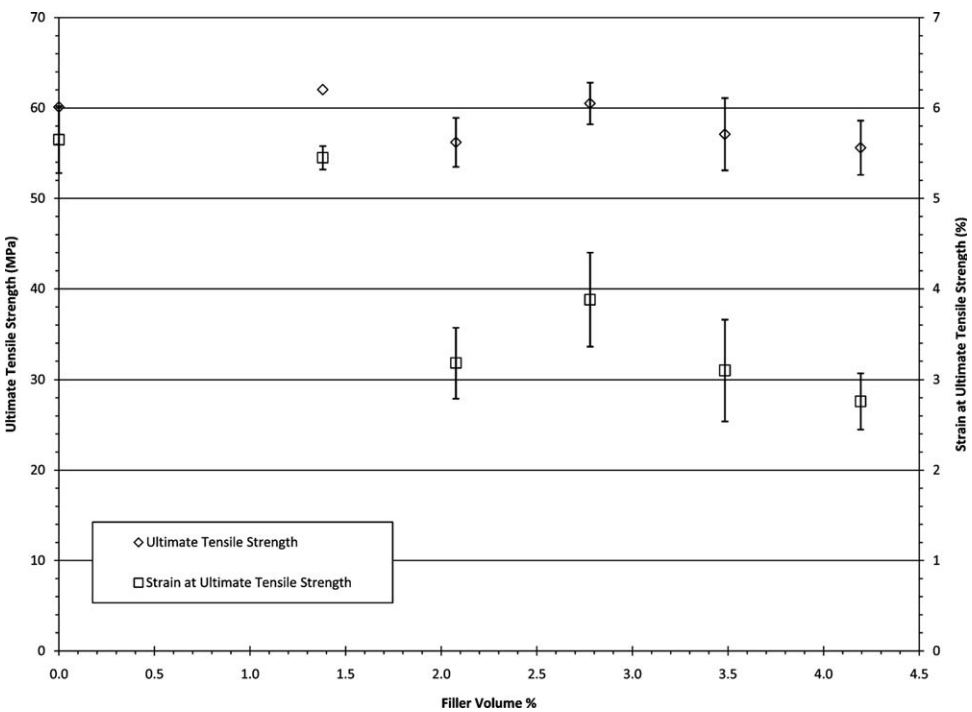

Figure 7 Ultimate tensile strength and strain at ultimate tensile strength for carbon nanotube/polycarbonate composites.

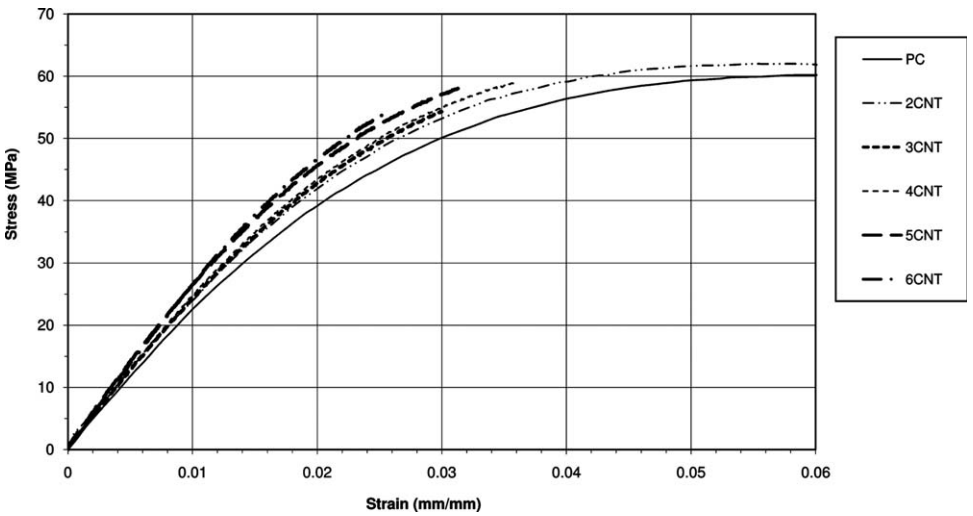

Figure 8 Tensile stress–strain curves for polycarbonate and carbon nanotube/polycarbonate composites.

CNT in PC. For example, Hornbostel et al.[15] reported a percolation threshold from 1.5 to 2.0 wt % CNT for melt extruded composites. Our results are more conductive than those reported by Chen et al. for multiwalled CNT's in PC.[19]

Thermal conductivity results

Figure 5 shows the mean through-plane thermal conductivity using the guarded heat flow meter for the composites containing only varying amounts of single fillers as a function of filler volume fraction. These formulations correspond to those shown in Table III.

Figure 5 shows that CNTs do increase the through-plane thermal conductivity of the polymer from 0.22 to 0.31 W/m · K for the composites containing 8 wt % (5.6 vol %) CNT. These values are similar to those previously reported by King et al. for CNTs in polypropylene[35] and are lower than those reported by Lee et al. for CNTs in room temperature vulcanizing silicone elastomer.[36]

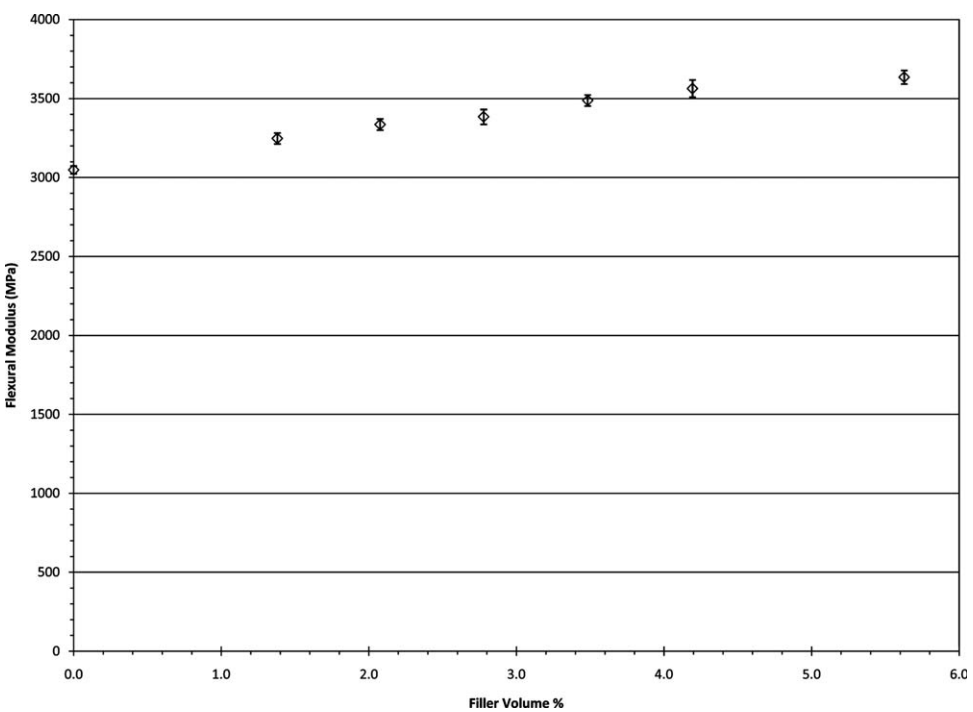

Figure 9 Flexural modulus for carbon nanotube/polycarbonate composites.

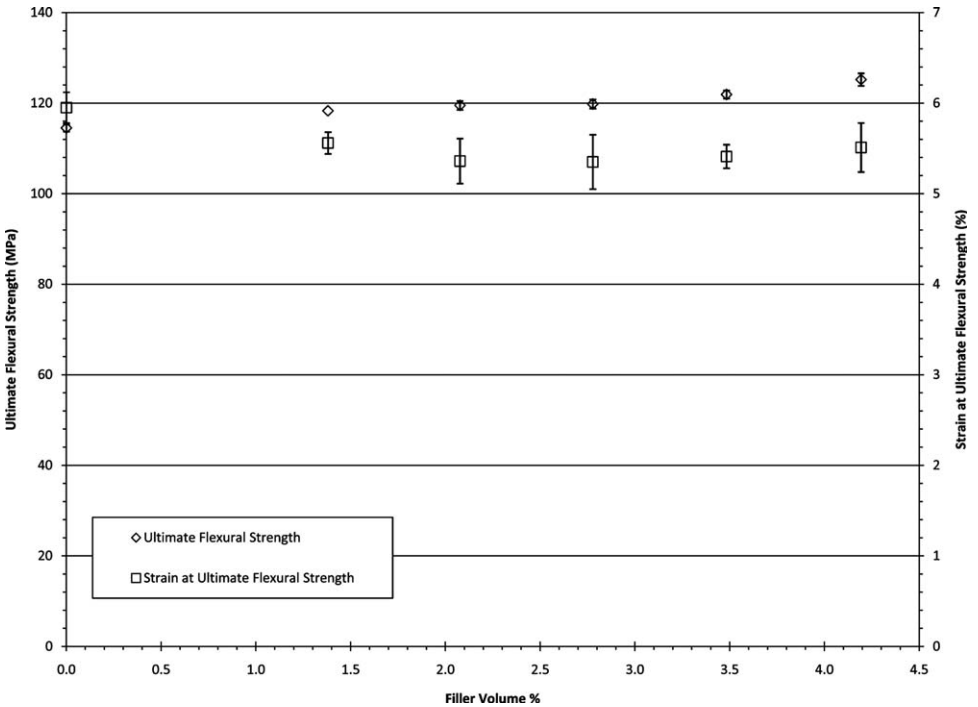

Figure 10 Ultimate flexural strength and strain at ultimate flexural strength for carbon nanotube/polycarbonate composites.

Tensile test results

Figures 6 and 7 show the tensile results (tensile modulus, ultimate tensile strength (UTS), and strain at UTS) (mean and ± one standard deviation) for composites containing varying amounts of single fillers as function of volume percent filler. These formulations correspond to those shown in Table III. If the standard deviation is smaller than the marker size, the error bars are not shown. The tensile modulus results are located in Figure 6. As expected, adding CNT caused the tensile modulus to increase from 2.2 GPa (neat polymer) to 2.9 GPa at 8 wt % (5.6 vol %) CNT. Figure 7 illustrates the UTS and strain at UTS results. The results for the 8 wt % CNT in PC composite are not shown as the sample broke prematurely (the resin was very viscous and was difficult to injection mold at this highest filler content). Figure 7 shows that the UTS is similar for all the composites containing $\leq$6 wt % CNT. This figure also shows that the strain at UTS generally decreases with the addition of CNT. These trends agree with those of Hornbostel et al.[15] for single-walled CNT in PC, Lopez Manchado et al.[21] for single-walled CNT in polypropylene, and Fornes et al. for single-walled and multiwalled CNT in PC.[37] Figure 8 shows a typical tensile stress–strain curve (up to 6% strain) for PC and for composites containing up to 6 wt % CNT in PC. This figure shows that in all cases, ductile behavior of the neat polymer is retained after the addition of CNTs.

Flexural test results

Figures 9 and 10 show the flexural modulus, ultimate flexural strength, and strain at ultimate flexural strength (mean and ±1 standard deviation) for composites containing varying amounts of single fillers as function of filler volume percent. These formulations correspond to those shown in Table III. If the standard deviation is smaller than the marker size, the error bars are not shown.

Figure 9 shows the flexural modulus for composites containing varying amounts of single fillers. Figures 6 (tensile modulus) and 9 (flexural modulus) show the same general trends. Adding CNT caused the flexural modulus to increase from 3.0 (neat polymer) to 3.6 GPa at 8 wt % (5.6 vol %) CNT. Figure 10 shows the ultimate flexural strength and strain at ultimate flexural strengh. Once again, the results for the 8 wt % CNT in PC composite are not shown as the sample broke prematurely. The addition of CNT caused an increase in ultimate flexural strength from 115 MPa for the neat polymer to 125 MPa with 6 wt % (4.2 vol %) CNT. The strain at ultimate flexural strength remained approximately the same at all loading levels.

CONCLUSIONS

The object of this research was to determine the effects of CNTs on the composite properties. Concerning electrical properties, the percolation

threshold is less than 1.4 vol % CNT, which is due to the high aspect ratio (1000) for CNT. Adding CNT decreased the electrical resistivity from 1.1×10^{17} (neat polymer) to 8 Ω-cm for the composite containing 8 wt % (5.6 vol %) CNT. Adding CNT increased the composite thermal conductivity from 0.22 (neat PC) to 0.31 W/m · K for the 8 wt % CNT in PC.

Concerning mechanical properties, adding CNT caused the tensile modulus to increase from 2.2 (neat polymer) to 2.9 GPa at 8 wt % (5.6 vol %) CNT. The UTS was similar, ranging from 56 to 62 MPa, for all the composites. The strain at UTS decreased with increasing CNT content. Ductile tensile behavior is noted in pure PC and in samples containing up to 6 wt % CNT. The addition of CNT also caused the flexural modulus to increase from 3.0 (neat polymer) to 3.6 GPa at 8 wt % (5.6 vol %) CNT and caused the ultimate flexural strength to increase from 115 (neat polymer) to 125 MPa (6 wt % = 4.2 vol % CNT). The strain at ultimate flexural strength remained approximately the same (at 5.4–6.0%) for all CNT loading levels.

The composite with 6 wt % (4.2 vol %) CNT in PC had a good combination of properties for electrical conductivity applications. The electrical resistivity and thermal conductivity were 18 Ω-cm and 0.28 W/m · K, respectively. The tensile modulus, UTS, and strain at UTS were 2.7 GPa, 56 MPa, and 2.8%, respectively. The flexural modulus, ultimate flexural strength, and strain at ultimate flexural strength were 3.6 GPa, 125 MPa, and 5.5%, respectively.

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