# Measurements on the Enhancement of the Thermal Conductivity of an Epoxy Resin when Reinforced with Glass Fiber and Carbon Multiwalled Nanotubes†

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Measurements of the enhancement of the thermal conductivity of an epoxy resin, when reinforced with plies of plain weave glass fabric and by carbon multiwalled nanotubes (C-MWNT) and with both, are presented. The transient hot-wire technique, as it was recently modified to measure the thermal conductivity of solids, was used with an uncertainty of less than ± 1 %. The results revealed for the epoxy reinforced with glass fibers, with volume fraction of 28 % (47 % weight fraction), the thermal conductivity increase was 27 % compared to plain epoxy resin. When reinforced with C-MWNT, the maximum enhancement observed was about 20 % at a concentration of 1.2 % by weight of C-MWNT. Similarly, when reinforced with both the C-MWNT and glass fibers, the maximum thermal conductivity enhancement observed was about 60 % at a concentration of 1.2 % by weight of C-MWNT.

## Introduction

High-density electronics, as used in communication satellites and avionics, require lightweight thermal management systems. Fiber reinforced composites are considered ideal candidates for these and other applications. Knowledge of both the mechanical and physical properties is pertinent for design. There is a plethora of established standards and/or procedures for the measurement of the mechanical properties of composites, but there are none at the time of writing this for the measurement of physical properties, including the effective thermal conductivity. The absence of reliable thermophysical data may hinder the full utilization of these composites. Empirical and semiempirical models as well as numerical methods, each with their own assumptions,[1−6] have been used to estimate the thermal conductivity of a fiber-reinforced composite, and these ultimately require validation with experimental data that is of paramount importance. Polymer matrices are often reinforced by nanoparticles because they enhance both the physical and the mechanical properties of the composite.[7] To calculate the effective thermal conductivity of nanocomposites with either the numerical or analytical methods adopted for conventional composites requires additional assumptions, and to validate these necessitates very precise and reliable experimental data for comparison.

Many different techniques have been employed in the past to measure experimentally the thermal conductivity of conventional fiber reinforced materials.[8−10] Nevertheless, no single technique exists to measure the thermal conductivity of composites that can provide the basis for establishment of a standard. Recently, we have shown the transient hot-wire technique can be further advanced to allow absolute, very accurate measurements of the thermal conductivity of solids.[11−15] In addition, the enhancement of the thermal conductivity of fluids in the presence of small amounts of nanoparticles has been reported elsewhere.[16]

Following the aforementioned analysis, in a recent paper the transient hot-wire technique was employed to measure the enhancement of the thermal conductivity of an epoxy resin reinforced with glass fiber and/or carbon multiwalled nanotubes (C-MWNT).[17] The measurements revealed for epoxy reinforced with glass fiber with a volume fraction of 28 % (47 % weight fraction) that the thermal conductivity increased by about 27 % compared to solely the epoxy resin. When reinforced with 2 % by mass C-MWNT, the enhancement of thermal conductivity was 9 %, and when reinforced with both C-MWNT and glass fiber, the enhancement was 48 %. In this work, we have extended this study to examine the effect of thermal conductivity enhancement arising from variations in the mass fraction of C-MWNT.

Initially, the thermal conductivity of plain epoxy-resin blocks was measured to obtain the value for the matrix and serve as a control. Specimen were then manufactured and the thermal conductivity determined for the following reinforcements: (a) glass fibers (GF); (b) carbon multiwalled nanotubes (C-MWNT); and (c) (GF + C-MWNT). The mass fractions of C-MWNT were 0.25 %, 0.5 %, and 2 % that had been studied previously.[17] In this work, the term "thermal conductivity" refers strictly to the homogeneous materials, and when applied to reinforced polymers, it is strictly the "effective thermal conductivity". In this article, for simplicity, the term "thermal conductivity" is used throughout.

## Materials and Sample Preparation

***Epoxy-Resin Polymer.*** The epoxy resin used was the EPIKOTE 816 LV and the curing agent EPIKURE 207, available from Hexion Specialty Chemicals, Inc., Ohio. The resin and curing agent are proprietary with the former a mixture

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of 4-[2-(4-hydroxyphenyl)propan-2-yl]phenol, 2-(chloromethyl)-oxirane, and oxiran-2-ylmethyl 2,2-dimethyloctanoate, while the curing agent is a modified polyamine. This resin is a common resin employed in the building and civil engineering industries, e.g., flooring compounds, adhesives, mortars, and grouts. The epoxy was formed from a mixture of resin and a curer of ratio 100/47 by mass that was mixed by a mechanical stirrer. The mixture was poured into molds with dimensions of 10 cm by 5 cm and cured at a temperature of 293 K for a time of 24 h followed by postcuring for a time of 2 h at a temperature of 363 K after which both sides of the samples were polished in an attempt to reduce contact electrical resistance for the measurements. Each set of measurements consisted of two such samples, and to examine the homogeneity of the samples, many such samples were prepared.

***Glass Fiber Reinforced Epoxy-Resin Polymer.*** For the preparation of the reinforced epoxy-resin samples, an elongated-glass plain weave fabric of thickness 0.15 mm was obtained from Hexcel, CT, USA. Its nominal weight is 206 $g \cdot m^{-2}$, and its nominal construction is 7.4 fils-yarn$\cdot cm^{-1}$. The plain weave fabric was chosen because it is characterized by the simplest repetitive geometry among all woven fabrics and would not introduce significant uncertainties to the measurements and thus our conclusions. The unit cell of a balanced plain weave consists of two yarns in both the fill and warp directions.

To fabricate the laminate, the wet lay-up method was employed. The laminate thickness was about 5 mm, with 20 layers of elongated-glass plain weave fabric placed in a stacking sequence $(0°/90°)_{20}$. The fiber volume fraction was about 28 % (47 % by weight) to minimize the effect the fibers may have on the enhancement of the thermal conductivity. The laminate was left to cure at room temperature for a time of about 48 h and postcured at a temperature of 363 K for a time of 2 h. After postcuring, the 5 cm by 10 cm samples were cut from the laminate using a diamond tipped rotary saw. Samples were cut in the direction of the 0° fibers in parallel with the longer axis of the sample. Previous measurements[17] revealed no noticeable dependence of the thermal conductivity on the direction of cut.

***C-MWNT Reinforced Epoxy-Resin Polymer.*** The C-MWNTs employed in this work were produced by Xintek Inc., North Carolina. Macroscopically, the specimen appeared as large individual formations with a portion surrounded by dust. Transmission electron microscopy of the sample was provided by the supplier indicating the mean outer diameter of the C-MWNT was, as illustrated in Figure 1, < 10 nm. The pristine material was examined by scanning electron microscopy, shown in Figure 2, and this revealed a homogeneous sample of well-defined nanotubes with lengths greater than 10 $\mu$m. Unfortunately, the arrangement of the specimen did not permit the determination of mean tube length. These C-MWNTs dimensions and type were chosen because of the results obtained in our previous experiments with liquids.[16]

Four samples of carbon multiwalled nanotubes (C-MWNT) reinforced epoxy resin were prepared. The first two consisted of epoxy resin with 0.25 % and 0.5 % by weight C-MWNT. The other two sets were similar to the first ones but containing also glass fabric with volume fraction of 28 %. It should be mentioned that a preliminary study has shown that increasing the weight percentage of the C-MWNT in the epoxy-resin formulation was limited by the dramatic increase in viscosity making the fabrication of composites unfeasible.

The C-MWNTs were added in the liquid epoxy, and the mixture was shaken by ultrasonic agitation (Bandelin electronics, GmbH, model HD 2200). This procedure was used successfully in our previous work.[16] Following ultrasonic agitation, the hardener was added, and the mixture was stirred mechanically. Sections cut from the composites were studied using scanning electron microscopy, SEM (JEOL model JSM-6300, operating at 20 keV), and the results are shown in Figures 3 and 4. The scope of the SEM analyses was to obtain information on the morphology of the composites. Multiple areas were examined, as well as different samples, and the concentration of C-MWNT was found to be homogeneous and similar in all specimens studied.

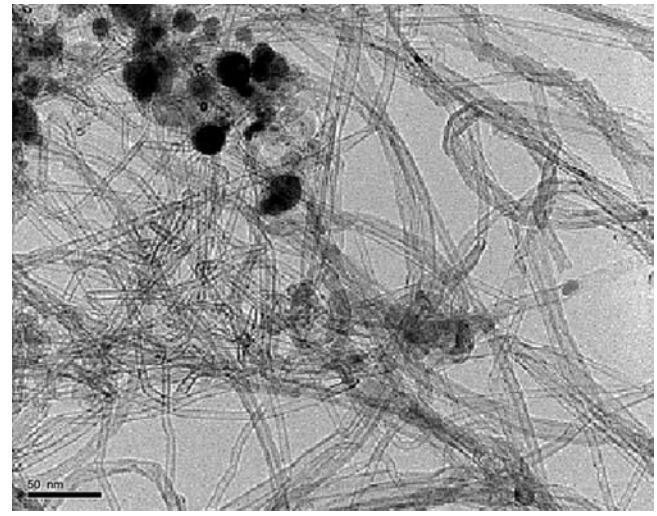


**Figure 1.** Typical transmission electron microscopy SAD images of the C-MWNT used.

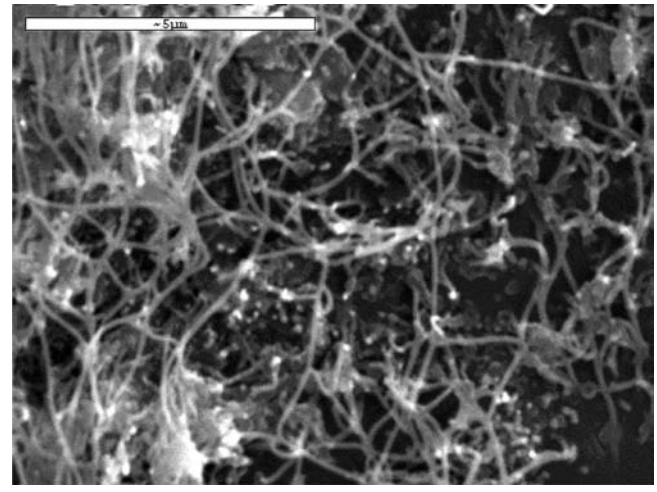


**Figure 2.** Typical scanning electron micrograph of the C-MWNT used.

In the sample with mass fraction of C-MWNT of 0.25 %, shown in Figure 3, two areas were identified: one with woven layers of the glass fabric easily distinguished; they were equally spaced throughout the sample. The resin with the carbon nanotubes located between the woven layers of the glass fabric was also easily identified. In addition, it was possible to observe air bubbles (well-defined circles), the formation of which was associated with the method of manufacture (wet lay-up) of the composites that leave behind a small void fraction. Figure 4 shows a typical high-magnification SEM image that reveals some agglomerated nanotubes at certain locations in the middle, while e-glass is shown on the sides.

Typically, the C-MWNTs are held together as bundles by intrinsic van der Waals forces in most solvents.[7] When mixed with the epoxy resin, the C-MWNTs exist as entangled

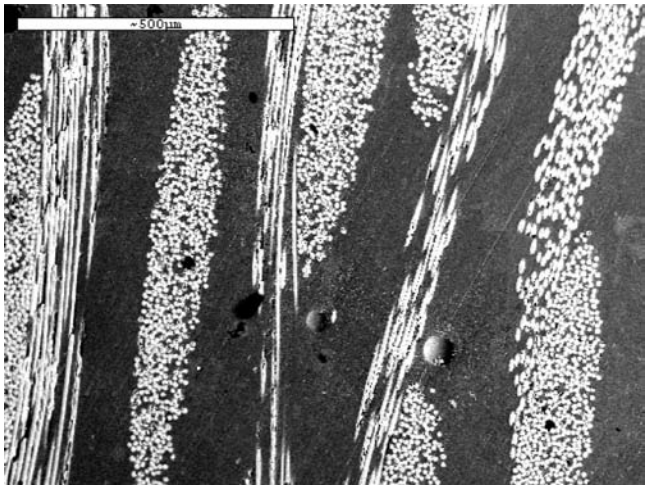


**Figure 3.** SEM image of samples with C-MWNT mass fraction of 0.25 %.

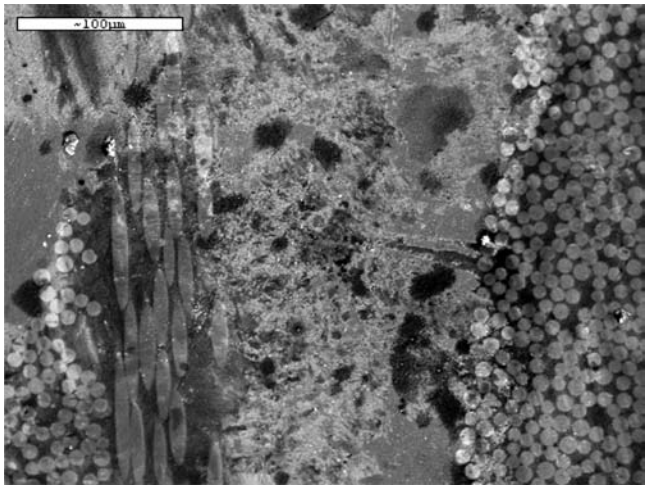


**Figure 4.** SEM image of samples with C-MWNT mass fraction of 0.25 %.

aggregates, which lead to an increased viscosity in the C-MWNT/epoxy system. Surfactant-assisted methods are reported to achieve a good dispersion, but surfactants are difficult to remove from composites.[18] Some studies have also reported that functionalization of the nanotubes can increase dispersion,[19] but in this study, nonfunctionalized nanotubes were used.

***Transient Hot-Wire Technique.*** The transient hot-wire apparatus developed for thermal conductivity measurements has been described elsewhere,[11−15,17] and only the important features are mentioned here. During a single run, 1000 temperatures are measured as a function of time from 20 $\mu$s up to 10 s with bridge that has been discussed elsewhere.[11] In general for this technique, there are the following three unknown parameters: (1) the thickness of the paste (which, in our sensor, is equal to 1 mm); (2) the paste's thermal conductivity and the product of density and specific heat capacity; and (3) the sample's thermal conductivity and the product of density and specific heat capacity. The properties of the paste are obtained from measurements at time between 20 $\mu$s and 0.3 s, and with these, the parameters related to the thermophysical properties of the sample can be obtained from measurements at times between 0.3 and 10 s.

To fully describe the complete geometry of the sensor, the COMSOL Multiphysics V.3.2b finite element package was employed. This permits modeling of the sensor, which is the wire and silicone paste, the Kapton film (which has a negligible effect), and the solid. In this model, the measured thermal response with time is matched by the program by adjusting the unknown parameters resulting in essentially perfect agreement between the measured and calculated temperatures with time from 20 $\mu$s upward. Hence, full advantage of the whole temperature versus time curve is obtained. In COMSOL, we assume the composite blocks are one material and the calculated "thermal conductivity" is strictly the "effective thermal conductivity".

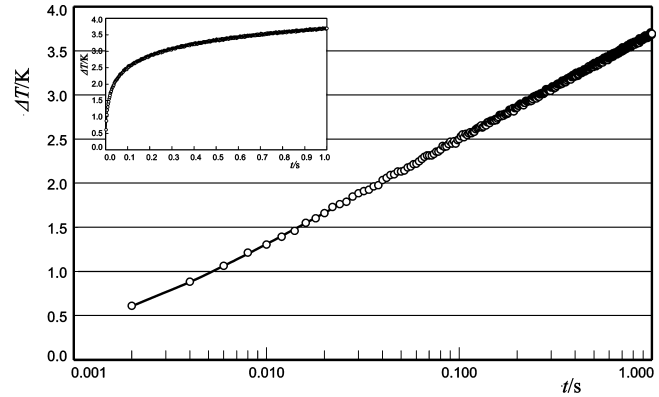


**Figure 5.** Typical temperature rise, $\Delta T$, as a function of time, $t$, for toluene at 302 K: ○, experimental points; −, COMSOL calculated points.

## Results and Discussion

***Validation of the Technique.*** Since there is no standard thermal conductivity solid known to be better than ± 5 %, the performance of the experimental apparatus was validated with measurements on liquid toluene that has been proposed by the Subcommittee on Transport Properties of the International Union of Pure and Applied Chemistry as a standard thermal conductivity liquid with an uncertainty of ± 0.6 %.[20] Indeed, it is a great advantage of the apparatus employed that it can perform measurements of thermal conductivity on both liquids and solids. The wires with their support were placed in toluene at a temperature of 302 K, and 1000 temperature measurements were made as a function of time after emitting a thermal pulse. To calculate the thermal conductivity of toluene, the COMSOL package was used, substituting the properties of the silicone layer, the Kapton film, and the solid for those of liquid toluene. The results for one measurement are shown in Figure 5. The thermal conductivity value obtained from the COMSOL model deviated by 0.4 % from the value proposed by the Subcommittee on Transport Properties of the International Union of Pure and Applied Chemistry, whereas the product of density and specific heat capacity was found to deviate by ± 0.3 % from literature values.[21,22]

The total absolute uncertainty of the measurement, derived from uncertainty in the variables involved, was estimated to be better than ± 1 %.[15,17] This estimate concurs with that obtained with the measurements of the thermal conductivity of toluene (and for that matter of Pyroceram 9606 elsewhere[15]). The estimated uncertainty in the measurement of the product density and specific heat capacity is about ± 5 %.

***Thermal Conductivity of the Epoxy-Resin Polymer Reinforced with Glass Fibers as a Function of Temperature.*** Following the validation procedure, the wires were placed in the silicone paste, and the sensor, prepared as described in the previous section, was placed between two blocks of the epoxy-resin polymer. Several different sets of epoxy-resin blocks were

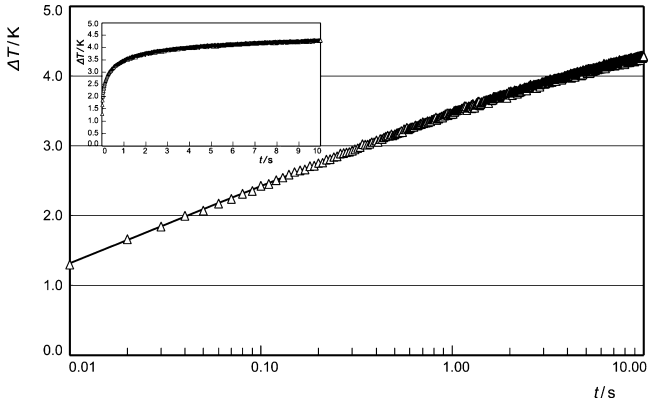


**Figure 6.** Typical temperature rise, $\Delta T$, as a function of time, $t$, for epoxy-resin polymer at 298.15 K: Δ, experimental points; −, COMSOL calculated points.

**Table 1. Measured Thermal Conductivity, $\lambda$, and Deviations, $\Delta\lambda^a$, for the Composites as a Function of the Temperature $T$**

| material | $T$/K | $\lambda$/(W·m$^{-1}$·K$^{-1}$) | $\Delta\lambda^a$/% |
|---|---|---|---|
| Epoxy-Resin Polymer | 298.15 | 0.252 | 0.83 |
| | 307.46 | 0.255 | −0.72 |
| | 320.32 | 0.267 | 0.22 |
| | 335.84 | 0.275 | −1.07 |
| | 348.78 | 0.290 | 0.84 |
| Epoxy-Resin Polymer + Glass Fabric $(0°/90°)_{20}$ | 298.15 | 0.320 | −0.38 |
| | 312.20 | 0.330 | −0.20 |
| | 320.12 | 0.338 | 0.61 |
| | 333.81 | 0.347 | 0.54 |
| | 348.50 | 0.353 | −0.57 |
| Epoxy-Resin Polymer + Glass Fabric $(0°/90°)_{20}$ (laminate cut at 60° angle from principal axis) | 298.15 | 0.324 | |
| | 306.93 | 0.332 | |
| | 320.27 | 0.345 | |

$^a$ $\Delta\lambda = 100\cdot[(\lambda - \lambda_{fit})/\lambda_{fit}]$, with $\lambda_{fit}$ from eq 1 and eq 2.

prepared by weighing as described above. The values of the thermal conductivity of these samples indicate that although the technique is accurate to ± 1 % the actual values measured differ by ± 2 % because of inhomogeneities in the samples probably attributed to factors like imperfect mixing or nonidentical composition. Figure 6 shows the measurements obtained at a temperature of 298.15 K for which there are about 1000 measurements of temperature at times between (0.01 and 10) s. The first part of the curve is related to the properties of the silicone paste and the second part to the properties of the epoxy-resin polymer. If the contact between the solid and the silicone was not excellent or there was an air gap present, it would have shown as a jump in the line.

The results for the thermal conductivity of the epoxy-resin polymer as a function of temperature are shown in Table 1 and plotted in Figure 7. The thermal conductivity, $\lambda$, was fit to a function of the absolute temperature $T$ by

$$\lambda/(\mathrm{W\cdot m^{-1}\cdot K^{-1}}) = \lambda(298.15\ \mathrm{K})/(\mathrm{W\cdot m^{-1}\cdot K^{-1}}) \cdot \left\{0.1118 + 0.880\left(\frac{T/\mathrm{K}}{298.15}\right)\right\} \quad (1)$$

where the value of the thermal conductivity, $\lambda$(298.15 K), is given in Table 1. The maximum deviation of the experimental measurements presented in Table 1 from eq 1 is −1.07 %, and the standard deviation of the fit at a 95 % confidence level is 0.69 %.

The measurements of the thermal conductivity of the glass fiber reinforced epoxy resin as a function of the temperature are also listed in Table 1 and shown in Figure 7 along with those for the epoxy resin for the purpose of comparison.

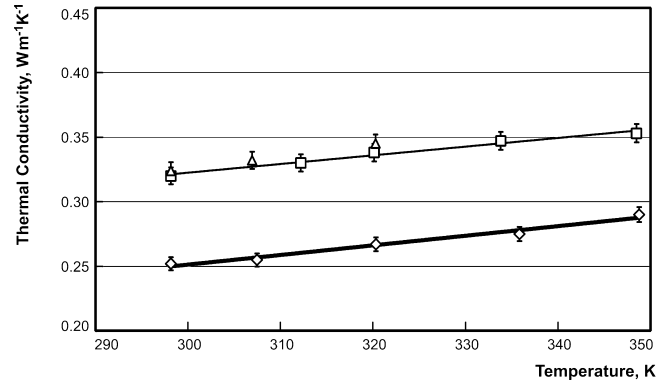


**Figure 7.** Thermal conductivity $\lambda$ measurements as a function of temperature T. ◇, epoxy-resin polymer measurements; -, eq 1; □, epoxy-resin polymer + glass fiber measurements; -, eq 2; Δ, epoxy-resin polymer + glass fiber (samples cut at 60° angle from principal axis) measurements.

**Table 2. Measured Thermal Conductivity, $\lambda$, Deviations, $\Delta\lambda_{enh}{}^a$, and Predicted Thermal Conductivity, $\lambda_{Pred}$, as a Function of the C-MWNT Concentration at $T$ = 298.15 K**

| C-MWNT mass fraction (%) | $\lambda$/(W·m$^{-1}$·K$^{-1}$) | $\Delta\lambda_{enh}{}^a$/% | $\lambda_{Pred}$/(W·m$^{-1}$·K$^{-1}$) |
|---|---|---|---|
| Epoxy-Resin Polymer | | | |
| 0 | 0.252 | − | |
| 0.25 | 0.265 | 5.1 | |
| 0.50 | 0.290 | 15.1 | |
| 2.0 | 0.275 | 9.1 | |
| Epoxy-Resin Polymer + Glass Fabric $(0°/90°)_{20}$ | | | |
| 0 | 0.320 | 27.0 | 0.335 |
| 0.25 | 0.367 | 45.6 | 0.340 |
| 0.25 | 0.363 | 44.0 | |
| 0.5 | 0.399 | 58.3 | 0.402 |
| 0.5 | 0.396 | 57.1 | |
| 2.0 | 0.373 | 48.0 | 0.371 |

$^a$ $\Delta\lambda_{enh} = 100\cdot[(\lambda - \lambda_o)/\lambda_o]$, with $\lambda_o$ = 0.252 W·m$^{-1}$·K$^{-1}$.

Reinforcing the polymer with the glass fabric results in a 27 % increase in the thermal conductivity that might be an artifact of the relatively high thermal conductivity of the glass fibers compared to the epoxy-resin matrix. The observed increase in thermal conductivity with temperature is a linear relationship that has resulted from the stronger dependence of the epoxy matrix with the temperature. A linear relationship was also obtained between the thermal conductivity and the temperature in the measurements of the plain epoxy blocks of Figure 7.

The thermal conductivities listed in Table 2 were obtained by placing the samples with the glass fibers parallel to the sample axis, thus parallel to the wires of the sensor. Measurements on samples with the 60° angle show that the orientation of the fibers had no significant effect on the thermal conductivity and support the conclusions presented in our previous work.[17] The isotropic nature of the solid that results from the amorphous character of the glass fibers probably nullifies the effect of orientation of the fibers in the composite.

The thermal conductivity, $\lambda$, values listed in Table 1, were fit as a function of the temperature $T$ to an equation

$$\lambda/(\mathrm{W\cdot m^{-1}\cdot K^{-1}}) = \lambda(298.15\ \mathrm{K})/(\mathrm{W\cdot m^{-1}\cdot K^{-1}}) \cdot \left\{0.3784 + 0.6254\left(\frac{T/\mathrm{K}}{298.15}\right)\right\} \quad (2)$$

where the value of the thermal conductivity, $\lambda$(298.15 K), is given in Table 1. The maximum deviation of the experimental points presented in Table 1 from eq 2 is 0.61 %, and the standard deviation at the 95 % confidence level is 0.29 %.

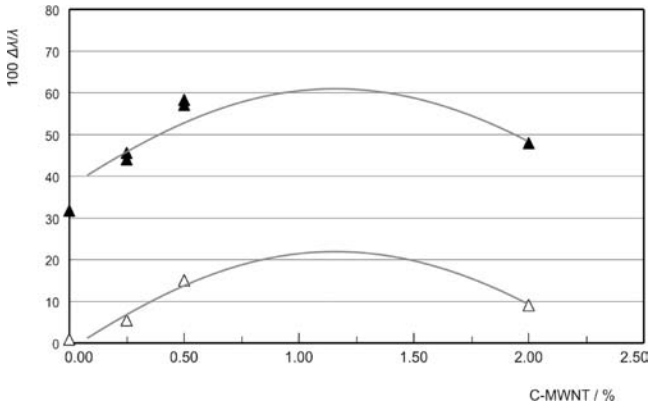


**Figure 8.** Enhancement of the thermal conductivity measurements, 100 $\Delta\lambda/\lambda$, as a function of the composition of the C-MWNT mass fraction: Δ, epoxy resin + C-MWNT; ▲, epoxy resin + C-MWNT + glass fibers. Black lines show trends only.

***Thermal Conductivity of C-MWNT Reinforced Epoxy as a Function of the C-MWNT Composition.*** Table 2 shows the thermal conductivity measurements of the epoxy resin reinforced with C-MWNT and the epoxy resin reinforced with C-MWNT and glass fibers as a function of the concentration of the C-MWNT at a temperature of 298.15 K. Values obtained on different samples are also included. In the same table, the enhancement of the thermal conductivity is also listed and shown in Figure 8. Values derived from a previous study[17] for the 2 % mass fraction of C-MWNT are also included solely for the purpose of comparison. From the trend in the figure, it seems that reinforcing the polymer only with C-MWNT resulted in a 20 % increase in the thermal conductivity with 1.2 % mass fraction C-MWNT. Reinforcing the epoxy resin with both C-MWNTs and glass fibers produced a thermal conductivity enhancement of about 60 % at the same concentration of C-MWNT. The apparent maximum observed in both curves can be attributed to the following: (a) the conductive path threshold limit between the plies of the glass fabric; (b) the breakage of the length of the nanotubes as a result of a stronger mixing to achieve uniformity; and (c) the difficulties observed in proper mixing at the high concentrations of 2 %. The observed maximum in enhancement of the thermal conductivity has been observed with C-MWNT in liquids,[16] and it was shown that the critical parameter was the ratio of the length to diameter of the nanotube. Further measurements in that region are necessary to better understand the mechanism.

***Thermal Conductivity Prediction of Composites.*** To conclude this study, a preliminary attempt to predict the thermal conductivity of the composites was made. It was assumed that the sample reinforced with glass fibers consisted of 21 layers of (resin + C-MWNT) of thickness of about 100 $\mu$m and 20 layers of glass fabric of thickness of 150 $\mu$m. The layers are considered to be consecutively placed one above the other starting with a layer of (resin + C-MWNT), followed by a glass fabric layer, etc., bringing the total thickness of the sample to about 5 mm. This geometry is simulated with the COMSOL software package, while the thermophysical properties of the (resin + C-MWNT) films used were obtained from the experiments. For the thermal conductivity of the glass fabric layer, as no value is given by the manufacturer, an average literature value equal to 0.5 $W \cdot m^{-1} \cdot K^{-1}$ was used. A triangular mesh was used with about 20 000 elements with the mesh being finer at the interfaces. The COMSOL package estimated the temperature response as a function of time. To obtain the thermal conductivity of the whole sample, the aforementioned estimated temperature response was compared with one determined by COMSOL for a uniform material of the same dimensions and equal heat flux.

The values predicted by COMSOL are listed in Table 2 and agree with the experimental values suggesting the model adopted was adequate at estimating the thermal propagation and is worth further investigation. The deviations obtained clearly follow those of the experimental values that are input for the thermal conductivity of the resin reinforced with glass fabric. If smoothed values had been used, the deviations would be smaller.

This estimation scheme is not very sensitive to the value obtained from the open literature for the thermal conductivity of the glass fiber because a 10 % increase in the value used resulted in a 4 % increase of the thermal conductivity estimated for the whole sample.

## Conclusions

This work focused on the precise measurement of the thermal conductivity of composites and nanocomposites using the transient hot-wire technique that is capable of measuring thermal conductivity of solid materials in an absolute way, with an uncertainty of better than 1 %. The experimental technique was validated by comparing the results obtained with liquid toluene with an accepted standard. For the composites, and in particular glass fiber reinforced laminates, the enhancement in the thermal conductivity was found to be 27 % over that obtained for the thermal conductivity of solely the epoxy-resin polymer. This result is satisfactory in view of the volume fraction (about 28 %) of the glass fibers used in the composite. When the epoxy was reinforced with C-MWNT, the maximum enhancement observed was about 20 % at a concentration of 1.2 % by mass of C-MWNT. The thermal conductivity obtained when reinforced with both the C-MWNT and glass fiber was about 60 % above that of the resin alone at a concentration of 1.2 % by mass of C-MWNT.

A method of predicting the thermal conductivity of the whole sample, based on the properties of each constituent layer, was obtained from COMSOL that was in good agreement with the measurements.

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