Studies of Electrical and Thermal Conductivities of Sheared Multi-Walled Carbon Nanotube with Isotactic Polypropylene Polymer Composites

Regular Paper

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Abstract

Polymer nanocomposite materials of higher thermal and electrical transport properties are important to nanotechnology applications such as thermal management, packaging, labelling and the textile industry. In this work, thermal and electrical conductivities in nanocomposites of multi-walled carbon nanotubes (MWCNT) and isotactic polypropylene (iPP) are investigated in terms of MWCNT loading, temperature dependence, and anisotropy caused by melt shearing. IPP/MWCNT nanocomposites show a significant increase in thermal and electrical conductivity with increasing MWCNT loading, reaching 17.5 W/m K and 10^{-6} S/m, respectively, at a MWCNT 5.0 weight percentage at 40°C. The increase in MWCNT/iPP is more than would be expected based on the additivity rule, and suggests a reduction of the interfacial thermal electrical resistance at nanotube-nanotube junctions and the nanotube-matrix interface. The anisotropy in both conductivities was observed to be larger at low temperature and to disappear at higher temperature due to isotropic electrical and thermal contact in both directions. Oriented MWCNT/iPP nanocomposites exhibit higher electrical and thermal conductivities, attributed primarily by orientation of nanotubes due to the shearing fabrication process.

Keywords MWCNT, iPP, thermal and electrical conductivity, percolation, shearing and anisotropy

1. Introduction

The modern history of human technology has been defined by the replacing of machines with suitable materials which can perform functions more efficiently and without much maintenance. Composite materials have continuously been substituted with nano-engineered and adapted polymer nanocomposite materials. Carbon nanotubes have been considered as ideal additive fillers for composite materials

to improve both electrical and thermal transport properties. In the past, experimental and theoretical work has shown a significantly high thermal conductivity with 3000 W/m K for multi-wall carbon nanotubes (MWCNT) [1-2]. Recent reports showed modest increases in the thermal conductivity of polymers at lower volume fraction of nanotubes loading [3-10]. Polymers have low thermal and electrical conductivities due to restriction of the phonon/electron motion through the composite matrix, and have larger interfacial thermal/electrical resistances at the polymer-nanotube interfacial surface [4, 11-12].

Here we focus on isotactic polypropylene (*iPP*) polymer, which is relatively easy to process, inexpensive, and non-toxic. *IPP* has liquid crystalline properties, which exhibit a smectic phase under shear stress. This smectic phase can be a precursor to an oriented fibrillar fibriller structure along the direction of shear alignment [13-14]. Recent experimental efforts showed that the orientation has a significant influence on thermal and electrical conductivity in SWCNT buckypapers, and that MWCNT thermal conductivity is higher for moderately aligned nanotubes as compared to randomly oriented nanotubes [15-20]. The induced nanoscopic orientation order of MWCNTs and complex polymer chains will affect effect the macroscopic property of electrical conductivity [17, 18]. The electrical conductivity of polymer composites is low at lower MWCNTs loading [21-22] and these nanocomposites should allow for tuneable material characteristics. The anisotropy of the carbon nanotubes is tuned into a global anisotropy in macroscopic properties of composites by orienting the nanotubes in the composites sample [4]. To achieve excellent electrical, thermal, optical and mechanical properties, we need to have excellent dispersion and stronger adhesive interaction of MWCNTs within the polymer matrix [23-31]. There are many fabrication methods to achieve good dispersion of nanotubes, such as molten processing [22–23, 32], or in solution [24, 33-35]. The melt-mixing fabrication process is the preferred method of nanocomposites. The aggregation of nanotubes can be reduced by an appropriate shearing process during the melting process. There are many ways of introducing nanotubes into polymer matrices, such as nanotubes hydrogel or aerogel followed by polymer infiltration [36]; MWCNT can be directly added to polymers during melt mixing, and commercially available polymers and nanotube composites can be used as a material. Concentration can be changed by adding a suitable amount of polymer into the melt-mixing fabrication process.

For this study, we probed the electrical and thermal conductivity of MWCNT/iPP nanocomposites by considering two factors to improve thermal and electrical conductivity: shearing and temperature. The aim of this study was to determine the shearing effect, temperature dependence, and anisotropy for both electrical and thermal conductivities.

2. Experimental methods

Isotactic polypropylene (iPP) with a molecular weight of about 38 kg/mol was obtained from Scientific Polymer Products, Inc., catalogue #130, in powder form with a crystallization temperature of 404.64 K [35] and a melting temperature of 436.1 K [35]. Multi-walled carbon nanotubes with diameter 140 ± 30 nm and length 7 ± 2 μm were purchased from MER Corporation and were produced by catalytic chemical vapour deposition (CVD) with a stated purity of greater than 90%. For further purification of MWCNTs, one gram of the MWCNTs was suspended in a mixture of concentrated sulphuric acid and nitric acid (3:1 vol. ratio). This solution was sonicated in a Misonix water bath sonicator for 24 h at 323 K. The resultant suspension was diluted with deionized water and filtered through a 400 nm pore membrane (PTFE) until the water passing through the filter had a pH between 6 and 7. The dispersions were subsequently filtered to the desired concentration. The resulting MWCNTs had a pH of between 3 and 3.5 and were stable, although the surface of the MWCNTs suffered oxidative damage. Nanocomposites were prepared by sonicating MWCNTs in Xylene at 323 K in a flask for 30 min. The iPP polymer was added to the xylene containing the MWCNTs to form different weight percentage concentrations of MWCNTs in the nanocomposites for the study. The solution was then heated and stirred by placing the flask in an oil bath on a hot plate. Once the iPP was completely dissolved, the solution was mixed slowly into a non-solvent (a polar compound such as alcohol) with about a 5× volume dilution. The resulting precipitate was dried and then pressed into a film using a compression-moulding hot press at a temperature of 473 K and pressure of 20.6 to 41.4 MPa. These non-sheared samples were then crystallized at 403 K for 30 min and cooled to room temperature for further sample preparation.
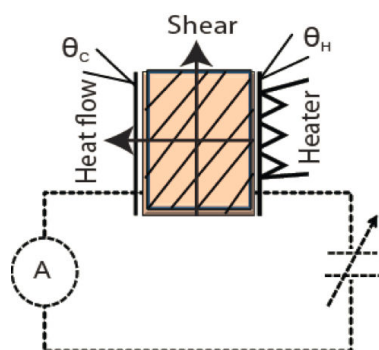
Figure 1. The thermal and electrical conductivity experimental setup is for polymer/MWCNT thin-film nanocomposites. The parallel-plate cell contains the thin-film sample that completely fills the gap (Area, A=0.41 mm²; thickness, d=0.09 mm). Note that the sheared samples have the shear direction in the plane of the parallel plates (perpendicular to the current flow). (θ_c and θ_h are the cold- and hot-side temperatures of the sample, measured with thermocouple readings).

Films were handled carefully and kept in dust-free environments, and the thin-film samples were sandwiched between

two metal plates. Two electrical leads were connected to both metal plates. The resistivity was measured using a Pico-ammeter with varying voltage across the thickness of samples, i.e., the direction perpendicular to the direction of the shear rotation in the Linkam CSS 450 shearing stage. The resistivity of the sheared sample gives its perpendicular component and the non-sheared samples provide a measure of the average resistivity. Voltages from 0 to 100 V were applied across the samples and the current was recorded with a Pico-ammeter. The linear electrical resistances were measured from the slope of the current-voltage graph. All electrical transport measurements were done at temperatures of 0°C to 50°C. The film had dimensions of length between 7 mm and 8 mm, and of width between 6 mm and 7 mm, for sheared and non-sheared samples. The thickness of the films was determined with a micrometer and had an error ranging from ± (0.04- 0.06 mm) for the non-sheared and sheared neat iPP films (0% MWCNT), and from ±(0.07- 0.09 mm) for the non-sheared and sheared nanocomposite samples. The electrical conductivity was then calculated using $R = d\sigma/A$, where σ is electrical conductivity, d is thickness of the sample and A is the cross-sectional area of the sample, A=wL, where w is the width and L is the length of the sample. In both samples, σ sheared ($\sigma_\perp$) and σ un-sheared (σ_{avg}), were determined. In this geometry, perpendicular to the film thickness, the contact area is maximized. The same setup was used for thermal conductivity measurements by attaching two thermocouples to measure temperature gradient across the two sides of a sample. As shown in Figure 1, a heater was attached to one side of the sample to apply voltage and then the linear thermal resistance was measured. All thermal transport measurements were done over a temperature of 0°C to 50°C.

The thermal and electrical conductivity were measured for all samples using the same procedure on at least three samples for each measurement.
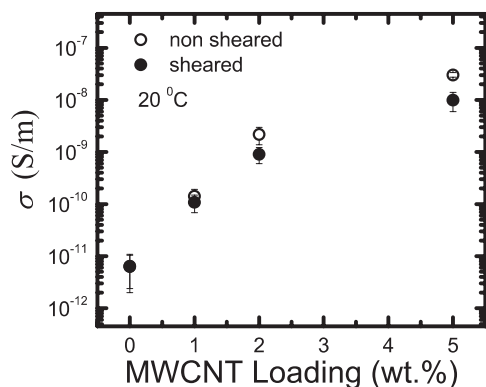
3. Results and discussion

A schematic illustration of CNT network formation is shown in Figure 2: random in un-sheared sample (2a), and aligned in sheared sample (2b). Polymer chains (black) are shown stretched alongside the nanotubes. The electrical conductivity σ, measured in this work, of neat iPP, iPP / MWCNT sheared and non-sheared films as a function MWCNT loading is shown in Figure 3. The electrical conductivity of pure iPP 10^{-11} S/m agrees well with the values reported in the literature [37]. The electrical conductivities of iPP/MWCNT nanocomposites do not exhibit typical percolation as a function of MWCNT loading; rather they show broader percolation due to shearing fabrication process. The trend is that the electrical conductivity increases by orders of magnitude with the addition of MWCNTs. For the nanocomposites, the conductivity for sheared composite samples is lower than for non-sheared composite samples (i.e., $\sigma < \sigma_{avg}$) due to 2D planar orientation of the CNTs at lower loading of MWCNT (less than 5wt %). In planar orientation, the average distance between the nanotubes is larger than in random orientation, which is expected to decrease the electrical conductivity in the sheared composite samples, shown schematically in Figure 2. For neat iPP, the effect is not present and this indicates that the effect of orientation on the conductivity of the nanocomposites is a novel property induced by adding MWCNTs.
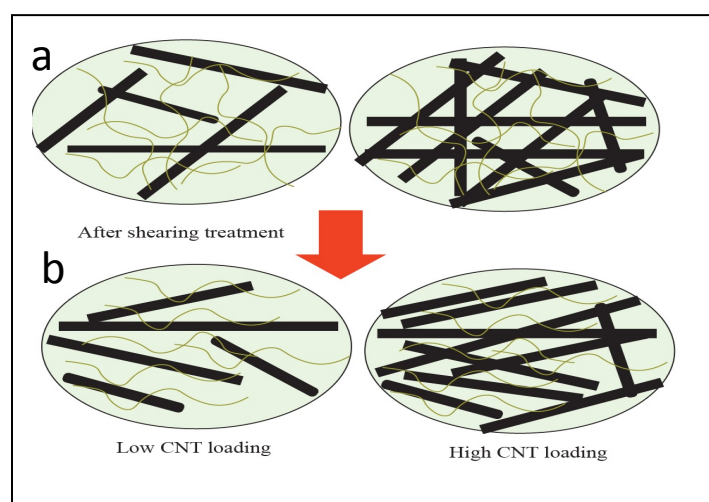
Figure 2. Schematic illustration of CNT network formation: random in un-sheared sample (a), and aligned in sheared sample (b). Polymer chains (black) are shown stretched alongside the nanotubes.

All these nanocomposites showed broader percolation threshold in electrical conductivity, which could be due to the melt-shearing process. The electrical conductivity significantly increased at higher loading of MWCNT (5.0 wt.%), which is attributed to lower contact resistance, lower interfacial resistance between the nanotubes-nanotube and nanotube-polymer matrixes. The polymer coating on nanotube junctions and on the nanotubes leads to lower

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Figure 3. Electrical conductivity of neat iPP and iPP/MWCNT composites as a function of MWCNT loading (wt.%) for sheared (filled circles) and non-sheared (empty circles) composites

interfacial resistance, which enhances electrical conductivity. Our previous study found that MWCNTs nucleate and improve the crystallization of *iPP* [35], such that MWCNT are partially covered with polymer crystallite. This formation of *iPP* crystallites does not damage the electrically conductive MWCNT network.

Electrical conductivity of neat iPP and 5.0 wt% MWCNT nanocomposites as a function of temperature for sheared and non-sheared composites is shown in Figure 4. The electrical conductivity for neat iPP remains the same in both sheared and non-sheared samples. It increases slightly at higher temperature due to increased polymer crystallite formation [38]. The electrical conductivity of 5.0 wt.% MWCNT composite is larger in non-sheared samples than in sheared samples. The electrical conductivity of 5.0 wt% MWCNT remains the same at lower temperature (up to room temperature) and increases from 10^{-8} to 10^{-6} S/m at a temperature of 325 K. The electrical conductivity of nanocomposites reaches approximately the same value at higher temperature for both sheared and non-sheared composite samples at the same loading of nanotubes. At higher temperature, the interfacial resistance becomes lower at the nanotube/polymer matrix interface, which leads to an increase in the electrical conductivity.
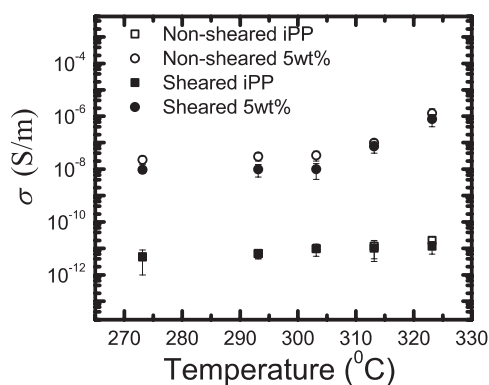
Figure 4. Electrical conductivity of neat iPP (squares) and 5.0 wt% MWCNT nanocomposites (circles) as a function of temperature for sheared (filled symbols) and non-sheared samples (empty symbols)

The thermal conductivity κ, measured in this work, of neat iPP, iPP /MWCNT sheared and non-sheared films as a function of MWCNT loading is shown in Figure 5. The thermal conductivity of the neat iPP is 0.26 W/m-K, agreeing well with the values reported in the literature [39]. For MWCNT/iPP non-sheared nanocomposites, the thermal conductivity increases with nanotube loading. At the highest loading of MWCNT (5wt%), the non-sheared composite thermal conductivity reaches 16 W/m-K at room temperature, an increase of 50 times compared to the neat iPP. The thermal conductivity is slightly lower in sheared composites due to planar orientation: the average distance between the nanotubes is larger than in random orientation (see Figure 2). For the neat iPP sample, the effect is not present. The behaviour of the thermal conductivity in nanocomposite samples is significantly different from the electrical conductivity. There is a significant increase in thermal conductivity at 5.0 wt.% MWCNT loadings, which is due to the low interfacial thermal resistance between the polymer matrix and MWCNT, as previously described. Strong thermal coupling between the MWCNT and polymer matrix causes low interfacial thermal resistance and higher phonon transport.
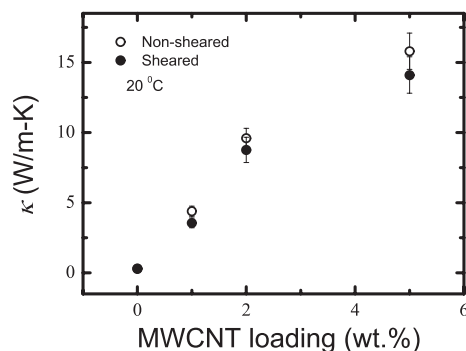
Figure 5. Thermal conductivity of neat iPP and 5.0 wt.% MWCNT composites as a function of MWCNT loading for sheared (empty circles) and non-sheared composites (filled circles)
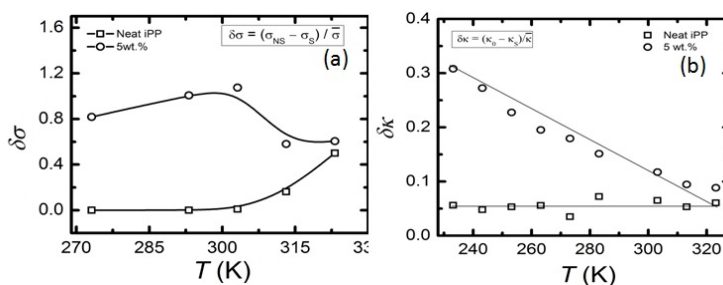
Thermal conductivity of neat iPP and 5.0 wt% MWCNT composites as a function of temperature for sheared and non-sheared composites is shown in Figure 6. At the higher MWCNT loading (5.0 wt.%) and higher temperature, the thermal conductivity for nanocomposites is significantly larger than the thermal conductivity in neat iPP (17.5 and 0.26 W/m-K, respectively), which implies that the contributions from the matrix and the MWCNT network are not simply additive, based on nanofiller loading. The observed increase could be the result of significant changes at the interface between polymer matrix and nanotubes. The thermal conductivity of neat *iPP* remains constant as a function of temperature. This study of nanocomposites calculates the contributions to the thermal conductivity from the amorphous and crystalline phases to be 0.091 and 0.593 W/m-K, respectively. So the thermal conductivity of the matrix itself is different with different MWCNT loading. Thus, we hypothesize that the *iPP* matrix reduces the interfacial thermal resistance relative to the other polymer matrices. We have previously observed that iPP can nucleate

with MWCNT loading in the melting state found that iPP nucleates on MWCNT are in the melt state, thereby locally increasing the *iPP* crystallinity at the polymer/MWCNT interface [35]. Thus, in *iPP* matrix, *iPP* crystallites are more likely to span between MWCNTs at higher MWCNT loadings. Bridging lamellae between MWCNTs could reduce the interfacial thermal resistance and consequently improve the thermal conductivity of the MWCNT network in *iPP* matrix at higher MWCNT loadings. Our results from polymer/nanotube nanocomposites suggest that the interfacial thermal resistivity can be reduced by increasing the nucleation of crystallites at the polymer/MWCNT interface. The observed increase in thermal conductivity for iPP/MWCNT composite is predominately caused by orientation of CNT due to the shearing treatment.
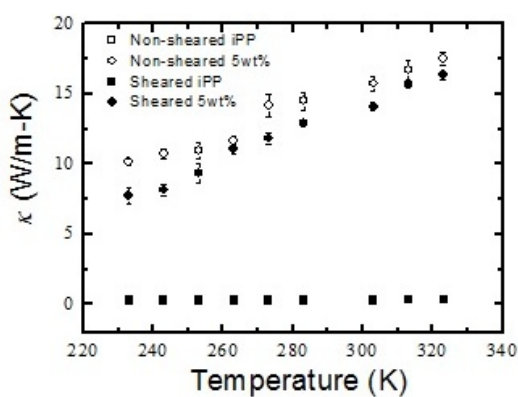
Figure 6. Thermal conductivity of neat iPP (squares) and 5.0 wt.% MWCNT composites (circles) as a function of temperature for sheared (filled circles) and non-sheared composites (empty symbols)

The orientation of *iPP* in neat and nanocomposite samples was varied from isotropic (re-crystallized in a melt sheared)

to be highly aligned with the shearing fabrication process. Considering alignment of the MWCNTs, given the tendency of MWCNT to nucleate *iPP* crystallization and to template *iPP* lamellae growth perpendicular to the MWCNT axis, both the polymer matrix and nanotubes are highly aligned after the shearing process.

Anisotropy in electrical conductivity of neat and 5.0 wt.% MWCNT composites is a function of temperature, as shown in Figure 7a. With respect to electrical conductivity [27], Du et al. have previously shown that MWCNT alignment increases the loading necessary to form a nanotube network sufficient for electrical percolation. Also, in-plane electrical and thermal conductivity of interface layers of graphite-nanoplatelet (GNP)-based composites is three orders higher than through-plane [40-41]. Our results suggest that alternative nanocomposite fabrication and processing methods that combine the effect of aligning a matrix and higher MWCNT loadings are likely to exhibit higher electrical conductivities with broader percolation threshold. The anisotropy in electrical conductivity is higher in 5.0 wt.% MWCNT composites at room temperature and becomes lower at higher temperature due to isotropic contacts in both directions.

Anisotropy in thermal conductivity of neat and 5.0 wt.% MWCNT composites as a function of temperature is shown in Figure 7b. The anisotropy in thermal conductivity is higher for 5.0 wt.% MWCNT composites and is absent in neat iPP. It disappears at higher temperature due to isotropic thermal contacts in both directions.

Figure 7. Anisotropy in electrical (a) and thermal (b) conductivity of neat (squares) and 5.0 wt.% MWCNT composites (circles) as a function of temperature. Anisotropy is defined as $\delta\sigma = (\sigma_{NS} - \sigma_S)/ \sigma_{Avg}$ and $\delta\kappa = (\kappa_{NS} - \kappa_S)/ \kappa_{Avg}$.

The composites showed a new property in polymer nanocomposites with an anisotropy in thermal and electrical conductivities. This can be tuned in degree of anisotropy and in absolute magnitude by controlling several parameters of the nanocomposites. The interfacial interactions of polymer and nanotubes, degree of alignment, aspect ratio of nanotubes, and the melt-shearing process are main factors to improve thermal and electrical

conductivities. These factors will significantly affect the electrical and thermal transport properties and improve the conductivities by orders of magnitude. The shearing effect and temperature dependence significantly improve thermal and electrical conductivities due to higher conductive path for electron and phonon transport. This property can be combined with other properties of polymer/carbon nanotube composites, like anisotropy and degree of

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conductivity, to achieve an ideal combination of these properties for several desired nanotechnology applications. Further improvement of the methods of dispersion and alignment of the CNTs can enhance electrical and thermal anisotropy for composite films.

4. Conclusion

IPP/MWCNT nanocomposites exhibit nonlinear behaviour of their thermal and electrical conductivities as a function of the CNTs' concentration, shearing effect and temperature dependence. The following results can be highlighted from our study. (i) The thermal and electrical conductivities of composites were higher in non-sheared composites than sheared composites due to planner 2D orientation. (ii) These nanocomposites show a significant increase in thermal and electrical conductivity with increasing MWCNT loading, reaching 17.5 W/m K and 10^{-6} S/m, respectively, at a MWCNT 5.0 weight percentage, at 40^0C. (iii) The anisotropy in both conductivities was observed to be larger at low temperature and to disappear at higher temperature due to isotropic electrical and thermal contact in both directions. Our sample fabrication shearing process method allows the creation of highly aligned polymer nanocomposites and is similar to the method used in industry, making our approach suitable for direct implementation for a wide range of nanotechnology applications including packaging, labelling and the textile industry.

5. Acknowledgements

6. References

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