

# Improving Electrical Conductivity and Thermal Properties of Polymers by the Addition of Carbon Nanotubes as Fillers

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## Abstract

The remarkable electrical and thermal conductivities of isolated carbon nanotubes have spurred worldwide interest in using nanotubes to enhance polymer properties. Electrical conductivity in nanotube/polymer composites is well described by percolation, where the presence of an interconnected nanotube network corresponds to a dramatic increase in electrical conductivity ranging from  $10^{-5}$  S/cm to 1 S/cm. Given the high aspect ratios and small diameters of carbon nanotubes, percolation thresholds are often reported below 1 wt%, although nanotube dispersion and alignment strongly influence this value. Increases in thermal conductivity are modest ( $\sim 3$  times) because the interfacial thermal resistance between nanotubes is considerable and the thermal conductivity of nanotubes is only  $10^4$  greater than the polymer, which forces the matrix to contribute more toward the composite thermal conductivity, as compared to the contrast in electrical conductivity,  $>10^{14}$ . The nanotube network is also valuable for improving flame-retardant efficiency by producing a protective nanotube residue. In this article, we highlight published research results that elucidate fundamental structure-property relationships pertaining to electrical, thermal, and/or flammability properties in numerous nanotube-containing polymer composites, so that specific applications can be targeted for future commercial success.

## Introduction

Carbon nanotubes possess three important characteristics relative to the numerous available fillers: phenomenal electrical conductivity, nanoscopic size, and high aspect ratio. This combination of properties can lead to electrical percolation at low concentrations and has naturally spurred considerable activity in producing value-added polymer nanocomposites. Electrical percolation occurs when the filler has an electrical conductivity vastly higher than the polymer matrix. The higher

electrical conductivities that are available in nanotube/polymer composites are being explored for a variety of potential applications, including housings for cell phones and computers, lightning-strike protection for airplanes, actuators, chemical sensors, photovoltaic devices (solar cells), electrical interconnects for plastic electronics, printable circuit wiring, transparent conductive coatings, and electromagnetic interference shielding. In fact, the first major commercial product from

Hyperion Catalysis International that used multiwall carbon nanotubes (MWNTs) provides improved electrical conductivity that facilitates electrostatic coating for painting automobile bumpers.<sup>1</sup> A variety of potential applications are also being developed that require higher thermal conductivities, including heat exchangers and heat dissipation materials for electronics packaging.

This article provides a brief synopsis of fundamental research in polymer nanocomposites containing carbon nanotubes focusing on electrical, thermal, and flammability studies. Note that activity by small, medium, and large companies is considerable in this area, but is largely omitted here because of a lack of public information. Interested readers are directed to several recent review articles for more complete presentations.<sup>2-8</sup>

A word of caution before proceeding: one must appreciate that all known preparations of nanotubes give mixtures of nanotube chiralities, diameters, and lengths along with different types and amounts of impurities and structural defects. These variations impact the electrical and thermal properties of the nanotubes, as well as how the nanotubes are arranged in a composite. Furthermore, these nanotube characteristics can vary somewhat from one batch to the next from the same provider, not to mention the significant variations among the numerous synthesis methods used across the world to grow carbon nanotubes. Thus, it is challenging to quantitatively compare results among different groups without accounting for variations in nanotubes. Ultimately, companies will select nanotube suppliers based on cost, consistency, compatibility with their composite fabrication methods, and composite properties.

## Electrical Conductivity of Polymers with Nanotubes

Electrical conductivity in filled polymers is typically discussed in terms of percolation phenomena when the filler has an electrical conductivity vastly higher than the polymer matrix. This is certainly the case with carbon nanotubes that have electrical conductivities ranging from semiconductive to metallic, whereas conventional polymers are insulating. The percolation threshold is the filler concentration at which the electrical conductivity increases sharply by orders of magnitude, indicating that conductive pathways span the macroscopic sample. Below the percolation threshold, electrons must travel through considerable amounts of insulating matrix between the conductive filler particles, whereas above the percolation concentration, electrons conduct predominantly along the

filler and move directly from one filler particle to the next.

Carbon nanotubes provide a new regime of fillers because of their high electrical conductivities, large aspect ratios, and nanometer-scale diameters and have revitalized the investigation of percolation phenomena. Significant increases in electrical conductivity have been reported at nanotube loadings considerably below the concentrations of traditional fillers in polymer matrices. For polymer composites with single-wall carbon nanotubes (SWNTs), the reported percolation thresholds range from 0.007 wt% to several wt%.<sup>9</sup> Percolation thresholds as low as 0.0025 wt% have been achieved in a polymer composite with long flexible bundles of aligned MWNTs.<sup>10</sup>

For composites with discrete fillers that are vastly more conductive than the matrix, the percolation threshold is determined by fitting experimental data to a power law,<sup>11</sup>

$$\sigma \propto (m - m_c)^{\beta_m}, \quad (1)$$

where  $\sigma$  is the electrical conductivity,  $m$  is the nanotube mass fraction,  $\beta_m$  is the critical exponent, and  $m_c$  is the concentration threshold. As an example, Figure 1a shows the electrical conductivity of polystyrene composites with functionalized SWNTs as a function of SWNT loading from 0 wt% to 10 wt%.<sup>12</sup> Nanotube concentrations of >0.3 wt% and >3 wt% SWNT loading are sufficient for applications in electrostatic painting and electromagnetic interference shielding, respectively. Fitting these data with the power law finds the concentration threshold to be 0.045 wt% (Figure 1b). When these authors used polycarbonate as the composite matrix, the concentration threshold increased to 0.110 wt%, demonstrating that electrical percolation might depend on the matrix as well as a variety of other factors, including processing, dispersion, and aspect ratio.

Whereas percolation concentration thresholds are frequently reported below ~1 wt%, there is considerable variability among the numerous nanotube/polymer composites. This observed range in percolation threshold stems primarily from the dispersion of the nanotubes, meaning the spatial distribution of the nanotubes. The van der Waals interactions between carbon nanotubes are notably larger than polymer-polymer van der Waals interactions because of the absence of hydrogen atoms, and have been calculated to be ~0.5 eV per nanometer.<sup>13</sup> This attraction coupled to their size leads to considerable aggregation of nanotubes, which typically reduces their aspect ratio, decreases the number of discrete filler bundles in a composite, and

thereby increases the percolation threshold.<sup>14</sup> Numerous processing methods and chemical modification strategies have been developed to improve dispersion within polymer nanocomposites, where better dispersion generally has reduced the percolation threshold.

In addition to nanotube dispersion, the percolation threshold for the electrical conductivity in nanotube/polymer composites is also influenced by the nanotube aspect ratio. Note that higher-aspect-ratio nanotubes are typically more difficult to disperse, and it can be difficult to separate the two effects. Bryning et al. prepared SWNT/epoxy composites with nanotubes from two different sources (high-pressure carbon monoxide conversion and laser oven) having aspect ratios of ~150 and ~380, respectively, and found a smaller percolation threshold with the higher-aspect-ratio nanotubes.<sup>9</sup> Bai and Allaoui found more than an eightfold decrease in the threshold concentration in MWNT/epoxy composites

when the MWNT length was increased from 1 to 50  $\mu\text{m}$ .<sup>15</sup>

It is widely accepted that chemical functionalization by covalent bonding disrupts the extended  $\pi$  conjugation of nanotubes and reduces the electrical conductivity of isolated nanotubes. However, we note that several researchers report that functionalization can improve the electrical properties of the composites. For example, Valentini et al. pointed out that amine-functionalized SWNTs in epoxy matrix enable migration of intrinsic charges, which contributes to the overall conductivity.<sup>16</sup> Tamburri et al. found that extensive functionalization of SWNTs with -OH and -COOH groups enhances the current in a conducting polymer (1,8-diaminophthalene) by factors of 90 and 140, respectively, whereas the untreated tubes showed an enhancement of only 20.<sup>17</sup> At present, it appears that the disadvantages of covalent chemical functionalization with respect to SWNT conductivity are outweighed by the improved dispersion enabled by functionalization. Similarly, noncovalent chemical functionalization, such as the addition of surfactant molecules, improves dispersion and generally improves electrical conductivity unless the dispersant remains between the nanotubes and increases the interfacial resistance.<sup>18</sup>

An underlying assumption in this discussion is that the composites are isotropic, that is, the nanotubes and/or nanotube bundles are randomly oriented in the sample. Alignment occurs at low concentrations when the polymer matrix flows, as in melt-fiber spinning<sup>19</sup> or shear deformation,<sup>20</sup> because the polymer chains are perturbed from random conformations to extended conformations along the flow direction by shear and extension forces and thereby align the nanotubes. When flow stops, entropic forces quickly drive polymers back to random conformations, whereas, in contrast, long nanotubes and nanotube bundles typically remain in their aligned arrangement. This nanotube alignment can remain indefinitely when the polymer matrix becomes glassy or semicrystalline upon cooling. Scanning electron microscopy readily detects nanotube orientation qualitatively, and quantitative measures of orientation have employed x-ray scattering from either the interlayer diffraction in MWNTs or the SWNT form-factor scattering.<sup>21</sup>

Nanotube alignment typically increases electrical conductivity in dry carbon nanotube mats, called "buckypaper," with high nanotube loadings (>30 vol%).<sup>22</sup> The situation is more complex with nanotube concentrations near the percolation concentration threshold. At low loadings, near-perfect alignment disconnects nanotubes,

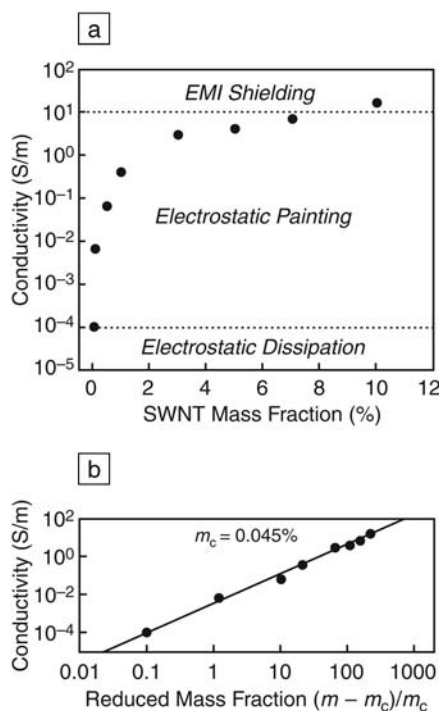


Figure 1. (a) Electrical conductivity of functionalized single-wall carbon nanotubes (SWNTs) in polystyrene composites as a function of SWNT loading. Dotted lines represent the conductivities required for various applications. (b) The power law dependence of the electrical conductivity according to Equation 1 gives a threshold concentration of 0.045 wt%;  $m$  is the nanotube mass fraction and  $m_c$  is the concentration threshold. (Reused with permission from Reference 12.)

thereby destroying the percolating pathways; the electrical conductivity approaches that of the polymer matrix. Du et al. studied the effect of nanotube orientation using a series of SWNT/poly(methyl methacrylate) (PMMA) composites in which the nanotubes were aligned using an extensional flow (Figure 2).<sup>23</sup> The extent of nanotube orientation was controlled by the melt-fiber processing conditions (extrusion rate, wind-up speed). The full width at half maximum (FWHM) refers to the azimuthal dependence of the form-factor scattering arising from the nanotube bundles, where  $0^\circ$  and  $180^\circ$  correspond to perfect alignment of SWNTs and isotropic distribution of SWNTs, respectively. At a fixed SWNT concentration (2 wt%), the electrical conductivity parallel to the alignment direction increases sharply with increasing FWHM (i.e., decreasing alignment). As with concentration, a power law was applied to determine the critical threshold for percolation with respect to alignment (inset in Figure 2). Figure 2 also indicates that there are intermediate levels of nanotube alignment (FWHM  $\sim 100^\circ$ – $140^\circ$ ), where the electrical conductivities are higher than the isotropic condition (FWHM  $180^\circ$ ) because of fewer redundancies in the network; this effect is more pronounced at lower SWNT loading. Given that typical industrial polymer processing methods (injection molding) use considerable shear forces, nanotube alignment can vary across a piece, so strategies will be required to address the accompanying variations in electrical conductivity.

Alternative approaches to high electrical conductivity create heterogeneous distributions of nanotubes in which the nanotube-rich phase is continuous within a macroscopic sample. One example of this approach starts with nanotube buckypaper that is prepared by filtering a nanotube suspension to yield a porous, flexible nanotube material consisting of interconnected nanotube bundles. Buckypaper can be infiltrated with polymers or with polymer precursors that are subsequently polymerized to give composites with  $>20$  vol% nanotubes.<sup>24,25</sup> Epoxy-based nanotube composites of this type are being developed for lightning-strike protection. A second example begins with a polymer latex that consists of an aqueous suspension of polymer particles prepared by emulsion polymerization. Nanotubes are added to the suspension, the solvent is removed, and as the latex particles consolidate into a dense film, the nanotubes are trapped between the particles and form a continuous minority phase that is electrically conductive.<sup>26</sup> This route is particularly well suited for preparing electrically conductive thick films with nanotube loadings of  $\sim 1$  wt% without compromising the processability of the latex.

The electrical conductivities in nanotube/polymer composites are readily understood in terms of a percolating network of highly conductive, high-aspect-ratio fillers with minimal tube-tube resistance. Consequently, research to date finds strong dependencies of the composite electrical

properties on the following nanotube characteristics: spatial distribution, orientation, length, and functionalization. Note that these characteristics are certainly not independent, that is, functionalization typically alters the spatial distribution. As these materials are developed, the correlations among processing, structure, and properties will be refined for specific fabrication methods, materials, and applications.

## Thermal Conductivity of Polymers with Dispersed Nanotubes

The excellent thermal conductivity of individual nanotubes led to early expectations that nanotubes will vastly enhance the thermal conductivity of polymers, as nanotubes do with the electrical conductivity. However, the thermal conductivities of nanotube/polymer composites have increased to only a few W/m K and fail to show a dramatic percolation threshold. There are two important differences between thermal and electrical conductivity in nanotube/polymer composites that provide insight into the relatively poor performance of the nanocomposites as thermal conductors: property contrast between nanotubes and polymers and interfacial thermal resistance. Of course, the thermal conductivity of nanotube/polymer composites also depends on the nanotube characteristics described in the section "Electrical Conductivity of Polymers with Nanotubes," including dispersion, alignment, and aspect ratio, but these are less important here.

First, there is the issue of contrast in thermal versus electrical conductivity. The highest reported nanotube thermal conductivities are on the order of  $10^3$  W/m K, whereas typical thermoplastics and thermosets have  $\sim 10^{-1}$  W/m K. Note that the thermal conductivity of isolated nanotubes is an active area of research, with some reports giving values as low as 30 W/m K and others illustrating the influence of nanotube size and type.<sup>27–28</sup> Thus, a larger fraction of phonons entering a nanotube/polymer composite are likely to travel through the matrix than electrons, because the contrast in thermal conductivity,  $\kappa_{\text{nanotube}}/\kappa_{\text{polymer}}$ , is at most  $\sim 10^4$ , in comparison with the electrical conductivity contrast,  $\sigma_{\text{nanotube}}/\sigma_{\text{polymer}}$ , of  $\sim 10^{14}$ – $10^{19}$ .

Second, atomic vibrations or phonons dominate the thermal conductivity of carbon materials, leading to high interfacial thermal resistance between nanotubes. This interfacial thermal resistance arises from the constraint that the energy contained in high-frequency phonon modes within the nanotubes must first be transferred to low-frequency modes through phonon-phonon coupling in order to be

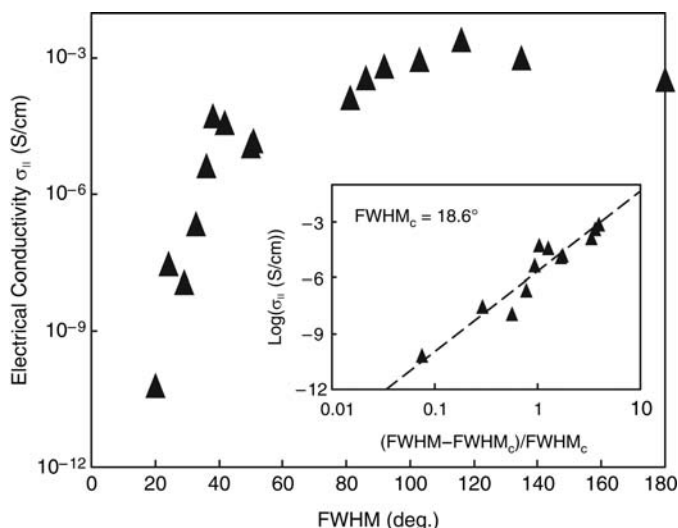


Figure 2. Electrical conductivity of poly(methyl methacrylate) composite fibers with 2 wt% single-wall carbon nanotubes (SWNTs) as a function of nanotube alignment as given by a full width at half maximum (FWHM) from scattering measurements. Electrical conductivity measured along the composite fibers at room temperature. Inset: log-log plot of conductivity versus reduced FWHM determines the critical alignment, FWHM<sub>c</sub>. (Reused with permission from Reference 23.)



exchanged with the surrounding medium. Huxtable et al. has estimated the thermal conductance to be  $\sim 12$  MW/m<sup>2</sup> K at a nanotube-polymer interface.<sup>29</sup> Simulations of heat transfer between two nanotubes without an intervening matrix material also show high thermal resistance.<sup>29</sup> This result is consistent with the low thermal conductivity of SWNT buckypaper ( $\sim 30$  W/m K for the unaligned buckypaper<sup>30</sup>) as compared to that of the individual SWNTs ( $\sim 6600$  W/m K, theoretical prediction<sup>7</sup>). From a comparative study of different nanotubes (SWNTs; MWNTs; and double-wall nanotubes, DWNTs) in epoxy composites, Gojny et al. suggest that MWNTs have the highest potential to improve the thermal conductivity of polymer composites, because of the relatively low interfacial area (therefore, less phonon scattering at the interface) and the existence of shielded internal layers that promote the conduction of phonons and minimize the matrix coupling losses.<sup>31</sup> Efforts are now focused on clever methods to reduce this interfacial thermal resistance by covalently bonding functional groups onto nanotubes<sup>32,33</sup> and by introducing extensive nanotube overlap.<sup>34</sup>

Here, we summarize some promising experimental findings. Biernick et al. prepared an epoxy composite with 1 wt% raw (not purified) laser-oven SWNTs that showed a 125% increase in thermal conductivity at room temperature.<sup>35</sup> Choi et al. reported a 300% increase in thermal conductivity at room temperature with 3 wt% SWNTs in epoxy, with an additional increase (10%) when aligned magnetically.<sup>36</sup> Du et al. developed an infiltration method that produced a bicontinuous morphology of epoxy and a nanotube-rich phase (Figure 3) and showed a  $\sim 220\%$  increase in thermal conductivity at  $\sim 2.3$  wt% SWNT loading.<sup>37</sup> As discussed previously for electrical conductivity, spatially heterogeneous or two-phase materials demonstrate methods that circumvent the need for good dispersion to produce favorable properties. Overall, improving the thermal conductivity of polymers using carbon nanotubes is proving more challenging than improving electrical properties, but progress is steady. Currently, the thermal conductivities of nanotube/epoxy composites fall far below commercial silver flake/epoxy composites (50–65 wt% silver content and several to 30 W/m K) that are widely used in packaging microprocessors.

## Flammability in Polymers with Dispersed Nanotubes

The flammability of polymeric materials can be significantly reduced by the addition of a small quantity of carbon nanotubes. During flammability experiments,

nanotube/polymer composites with well-dispersed nanotubes form a freestanding nanotube network that remains robust after burning, thus serving as a protective layer and providing good flame-retardant effectiveness.<sup>38,39</sup> The nanotube concentration and dispersion are critical for producing this protective nanotube network. The size of the carbon filler (SWNTs; MWNTs; and carbon nanofiber, CNF) also has an impact on the physical structure of the protective layer and the flame-retardant effectiveness, as detailed later.<sup>40</sup> CNF is graphite fiber with small diameters of 60–200 nm and lengths in the range of tens to hundreds of microns.

The mass loss rate curves of samples having a mass fraction of 0.5 wt% of SWNTs, MWNTs, or CNF under an external radiant flux of 50 kW/m<sup>2</sup> in nitrogen (no flaming but sample heating similar to fire conditions) are shown in Figure 4. The liquid-like PMMA bubbled violently, and no residue remained in the container at the end of the test. The 0.5% SWNT/PMMA nanocomposite was solid-like throughout the test and effectively suppressed bubbling. The residue is a continuous nanotube network (see Figure 4) and effectively

reduced the mass loss rate of the PMMA by roughly 70%. The 0.5% MWNT/PMMA composite formed numerous discrete structures that agglomerated as the test progressed, leading to the formation of a discontinuous residue. The mass loss rate of the 0.5% MWNT/PMMA was significantly lower than that of the PMMA but not as low as that of the 0.5% SWNT/PMMA. The 0.5% CNF/PMMA composite is a slightly viscous liquid that bubbled vigorously during the test and left only a thin, fragile residue. The mass loss rate of the 0.5% CNF/PMMA was not significantly different from that of the PMMA. These results indicate that the order of flame-retardant effectiveness is SWNTs, MWNTs, and CNF at 0.5 wt% loading.

The development of a mechanically and thermally robust filler network provides critical protection during burning and improves the flammability properties of the polymer nanocomposites. Such a structure appears to form when a filler network exists in the initial sample. The presence of a network can be detected by viscoelastic measurements on the samples as a function of filler type and concentration. The frequency dependence of the shear-storage modulus ( $G'$ ) indicates whether a composite behaves like a liquid ( $G' \sim \omega^2$ ) or like a solid ( $G' \sim \omega^0$ ), where  $\omega$  is the frequency of the dynamic experiment. As the filler concentration increases, a transition from a Newtonian liquid to an ideal Hookean solid accompanies the formation of a filler network structure in the composite (Figure 5). For larger fillers (e.g., CNF), a higher loading is necessary to exhibit solid-like behavior in the composite. Solid-like behavior in the composite at 200°C corresponds to an ability to form a protective layer during burning. This strong correlation between viscoelastic properties and flame-retardant effectiveness of polymer nanocomposites provides a convenient method for screening materials. For example, the 4% CNF/PMMA composite is solid-like at 200°C and has a mass loss rate of the PMMA similar to the 0.5% SWNT/PMMA (see Figure 4), because the higher concentration enabled the formation of a CNF network.

## Remaining Challenges and Outlook

The high aspect ratios and small diameters of carbon nanotubes permit network structures to form within polymer matrices at remarkably low concentrations. The specific threshold concentration depends on the size of the individual nanotubes (length and diameter); the extent of nanotube dispersion into isolated nanotubes, bundles, ropes, or fiber; the spatial distribution of these objects; and their local

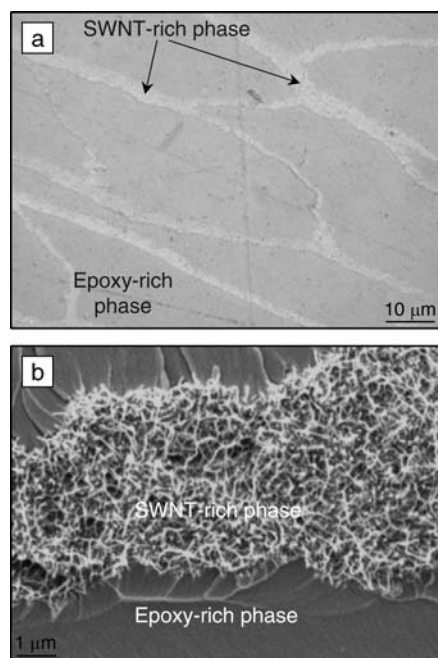


Figure 3. (a) Optical and (b) scanning electron microscopy images of a SWNT/epoxy composite fabricated by infiltrating a freestanding nanotube network. This composite exhibits a heterogeneous morphology in which the nanotube-rich phase is both the minority phase and continuous across the sample. (Modified from Reference 37.)

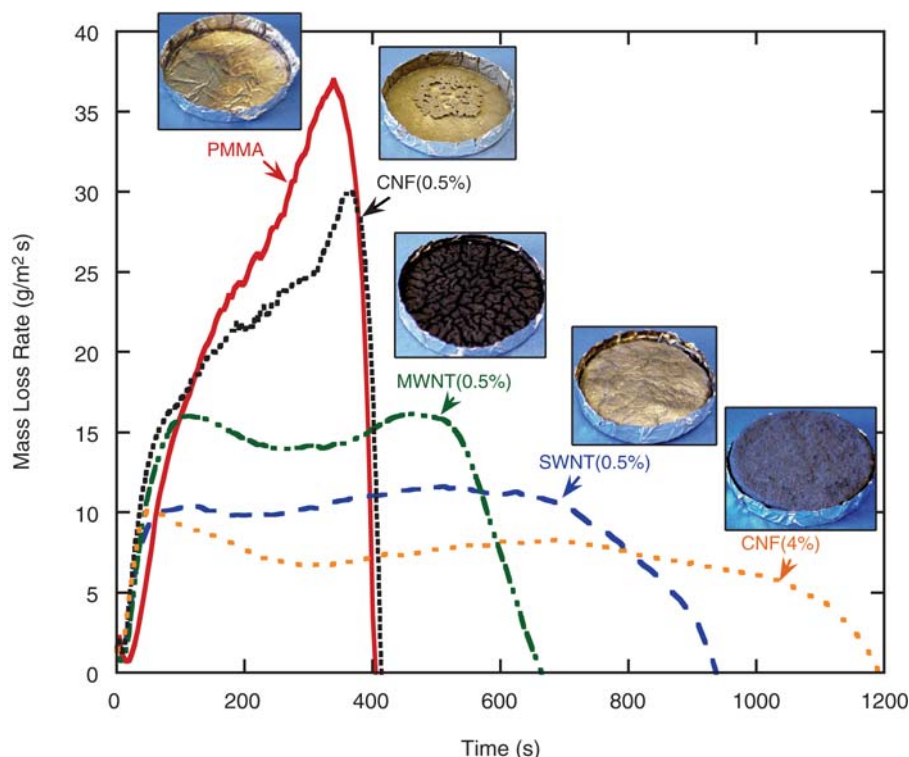


Figure 4. Effects of filler type on mass loss rate of poly(methyl methacrylate) and (insets) residue photographs collected at the end of the flammability tests. The tests were conducted at an external radiant flux of 50 kW/m<sup>2</sup> in nitrogen. Abbreviations: PMMA is poly(methyl methacrylate), CNF is carbon nanofiber, MWNT is multiwall carbon nanotube, and SWNT is single-wall carbon nanotube. (Modified from Reference 40.)

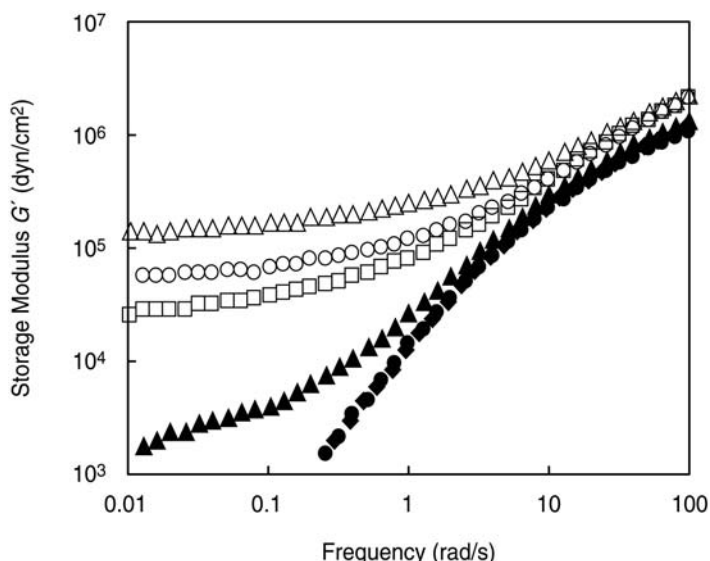


Figure 5. Rheological response transitions from liquid-like behavior ( $G' \sim \omega^2$ ) to solid-like behavior ( $G' \sim \omega^0$ ) as a function of increasing SWNT loading in poly(methyl methacrylate): 0 wt% (solid diamonds), 0.1 wt% (solid circles), 0.2 wt% (solid triangles), 0.5 wt% (open squares), 1.0 wt% (open circles), and 2.0 wt% (open triangles). (Modified from Reference 14.)

and global orientation. Above a critical concentration, a nanotube network can (1) sufficiently constrain the motion of thermoplastics to produce solid-like behavior; (2) form a protective layer that significantly improves flame-retardant efficiency; and (3) provide pathways for electrical conduction, leading to dramatic increases in electrical conductivity.

Carbon nanotubes have unequivocally demonstrated their ability to convert an insulating polymer matrix into a conductive nanocomposite. Electrical conductivity is likely to continue to increase as sizable quantities of metallic (or at least, predominantly metallic) nanotubes become available either by controlling the nanotube synthesis or by removing the semiconductive nanotubes. It remains to be seen whether well-dispersed nanotubes or spatially heterogeneous nanotube distributions will be favored for specific applications, given the complex interplay of cost, processability, and properties. For applications requiring the processability of thermoplastic or thermoset composites with improved electrical conductivity, the outlook is excellent both in terms of performance and the number of potential applications. The improvements in flammability properties also indicate a positive trajectory for nanotube/polymer composites. In contrast, thermal conductivity in nanotube/polymer composites requires a substantial scientific breakthrough before its future is assured.

Emerging areas of opportunity in nanotube/polymer composites include combining nanotubes with conventional fillers or other nanoscale fillers. The motivations for combining fillers include the goals of improving multiple properties simultaneously, incorporating nanotubes into existing commercial processing methods, and making the price point of the composite attractive to new applications. The principles involved will remain the same, but the level of complexity increases dramatically with two or more fillers. Another emerging area will be the incorporation of nanotube fibers into polymer matrixes. Nanotube fibers have been wet-spun from acid suspensions of SWNTs,<sup>41</sup> spun from chemical vapor deposition-grown nanotubes,<sup>42</sup> and twisted from densely grown arrays of MWNTs,<sup>43</sup> and will provide a new class of carbon-based fibers to the field of continuous fiber composites.

Looking beyond carbon nanotubes, electrical and thermal conductivities and flammability properties of polymer nanocomposites may benefit from other nanoscale fillers, particularly nanowires and nanorods. Nanowires and nanorods are high-aspect-ratio particles ( $l/d > 100$ )

prepared by using catalytic nanoparticles in a chemical vapor deposition or solution method and by electroplating material into porous templates. Metals, semiconductors, and ceramics have been made as nanowires and can be either single-crystal or polycrystalline. The dispersion, fabrication, characterization, and testing methods developed for nanotube/polymer composites will readily be adapted for nanowire- or nanorod-based polymer nanocomposites.

In summary, we have highlighted research results that elucidate fundamental structure-property relationships pertaining to electrical, thermal, and/or flammability properties in numerous nanotube-containing polymer composites. MWNTs are already established commercially as fillers that improve electrical conductivity in polymers. As research continues, specific potential applications will define materials properties and materials scientists will endeavor to design nanotube-polymer composites that meet both the targeted materials and cost criteria. In the coming decade, the number of commercial nanotube-polymer composites with improved electrical, thermal, or flammability properties will certainly increase.

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