Carbon nanotube polymer composites

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Abstract

The state of research into carbon nanotube/polymer–matrix composites for mechanical reinforcement is critically reviewed with emphasis on recent advances in CNT composite toughness. Particular interest is also given to interfacial bonding of carbon nanotubes to polymer matrices as it applies to stress transfer from the matrix to the CNT. Potential topics of oncoming focus are highlighted.

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1. Introduction

Since the documented discovery of carbon nanotubes (CNTs) in 1991 by Iijima [1] and the realization of their unique physical properties, including mechanical, thermal, and electrical, many investigators have endeavored to fabricate advanced CNT composite materials that exhibit one or more of these properties [2,*3,*4]. For example, as conductive filler in polymers, CNTs are quite effective compared to traditional carbon black microparticles, primarily due to their large aspect ratios [5]. The electrical percolation threshold was recently reported at 0.0025 wt.% CNTs and conductivity at 2 S/m at 1.0 wt.% CNTs in epoxy matrices [6]. Similarly, CNTs possess one of the highest thermal conductivities known [*7], which suggests their use in composites for thermal management [2]. The main focus of this paper, however, will be on the use of CNTs as discontinuous reinforcement for polymer matrices. The CNT can be thought of as the ultimate carbon fiber with break strengths reported as high as 200 GPa, and elastic moduli in the 1 TPa range [8,**9]. This, coupled with approximately 500 times more surface area per gram (based on equivalent volume fraction of typical carbon fiber) and aspect ratios of around $10^3$, has spurred a great deal of interest in using CNTs as a reinforcing phase for polymer matrices.

However, after nearly a decade of research, their potential as reinforcement for polymers has not been fully realized; the mechanical properties of derived composites have fallen short of predicted values. Yet given the magnitude of the CNT’s mechanical properties, significant improvement on current composites should be possible provided means to harness the CNT’s unique attributes exhibited at the nanoscale can be transferred to the macroscale. This essentially defines the fundamental challenge for applied CNT/polymer composites research. How does one effectively manipulate nanoscale building blocks to assemble useful macroscale materials? Some have suggested a bottom-up approach possibly utilizing non-continuum effects at the CNT-polymer interface [*10,*11,*12], while macroscale composite synthesis typically utilizes more traditional continuum ideas. A better understanding of the relationships between processing, interfacial optimization, and composite properties is a major goal of this area of research, which may lead to optimal reinforcement of polymer matrices with CNTs.

The microscale analogue to the current nanocomposite material is a carbon fiber/epoxy laminate: essentially the same materials in dimensions 1000 times larger. A 20 nm thick polymer/aligned CNT ‘nanolamina’ may in fact be desirable, but currently extremely difficult to scale-up or make reproducibly. Lacking direct manipulation, when used as reinforcement in polymers, CNTs are typically first randomly dispersed in a solvent or polymer fluid/melt by sonication or shear mixing followed by further processing to create the composite. It should be noted that the energy input to
disperse the CNTs tends to break them into shorter segments [2,13] decreasing their aspect ratio in the final composite while simultaneously increasing their dispersibility. However, insufficient dispersion is often cited as a process limitation [14] and the key diminishing factor [11,13,15] on the composite’s mechanical properties. Efforts to improve CNT dispersion include: the use of surfactants [16], and the oxidation or chemical functionalization of the CNT surface [17,18].

2. CNT functionalization

Recently, functionalization has been achieved via exposure of vapor grown Pyrograf™ III carbon nanofibers (Applied Sciences Inc.) to a CO2/Ar plasma optimized with respect to time, pressure, power, and gas concentration resulting in 14.5 at.% oxygen in the first 10 atomic layers [19]. The presence of functionalities such as hydroxyl, carbonyl, and carboxyl groups were detected by XPS. In this case, surface defects in the curved graphene planes of the nanofibers may have increased reactivity promoting the formation of the functionalities. Nitric acid treatment has also been reported to successfully oxidize the surface of multiwall carbon nanotubes (MWNTs) as detected using diffuse reflection infrared Fourier-transform (DRIFT) spectroscopy [18]. Functionalization of the CNT surface cannot only lead to increased dispersibility of the CNTs in various organic solvents and polymers [4,20], but also to increase the strength of the interface between the CNT and the polymer matrix [20]. However, chemical functionalization may disrupt the bonding of the graphene sheet, and thereby reduce the mechanical properties of the functionalized CNT in the final composite.

3. CNT/polymer interfaces

Increasing attention is being focused on the CNT surface, namely the interface between the CNT and surrounding polymer matrix. From micro-mechanics, it is through shear stress build-up at this interface that stress is transferred from the matrix to the CNTs. Numerous researchers have attributed lower-than-predicted CNT-polymer composite properties to a lack of interfacial bonding [4,15,21]. If one considers the surface of a CNT, essentially an exposed graphene sheet, it is not surprising that interfacial traction is a concern. It is the weak inter-planar interaction of graphite that provides its solid lubricant quality, and resistance to matrix adhesion. This is exaggerated by the chemically inert nature of graphene structures. A June 2002 publication on CNT composites noted ‘In depth study on the stress transfer mechanism of the nanotube composites with different chemical and geometrical properties, matrix environments and loading conditions are essential ...’ [8]. Since then, some interesting published results have described progress on addressing this issue. The pull-out force necessary to remove a given length of an individual MWNT embedded in polyethylene–butene using an AFM was measured [22]. The calculated average interfacial shear strength using a Kelly-Tyson approach of 47 MPa (typical values for carbon fiber/epoxy are in the 30–80 MPa range depending on fiber modulus and surface treatment [23]) was sufficiently high to suggest that covalent bonding between defects in the outer shell of the MWNT and the polymer was occurring. It also suggested that the polymer chains close to the interface behaved differently than the bulk, a logical result when considering the CNT outer diameter is of similar magnitude to the radius of gyration for the polymer. In a report corroborating an interfacial region of non-bulk polymer, a ‘sheathing layer’ of polycarbonate on pulled out MWNTs was imaged, which gave further evidence of significant interfacial interaction between MWNTs and a polymer [44]. It was also found that chemical functionalization of the MWNTs augmented the diameter of the polymer sheath suggesting chemical augmentation of interfacial bonding. Acid oxidation of MWNTs was again reported to attach carboxylic groups on the surface, which were then reacted with epoxide-terminated molecules up to 12 wt.% by TGA [20]. These types of functionalized tubes could

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**Abbreviations**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>CNT</td>
<td>carbon nanotube</td>
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<tr>
<td>MWNT</td>
<td>multiwall carbon nanotube</td>
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<tr>
<td>SWNT</td>
<td>singlewall carbon nanotube</td>
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<tr>
<td>CNF</td>
<td>carbon nanofiber</td>
</tr>
<tr>
<td>ISS</td>
<td>interfacial shear strength</td>
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<tr>
<td>TEM</td>
<td>transmission electron microscope</td>
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<tr>
<td>SEM</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>AFM</td>
<td>atomic force microscope</td>
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<td>XPS</td>
<td>X-ray photoelectron spectroscopy</td>
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<td>TGA</td>
<td>thermal gravimetric analysis</td>
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<tr>
<td>PE–B</td>
<td>polyethylene–butene</td>
</tr>
<tr>
<td>PAN</td>
<td>polyacrylonitrile</td>
</tr>
<tr>
<td>PS</td>
<td>polystyrene</td>
</tr>
<tr>
<td>PMMA</td>
<td>polymethylmethacrylate</td>
</tr>
<tr>
<td>PVA</td>
<td>polyvinylalcohol</td>
</tr>
<tr>
<td>UHMWPE</td>
<td>ultra-high molecular weight polyethylene</td>
</tr>
<tr>
<td>PMEMA</td>
<td>methyl–ethyl methacrylate copolymer</td>
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</tr>
</tbody>
</table>

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enhance reinforcement of epoxy resins. Carboxylated tubes have also been reported to augment the cure rate of epoxy resins at lower temperatures [18]. Unfortunately, direct and indirect measurement of polymer–CNT interfacial shear strength suggesting good bonding exists conflict with other reports which often cite clean pull-out of CNTs and poor interfacial bonding [*4,15,*24]. The magnitude of CNT strength (>10 times that of typical carbon fiber) may preclude embedded CNT tensile failure in large numbers resulting in the dominant failure mode to be CNT pull-out. Order of magnitude increases in interfacial shear strength may be required for the most efficient strengthening of polymers with CNTs. Optimizing the polymer–CNT interface for nanoscale mechanical reinforcement remains unclear, but the evidence available indicates that chemical means can be effective, and this is likely to be a major focus in the near term.

Theoretical treatments of CNT pull-out were recently reported [*24,25]. Including one in which a (10, 10) single-wall carbon nanotube (SWNT) pulled out from a polyethylene matrix was modeled via molecular dynamics simulations, and quite a low effective viscosity of 0.2 cP was found for interfacial sliding [*24]. A force of approximately 0.1 nN was required for pull-out to initiate for a single tube.

4. Embedded CNT strain characterization by micro-Raman spectroscopy

Earlier publications have associated Raman peak shifting up (to higher wavenumbers) or down (to lower wavenumbers) from the peak near 2700 cm\(^{-1}\) with compressive and tensile strain in the CNT respectively [26,27]. A recent report monitored tensile load transfer to MWNTs in a UHMWPE matrix by the Raman peak shifting at 2691 cm\(^{-1}\) [**28], and found four regions of strain behavior outlined in Table 1. Another report [*29] related the Raman peak shift at 1594 cm\(^{-1}\) to the axial strain in SWNTs embedded in epoxy by:

\[
\Delta \omega(1594 \text{ cm}^{-1}) / \omega_0 = -\gamma(1 - \nu_e)\varepsilon_z
\]

where the Gruneisen parameter \(\gamma = 1.24\) and the Poisson ratio \(\nu_e = 0.28\).

5. CNT reinforcement of polymers

Significant toughening of polymer matrices through the incorporation of CNTs has been reported [*4,**16, **28]. A loading of 1 wt.% MWNTs, randomly distributed in an ultra-high molecular weight polyethylene film, was reported to increase the strain energy density by 150% and increase the ductility by 140%. Secondary crystallities, which nucleated from the MWNTs, were attributed a higher mobility and hence the increase in strain energy [**28]. A similar effect was found in aligned MWNT/polyacrylonitrile fibers containing 1.8 vol.% MWNTs with an approximately 80% increase in energy to yield and energy to break [*4]. A process of spinning 60 wt.% SWNT/polyvinylalcohol fibers with pre-drawn energy absorbing capacity nearly 3.5 times spider silk (165 J/g) was reported [**16]. Slippage between SWNT bundles was suggested as the mechanism responsible for the enhancement in the toughness. The addition of 1 wt.% MWNTs to isotactic polypropylene (iPP) was shown to affect crystal nucleation from differential scanning calorimetry and X-ray diffraction measurements [*30]. Compared with neat iPP, there was an increase in crystallization rate for the composite material with evidence of fibrillar crystal growth rather than spherulite growth (Fig. 1). These modifications in the morphology of a polymer matrix combined with the energy required for CNT debonding and pull-out suggest CNTs may augment the energy absorption or toughness characteristics of the composite.

A twofold increase in the tension–tension fatigue strength for an aligned SWNT/epoxy composite was found in comparison to typical carbon fiber/epoxy composites (Fig. 2) [**31]. Embedded CNTs may effectively prolong the formation of and/or bridge micro-cracking/crazing that can propagate and lead to fatigue failure. CNT reinforced polymer composites are seen as a potentially fruitful area for new, tougher or fatigue-resistant materials. Further investigations into the toughness and fatigue properties of these composites are

<table>
<thead>
<tr>
<th>Region</th>
<th>% (\varepsilon)</th>
<th>Shift</th>
<th>Interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0–1</td>
<td>Clear shift down</td>
<td>Tensile loading of MWNTs Elastic response</td>
</tr>
<tr>
<td>2</td>
<td>1–10</td>
<td>Much less apparent or intense shift down</td>
<td>Interfacial ‘stick and slip’ yielding matrix</td>
</tr>
<tr>
<td>3</td>
<td>10–15</td>
<td>Somewhat more apparent shift down</td>
<td>Tensile loading of MWNTs; MWNT ‘knots’ preventing further PE chain stretching</td>
</tr>
<tr>
<td>4</td>
<td>&gt;15</td>
<td>A shift up</td>
<td>Compressive loading of MWNTs Elastic recovery from local matrix failure</td>
</tr>
</tbody>
</table>

Four regions of behavior were found for D* Raman band shifting as a function of applied tensile strain to an ultra-high molecular weight polyethylene/MWNT composite film. Data and interpretation from [**28].
Tensile strength and modulus enhancements are continually reported [32–34]; very little of the data achieve reinforcement predicted by a rule-of-mixtures approach especially at loadings beyond 10 vol.% [4,17,35] (For aligned, discontinuous fiber reinforcement, the Halpin–Tsai model [36] is often used, which approaches the rule of mixtures for large $E_f/E_m$ or $l/d$). For $E_{NT}/E_m = 100$ and $l/d = 500$, the Halpin–Tsai approach is 95% that of the rule of mixtures for aligned composites. An effective $l/d$ of the CNT, deduced from these sort of comparisons, can shed light on stress transfer to the CNTs [37]. Some have offered new explanations and factors contributing to understanding this discrepancy between experimental and predicted results. In a two paper series using finite element analysis and micro-mechanical methods, it was proposed that observed curvature of embedded CNTs or ‘waviness’ significantly reduced their reinforcement capabilities (by factors from 50 to 200) compared to straight CNTs [12,35]. It was also noted that other indistinguishable factors contribute to the low values measured in experimental data: including weak interfacial bonding, insufficient dispersion, and degradation of the CNTs due to processing [35]. At very low strains, it was suggested that the effect of poor interfacial shear strength should not affect the composite’s modulus, implying a measurable elastic response below the strain detrimental to the CNT interface [10]. It was also suggested that the diameter distribution of embedded MWNTs would have

needed to understand the reinforcing mechanism at work.

Fig. 1. SEM images crystalline morphology of isotactic polypropylene. The difference in crystalline morphology for (a) neat isotactic polypropylene (iPP), and (b) iPP/1 wt.% MWNT composite is shown in these SEM images. Fibriilar morphology is observed for the composite while the neat iPP has a spherulite morphology [*30, p. 525, 526]. Nucleation Ability of Multiwall Carbon Nanotubes in Polypropylene Composites, Assouline E, Lustiger A, Barber AH, Cooper CA, Klein E, Wachtel E, Wagner HD, Copyright (2003) Wiley Periodicals Inc., Reprinted by permission of John Wiley and Sons Inc.

Fig. 2. SWNT fracture surface bridging. SWNT ropes are observed bridging a fatigue fracture surface in an epoxy matrix. Twofold increases in tension–tension fatigue strength over typical carbon fiber/epoxy composites were observed [*31, p. 2178]. Reprinted form Carbon 41, Ren Y, Feng L, Cheng HM, Liao K, Tension–Tension Fatigue Behavior of Unidirectional Single-Walled Carbon Nanotube Reinforced Epoxy Composite, pg 2177–79, Copyright (2003) with permission from Elsevier.

Fig. 3. Effect of MWNT outer diameter on aligned MWNT composite tensile modulus. The tensile modulus of a MWNT composite is modeled as a function of embedded MWNT outer diameters for various MWNT lengths and volume fractions. The strong dependency on MWNT outer diameter results from using an effective MWNT modulus that accounts for only the outer shell carrying any of the tensile load [*10, p. 581]. Thostenson ET, Chow TW, On the Elastic Properties of Carbon Nanotube-Based Composites: Modelling and Characterization, Copyright (2003) IOP publishing limited, Reprinted with permission.
a very strong effect on the composite’s modulus (Fig. 3) [*10]. In the case of MWNTs, an effective modulus was deduced assuming that the outer shell carries essentially all the load [26] such that its modulus, \( E_{\text{NT}} \), was reduced in proportion to the ratio of the area of the annular outer shell thickness to the total cross-sectional area. A MWNT diameter distribution was therefore essential for accurate prediction of the composite’s modulus [*10].

In another study, increased inter-tube friction of SWNT bundles by the introduction of twist, which served to ‘flatten’ the SWNTs, was predicted by molecular mechanics modeling to have increased the bundle’s load carrying capacity [38]. Some recent mechanical property results for CNT/polymer composites are summarized in Table 2.

6. A look forward

The ultimate goal remains macroscale CNT/polymer composites that are optimally reinforced. Increases in composite moduli are almost always observed even for very low loadings of CNTs. It should be feasible to increase the modulus of a polymer/CNT composite up to and beyond that achievable with high modulus graphite fiber. Alignment of CNTs has been achieved in polymeric matrices most often by means of shear or elongational flow [*4,14,*,16,17,**28], and recently by a magnetic field [40] resulting in film and fiber geometries. However, strengthening of polymer/CNT composites is less commonly reported. Due to the small number of defects per unit length, the most impressive mechanical property of the CNT is its tensile strength with recent experimentally measured values up to 150 GPa for MWNTs **9]. These high strengths imply break strains of approximately 10% with even higher break strains reported in the literature [8,41,42]. Recent reports indicate that tensile failure of MWNTs that carry all of the load within the outer shell is initiated by the formation of a Stone–Wales defect in which 2 C–C bonds are broken and 2 new C–C bonds are formed such that a pair of pentagons and a pair of heptagons results (Fig. 4) [41,42]. Increasing numbers of these dislocations leads to necking and ultimately, failure of the CNT [42]. Often, however, in a polymer/CNT composite a reduction in strength is observed suggesting that the CNTs

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**Table 2**

Selected mechanical properties of CNT/polymer composites

<table>
<thead>
<tr>
<th>CNT</th>
<th>Matrix</th>
<th>Conc. (%)</th>
<th>Loading</th>
<th>Modulus</th>
<th>Yield stress</th>
<th>Strength</th>
<th>Toughness</th>
<th>Max. strain</th>
<th>ISS</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>MWNT</td>
<td>None</td>
<td>100</td>
<td>Tensile</td>
<td>910 GPa</td>
<td>150 GPa</td>
<td>11–200 GPa</td>
<td>12%</td>
<td>~100%</td>
<td>12%</td>
<td>[**9]</td>
</tr>
<tr>
<td>MWNT</td>
<td>None</td>
<td>100</td>
<td>Tensile</td>
<td>270–950 GPa</td>
<td>+46%</td>
<td>+31%</td>
<td>+80%</td>
<td>1.8 GPa</td>
<td>570 J/g</td>
<td>[**4]</td>
</tr>
<tr>
<td>MWNT</td>
<td>PAN</td>
<td>1.8 vol.%</td>
<td>Tensile</td>
<td>80 GPa</td>
<td>(aligned) +10%</td>
<td>(random)</td>
<td></td>
<td></td>
<td></td>
<td>[**16]</td>
</tr>
<tr>
<td>SWNT</td>
<td>PVA</td>
<td>60 wt.%</td>
<td>Tensile</td>
<td>80 GPa</td>
<td>+49%</td>
<td>+150%</td>
<td>+60 to +140%</td>
<td></td>
<td></td>
<td>[**28]</td>
</tr>
<tr>
<td>MWNT</td>
<td>PS</td>
<td>5 wt.%</td>
<td>DMA (25 °C)</td>
<td>+49%</td>
<td>+25%</td>
<td>+10%</td>
<td>47 MPa</td>
<td></td>
<td></td>
<td>[**22]</td>
</tr>
<tr>
<td>MWNT</td>
<td>PE–B</td>
<td>1 wt.%</td>
<td>Tensile</td>
<td>+25%</td>
<td>+48%</td>
<td>+25%</td>
<td>+150%</td>
<td>+60 to +140%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SWNT</td>
<td>PAN</td>
<td>4 wt.%</td>
<td>AFM</td>
<td>~+100%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>[14]</td>
</tr>
<tr>
<td>CNF</td>
<td>PMMA</td>
<td>5 wt.%</td>
<td>Tensile</td>
<td>+50%</td>
<td>+200%</td>
<td></td>
<td>−37.5%</td>
<td></td>
<td></td>
<td>[34]</td>
</tr>
<tr>
<td>MWNT</td>
<td>PMEMA</td>
<td>1 wt.%</td>
<td>DMA</td>
<td>+200%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>[33]</td>
</tr>
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</table>

(*+/−) indicates an enhancement/diminishment from the neat matrix. Recently reported mechanical property enhancements for various CNT/polymer composites are presented with additional information on the method used to collect the data. Experimentally measured values of MWNT mechanical properties are also presented.

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may promote crystalline defects in the matrix or, considering their size, act as defects themselves. This suggests a critical volume fraction of CNTs is required for hindering matrix strain allowing strengthening to occur as typically observed in composites. Classically this results from the composite’s failure criterion being fiber rupture at the fiber break strain, which is typically smaller than the matrix break strain. It remains to be seen whether or not a 10% elastic tensile strain can be transferred to CNTs embedded in a polymer, fully exploiting their load carrying capacity, and at this strain, most polymeric matrices will have begun to flow plastically. Furthermore, many authors report higher loadings of CNTs in a composite do not perform as well as lower loadings [*4,34] suggesting an increase in voids or other defects as the loading of CNTs increases, likely due to the difficulty of homogeneously dispersing concentrated CNT/polymer melts or solutions. All this begs the question as to what is the failure criterion applicable from the composite’s failure criterion being fiber rupture at the fiber break strain, which is typically smaller than the matrix break strain. A process for making MWNT/PAN fibers is presented with data pointing to an 80% increase in composite fiber toughness.

Acknowledgement

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The papers of particular interest have been highlighted as:
* of special interest;
** of outstanding interest.

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trated (60 wt.%) SWNT/PVA fibers that display toughness values that are incredibly large in magnitude.


[24] Frankland SJV, Harik VM. Analysis of carbon nanotube pull-out from a polymer matrix. Surf Sci 2003;525:L103–8. The pull-out of a SWNT from PE was simulated using molecular dynamics and an interfacial viscosity for pull-out to occur was found to be 0.2 cP.


[28] Ruan SL, Gao P, Yang XG, Yu TX. Toughening high performance ultra-high molecular weight polyethylene using multiwalled carbon nanotubes. Polymer 2003;44(19):5643–54. This work reports 150% increases in strain energy density for 1 wt.% MWNTs in UHMWPE. Secondary crystals nucleating from the MWNTs are attributed higher mobility under stress. Raman spectroscopy is used to characterize both the embedded MWNTs and the matrix indicating 4 regions of strain that occur.

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