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The reduction of carbon nanotube (CNT) length during the manufacture of CNT/polymer composites and a method to simultaneously determine the resulting CNT and interfacial strengths

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ABSTRACT

Carbon nanotube/epoxy composites with an excellent dispersion of carbon nanotubes (CNTs) were prepared using a three-roll calendering technique. CNT length after processing of composites is measured and then characterized using a two-parameter Weibull distribution function. Significant reduction of the CNT length is observed as a result of the processing and it is thus suggested that great attention should be paid to the retention of CNT length after processing in order to obtain good mechanical properties. Because of the difficulties in manipulating nanometer sized CNTs during measurement of CNT strength and CNT–polymer interfacial strength, CNT strength and CNT–polymer interfacial strength have previously been determined using complicated methods with expensive or specially designed equipments. In this work a simple methodology based on the modified rule of mixtures is proposed to simultaneously determine the CNT strength and CNT–polymer interfacial strength.

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1. Introduction

Carbon nanotubes (CNTs) are long cylinders of covalently bonded carbon atoms with a diameter ranging from a few angstroms to several tens of nanometers across. There are two basic types of CNTs, namely single-walled CNTs (SWCNTs) and multi-walled CNTs (MWCNTs). CNTs are among the stiffest [1,2] and strongest [2–4] materials known to mankind. Moreover, CNTs possess high flexibility, low mass density, and large aspect ratio (typically ca. 300–1000). For these reasons, they have been suggested as ideal reinforcements for the mechanical reinforcement of various polymers for making composites. It was thought that smaller grains would result in harder metals. It turns out that they are harder but also more brittle [5]. Intuitively there is reason

to suspect that CNTs might be brittle since they are very stiff. It is well known that CNTs exhibit an enormous surface area which is several orders of magnitude larger than the surface of conventional carbon fibers. This large surface area leads to a strong tendency of CNTs to form agglomerates. Any efficient exfoliation of CNT agglomerates would rely on high external forces during processing of CNT/polymer composites. If CNTs were brittle, CNTs would be severely damaged by external forces during processing. This supposed brittle characteristic of CNTs has not been reported yet and will be verified herein by observation of the significant reduction of CNT length after processing.

Materials scientists predict that composites containing CNTs will have exceptional mechanical properties [6]. However, the results obtained so far for the strength and modulus

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of polymer composites are disappointing, particularly when compared to advanced composites reinforced with high performance continuous fibers [6]. It was regarded that inadequate dispersion, misalignment, low content and poor interfacial bonding were responsible for the unexpected low mechanical properties of composites [6]. Nevertheless, the expected enhancement in the mechanical properties of the composites has still not entirely been realized though many studies have been conducted to achieve excellent CNT dispersion [7–9], good CNT alignment [10,11], high CNT content [12,13], and excellent CNT–polymer interfacial bonding [14–17]. Therefore, there must be other unclear reasons for the unexpected low mechanical properties.

CNTs are considered short fibers, and polymer composites with nanotube fillers are always analogues of short fiber composites [18]. The theories for the strength and modulus of short fiber reinforced polymer (SFRP) composites can thus be extended to the case of CNT reinforced polymer composites [19–23]. According to the theories for the strength and modulus of SFRP composites [24–28], residual short fiber length within polymer matrix after processing is one of the key factors determining the mechanical properties of the SFRP composites. As mentioned above, CNTs might be brittle and thus might be damaged during processing due to exertion of high external forces on CNTs. Previous studies on the mechanical damage of carbon nanotubes by ultrasound [29] and the effect of polymer and solvent on purification and cutting of carbon nanotubes [30] indicate that carbon nanotube length might be shortened by external forces. However, the reduction of carbon nanotubes during the manufacture of CNT/polymer composites has not been reported so far. If a significant reduction of CNT length during processing indeed occurs and is considered, the observation of the unexpected low enhancement in the strength and modulus can then be reasonably understood in terms of the theories for the strength and modulus of SFRP composites [24,25,28]. Retention of residual CNT length is thus very important to obtain high mechanical performance composites.

Moreover, determination of CNT strength and CNT–polymer interfacial strength is of great importance since they are two important factors in determining the mechanical properties of polymer composites. Due to the difficulties in manipulating nanometer sized CNTs, they have been determined previously via complicated methods using expensive equipments or specially designed devices. The measurement of CNT strength was carried out by Yu et al. [3] when they managed to do stress–strain measurements on individual arc-MWCNTs inside an electron microscope. The measured strength was in the range 11–63 GPa. Demczyk et al. [31] conducted pulling and bending tests on individual arc-MWCNTs in situ in a transition electron microscope. Based on their observation of the force required to break the tubes, a tensile strength of 0.15 TPa was computed. The first measurement on SWCNTs was carried out by Salvétat et al. [32] using the atomic force microscope (AFM) method. They observed a tensile strength of about 1 TPa for small diameter SWCNT bundles by bending methods. Xie et al. made stress–strain measurements on bundles of chemical vapor deposition (CVD)-MWCNTs with a specially designed stress–strain puller [33] and they measured a tensile strength of about 4 GPa. On the

other hand, CNT–polymer interfacial strength has also been measured independently. Wagner et al. [14] examined the fragmentation of MWCNTs in the polymer films and concluded that the interfacial bonding stress between the nanotubes and polymer matrix could be as high as 500 MPa. Cooper et al. [15] directly measured the interfacial strength by drawing out individual SWCNT ropes in an epoxy matrix using a scanning probe microscope tip. Based on their experiments, the interfacial strength between the MWCNTs and the epoxy matrix were calculated to be in the range of 35–376 MPa. Barber et al. [16] also measured the adhesive interactions between the MWCNTs and the polyethylene–butene matrix by performing reproducible nano-pullout experiments using atomic force microscope (AFM). Their experimental data resulted in an interfacial separation stress of 47 MPa. Moreover, Barber et al. [17] mounted MWCNTs onto an AFM tip before pushing it into a heated polymer film and on cooling they measured the force required to pull the CNTs out, obtaining the values between 20 and 90 MPa.

In this paper, CVD-grown MWCNT–epoxy composites are prepared using the three-roll calendering technique. This technique, which applies intensive shear forces on the processed compounds, has been considered as an effective method for achieving the excellent dispersion of CNTs in polymers such as epoxy resins [7,8] and polyester resin [9]. It was regarded that this dispersion technique would not damage nanotubes to substantially reduce carbon nanotube length after processing [7,8]. However, no data for length of residual carbon nanotubes after processing of composites are available. As mentioned above, carbon nanotubes might be damaged by external forces and a direct measurement would thus be of significance to examine if the reduction of carbon nanotube length occurs or not after processing of composites. In this work the residual CNT length after processing is measured using a computer software SemAfore 4.0 on SEM images of collected CNTs after removing epoxy resin. The significant reduction of the length of CNTs is observed for the first time, indicating that CNTs are indeed brittle to be easily broken during processing. The residual CNT length plays a critical role in determining the mechanical properties and is thus mainly responsible for the unexpected low reinforcing efficiency. This also suggests that a great attention should be paid in future to retention of CNT length during processing in order to obtain high mechanical performance polymer composites. Moreover, the CNT strength and the CNT–epoxy interfacial strength are simultaneously determined in terms of a simple methodology based on the modified rule of mixtures for SFRP composites [24,28]. The CVD-MWCNT strength obtained is close to that directly measured from a specially designed stress–strain puller [33] and the CNT–epoxy interfacial strength obtained here is within the reported values measured using a scanning probe microscope [15], indicating that this methodology is appropriate for determination of CNT strength and CNT–polymer interfacial strength.

2. Experimental

Diglycidyl ether of bisphenol-F (DGEBF, D.E.R.354, the Dow Chemical Co., USA) with the epoxide weight equivalence in

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