



On the mechanical characterization of carbon nanotube reinforced epoxy adhesives



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ABSTRACT

In this work, the mechanical properties of carbon nanotube reinforced epoxy adhesives are investigated experimentally. The investigations are intended to characterize the physical and mechanical properties of nano-reinforced structural epoxy adhesives and to further highlight some of the complex phenomena associated with these materials. We describe the dispersion methodology used to disperse the carbon nanotubes into the considered adhesive and provide details pertaining to adherent surface preparation, bondline thickness control and adhesive curing conditions. Furthermore, the following tests are described: (i) dogbone tensile testing, (ii) tensile bond testing, (iii) double lap shear and (iv) double cantilever beam fracture toughness testing. The experimental observations indicate a critical carbon nanotube concentration in the vicinity of 1.5 wt% that results in the largest improvements in the measured properties. At concentrations exceeding this critical value, the properties begin to degrade, in some cases, to levels below that of the pure epoxy. Advanced electron microscopy techniques and rheological assessments indicate that this is mainly due to the agglomeration of the carbon nanotubes at higher concentrations as a result of increased resin viscosity and the consequent resistance to dispersion.

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1. Introduction

It has been recognized for some time that the mechanical properties of polymeric materials can be engineered by fabricating composites that are comprised of different volume fractions of one or more reinforcing phases. A number of techniques have been considered to improve the mechanical properties of structural adhesives. They included the addition of carbon, nylon or glass micro-fibres, rubber and liquid rubber precipitates, reactive ductile diluents and inorganic hybrid particles, among others. Whilst some improvements in bond properties have been observed [1,2], these additives also led to reductions in high temperature service capabilities, low impact strength, and poor shrinkage characteristics [3]. As time has progressed, practical realization of composites has begun to shift from micro-scale composites to nanocomposites, taking advantage of the unique combination of mechanical and physical properties of *nanofillers* – fillers with a characteristic dimension below 100 nm. There are a number of advantages associated with dispersing nanofillers in polymeric materials. While some credit can be attributed to the intrinsic properties of the fillers, most of these advantages stem from the extreme reduction in

filler size combined with the large enhancement in the specific surface area and interfacial area they present to the matrix phase. In addition, whereas traditional composites use over 40 wt% of the reinforcing phase, the dispersion of just a few weight percentages of nanofillers into polymeric matrices could lead to dramatic changes in their mechanical properties with added functionalities.

In this work, we propose to reinforce the adhesive layer through the homogeneous dispersion of only a small fraction of carbon nanotubes (CNTs). CNTs are regarded as one of the most promising reinforcement materials for the next generation of high-performance structural and multifunctional composites [4]. These molecular scale tubes of graphitic carbon have outstanding mechanical, thermal and electrical properties. In fact, some CNTs are stronger than steel, lighter than aluminum, and more conductive than copper [5]. Theoretical and experimental studies have shown that CNTs exhibit extremely high tensile modulus (1 TPa) and strength (150 GPa). In addition, CNTs exhibit high flexibility, low density (1.3–1.4 g/cm³), and large aspect ratios (1000 s). Due to this unique combination of physical and mechanical properties, CNTs have emerged as excellent candidates for use as tailoring agents in polymeric materials to yield the next generation nanocomposites.

Recent work in this area shows that the scientific community is adopting a variety of different methods to develop these nano-reinforced composites with varying levels of success. The

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properties of CNT-based nanocomposites are influenced by a number of factors that include the CNT synthesis and purification process, the geometrical and structural properties of the CNTs, their alignment in the matrix, the dispersion process, and the fabrication process.

Perhaps the most remarkable improvement in the tensile modulus and yield strength of a polymer through the dispersion of CNTs was observed by Liu et al. [6]. By dispersing only 2 wt% of multi-walled carbon nanotubes (MWCNTs) in a nylon-6 matrix Liu et al. observed an increase of approximately 214% in the tensile modulus and 162% in the yield strength. They attributed these impressive improvements in the stiffness and strength to a uniform and fine dispersion of the CNTs and good interfacial adhesion between the nanotubes and matrix which were assessed using SEM. Montazeri and Chitsazzadeh [7] studied the effects of the ultrasonication dispersion processing parameters on the Young's modulus and tensile strength of MWCNT epoxy composites. Specifically, they fabricated samples containing 0.5 wt% MWCNTs and investigated the effects of sonication time and output power. They observed a maximum increase of only 17.5% in the strength and 46.9% in Young's modulus for the case involving 45 min sonication time and output power in the range of 25–50 W. In contrast, Liu et al. [8] also used the melt compounding method to fabricate MWCNT/PA6 composite specimens. The MWCNTs used in their study were synthesized in ethanol flame which was shown, through the use of a variety of microscopy techniques, to produce active functional groups on the surface of the MWCNTs. These MWCNTs were further functionalized with *n*-Hexadecylamine molecules. Through the use of SEM, they concluded a uniform dispersion and good wetting with the PA6 matrix. However, the observed improvements in the tensile modulus and tensile strength were only approximately 29% and 6%, for cases involving 1 wt% CNTs, respectively. In another recent study, Sahoo et al. [9] dispersed carboxyl-functionalized MWCNTs in a PA6 matrix. They used two both an internal mixer and an extrusion process to disperse the CNTs in the PA6 matrix. They observed a maximum increase of approximately 126% in the tensile strength when 10 wt% of the functionalized MWCNTs were incorporated in the PA6 matrix. This clearly demonstrates a significant variability in the results of three separate experimental studies that used the same fabrication method, constituent materials and both claiming uniform dispersions and good wetting with differences only in the pretreatment processes of the MWCNTs.

Gojny et al. [10] investigated the mechanical properties of an epoxy reinforced with both non-functionalized and amino-functionalized CNTs. One of the objectives of the study was to identify the best nanofiller when single-walled carbon nanotubes (SWCNTs), double-walled carbon nanotubes (DWCNTs), and MWCNTs were considered. In all cases amino-functionalized CNTs produced better results when compared with non-functionalized CNTs which can be attributed to the better interfacial adhesion of the CNTs and epoxy and the improved dispersion. The results showed that the greatest improvements in both tensile modulus and strength were observed for the amino-functionalized DWCNTs at concentrations of about 0.5 wt% with improvements of approximately 14.5% and 8.4%, respectively. It was expected that SWCNTs would provide the highest improvement in these properties given that these fillers have the largest specific surface area and aspect ratio, however, the DWCNTs did not agglomerate as pronounced as SWCNTs. In general, MWCNTs, whether functionalized or not, actually degraded the tensile modulus and tensile strength over that of the pure epoxy. This was attributed to the absence of stress transfer between the internal layers of the MWCNTs, and the relatively low specific surface area and aspect ratio present. Fig. 1(a–d) summarize these results.

Several experimental efforts have also been directed to improve the fracture toughness of polymer systems through the

homogeneous dispersion of CNTs. Gojny et al. [11] examined the fracture toughness of CNT/epoxy composites containing functionalized and non-functionalized DWCNTs dispersed through shear mixing in an epoxy matrix. The fracture toughness for functionalized DWCNTs was found to increase by as much as 26% compared to the pure epoxy matrix, when a filler content of 1 wt% was used. Lachman and Wagner [12] investigated a multitude of different CNTs and other nanofillers as a means of improving the fracture toughness of a common epoxy. These included carbon black, carbon nanofibers, pristine MWCNTs, and two different forms on functionalized MWCNTs, namely, carboxylated and aminated. The nanofillers were dispersed in the epoxy by means of mechanical stirring and sonication. Fig. 2 presents the results of their test. As can be seen, the carbon black and carbon nanofiber based composites showed no significant improvement in fracture toughness, whereas all CNT polymer composites displayed significantly higher fracture toughness values when compared with the pure epoxy. The toughening effect was the highest in the NH_2 -CNT nanocomposites for which it is approximately twice the pure epoxy value when the CNTs were dispersed in ethanol prior to mixing with the epoxy.

Existing experimental results indicate that significant increases in the stiffness, strength and fracture toughness of CNT based composites can be achieved at very low CNT concentrations. However, as with most of the other mechanical properties, there exists a critical CNT concentration above which the fracture toughness begins to degrade which can be attributed to the inability of present dispersion techniques to ensure de-agglomeration of CNTs at large concentrations. In this study, we describe our initial efforts to improve the mechanical properties of carbon nanotube reinforced epoxy adhesives. Specifically, we evaluate the strength and stiffness, tensile and shear bond strengths, and fracture toughness of the newly developed adhesives for CNT concentrations spanning from 0 to 3 wt%. We identify the critical CNT concentration for our material system and provide an assessment of the dispersion quality and its effect on the observed properties.

2. Fabrication of nano-reinforced epoxy adhesives

This section describes the experimental methodology adopted in evaluating the constitutive response and mechanical properties of the newly developed nano-reinforced epoxy adhesives. Specifically we provide details of the materials used in the study, the dispersion methodology, and the general processing conditions.

2.1. Materials used

2.1.1. Carbon nanotubes

The CNTs were obtained commercially and synthesized using the electric arc technique. The resulting CNTs are extremely high purity (~99.9%) MWCNTs with an outer diameter of 13–18 nm and a length in the range of 1–12 μm . Fig. 3(a–c) shows scanning transmission electron micrographs (STEM) of the as-received MWCNTs at three different magnifications taken with a Quanta 250 field emission gun environmental scanning electron microscope. As can be seen, the CNTs exist as large agglomerates (~5 μm) of MWCNTs that need to be broken up and homogeneously dispersed in the adhesive. Fig. 4 shows a TEM micrograph of the MWCNTs at a very high resolution allowing one to see the individual fringes formed from the concentric walls of the CNT. Table 1 summarizes some of the pertinent properties of the CNTs.

2.1.2. Substrate materials

Unidirectional carbon fiber sheets and aluminum 6061-T4 were selected as the substrate materials for the different tests programs.

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