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# Experimental torsional shear properties of carbon fiber reinforced epoxy composites containing carbon nanotubes



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#### ABSTRACT

The in-plane and torsional shear properties directly relevant to specific applications of carbon fiber-reinforced composites (CFRPs), such as sporting goods and wind turbine blades, are studied. Multiscale composites are fabricated using carbon fiber prepregs containing carbon nanotube (CNT) modified epoxy matrix. The interlaminar shear strength (ILSS) of the composites with 0.5 wt.% CNTs increases by 12% compared to the neat CFRP composites, whereas the torsional shear modulus and strength determined using tubular specimens present corresponding improvements of 17% and 19.5%, respectively. The enhanced interfacial adhesion between the modified matrix and carbon fibers, and the increases in matrix modulus/strength arising from the well-dispersed functionalized CNTs are mainly responsible for the above observations. An increase in CNT content beyond 0.5 wt.% has moderately negative effects on these properties, indicating potential agglomeration at a high CNT content.

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# 1. Introduction

Incorporation of nanoparticles in polymers creates new composite materials with tailored properties for potentially new applications. Carbon nanotubes (CNTs) possessing excellent mechanical and functional properties [1] have been considered as one of the most promising nanofillers for nanocomposites based on various types of polymer [2–9]. The interest in the opportunity to produce hybrid fiber-reinforced polymer (FRP) composites consisting of CNTs and continuous fibers as the main reinforcements has also been growing as these multi-scale composites can offer significant flexibilities in the design, manufacturing, choice of material and properties [10,11].

The exploitation of unique properties of CNTs in a composite depends strongly on how the CNTs are integrated into it. The incorporation of nanoscale CNTs with conventional microscale fiber reinforcements in a common polymer matrix can be achieved by (i) modifying the matrix resin [12–17] using CNTs; (ii) attaching, grafting or growing CNTs directly on the fiber reinforcement or fabric [18–22]; or (iii) interleaving bucky papers made from CNTs at certain laminar interfaces [23,24]. Modifying the matrix resin by dispersing CNTs in the polymer offers the most conventional, facile and industrially compatible route. Several methods have been devised for CNT dispersion into a polymer matrix, which includes shear mixing, calendaring, extrusion, ultrasonication and ball milling, either in the presence or absence of solvents [25].

The existing FRP fabrication techniques can be directly employed to produce multiscale FRP composites using CNT-polymer nanocomposites as the bulk matrix material. The process has been often combined together with other manufacturing techniques, such as resin transfer molding (RTM) and vacuum assisted resin transfer molding (VARTM) [12–17]. However, these manufacturing routes have limitations, such as the difficulty to uniformly disperse the CNTs in the laminate due to the phenomenon called 'filtration' of CNTs, leading to large CNT agglomerates [26]. Another major issue is that the viscosity of the CNT-modified matrix increases drastically with increasing CNT content, even with a content as low as 1 wt.%, causing more severe CNT agglomerates makes it difficult to incorporate high CNT contents into the system whether solvents are used or not.

An alternative route is to produce prepregs in which fibers are pre-impregnated with a resin containing a desired amount of CNTs in a latent or beta state. Since the prepregging method involves dipping of the fibers in the resin in a continuous process, rather than large flow through tubes or channels into the preforms, it is expected that the problem of 'filtration' of CNTs be minimized. Very few studies have so far reported the fabrication of multiscale CFRP composites using prepregs containing CNTs [27,28] although it is a well established processing technique to produce composites for high end structural applications. Apart from the above fabrication process using the CNT-containing resin as the bulk matrix, direct growth/grafting of CNTs on the microscale fibers/fabrics or interleaving of CNT bucky papers have major advantages to incorporate high CNT contents and to place them at desired locations to maximize the synergy arising from CNTs.





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The presence of a small amount of CNTs in the matrix, or along the fiber-matrix interface or interleaved laminar interfaces results in improvements of various mechanical properties of the multiscale composites [11]. Particularly large improvements were noted in interlaminar properties, including the interlaminar fracture toughness in various loading modes [18,27] and the interlaminar shear strength [18,29], as well as the impact damage resistance [27,30]. All these improvements are a direct reflection of the enhancement in the mechanical properties, fracture toughness in particular, and the interface properties of CNT-polymer nanocomposites [14,31]. This paper is part of a larger project aiming to develop high performance, multiscale CFRP composites containing nanofillers, such as nanoclay and CNTs [28,32-35] for specialty structural applications. Important mechanical properties of the epoxy-based CFRP composites containing CNTs, produced by a solvent-less prepregging process were evaluated. A special emphasis was placed on the evaluation of the in-plane and torsional shear properties of the composites affected by the incorporation of CNTs into the prepregs. These shear properties are of paramount importance in structural performance and durability of many components made from FRP composites, such as sporting goods like golf shafts and tennis rackets, rotor blades in helicopters and wind turbine blades.

# 2. Experimental

# 2.1. Materials and preparation of CNT modified matrices

The materials and processes used to produce hybrid composites in this work were essentially the same as those presented elsewhere [28]. The multi-walled carbon nanotubes (MWCNTs) (NK-50 supplied by Nanokarbon) used in this work were produced by a vapor grown method and had a bamboo-like structure. The outer diameters ranged between 40 and 60 nm and the lengths were about 20  $\mu$ m. Typical transmission electron microscopic (TEM) images of these CNTs are presented in Fig. 1. The carbon fiber roving (Pyrofil TR 30S, supplied by Mitsubishi Rayon, Japan) with a filament count of 6 K was used as the main reinforcement. Araldite LY556 (a reaction product of bisphenol A (epichlorohydrin)) resin system was selected to prepare prepregs, which was composed of Araldite LY556, Aradur 5021 and hardener XB 3471 (all supplied by Huntsman), mixed in the ratio of 100:25:12 parts by weight.

The CNTs were functionalized using a combination of processes chosen based on previous studies [8,31]. The CNTs were first subjected to oxidation in a UV ozone chamber (Jelight 144AX-220) for 30 min, followed by a treatment with surfactant. A non-ionic surfactant, polyoxyethylene phenyl ether (Triton X-100, supplied by VWR International) with the critical micelle concentrations (CMC) value of 0.2 mM at 25 °C was used to treat the CNTs to improve the dispersion in the resin. The details of procedure adopted for the treatment was basically similar to the previous report [28]. A desired amount of CNTs was dispersed in acetone containing 10CMC of surfactant, equivalent to a Triton weight to acetone volume ratio of approximately 12.5 mg/l000 ml. The mixture was subjected to sonication in a bath (Branson 150) for 120 min. A required amount of epoxy was heated to 75 °C to lower the viscosity and was added into the CNT/acetone mixture. The mixture was subject to sonication at 60 °C for 30 min, followed by further mixing using a high speed shear mixer (Ross) at 4000 rpm for 30 min. The dispersion was degassed in a vacuum oven at 70 °C overnight to ensure complete removal of acetone.

### 2.2. Prepregging process and composite fabrication

The schematic showing the steps involved in the processing and fabrication of the hybrid composites is given in Fig. 2a. CNT-CFRP hybrid composite prepregs were prepared on a lab-scale, hot-melt prepregger (Model 40 Research Tool Corp., USA), as shown in Fig. 2b. The temperature of the resin bath was set at 37 °C, which was optimised to maintain the viscosity of the CNT-resin mixture within the required limits (1.5-2.5 Pa S) and to achieve good wetting of the carbon fiber tow, according to our previous study [28]. Composite prepregs containing 0, 0.5, 0.7 and 1.0 wt.% CNTs were prepared with average fiber volume fractions of 41.2%, 40.3%, 40.7% and 40.8%. The thickness and the average areal weight of the prepregs were 0.2 mm and 1.60 kg/m<sup>2</sup>, respectively. The composites with low fiber volume fractions used in this study were a direct consequence of the hot melt prepregging process adopted without using solvents where the viscosity of resin was inevitably high and thus high resin contents. It was also hoped that the bulk properties of the composites might become more sensitive to the matrix properties when a relatively low fiber volume fraction was used. It was noted that the fiber distributions were consistently uniform in all types of FRPs, partly confirming the adequate prepregging process.

# 2.3. Fabrication of specimens and mechanical tests

The flexural properties of the hybrid composites were determined according to the specification, ASTM standard D790. 2.5 mm thick laminates consisting of nine layers of unidirectional prepregs [0]<sub>9</sub> were fabricated by hand lay-up. The curing of all



Fig. 1. TEM micrographs of the CNTs used in this study.

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