



# Mechanical, thermal and interfacial performances of carbon fiber reinforced composites flavored by carbon nanotube in matrix/interface



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## ABSTRACT

The introduction of carbon nanotubes (CNTs) into fiber-reinforced polymer composites has been achieved predominantly via two routes: mixing CNTs entirely throughout the matrix (matrix modification) or attaching CNTs onto reinforcing fibers (interface modification). We studied unidirectional carbon fiber/epoxy composites where CNTs were introduced to enhance the hierarchical composites by two alternative strategies: mixing into the epoxy or attaching onto carbon fibers by electrophoretic deposition. Single-fiber fragmentation test combined with energy dispersive X-ray spectroscopy was creatively applied to calculate interfacial shear strength and improvements of 45.2% and 10.14% were achieved for hierarchical composites based on CNT-modified fibers (CF-CNTs/EP) and composites based on CNT-reinforced matrix (CF/EP-CNTs), respectively. Increases of 24.42% for CF-CNTs/EP and 10.41% for CF/EP-CNTs in tensile strength were achieved separately. The superiorities of CF-CNTs/EP were derived from that gradient interface layer formed due to the introduction of CNTs in interface and it was not obvious in CF/EP-CNTs.

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## 1. Introduction

Carbon fiber-reinforced polymer composites with chemical and environmental resistance have superior strength-to-weight and stiffness-to-weight ratios, which make them ideal for many structural applications in many fields, such as aerospace, automotive, sports equipment and so on. The properties of fiber-reinforced polymer composites are, to a great extent, controlled by the performance of the interface which works as a bridge between fiber and matrix. Preeminent interfacial bonding ensures efficient load transfer from matrix to the fibers and internal crack propagation, which helps reduce stress concentrations and improve the ultimate performance of composites [1–3]. Due to their unique structures, excellent mechanical properties where the strength, modulus and resilience are equal or superior to any current materials, brilliant electrical and thermal properties, carbon nanotubes (CNTs) have been employed to enhance the hierarchical composites, which is a topic of significant interest in recent years [4,5].

Carbon fibers (CFs) show crystallized graphitic basal planes with non-polar surface and the chemical inertness due to the high

temperature carbonization/graphitization step during manufacturing process [6]. Excessive smoothness and few polar groups of CFs lead to weak bondings with matrices [7,8]. According to the characteristics of CFs, the introduction of CNTs into carbon fiber-reinforced polymer composites has been attained, which was fundamentally based on two techniques: mixing CNTs entirely throughout the matrix (matrix modification) or attaching CNTs directly onto fiber surface (interface modification) (Fig. 1.). To date, direct mixing by mechanical [9], shear mixing involving a three-roll mill [10,11], or ultrasonic techniques was generally used to mix CNTs into low-viscosity thermosetting matrices. And, generally, five techniques to graft or attach CNTs onto carbon fiber surface have been reported so far: (1) direct growth of CNTs on fibers by chemical vapor deposition (CVD) [12–14]; (2) chemical reactions between functionalized CNTs and fibers [3,15,16]; (3) electrophoretic deposition (EPD) of CNTs on fiber surface [17]; (4) coating of fibers with CNT-containing sizing [2,18,19]; (5) immersing fibers in the solution of CNTs [19].

The most simple and straightforward manufacturing process for introduction of CNTs involves matrix modification, i.e. direct mixing CNTs into the matrix. Functionalized CNTs, treated by either chemical or physical treatments, can make a favor for promoting the CNT dispersion and stress transferring between CNTs and matrices. Matrix modification commonly has the advantages of

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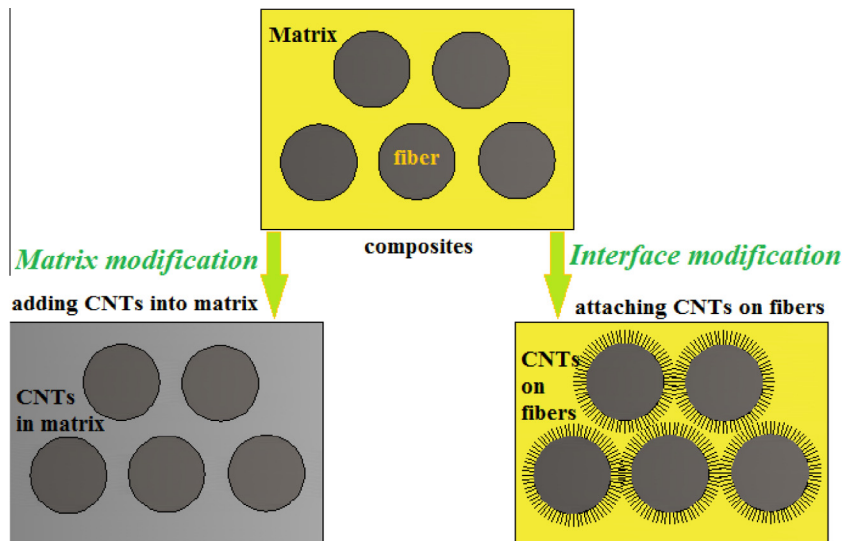


Fig. 1. Schematic diagrams of fiber-reinforced polymer composites and CNT-based hierarchical polymer composites.

simplicity and compatibility with standard industrial techniques. This technique has been applied by a number of commercial companies and a variety of CNT-modified matrix products are now commercially available. Grafting CNTs onto fiber surfaces with proverbial superiority, efficaciously, could acquire higher loadings of CNTs with a radial orientation [20], which is expected to be optimal for transverse reinforcement [5,21]. Researchers [3,15,16,22] have applied chemical methods to covalently graft functionalized CNTs onto functionalized CFs. Thostenson [12] firstly synthesized CNTs via thermal chemical vapor deposition on the surfaces of CFs. Bekyarova et al. [17] authenticated that a uniform distribution of CNTs on the surface of carbon fabric could be obtained using EPD technique, in which both as-received and oxygenated CNTs were deposited onto CFs. EPD technology has the advantages of simplification and uniform deposition which could be applied to continuous processing of carbon fiber tows. Alternatively, CNTs have been attached onto CFs by using a nanocomposite polymer sizing [18,23,24]. Repetitious sizing treatment was used to modify the carbon fiber surfaces with CNTs for improving interfacial properties of CF/epoxy composites [19].

However, up to present, few systematic investigations and comparisons of matrix modification and interface modification have been reported. So, in this work, we focused on two alternative ways to introduce CNTs to composites: mixing CNTs in the matrix using ultrasonic technique (matrix modification) or depositing on carbon fiber surface using EPD technology (interface modification); meanwhile strength and weakness of the two alternative strategies were contrasted. Morphologies of carbon fiber surface and fracture structures obtained from mechanical tests were characterized by scanning electron microscopy (SEM). Moreover, the distribution of CNTs on carbon fiber surface and in matrix was detected by transmission electron microscope (TEM). Common mechanical tests, including flexural test and tensile strength test, were carried out. Simultaneously, the thermal properties of the neat epoxy (EP) and hierarchical composites were characterized by dynamic mechanical thermal analysis (DMA). In addition, the interlaminar shear strength (ILSS) and interfacial shear strength (IFSS) which indicate the interfacial properties of hierarchical composites were also investigated, respectively, using short beam shear test and single-fiber fragmentation test. For matrix modification, the presence of CNTs in the resin led to the opaque of matrix (Fig. S1). Therefore, during single-fiber fragmentation test, fragments and birefringence phenomenon cannot be caught and the IFSS was unable to be measured. We made great efforts to conquer this

problem and adopted energy dispersive X-ray spectroscopy (EDS) equipped on SEM to assist single-fiber fragmentation test. Finally, EDS, force modulation atomic force microscope (f-AFM) and TEM techniques were used to survey the microstructures of interface and explore the enhancement mechanism of CNTs.

## 2. Materials and experimental methods

### 2.1. Materials

Commercially-available T700S CFs (Toray) with the diameter of 7  $\mu\text{m}$  were employed in this study. As-received multi-wall CNTs (Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences) with a diameter of 30–50 nm, length of 10–20  $\mu\text{m}$  and purity of more than 95wt% were adopted in this study. JC-02A modified epoxy and JH-0511 modified 2-ethyl-4-methylimidazole, used as accelerator, were purchased from Changshu Jia Fa Chemical Co. Ltd., China. We selected tetrahydrophthalic anhydride as the curing agent.

### 2.2. Introduction of CNTs onto carbon fiber surface and into matrix

In order to better appraise the influences of CNTs on the modification of composites, virgin CFs were refluxed by acetone in the soxhlet apparatus at 70  $^{\circ}\text{C}$  for 48 h to remove the commercial sizing. Subsequently, CFs were dried 3 h under vacuum at 50  $^{\circ}\text{C}$  and the unsized CFs were also designated with CFs. And CFs were on behalf of unsized CFs in the following statements, if there is no special note. As-received CNTs were treated with a mixture of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ )/concentrated nitric acid ( $\text{HNO}_3$ ) (3:1 v/v) to introduce carboxylic acid groups ( $-\text{COOH}$ ) to CNTs [25]. Similarly, the abbreviation of CNTs stands for functionalized CNTs in the following descriptions.

#### 2.2.1. Electrophoretic deposition of CNTs onto carbon fiber surface

Referring to references [26–30] and our previous research [19], we chose these EPD parameters: concentration of CNTs 0.3 mg/ml, deposition voltage 24V, deposition time 5 min, electrode materials metal plate and electrode separation 50 mm. 300 mg CNTs containing carboxyl groups were initially dispersed in 1000 ml of deionized water by an ultrasonic bath for 2 h to achieve a homogeneous suspension. After that, the unsized carbon fiber tow was immersed into the suspension, and a succedent process of EPD was shown in Fig. 2(a).

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