



Comparison of carbon nanotubes and graphene oxide coated carbon fiber for improving the interfacial properties of carbon fiber/epoxy composites



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ARTICLE INFO

Article history:

Received 20 November 2016

Received in revised form

5 September 2017

Accepted 6 September 2017

Available online 6 September 2017

Keywords:

Carbon fiber

Laminates

Nano-structures

Interface/interphase

Fiber/matrix bond

ABSTRACT

By designing the fiber/matrix interface, the properties of the carbon fiber-reinforced composite can be changed significantly. In this work, carboxylic-functionalized carbon nanotubes (CNTs) and graphene oxide (GO) were separately deposited on the carbon fiber (CF) surface to increase the interfacial properties of composites. The properties of fibers such as the surface topography, surface chemical composition and surface energy were examined, and the interfacial shear strength between the fibers and the matrix was examined using a microdroplet test. In addition, CNT-coated, GO-coated and pristine unidirectional carbon fiber fabrics were used to manufacture CF/epoxy composites via the vacuum-assisted resin transfer molding method, and their moisture absorption and interlaminar shear strength were measured. The results reveal that both CNTs and GO can significantly improve the interfacial properties of composites. The CNT/CF-reinforced composites have higher shear strength, and the GO/CF-reinforced composites have better humidity resistance. The interfacial reinforcing mechanisms of the composites with CNTs and GO were compared.

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1. Introduction

Because of their excellent characteristics, such as high specific strength, good corrosion resistance and outstanding design flexibility, carbon fiber-reinforced polymer composites are widely used in automobile, sports and aerospace applications [1–5]. The physical and mechanical properties of the composites strongly depend on the polymer matrix, fiber reinforcement, and their interfacial interactions. It is well known that the fiber/matrix interface guarantees the stress transfer from the weak matrix to the strong fiber, so the composite performance is largely depended on the fiber/matrix adhesion [6–9]. However, a long existing issue to overcome is the low interfacial bonding strength between the carbon fibers (CFs) and the polymer matrix due to the intrinsically smooth, hydrophobic and chemically inert CF surface [10,11]. As a result, over the last few decades, significant scientific effort has been focused on the surface modification of CF to improve the interfacial

properties of composites using many methods such as plasma etching treatment [12], the sizing technique [13], chemical treatment [14,15], and electrochemical oxidation treatment [16,17]. These methods improve the interfacial properties of the composites, but because the fiber and polymer matrix have different scales, these methods have not achieved satisfying results.

In recent years, with the production and popularity of nanomaterials, there has been significant interest in developing micro/nanocomposites, where traditional fibers are integrated with diverse nanoparticles [18–21]. Nanoscale reinforcements have critical dimensions that are three orders of magnitude smaller than the traditional fiber reinforcements. This arrangement will have the advantage of a complex, hierarchical structure because of the bond between the matrix and the traditional fibers in addition to the smaller-scale bond between the nanoparticles and the polymer matrix [22]. Many methods have incorporated chemical vapor deposition (CVD) [23–25], chemical grafting [26,27], electrophoretic deposition (EPD) [28–30], etc. These methods effectively attach nanoparticles to the surface of the fiber. However, the chemical grafting method is not practically applicable due to its long processing time and violent chemical reaction, and CVD requires high temperatures and pre-deposited catalysts. The EPD

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technique has been widely used for surface modification to produce a large-scale nanoparticle reinforcement material for composite applications. EPD has multiple advantages over other techniques, such as the effortless control of the film thickness, good surface homogeneity, high deposition rate, and simplicity of up-scaling [31–35].

As typical one-dimensional and two-dimensional carbon-based nanomaterials, carbon nanotubes and graphene nanoplatelets are widely used as nanoscale reinforcements in the interphase region of composites to cause the interfacial mechanical interlocking between the fibers and the matrix to improve the interfacial properties of the composites [36–39]. Functionalized CNTs and GO are always used to modify CF because the oxygen-containing polar groups can improve the surface wettability of the CF. In addition, these polar groups are helpful in enhancing the interfacial adhesion by establishing covalent bonds with the matrix in the curing step [40]. In this work, carboxylic-functionalized CNTs and GO were separately deposited on the surface of CF using the EPD technique. Although some work has been reported in the literature with these two separate nanomaterials, they have not been compared. This paper compares the interfacial properties of CF/epoxy composites with CNT- and GO-modified CF.

2. Experimental

2.1. Materials

The commercial PAN-based CF tow (T700) with a mean diameter of 7 μm was supplied by Toray Industries. Commercially available multiwall carbon nanotubes (length 10–20 μm ; diameter approximately 10–20 nm; 95% purity) were purchased from Shenzhen Nanotech Port. Co., Ltd. The natural graphite flakes (with a purity of 99% and a mean diameter of 25 μm) were purchased from Qingdao Huatai Co., Ltd. The matrix in this work was bisphenol A epoxy resin (E-51), which was provided by Nantong Xingchen Reagent Co., Ltd. The hardener was 3,3'-diethyl-4,4'-diaminodiphenylmethane (H-256) and was provided by Jiangsu Huifeng Co., Ltd.

The CF was refluxed in acetone for 48 h at 60 °C to remove the sizing agents [41]. Then, it was repeatedly washed in deionized water and dried at ambient temperature to obtain the desized CF. All CFs used in this study were desized CF unless otherwise specified.

The carboxylic-functionalized CNTs were prepared by a mixed-acid treatment method [42]. The received CNTs (0.1 g) were refluxed in a mixture of concentrated sulfuric acid (98%, 25 mL) and concentrated nitric acid (65%, 75 mL) and stirred at 98 °C for 4 h. Then, the mixture was washed with deionized water several times to neutralize it. The carboxylic-functionalized CNTs were dispersed in deionized water by sonication for 1 h to obtain the CNT solutions. All CNTs utilized in this study were carboxylic-functionalized CNTs unless otherwise specified.

The GO was made by a modified Hummers method [43,44]. Briefly, graphite flakes (2 g) were mixed with concentrated sulfuric acid (98%, 50 mL) and sodium nitrate (2 g). Then, potassium permanganate (4 g) was slowly added into the system. The mixture was magnetically stirred at 30 °C for 2 h; then, deionized water (100 mL) was added and stirred at 96 °C for 1 h. Hydrogen peroxide (5%, 100 mL) was added, and the mixture was washed with deionized water several times. The purified sample of GO was dispersed in deionized water by sonication for 1 h to yield GO dispersions. The typical transmission electron microscope images of the carboxylic-functionalized CNTs and GO in this work are presented in Fig. 1.

2.2. Surface modifications of the CF

The EPD process was performed using a DC power supply (HYELEC, HY3005MT, China). The CF was used as the positive electrode, and two graphite plates were placed opposite the CF as the negative electrodes. The constant voltage, solution concentration and solution pH were 20 V, 0.1 mg/mL and 10, respectively. The pH value was obtained by adding a 0.2 M sodium hydroxide solution. The deposition process was performed for 20 min with an electrode distance of 2 cm. During the deposition process, ultrasonication was used to remove the bubbles produced from the water electrolysis [45]. After the deposition, the CFs were dried in an oven at 90 °C for 12 h. The carboxylic-functionalized CNTs and GO were deposited on the CF to obtain the fibers, which were labeled CNTs/CF and GO/CF, respectively.

2.3. Fabrication of CF/epoxy composites

Composite laminates were prepared using the vacuum-assisted resin transfer molding (VARTM) method [46]. Sixteen plies of the desized, CNT-coated and GO-coated unidirectional carbon fiber fabrics (300 g/m²) were laid up to the required thickness of the testing standard in the unidirectional fiber orientation. VARTM was used with the epoxy resin and hardener at a mass ratio of 100:33 to saturate the CF preforms with epoxy resin. The epoxy was infused into the CF fabrics in vacuum during the VARTM. After the epoxy resin infusion, the composites were cured at 80 °C for 12 h. The manufactured composite laminates had similar thickness values of 3.5 mm, and the fiber volume fraction in the final composite laminates was ~52%.

2.4. Characterizations

The morphologies of the carboxylic-functionalized CNTs and GO were observed using a transmission electron microscope (TEM, FEI, Tecnai-F30-G2, USA). The surface morphologies of the CFs were characterized by a scanning electron microscope (SEM, JOEL, JCM-6000, Japan). The SEM was set to a backscattered electron image (BEI) with 15 kV accelerating voltage, high vacuum, and high probe current.

The chemical structures of the carboxylic-functionalized CNTs, GO and CFs were examined based on the Fourier transform infrared spectra (FT-IR, Nicolet, 20DXB spectrophotometer, USA) obtained using a KBr pellet in the range of 500–4000 cm^{-1} .

A monochromated Al K α source (1486.6 eV) was used to perform X-ray photoelectron spectroscopy (XPS, Physical Electronics, PHI-5400, USA) to study the surface chemical elements of the CFs. An energy step size of 0.05 eV and a pass energy of 20 eV were used to acquire the detailed spectra. Fibers were suspended across a custom designed frame attached to standard template. The base pressure was approximately 2×10^{-6} Pa. Charge neutralization was required for all samples, the surface charging was compensated using a flood gun operating at 0.8 eV. A curve fitting program (Avantage software) with a Shirley background subtraction and a Gaussian-Lorentzian mix function was used to fit the XPS peaks. Each peak was fitted by Gaussian 70%–Lorentzian 30% mixture curves that were constrained in location and FWHM to realistically model the surface composition of the fiber. The entire binding energies refer to the C 1s peak at 284.6 eV [47].

A dynamic contact angle meter (DCA, Data Physics Instruments, DCAT21, Germany) was used to perform dynamic contact angle tests. Deionized water and glycol were used as the test liquids. According to the Owens-Wendt method [48,49], the polar components, dispersive components and total surface energy can be

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