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Directly grafting graphene oxide onto carbon fiber and the effect on the mechanical properties of carbon fiber composites

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ABSTRACT

Amino-functionalized graphene oxide (GO-NH_2) was directly grafted onto carbon fiber surface by covalent bonding in an attempt to improve the mechanical properties of carbon fiber composites. The effect of surface modification on the properties of carbon fiber and the resulting carbon fiber composites was investigated by X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and interfacial shear strength (IFSS). As revealed by Fourier transform infrared spectroscopy (FT-IR), GO-NH_2 was successfully grafted on the carbon fiber surface. From dynamic contact angle analysis (DCAT) and XPS values, such GO-NH_2 grafted carbon fiber exhibited significant improvements in surface energy and functional groups of the carbon fiber surface, which lead to an increase of 36.4% in the IFSS of its composites. Such hierarchical reinforcement shows great potential for enhancing interfacial properties in carbon fiber-reinforced composites.

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1. Introduction

Excellent mechanical and thermal properties make carbon fiber (CF) reinforced composites become an ideal structure material [1–3]. Considerable efforts have been focused on improving mechanical properties of fiber-reinforced polymer composites [4–6]. Recently, hybridizing carbon fiber with nanoparticles arises as a novel method to produce highly reinforced composites [7–9]. Such polymer composites exhibit effectively improved interfacial strength and inter-laminar properties by the hierarchical skeleton [10–12].

As a novel carbon materials, graphene oxide (GO) possesses a two-dimension structure and exhibits extraordinary mechanical [13], electrical [14], thermal [15,16] properties. More importantly, it is practical to produce GO, which contains various oxygen functional groups such as epoxide, hydroxyl and carbonyl groups, in large quantities at a relatively low cost [17,18]. In the last few years, a new hierarchical reinforcement by chemically grafting graphene oxide onto carbon fibers through the bridge of PAMAM was investigated [19,20]. In order to replace the expensive PAMAM, researches have been carried out to graft a graphene oxide thin film on carbon fiber surface by the electrophoretic technique [21,22]. However, the IFSS values of carbon fiber deposited with graphene oxide were significantly lower than that of chemical grafting. Similarly, this phenomenon exists in the hierarchical reinforcement system as CNTs on the carbon fiber surface [23–26]. As a result, to fully explore the potential of GO in improving the load-transfer capacity in a hierarchical composite without the use of costly PAMAM, it is necessary to directly graft GO onto the carbon fiber.

In the work reported here, we developed a method of directly grafting functionalized GO onto the surface of carbon fiber. The mechanism of directly grafting functionalized graphene oxide onto carbon fiber and the effects on mechanical properties of the advanced composites were clarified. X-ray photoelectron spectroscopy (XPS) and SEM were applied to unveil the functional groups and microstructures of fiber. Meanwhile, IFSS was performed to measure the interfacial properties of carbon fiber composites.

2. Experiments

2.1. Materials

Carbon fiber (T-300, average diameter 7 um, the density 1.78 g/cm^3) used in this study was obtained from Toray, Japan. Except Ethylenediamine (EDA, Aldrich), all the other chemicals (analytical grade) were purchased from Tianli Chemical Reagent Co. Ltd., China and used as received.

2.2. Preparation of the carbon fiber grafted with GO

The carbon fiber was extracted with supercritical ace-tone/water at 633 K for 20 min [27,28], and were denoted as untreated carbon fiber. Then, the carbon fiber was oxidized by nitric acid at 60 °C for 2 h. After that, the carbon fiber was reacted with the mixture solution of 100 mL $SOCl_2$ and 5 mL DMF at 78 °C for 48 h, the products were finally dried in vacuum at 100 °C for 6 h.

The procedure adopted for synthesizing GO-NH_2 was reported by Isis E. Mejias Carpio et al. [29]. Next, the acyl chloride functionalized carbon fiber (CF-COCl) was first reacted with 0.2 mg of amino-

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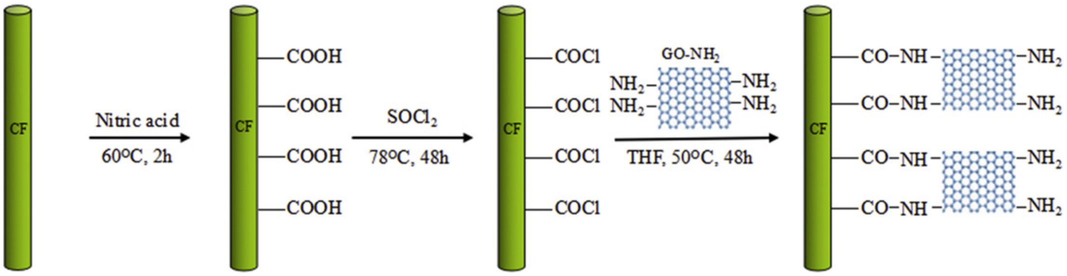

Fig. 1. Schematic of the grafting procedures.

functionalized GO (GO-NH_2) in 100 mL THF at 50 °C for 48 h. The resulted carbon fiber grafted with GO-NH_2 (CF-GO) was then washed with THF several times and dried at 80 °C under vacuum. The schematic illustration of CF-GO preparation was shown in Fig. 1.

2.3. Characterization

The chemical groups on the carbon fiber surface was identified by Fourier transform infrared spectroscopy (FT-IR) on a Perkin Elmer spectrometer (Spectrum one, USA) from 500 to 4000 cm^{-1}. The pellets for IR characterization consist of 0.1 mg grafting fibers and 100 mg KBr.

Thermogravimetry (TG) analysis of carbon fiber grafted with GO was performed with a simultaneous thermal analyzer (TGA/DSC1/1600LF, Switzerland) at a heating rate of 10 °C/min from 100 °C to 600 °C in nitrogen atmosphere.

Scanning electron microscopy (SEM, Royal Dutch Philips Electronics Ltd., Netherlands) was applied to observe the surface morphologies of carbon fiber as well as the fracture surface of composites on FEI Sirion 200 scanning electron microscope. Prior to SEM characterizations, all samples were sputtered by gold to improve the electrical conductivity of samples.

The chemical composition of carbon fiber without and with GO were analyzed by X-ray photoelectron spectroscope (XPS, Kratos Axis Ultra VG) with a monochromated Al Ka X-ray source ($hm = 1486.6$ eV) at 1 keV and an emission current of 0.6 μA. The total pressure was below 10^{-8} mbar in the main vacuum chamber. C1s, O1s and N1s high resolution spectra were recorded to investigate the detailed information about chemical structure.

The surface energies (γ) as well as its dispersive (γ^d) and polar (γ^p) components of different carbon fibers were measured by dynamic contact angle analysis test (DCAT-21, German). The carbon fibers were connected to a paper ($25 * 5$ mm) with adhesive in a parallel space of 5 mm. The wettability of carbon fibers was evaluated in two immiscible liquid (deionized water/non-polar diiodomethane) systems.

The tensile strength of carbon fiber was measured by the single-fiber tensile testing machine according to the standard ASTM D3379 specification at a crosshead speed of 5 mm/min. A single fiber was separated from the carbon fiber bundles and attached to paper tabs at a distance of 20 mm using an instant adhesive. A total of 100 data points were collected for a single carbon fiber.

Interfacial shear strength (IFSS) between carbon fiber and matrix was examined by single fiber micro-debond test (Tohei Sayon Corporation, Japan), which was performed by pulling a fiber out from the cured epoxy resin droplets with a loading rate of 0.5 μm/s. The values of IFSS were calculated according to Eq. (1),

$$IFSS = \frac{F_{\max}}{\pi \cdot \mathrm{d_f} \cdot \mathrm{l_e}} \tag{1}$$

where F_{max} is the maximum load recorded, d_f is carbon fiber diameter, and l_e is the embedded length [24].

3. Results and discussion

3.1. Chemical groups of carbon fiber surface

The carbon fiber functionalization was characterized by Fourier transform infrared spectroscopy (FT-IR). Fig. 2 presented FT-IR spectra of untreated and functionalized carbon fibers showing different absorption peaks. Compared with untreated carbon fiber, the new feature of CF-GO at 2930 cm^{-1} was assigned as the stretching vibration of —CH_2 [30] and the new peaks at 3440 cm^{-1} and 1540 cm^{-1} were related to the N—H stretching modes of amino groups(—NH_2) [31]. The new features at 1640 cm^{-1} and 1540 cm^{-1} of CF-GO were attributed to amide I (—CO—NH—) and II (—CO–NH_2) [32]. All the results indicated that the amino groups were introduced onto the GO surface and partial amino groups of GO-NH_2 were reacted with the acyl chloride groups on the carbon fiber surface. In a word, the formation of CF-GO could be confirmed by the appearance of —NH_2 peak at 3440 cm^{-1}, 1540 cm^{-1}, —N—C═O peak at 1640 cm^{-1} [33,34].

In order to obtain overall quantitative picture of carbon fiber, TG analysis measurements were carried out. Fig. 3 presented the TG analysis results of the untreated carbon fiber and the CF-GO. Typically, the weight loss (about 0.7%) between 200 °C and 600 °C was mainly attributed to the decomposition of amide bond groups [35,36]. The ratio of GO to CF in treated carbon fiber can be assessed via the TG spectra values. These results were consistent with the previous FT-IR research results.

3.2. Surface chemical elemental composition

XPS spectra show changes in the binding energy of the ejected photoelectrons, which corresponds with changes in the carbon

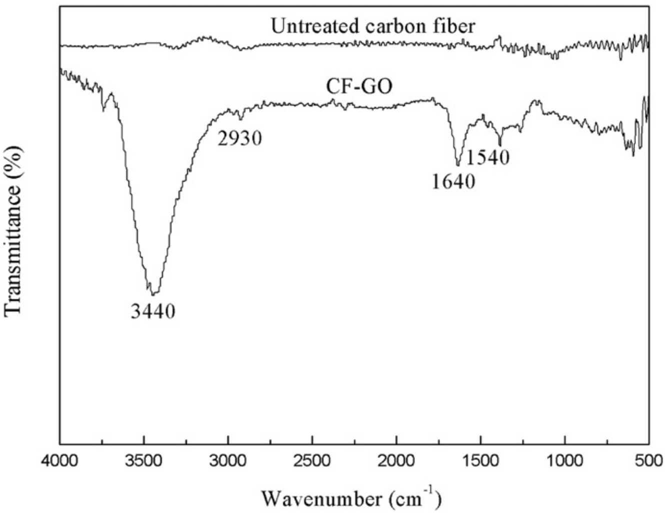

Fig. 2. FT-IR spectra of untreated and grafted carbon fiber.

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