



Effects of degree of chemical interaction between carbon fibers and surface sizing on interfacial properties of epoxy composites

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ARTICLE INFO

Keywords:

- A. Carbon fibers
- A. Polymer-matrix composites (PMCs)
- B. Surface treatments
- B. Interphase
- B. Interfacial strength

ABSTRACT

This paper offers a simple approach to increase the interfacial bonding strength of carbon fiber/epoxy composites by optimizing the degree of chemical interaction among functional groups of fiber surface, sizing agent and resin via heat treatments on carbon fibers. 130 °C treatment on CF1 and 150 °C treatment on CF2 results respectively 71.1% and 15.1% improvements on interfacial shear strength (IFSS), as compared with untreated CF1 and CF2 composites. Variations of IFSS for heat treated carbon fibers correspond well to dispersion component to polar component (γ^d/γ^p) tendency of desized heat treated fibers. The work of adhesion between heat treated fibers and resin matrix only reveals close correlation with IFSS trend for CF1/epoxy composites, while not applicable for CF2/epoxy composites. With increasing heat treatment temperature, the decreasing γ^d/γ^p of desized fibers indicates increasing degree of chemical interaction between fibers and surface sizing, which was also proven by the results of Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy. High degree of chemical interaction between fibers and surface sizing is adverse to the interaction between sizing and resin matrix, and thus lower the IFSS.

1. Introduction

Carbon fibers are increasingly used in various industries of aerospace, energy and transportation due to their outstanding features, such as high specific strength and modulus, environmental stability, heat and oxidation resistance [1,2]. However, as carbon fibers are brittle, fuzzing and filament breakage usually arise during their manufacturing process. In addition, since the pristine surface of carbon fiber is non-polar and has highly crystallized graphitic basal planes, the interfacial adhesion between carbon fibers and resin matrix is weak, which in turn negatively affects the overall performance of composites [3–5]. Therefore, numerous methods [6–8], including oxidation, chemical grafting, plasma treatment, high-energy irradiation and self-assembly, have been proposed to activate the fiber surface and thus improve the interfacial adhesion of the resulting composites.

During manufacture process of commercial carbon fibers, they are almost invariably coated with a polymer based sizing agent of about 100 nm thick for the original purpose to protect fibers from damage and enhance the handleability [9]. Subsequently, sizing agent has been proved to be effective in improving the fiber-matrix adhesion [10–12], and the main reasons can be categorized into three aspects: (1)

enhancing the content of oxygen-containing functional groups on fiber surface and thus promoting the chemical reactions between fiber and matrix; (2) increasing the mechanical interlocking between fiber and matrix; (3) improving the wettability of fiber with matrix. Recently, incorporation of nanoparticles in sizing agent, such as graphene oxide [13], carbon nanotube [14], silicon dioxide [15], zirconium dioxide [16] and titanium oxide [17], to improve the interfacial adhesion between fiber and matrix has become a subject craze. Enhancement mechanisms by adding nanoparticles are: (1) a stiffer interphase with a higher shear modulus is formed, which promotes the stress transfer from the matrix to the fiber; (2) the presence of nanoparticles in the interphase assists in holding back excessive stress spreading in the flaw and provides a crack deflection mechanism; (3) chemical interaction among fiber, sizing and resin matrix can be improved when the nanoparticles are modified with surface modifier. Although advances in interfacial improvement have been made by sizing treatment on carbon fibers, the exact role of sizing on the formation of interphase, especially the effects of the degree of chemical interaction between fibers and sizing on the level of interfacial adhesion have not been clearly understood.

Before sizing, carbon fibers are electrolytically oxidized to create

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oxygen-containing functional groups (i.e. $-\text{COOH}$, $-\text{OH}$, $-\text{C}=\text{O}$) on fiber surface to promote the chemical bonds at the fiber/sizing interface, which could further influence the interaction of sizing with resin matrix. Kafi A. et al. [18] correlated the interlaminar shear strength with the surface properties of carbon fibers collected after three stages of production, namely carbonization, electrolytic oxidation and epoxy sizing. Ma et al. [19] studied the impacts of anodic oxidation and sizing treatment on the surface properties of carbon fiber and the interfacial bonding strength of composites. Guo et al. [20] discussed the effects of physicochemical interaction between carbon fibers and sizing agent on tensile strength of carbon fibers. Dai et al. [21] investigated the chemical reactions between carbon fibers and surface sizing during the curing process. However, there are few published papers focusing on the effects of the degree of chemical interaction between fibers and sizing on the chemical interaction at sizing/matrix interface and the fiber-matrix adhesion. Considering irreversible interaction between sizing and active functional groups on surface of carbon fibers, understanding these effects is favorable for composite preparation and interphase optimization.

In this work, referring to the curing process of epoxy matrix, different heat treatment temperatures on carbon fibers were carried out to cause different degree of chemical reactions between fibers and sizing. Its effects on interfacial adhesion of the resulting composites were investigated. The degree of chemical interaction between fibers and sizing with increasing temperature were studied by Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and dynamic contact angle test (DCAT). Meanwhile, the chemical interaction between sizing and resin was also discussed. Proper heat treatment temperature on carbon fibers before they compositing with resin is in favor of improving the interfacial adhesion, which may provide a simple and promising alternative to prepare high performance polymer composites.

2. Experimental

2.1. Materials

Two Toray T800H carbon fibers (12 K, 1.81 g/cm^3), namely CF1 and CF2, coated with different sizing agents were used in this work. The average diameters of the two fibers are $5.5 \mu\text{m}$. CF1 and CF2 were refluxed in acetone at 80°C for 24 h to remove the original sizing by Soxhlet method. The obtained fibers were designated as D-CF1 and D-CF2, and the obtained sizing agents were marked as CF1-sizing and CF2-sizing. FTIR spectra of extracted sizing agents were presented in Fig. 1. Both two sizing agents are epoxy type due to the characteristic

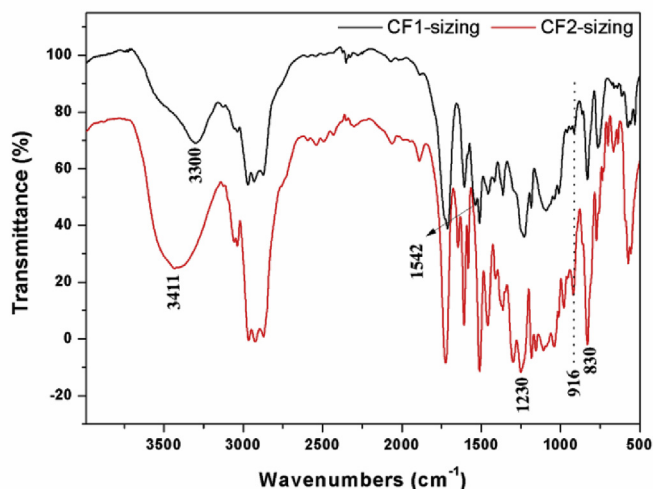


Fig. 1. FTIR spectra of CF1-sizing and CF2-sizing.

vibration peaks of epoxide group at about 1230 cm^{-1} , 916 cm^{-1} and 830 cm^{-1} , while the differences lie in the asymmetric stretching and bending vibrations of amino group ($-\text{NH}_2$) at approximately 3300 cm^{-1} and 1542 cm^{-1} [22,23] for CF1-sizing only. This may greatly affect the chemical interaction among fibers, sizing agent and polymer matrix. The band at 3411 cm^{-1} of CF2-sizing is ascribed to the stretching vibration of hydroxyl group ($-\text{OH}$).

The high-temperature curing epoxy resin (5228), with tensile strength of 86 MPa , tensile modulus of 3.5 GPa and density of 1.26 g/cm^3 , were supplied by Beijing Institute of Aeronautical Materials, China. Acetone, diiodomethane and formamide were analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd.

2.2. Heat treatment

Refer to the standard curing cycle of 5228 epoxy resin, that is 130°C for 1 h, 180°C for 2 h and 190°C for 3 h, both two fibers were treated at respectively 130°C , 150°C , 180°C and 200°C for 2 h. CF1s (or CF2s) after heat treatment were designated as CF1(2)-130, CF1(2)-150, CF1(2)-180 and CF1(2)-200, respectively. Correspondingly, the sizing agents from heat treated fibers were labeled as CF1(2)-130-sizing, CF1(2)-150-sizing, CF1(2)-180-sizing and CF1(2)-200-sizing. After sizing extraction, the fibers were marked as D-CF1(2)-130, D-CF1(2)-150, D-CF1(2)-180 and D-CF1(2)-200. For comparison, CF1-sizing and CF2-sizing were also experienced the same heat treatment temperatures at 130°C , 150°C , 180°C and 200°C for 2 h, and marked as CF1(2)-sizing-130, CF1(2)-sizing-150, CF1(2)-sizing-180 and CF1(2)-sizing-200, respectively.

2.3. Characterization

2.3.1. Analysis of chemical interaction

Functional groups of sizing and mixture of sizing/matrix were evaluated by VECTOR-22 FTIR spectroscopy (Bruker, Germany) using powder pressed KBr pellets. FTIR spectra were acquired in the wavenumber range of $400\text{--}4000 \text{ cm}^{-1}$ with the resolution of 4 cm^{-1} . Chemical interaction was estimated by concentration differences of the functional groups before and after heat treatment. The relative concentration of typical functional group (C_x) could be calculated by $C_x = I_x/I_{\text{reference}}$ [24,25], where I_x is the peak intensity of functional group x, and $I_{\text{reference}}$ is the peak intensity of the benzene ring vibration at 1610 cm^{-1} .

XPS (AXIS Supra, Kratos, Britain) was carried out to study the chemical composition of fiber surface with a monochromatic $\text{Al K}\alpha$ X-ray source at 120 W ($15 \text{ kV} \times 8 \text{ mA}$). Samples were analyzed at a take-off angle of 90° and at a base pressure of less than 10^{-8} Torr. Peak binding energies were calibrated upon $\text{C}1s$ peak at 284.6 eV . The integral background of $\text{C}1s$ spectra was deducted by XPSPEAK software and the $\text{C}1s$ spectra were fitted according to Gaussian-Lorentzian function with gaussian content of 70%.

Advancing contact angles between fibers and reference liquids (water, diiodomethane and formamide) were measured using a dynamic contact angle and surface/interfacial tension instrument (DCAT21, Dataphysics, Germany) at an immersion speed of 0.01 mm/s . The surface detection threshold is 0.15 mg and immersion depth is 3 mm . The experiment was tested with eight monofilaments at the same time, and each test was repeated five times to get an average value. The surface energy (γ), including polar γ^p and dispersive γ^d components, were calculated based on OWRK method [26]. The work of adhesion (W_{ad}) between sizing and matrix was estimated based on the Young-Dupré equation [27,28] and Fowke's theory [29].

2.3.2. Interfacial shear strength

The interfacial shear strength (IFSS) of carbon fiber/5228 composites was measured by micro-debonding method on MODEL HM410 device (Japan's East Wing Industrial) with moving speed of 0.03 mm/

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