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Effects of surface characteristics of F-12 polymeric fibers on the mechanical properties of F-12/epoxy composites

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Abstract

The effects of the surface properties and skin-core structure of F-12 high-performance polymeric fibers on the mechanical properties of F-12 fiber-reinforced epoxy composite were investigated. Atomic force microscopy, scanning electron microscopy and X-ray photoemission spectroscopy were employed to characterize the microstructure and surface-elemental analysis of the F-12 fiber. Mechanical properties of the composite, such as interfacial shear strength, interlaminar shear strength, flexural modulus and flexural strength were tested. The results clearly show the skin-core structure and surface properties of F-12 fiber have great effects on the water absorption rate and mechanical performances of the composites.

Keywords: F-12 fiber; Interfacial bonding; Surface characteristics; Mechanical property

1. Introduction

In the recent years, the high-performance polymeric fiber 'F-12 fiber' has become an important reinforcing material because of its extremely high strength, modulus and good high temperature resistance [1,2]. However, it is well known that the good structure and surface properties of fibers are crucial to ensure good mechanical performances due to the existence of physical or secondary interaction, i.e., van der Waals attraction and hydrogen bond, at the interfaces between fibers and matrix. Moreover, the degree of adhesion at the interfaces is vital to the strength and durability of the composites [3,4]. Up to now, it is not clearly understood how the surface characteristics and microstructure of F-12 fiber affect the mechanical properties of F-12 fiber/epoxy composites. It is nevertheless essential that the mechanical properties and environmental resistance of the composite can be controlled and improved [5–7].

In this paper, the effects of the surface properties and microstructure of F-12 fibers, which are characterized by scanning electronic microscopy (SEM), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS), on the mechanical properties and environmental resistance of F-12 fiber/epoxy composite are investigated.

2. Experimental

2.1. Material preparation

All the starting materials in this work were obtained from commercial sources and used as received. The F-12 fiber is a kind of polyamide fiber with high performances. The matrix is a middle-curing-temperature resin system comprised of multifunctional epoxy resin and a curing agent of the aromatic amine type. Unidirectional laminates were wound by laying up 18 plies of each prepreg with a fiber volume fraction of 61–65%. Laminates were cured at 90 °C for 2 h, 120 °C for 3 h and 150 °C for 4 h. After being cured, laminates were cut along 0° direction into a shape adequate for mechanical tests.

2.2. Characterization

Surface chemical analysis of F-12 fiber was carried out on a PHI5400 instrument (Perkin Elmer Co.) using Al Kα X-rays at a power of 200 W and in a residual vacuum of 10^{-7} mbar. Measurements were made with the analyzer in fixed transmission

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mode and normal to the plane of the sample. An analyzer passing energy of 100 eV was used to collect broad scan spectra (0–1100 eV).

The morphologies of F-12 fibers were examined by SEM (Model JSM-5800 instrument, Japan Electron Co.) at 15 kV with gold plating. A MultiMode atomic force microscope SPM-9500J3 (SHIMADZU LIMITED) was used to investigate the surface topography of the F-12 fibers.

The debonding strength between a F-12 fiber and epoxy resin was evaluated from the interfacial shear strength (IFSS) using a micro-debonding test. The test was performed with an in-house made micro-debonding machine using a stereoscope light microscope, which allows visual observation of the sample during the testing procedure. The force was measured to calculate the IFSS when the resin ball was pulled out from the filament.

The interlaminar shear strength (ILSS) was measured by the short beam method according to a Chinese National Standard GB2791-81. The composites were machined along the fiber direction into 20 mm × 6 mm short beam-shear specimens with 3 mm thickness. The crosshead speed was 10 mm/min. More than five specimens on average were tested for every mechanical datum.

Flexural properties were determined in a three-point bending test according to a Chinese National Standard GB1449-83. For each material, five specimens with a length of 80 mm and width of 15 mm were tested. The support span was 64 mm and the crosshead speed was 2 mm/min. Deflection was measured as crosshead traveled. All tests were performed in an Instron machine 4505.

2.3. Moisture absorption test

Moisture uptake was determined by placing samples in a constant humid-heat instrument (Model SH05N, Chongqin Instrument, China) at 35 °C–80%RH for 400 h, 700 h and measuring the weight periodically. Prior to absorption experiments, the samples were dried in a vacuum oven at 70 °C until the weight was constant.

3. Results and discussion

3.1. Surface characteristics of F-12 fiber

X-ray photoelectron spectroscopy, also known as ESCA, is a sensitive analytical method of the investigation of polymer surface, thus indicating the composition of functionalities on the fiber surface [8,9]. Table 1 shows the XPS data of the F-12 fiber. It can be seen that the atomic ratio of N to C is as high as 0.038, suggesting that large numbers of polar functional groups containing nitrogen exist on the surface of a F-12 fiber, beneficial to the interfacial strength between fiber and matrix. The results are in good agreement with the high-resolution N1s spectra given in Fig. 1. High-energy resolution studies of the N1s peak envelopes have been used to identify the types of nitrogen functional groups [10]. As shown in Fig. 1, the N1s peak is quite broad (3.0 eV width at half max), which indicates that nitrogen on the surface is bound in more than one binding state. The N1s peak is composed of three sharp peaks corresponding to carbon nitride groups (C–N; 397.4 eV), amide groups (–NH_2; 399.3 eV) and also aromatic imines groups (Ar–NH; 401.1 eV) in Fig. 1. It indicates that the concentration of amine groups on the F-12 fiber's surface is very high.

Table 1
XPS data of F-12 fiber

Elementary composition (%)					Atomic ratio	
C	N	O	Si	P	O/C	N/C
76.6	2.9	17.4	2.6	0.5	0.23	0.038

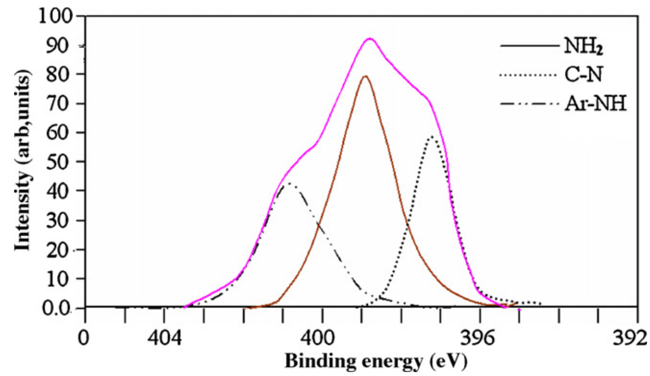

Fig. 1. High-resolution XPS N1s photo peaks of F-12 fabrics.

The SEM micrograph and AFM image of a F-12 fiber in Fig. 2 show that the surface of the F-12 fiber is very rough, including many grooves and protuberances. The morphology favours strong interfacial bonding between matrix and fibers.

3.2. Mechanical properties of F-12/epoxy composite before and after environmental exposure

It has been reported that, because absorbed moisture will weaken the interface bonding strength, the mechanical performances of fiber-reinforced polymer-based composite decrease as the moisture absorption rate of the composite increases [11,12]. The moisture absorption content of F-12 fiber-reinforced epoxy resin composites is plotted as a function of the square root of time in Fig. 3.

It can be seen from Fig. 3 that a nonlinear curve exists for the composite, and that it deviates from Fick's second law. The result is consistent with a wicking mechanism. The moisture content gradually increases due to the diffusion of moisture into the matrix and interphase via wicking, and causes the combined absorption curve to be nonlinear.

The moisture absorption has a great effect on the mechanical properties of fiber-reinforced polymer-based composites [13,14]. The changes of IFSS, ILSS, flexural modulus and flexural strength on moisture absorption are shown in Table 2. Before the moisture is absorbed, the IFSS and ILSS of the F-12