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Effects of carbon fiber surface characteristics on interfacial bonding of epoxy resin composite subjected to hygrothermal treatments

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ABSTRACT

The changes of interfacial bonding of three types of carbon fibers/epoxy resin composite as well as their corresponding desized carbon fiber composites subjecting to hygrothermal conditions were investigated by means of single fiber fragmentation test. The interfacial fracture energy was obtained to evaluate the interfacial bonding before and after boiling water aging. The surface characteristics of the studied carbon fiber were characterized using X-ray photoelectron spectroscopy. The effects of activated carbon atoms and silicon element at carbon fiber surface on the interfacial hygrothermal resistance were further discussed. The results show that the three carbon fiber composites with the same resin matrix possess different hygrothermal resistances of interface and the interfacial fracture energy after water aging can not recovery to the level of raw dry sample (irreversible changes) for the carbon fiber composites containing silicon. Furthermore, the activated carbon atoms have little impact on the interfacial hygrothermal resistance. The irreversible variations of interfacial bonding and the differences among different carbon fiber composites are attributed to the silicon element on the carbon fiber bodies, which might result in hydrolyzation in boiling water treatment and degrade interfacial hygrothermal resistance.

1. Introduction

Fiber reinforced polymer matrix composites are widely used as aircraft structure materials due to their superior mechanical properties and low density. For the safety of long-term service, their responses to environmental exposure attract much attention. The moisture absorption in polymer composites exposing to hot/wet conditions would result in the degradation of composite mechanical properties, especially fiber–matrix interface-dominated properties. And the fiber–matrix interphase region plays an important role in resisting enviro-mechanical loads [1].

Carbon fiber surface properties significantly affect interfacial adhesion and mechanical performances of composites. The surface chemical properties depend greatly on the chemical structure of carbon fiber sizing, which plays an important role in the formation of interphase through interacting with resin matrix [2–4]. Different sizing agents may result in distinguished surface properties of carbon fiber and affect interfacial adhesion. Most works focused on the effects of carbon fiber surface property on interfacial adhesion in dry condition [5–9]. Due to the importance of interfacial bonding after water aging, it is essential to study the influence of carbon fiber surface characteristics on interfacial hygrothermal resistance.

Macroscopic mechanical approaches, such as transverse tensile test [10], flexural test [11,12] and interlaminar shear test [11,13–15], are often employed to evaluate the fiber–matrix interface related performances in dry and hygrothermal environments. However, the characterizations in laminate tests are significantly influenced by the matrix properties, and the interfacial bonding property and the mechanical properties of matrix are difficult in distinguishing. In recent decades, a series of microscopic single-fiber specimen tests are developed, which can obtain fundamental mechanical information of interphase, including interfacial strength, toughness and load transfer efficiency. These microscopic methods can be used to understand macroscopic mechanical behavior of composite.

In single fiber specimen methods, single fiber fragmentation test (SFFT) is commonly adopted to research hygrothermal effects (water aging) on the interfacial adhesion of composite materials. In this test, bulk matrix sample containing fiber filament is used and hygrothermal effect is easily applied on the sample [5]. For SFFT, fiber/matrix adhesion is quantified by interfacial shear strength derived from typical stress-based model (Kelly–Tyson model) [16]. In addition, SFFT can give interfacial toughness, which is an important parameter to assess the crack-expand resistance at the interphase region. Wagner et al. [17,18] introduced an energy-based model (called WND model) to characterize the interfacial

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Table 1
Mechanical property of different carbon fibers.

Parameters	T300	CF-1	CF-2
Tensile strength (MPa)	3530	3800	3750
Young's modulus (GPa)	230	250	230
Poisson's ratio	0.2	0.2	0.2
Fracture energy (J/m^2)	10	10	10
Fiber radius (μm)	3.5	3.5	3.5
Thermal expansion coefficient (10^{-6}/°C)	−0.5	0	0

toughness in SFFT. Based on this model, the water degradation of interfacial toughness of glass fiber/vinylester composite was studied by Ramirez and Carlsson [19,20]. However, there are few works on the water degradation of interfacial bonding in carbon fiber composite by means of SFFT, and the effect of carbon fiber surface on the interfacial hygrothermal resistance is still not understood.

In our previous work, the energy-based model with SFFT was modified to characterize the interfacial toughness of T300 carbon fiber/epoxy resin under hygrothermal treatment [21]. In this paper, the same method was used to investigate the changes of interfacial toughness of different carbon fiber/epoxy resin composites under hygrothermal condition. To find out the key factors affecting the interfacial hygrothermal resistance, the fiber surface element compositions, surface activity and sizing agent were characterized and analyzed. The effects of fiber surface characteristic on the interfacial hygrothermal resistance were verified by means of changing carbon fiber surface characteristics.

2. Experimental

2.1. Materials

Three types of polyacrylonitrile (PAN) based carbon fibers were used as the reinforcement in single-fiber composite, including T300 carbon fiber (Toray Inc.), CF-1 and CF-2 carbon fibers supplied by the Beijing Institute of Aeronautical Materials (BIAM). The mechanical properties of these carbon fibers are listed in Table 1. To investigate the carbon surface characteristics, the desizing treatment (removing the sizing agent on carbon fibers) was performed by means of Soxhlet extraction using acetone solvent at 75 °C. The corresponding desized carbon fibers were named DT300, DCF-1 and DCF-2. The matrix for the single-fiber specimens was a kind of epoxy resin, named 5228 supplied by BIAM. The cure cycle for this epoxy resin was 1 h at 130 °C, 2 h at 180 °C, and 3 h at 190 °C. In order to analyze the effect of carbon fiber sizing on the hygrothermal resistance of interphase, the mixture of extracted carbon fiber sizing and epoxy resin 5228 with the ratio of 1:1 was prepared, and then was cured with the same cure schedule for pure 5228 epoxy resin. The blend was used to simulate the component in the interphase region of 5228 matrix composite.

2.2. Carbon fiber treatment

In order to change the surface chemical property, T300 carbon fiber bundle was heat treated in a vacuum chamber. Two kinds of temperature cycles were adopted, including 2 h at 150 °C followed 2 h at 180 °C heat treatment and 2 h at 150 °C, 2 h at 180 °C accompanied with 4 h at 200 °C. The heating rate was 2 °C/min for the two processes. The same method was used in our previous work for studying the chemical change of carbon fiber sizing under high temperature [9].

Silane coupling agent KH550 (Gaizhou Chemical industry Co., Ltd.) was coated on the surface of desized T300 carbon fiber (DT300) to investigate the effect of silicon element on the hygrothermal resisting of interface. A single desized T300 fiber was drawn through a liquid drop of KH550 and dried in an oven at 70 °C for 12 h. The molecular structure of KH550 is shown in Fig. 1.

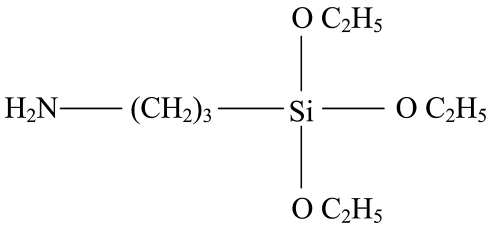

Fig. 1. Molecular structure of KH550.

The treated T300 carbon fibers were used to prepare SFFT specimens and the corresponding interfacial fracture energy was obtained.

2.3. Characterization

X-ray photoelectron spectroscopy (XPS) was employed to evaluate the chemical properties on the studied carbon fiber surfaces. A Physical Electronics ESCALAB 250 system (ThermoFisher Scientific) with a concentric hemispherical analyzer and a monochromatic Al *K*a X-ray source (1486.6 eV) was operated in an evacuated chamber at approximately 5.0×10^{-9} mbar. An electron take-off angle of 45° with respect to the sample plane was employed. In all measurements 150 eV of pass energy for survey scan and 30 eV for high resolution scan were used. A seven-parameter curve fitting was conducted for the C1s spectra by taking 284.6 eV as the reference peak. The three studied carbon fibers, their responding desized carbon fibers and thermal-treated T300 carbon fibers were analyzed using this technique.

A Nicolet 560 Fourier transform infrared (FTIR) spectroscopy (Thermo Nicolet) was used to examine the chemical reactions of 5228 resin and the mixture of sizing agent and 5228 resin before and after the treatment of boiling water immersion for 3 days. The thickness of samples was very small (less than 0.2 mm), so the water contents of samples reached saturated state within 3-d hygrothermal treatment. Spectra were obtained in an optical range of 400–4000 cm^{-1}. The hygrothermal treated samples were dried in an oven at 70 °C 3 d before testing.

2.4. SFFT procedure

Cured 5228 resin is not enough ductile (low elongation) for ensuring the saturation state in the measurement of interfacial shear strength during SFFT. However, the information of interfacial debonding at first fiber break can be obtained for this resin in SFFT, which was used to calculate interfacial fracture energy (Γ_i). Γ_i indicates interfacial toughness and the bonding between fiber and resin matrix. The calculations of Γ_i are shown as follows:

$$\Gamma_i = \frac{\sigma_{f\infty}^2 r_f}{2E_f L_d}\left[\frac{L_d}{2} + \frac{1}{\beta} - \frac{\beta E_f r_f^2}{16 G_f}\right] - \frac{r_f \Gamma_f}{2L_d} \quad (1)$$

$$\beta = \frac{1}{r_f}\sqrt{\frac{2G_m}{E_f \ln(r_m/r_f)}} \quad (2)$$

where E_f is the Young's modulus of the fiber, $\sigma_{f\infty}$ is the far field fiber stress, L_d is the length of the interfacial debonding zone, Γ_f is the fiber fracture energy, r_f is the radius of the fiber, r_m is the effective radius of the matrix, G_f and G_m are the shear modulus of the fiber and matrix, respectively. The shear modulus G of the materials is equal to $E/2(1+\mu)$, where μ is the Poisson's ratio and E is the Young's modulus of the materials.