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Short communication: test equipment

An improved microbond test method for determination of the interfacial shear strength between carbon fibers and epoxy resin

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ABSTRACT

An improved microbond method, with a corresponding testing device, was developed to measure the interfacial shear strength (IFSS) between carbon fibers and epoxy resin. Compared to other methods, this proposed approach is both highly efficient and easy to operate. As a case study for this new method, we measured the IFSS between carbon fibers and epoxy resin. Although the average IFSS obtained was only 7.08 MPa, which is much lower than values documented in several previous studies, the displacement-load curves demonstrate the strong reliability of this method. The lower IFSS could be explained by the highly inert surface of the carbon fibers, which was highly graphitized and had no sizing treatment. Therefore, this method has high potential in applications for screening the sizing agents of carbon fibers or optimizing the surface sizing processes.

1. Introduction

Due to their high modulus and strength, light weight, good thermal stability and corrosion resistance, carbon fiber reinforced epoxy composites (CFREC) have been widely used in aviation, automotive and other high-end industrial fields [1]. It is well known that the interfacial properties in a CFREC play a significant role in almost all the mechanical properties of the composite [2,3]. Furthermore, the inherent weak interfacial bonding between carbon fibers and epoxy in the composite must be improved through various modification methods, such as oxidization and grafting of chemically active polar groups onto the fiber surface. Therefore, improved knowledge of the bond strength between carbon fibers and matrix, as well as the development of a highly efficient characterization method, are of great importance for ensuring the optimal design of composites.

In order to experimentally measure and evaluate the interfacial properties of fiber-reinforced composites, various macro- and micro-mechanical test methods, such as the fragmentation test [4,5], pull-out test [6–8], push-out test [9] and microbond test [10,11] have been developed and used for steel fiber, glass fiber, carbon fiber and ceramic fiber reinforced polymer composites. Due to its simplicity and versatility, the microbond test, based on the well-known pull-out methodology, has been one of the most widely used methods to measure the interfacial shear strength (IFSS) between reinforcing fibers and polymers [10–14]. In a traditional microbond test, two blades are arranged so that an individual reinforcing fiber can be pulled out from a droplet of matrix material. Several key parameters, including embedded fiber length, fiber diameter and the debonding peak force are measured, with IFSS then calculated using the following equation:

$$IFSS = \frac{F}{d\pi L} \tag{1}$$

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where F is the pull-out force (N); d is the fiber diameter (μm); and L is the embedded fiber length (μm) in the matrix.

Shelestova et al. [15] employed the microbond test to measure the IFSS between polytetrafluoroethylene(PTFE) and carbon fiber, thus evaluating the effect of the fluoropolymer coating on the bonding strength. Using the same method, Zhang et al. [16] concluded that dispersing 5wt% graphene oxide sheets in the fiber sizing onto the surface of individual carbon fibers significantly enhanced the IFSS between the carbon fiber and epoxy in composites. However, as the traditional microbond test is not instrumented and time-consuming, it has not been applied extensively. Some efforts have been made to improve its efficiency and reliability. For example, Morlin et al. [17] proposed a cylinder test as a modification of the traditional microbond method to overcome to some extent the uncertainty in calculating IFSS due to the droplet shape, blade angle and the operational procedures of this method.

Therefore, the aim of this paper is to present an improved microbond test method that has higher accuracy and speed than traditional approaches. The method uses a custom-built microtensile device, which was initially developed to measure the mechanical properties of single short natural plant fibers, such as bamboo or wood fibers [18]. This new method can quickly and quantitatively measure the IFSS between carbon fibers and epoxy resin, enabling researchers to rapidly screen surface sizing agents or sizing processes in carbon fibers. This has the potential to significantly improve mechanical properties of CFREC.

2. Experimental

2.1. Materials

The Materials College of Hunan University provided the carbon fibers used in this paper. These fibers were highly graphitized, with a carbon content of more than 99%. The tensile strength and elastic modulus of the carbon fibers were as high as 2 GPa and 400 GPa, respectively. No sizing treatment was applied to the fibers. A fast-curing two-component epoxy resin (HY-914), consisting of epoxy resin and amine curing agent, with a weight ratio of 5 to 1, was supplied by Tianjin Yan Hai Chemical Co. Ltd.

2.2. Sample preparation

Under a microscope, carbon fibers with minimal damage were carefully selected and placed across a 2 mm wide gap in an organic glass panel. Using super-fine tweezers, two epoxy droplets of approximately 300 μm diameter were then placed on each fiber, with an approximate spacing of 1 mm (Fig. 1). The epoxy resin was allowed to solidify at room conditions for 24 h to reach full strength.

2.3. Experimental apparatus

An overview of the custom-built microtensile device is shown in Fig. 2A. A special fiber gripping system (Fig. 2B) was developed and combined with a small high-resolution commercial mechanical tester (Microtester 5848, Instron,

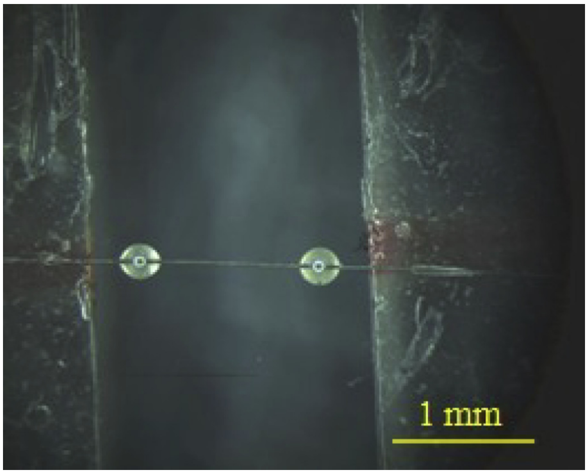

Fig. 1. The prepared sample under a stereo microscope.

USA). The capacity of the load cell used was 5 N. This device was initially developed to measure the mechanical properties of single short plant fibers. The key part of the fiber gripping system is a pair of innovative fiber clamps that can effectively grasp the epoxy droplets at the ends of fibers during tension. The fiber clamps were designed to deal with fibers as short as 1.0 mm in length [18–20]. Fig. 2C gives a detailed schematic representation of the fiber clamps. A high-resolution laser cutting technique was adopted to produce slots ranging from 100 to 120 μm in width. The angle and the length of the shorter side of the trapezoid, namely "θ" and "a", as shown in Fig. 2C, are 60° and 200 μm, respectively. Both values can be adjusted in accordance with the acceptable minimum distance allowed between the two epoxy droplets and the diameter of the fibers.

A microscopic imaging system, consisting of a vertical microscope and a horizontal microscope, was used to observe the full process of microbond pull-out, as well as record relevant images to calculate the fiber diameter and the embedded fiber length in the matrix.

2.4. Experimental procedure

A well-prepared sample, as indicated in Fig. 1, was installed in the fiber clamps with the aid of the vertical microscope. The fiber was then pre-tensioned at 5 mN. Due to the small size of the sample, it is often difficult to place a fiber with its axis perfectly aligned in the tensile direction. Therefore, an integrated 3D positioning stage with a fine tuning system (Fig. 2B) was used to ensure the tensile direction aligned exactly with the fiber axis. Before testing, the initial image (Fig. 3A) was taken using the vertical microscope (Fig. 2B). All pull-out tests were performed with a constant crosshead movement rate of 0.048 mm/min and under an environment of 22.5 °C and 15% relative humidity (RH). During the test, force-displacement curves were recorded and a second image was simultaneously taken, as shown in Fig. 3B. From each curve, the debonding peak force, *i.e.* F, was recorded. When the pull-off process was finished, a