Letter

Load transfer from fiber to polymer matrix, studied by measuring the apparent elastic modulus of carbon fiber embedded in epoxy

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Abstract—Load transfer from a single carbon fiber to the surrounding epoxy matrix was studied by measuring the apparent tensile modulus of the fiber while the fiber was embedded in epoxy and comparing the apparent modulus (1650 GPa) with the real modulus (230 GPa). Thus, it was found that 87% of the tensile load applied to the fiber was transferred to the epoxy.

Keywords: Composites; polymer; carbon fiber; modulus; electrical resistance; epoxy.

1. INTRODUCTION

The interfacial shear strength (IFSS) between fiber and matrix in composite materials is one of the critical factors that control the effectiveness of the fiber as a reinforcement [1]. If IFSS is too low, the performance of reinforcing fibers may not be reflected in the composites. On the other hand, if IFSS is too high, an external stress can be transferred easily to the interphase in the composite material. Several micromechanical techniques have been proposed for measuring the interfacial properties in various fiber reinforced composites [2–7]. The IFSS can be assessed conventionally by single-fiber pullout testing [8–12], single-fiber fragmentation testing [13–18], microindentation testing [19], off-axis tensile testing [20] and related methods, which provide information on the interfacial properties [7, 20]. The single-fiber pullout test has been used because it can measure IFSS between the fiber and matrix directly and simply. It is also not limited by the material properties of the fiber or matrix, though it suffers from data scattering. The single-fiber fragmentation testing can provide statistical information as well as the interfacial failure

modes and IFSS, using only several specimens. Although the statistical analysis is rather complex, the configuration of single-fiber fragmentation testing is quite similar to that of a real composite system.

Measurement of the elastic modulus of a unidirectional composite in the fiber direction and comparing the measured value to the calculated value obtained by using the Rule of Mixtures in principle provides information on the extent of load transfer. However, in practice, this is difficult because the fibers are not perfectly straight and the imperfect arrangement diminishes the measured modulus of the composite.

This paper provides a method for directly measuring the load transfer from fiber to matrix. This method involves measuring the apparent modulus of a single fiber under tension while the fiber is embedded in the matrix. The tension is applied to the fiber, which protrudes at both ends from the matrix for the purpose of load application.

In a continuous fiber composite, the fibers usually carry most of the load, since the modulus of the fibers is much higher than that of the matrix. As a consequence the term 'load transfer' is conventionally used to refer to that from matrix to fiber [21]. However, in this paper, load transfer from fiber to matrix (i.e. fraction of the load applied to the fiber that is transferred to the matrix) is addressed. The stronger the bond between fiber and matrix, the more effective is the load transfer in either direction.

In order to help distinguish elastic deformation from plastic deformation and damage, the method of this paper is supplemented by measurement of the electrical resistance of the fiber during the deformation. As the fiber is conducting and the matrix is insulating, resistance measurement provides information that is focused on the fiber. Dimensional changes during elastic deformation are known to cause a reversible change in the resistance, such that the fractional change in resistance per unit strain is 2 [22]. In contrast, plastic deformation and damage are associated with at least partial irreversibility of the resistance change and, due to the change in resistivity, the fractional change in resistance exceeds 2.

The reversibility in strain also indicates elastic deformation, but the small strain during the deformation of a carbon fiber makes the strain a less sensitive indicator of the nature of the deformation than the resistance.

This work is a continuation of earlier work [22], in which the same single fiber electromechanical testing was performed, except that the carbon fiber (same type as in this work) was bare (without any matrix material). Comparison of the results of this paper and those of Ref. [1] provides information on the effect of the matrix material. Reference [22] gives the real modulus of the fiber, whereas this paper gives the apparent modulus (much higher than the real modulus) of the fiber when the fiber is embedded in the matrix. The difference between the real and apparent moduli gives the extent of load transfer from fiber to matrix.

In another earlier paper [23], the same single carbon fiber electromechanical testing was performed with the presence of a polymer matrix, as in this work,

except that the carbon fiber was under a residual compressive stress prior to the electromechanical testing. Hence, during the electromechanical testing under tension, the residual stress was diminished, thereby decreasing the fiber resistance. In contrast, the carbon fiber was essentially not under a residual stress in this paper, which was made possible by using a different experimental condition. Due to the essential absence of residual stress in the fiber, in this paper, during electromechanical testing under tension, the resistance change was not due to the reduction in residual stress, but was due to fiber deformation.

2. EXPERIMENTAL METHODS

The carbon fiber used was 10E-Torayca T-300 (unsized, PAN-based), of diameter 7 μm, density 1.76 g/cm^3, tensile modulus 221 $\pm$ 4 GPa, tensile strength 3.1 $\pm$ 0.2 GPa and ultimate elongation 1.4%. The electrical resistivity was $(2.2 \pm 0.5) \times 10^{-3} \Omega$ cm, as measured by using the four-probe method and silver paint electrical contacts on single fibers. The epoxy used was EPON(R) resin 9405 (a bisphenol A epoxy resin modified with a reactive monomer) together with curing agent EPI-CURE 9470 (a non-MDA aromatic amine modified with a reactive monomer), both from Shell Chemical Co. (Houston, TX), in weight ratio 70:30. The recommended curing temperature is 150–180°C for this epoxy. Curing was performed in this work at 170°C, without pressure, for 2 h.

A single fiber was embedded in epoxy for a length of 80 mm, an epoxy overlay width of 4 mm in the plane of Fig. 1 and an epoxy overlay thickness of 0.5 mm on either side of the fiber in the direction perpendicular to the plane of Fig. 1, such that both ends of the fiber protruded and were bare in order to measure the apparent fiber modulus upon tension and to allow electrical contacts to be made on the fiber using silver paint. Six samples were tested for the apparent modulus without resistance measurement. Four samples were tested for the apparent modulus while the resistance was measured. For samples involving resistance measurement, four electrical contacts (labeled A, B, C and D in Fig. 1) were made. The outer two contacts (A and D) were for passing a current, whereas the inner two contacts (B and C, 85 mm apart) were for measuring the voltage. A Keithley 2001 multimeter was used for the DC electrical resistance measurement. A screw-action mechanical testing system (Sintech 2/D, Sintech, Stoughton, MA) at a crosshead speed of 0.1 mm/min was used for apparent modulus measurement. A 'blank' test was performed using an alumina strip instead of a single carbon fiber. The dimension of the alumina strip was $100 \times 10 \times 0.5$ mm. The alumina strip was chosen for the blank test due to its rigidity. Under the load that could be taken by a single carbon fiber (less than 20 g), the extension of the alumina strip was negligible. Thus the blank test provided information on the extension due to the machine. The extension between the two glue points was obtained by deducting the extension determined by the blank test from the total extension measured by the machine. There was a small portion (about 1 mm) of bare fiber between each of the two glue points and
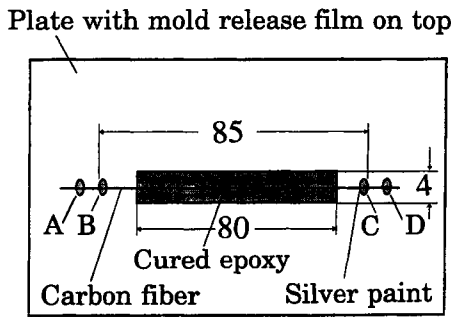
Plate with mold release film on top

Figure 1. Sketch of the resistance measurement set-up for single carbon fiber embedded in epoxy. A, B, C and D are four probes. A and D are for passing current; B and C are for voltage measurement. Dimensions are in mm.

the edge of the epoxy close to it. The extensions of these bare fiber portions were calculated by the equation

$$\Delta \ell_f = \ell_f \frac{F / \left(\frac{1}{4} \pi d_f^2 \right)}{E_f}, \tag{1}$$

where $\Delta \ell_f$ is the extension of the two bare fiber portions together, ℓ_f is the length of the two bare fiber portions together, d_f and E_f are respectively the diameter and modulus of the carbon fiber ($d_f = 7 \mu m$, $E_f = 221$ GPa), and F is the load. The extension of the part of the carbon fiber embedded in the epoxy was obtained by subtracting the extension of the bare fiber portions, calculated by using equation (1), from the extension between the two glue points. This extension, divided by the length of the part of the carbon fiber in the epoxy, gave the average strain of that part of the fiber. From this strain and the stress calculated from the load and the cross-sectional area of the fiber, we obtained the apparent tensile modulus. The maximum strain was about 70% of the failure strain.

3. RESULTS AND DISCUSSION

Measurement of the fiber resistivity before and after the curing of the epoxy surrounding the fiber showed negligible change (0.07%) in the fiber resistance after curing, indicating the essential absence of the residual stress reported in Ref. [23]. (Reference [23] reported a 10% irreversible resistivity increase after the curing cycle.)

Figure 2 shows the stress–strain curve for a fiber, as determined after curing. The strain change was reversible. The curve is linear, with a slope of 1620 GPa (the apparent modulus). The values of the apparent modulus of the ten samples tested ranged from 1440 to 1980 GPa. The average value is 1650 GPa.

Figure 3 shows the fractional change in fiber resistance and fiber strain during repeated tensile loading of the fiber of Fig. 2. The resistance and strain increased
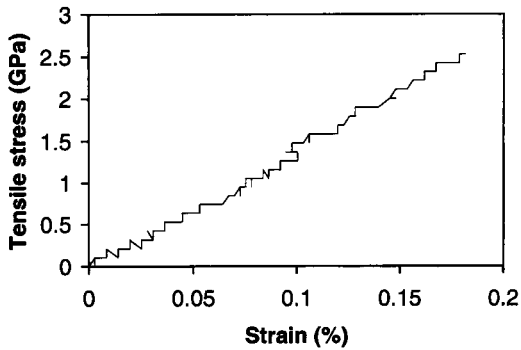
Figure 2. Tensile stress–strain curve of a single fiber embedded in epoxy except for the ends.
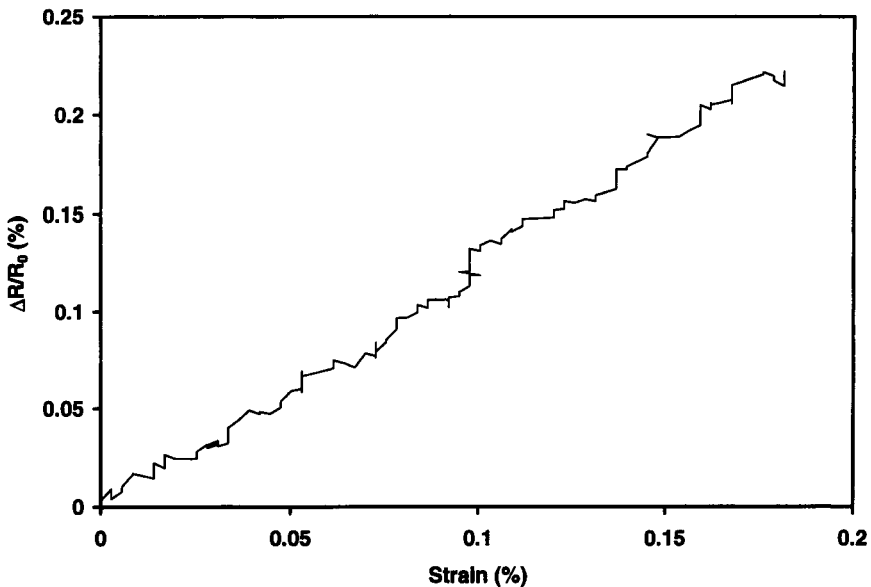

Figure 3. Tensile strain and fractional change in electrical resistance during tensile loading of the sample of Fig. 2.

reversibly upon tensile loading. The fractional change in resistance per unit strain ranged from 1.22 to 1.64 for the four samples tested. This indicates elastic deformation.

From Ref. [22], the real modulus of the fiber was 230 GPa, as measured by using the same method as this paper, except that the fiber was bare. That the apparent modulus is much higher than the real modulus is because much of the applied load was transferred from the fiber to the matrix. Comparison of the real modulus and average apparent moduli shows that 87% of the load applied to the fiber was transferred to the matrix. This percentage of load transfer applies to the particular combination of interface quality and epoxy thickness used. The epoxy thickness used approached infinite thickness, relative to the fiber diameter. A decrease in

epoxy thickness was observed to decrease this percentage, since the stress in the epoxy decreases with increasing distance from the fiber. A decrease in bond strength is expected to decrease this percentage.

The method of the paper can be used for fiber–matrix interface characterization. The electrical resistance measurement in this method serves to confirm the occurrence of elastic deformation. If this confirmation is already in place, the electrical resistance measurement is not necessary for the purpose of measuring the apparent modulus. Without the electrical measurement, the method will be much simpler, as it will just involve determining the stress–strain curve.

4. CONCLUSION

Load transfer from a single carbon fiber to the surrounding epoxy matrix was studied by measuring the apparent tensile modulus of the fiber while the fiber was embedded in epoxy and comparing the apparent modulus (1650 GPa) with the real modulus (230 GPa). Thus, it was found that 87% of the tensile load applied to the fiber was transferred to the epoxy. That the tensile loading indeed caused elastic deformation of the fiber was confirmed by measuring the electrical resistance of the fiber during loading.

Acknowledgement

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