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Letter

Residual stress in carbon fiber embedded in epoxy, studied by simultaneous measurement of applied stress and electrical resistance

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Abstract—Due to the thermal contraction of the epoxy embedding a carbon fiber after curing, the fiber exhibited a compressive residual stress which caused the electrical resistivity of the fiber to increase by 10%. Subsequent application of tension to the exposed ends of the embedded fiber reduced the residual stress, thereby causing the resistivity to decrease reversibly back to its value prior to embedding. The tensile stress (1320 MPa) required for this to happen was the residual stress in the fiber direction. Excessive tensile stress caused the resistivity to increase, due to fiber damage.

Keywords: Residual stress; carbon fiber; polymer; epoxy; electrical resistance; resistivity.

1. INTRODUCTION

Due to the shrinkage of the matrix during composite fabrication and/or the thermal contraction mismatch between fiber and matrix during cooling near the end of composite fabrication, the fibers in a composite can have a residual compressive stress [1]. This stress may affect the structure of the fiber so that the fiber properties are affected, often adversely. It may also cause fiber waviness, which degrades the mechanical properties of the composite.

The measurement of the fiber residual strain by X-ray diffraction, Raman scattering and other optical techniques is difficult due to the anisotropy of the fiber strain and the necessity of embedding the fiber in the matrix. To help alleviate this problem, this paper presents a new method, which involves simultaneous electrical and mechanical measurements on the same sample under load, in contrast to the separate electrical and mechanical measurements in previous work. This electromechanical testing provides a simple and effective method for measuring the fiber residual stress along the fiber direction, as illustrated in this paper for the case of carbon fiber in epoxy. Carbon fiber epoxy-matrix composites are the most widely used form of carbon fiber composites due to the good adhesion between fiber and epoxy.

2. EXPERIMENTAL

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±4 GPa, tensile strength 3.1±0.2 GPa and ultimate elongation 1.4%. The electrical resistivity was $(2.2 \pm 0.5) \times 10^{-3}$ Ω 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 together with curing agent 9470, both from Shell Chemical Co., in weight ratio 70 : 30. The recommended curing temperature is 150–180°C for this epoxy.

The electrical resistance of a carbon fiber embedded in epoxy before and after the curing of the epoxy (at 180°C, without pressure, for 2 h), as well as during subsequent tensile loading, was measured using the sample configuration of Fig. 1. A single fiber was embedded in epoxy for a length of 60 mm and an epoxy coating thickness of 5 mm, such that both ends of the fiber protruded and were bare in order to allow electrical contacts to be made on the fiber using silver paint. Four 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, 80 mm apart) were for measuring the voltage. A Keithley 2001 multimeter was used for DC electrical measurements.

3. RESULTS AND DISCUSSION

Table 1 shows the electrical resistivity of carbon fiber before curing of epoxy and after both curing and subsequent cooling. Data for six samples are consistent, indicating that the resistivity of fiber increased by ~ 10% after curing and subsequent cooling. The fractional resistance increase was also ~ 10%.

It is known that the disparate thermal expansion properties of carbon fiber and epoxy leads to an inevitable build-up of residual thermal stress during the matrix (epoxy) solidification and subsequent cooling. Here we consider only the residual stress along

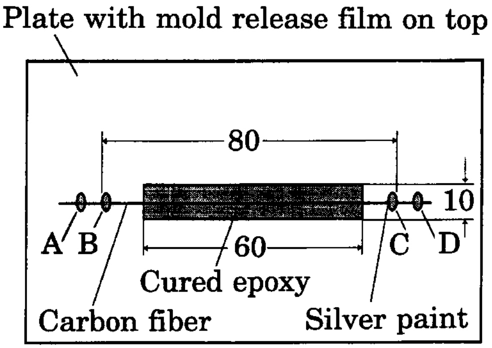

Figure 1. Sketch of the resistance measurement set-up for single carbon fiber embedded in epoxy. A, B, C and D are four probes. A and D are for passing current; B and C are for voltage measurement. Dimensions are in mm.

Table 1.
Electrical resistivity of carbon fiber before and after epoxy curing

Resistivity before curing (10^{-3} Ω cm)	Resistivity after curing (10^{-3} Ω cm)	Fractional change in resistivity
2.24	2.46	9.8%
2.43	2.69	10.5%
2.17	2.39	10.2%
2.11	2.34	11.0%
2.58	2.83	9.6%
2.36	2.61	10.7%

the fiber direction (one dimension). Since the strain of matrix and fiber is the same (if adhesion is perfect),

$$\frac{\sigma_f}{E_f} + \alpha_f \Delta T = \frac{\sigma_m}{E_m} + \alpha_m \Delta T, \tag{1}$$

where σ_f is the longitudinal residual stress built up in the fiber, σ_m is the residual stress built up in the matrix, E_f is the modulus of fiber, E_m is the modulus of matrix, α_f is the coefficient of thermal expansion of fiber, α_m is the coefficient of thermal expansion of matrix, and ΔT is the temperature change.

Since there is no external force on the specimen,

$$\sigma_f V_f + \sigma_m V_m = 0, \tag{2}$$

where V_f is the volume fraction of fiber, and V_m is the volume fraction of matrix.

Combining equations (1) and (2), we have the following equation for calculating the residual stress in the fiber.

$$\sigma_f = \frac{E_f E_m V_m (\alpha_m - \alpha_f) \Delta T}{(V_m E_m + V_f E_f)}. \tag{3}$$

In this work, $E_f = 221$ GPa, $E_m = 3.7$ GPa, $\alpha_m = 42 \times 10^{-6}$ K^{-1}, $\alpha_f = 0.09 \times 10^{-6}$ K^{-1} and $\Delta T = 155$ K. From equation (3), the residual thermal stress built up in the fiber reaches 1438 MPa. In this case of a single fiber in epoxy, the high residual stress is built up during curing and subsequent cooling. The observed resistance increase after curing and cooling is attributed to this residual stress.

Electromechanical testing of a single fiber in cured epoxy was conducted using the configuration of Fig. 1 during tension under load control, as provided by a screw-type mechanical testing system (Sintech 2/D). The crosshead speed was 0.1 mm/min. The strain was obtained from the crosshead displacement.

Figure 2 shows the fractional change in resistance ($\Delta R/R_o$) of fiber in cured epoxy upon static tension up to fiber fracture. Due to the small strains involved, $\Delta R/R_o$ was essentially equal to the fractional change in resistivity. The $\Delta R/R_o$ decreased by up to $\sim 10\%$ upon tension to a strain of $\sim 0.5\%$ (a stress of 1320 MPa) and then increased upon further tension. The magnitude of resistance decrease of carbon fiber in initial tension is close to the value of the prior resistance increase during curing and cooling of epoxy. The stress at which the resistance decrease was complete (1320 MPa) is close to the value of 1438 MPa obtained from equation (3). Therefore, the initial decrease in $\Delta R/R_0$ in Fig. 2 is attributed to the reduction of the residual compressive stress in the fiber. The later increase in $\Delta R/R_0$ in Fig. 2 is attributed to damage in the fiber. Previous work on the electromechanical behavior of a bare carbon fiber of the same type as this work has shown that damage causes the resistivity of the fiber to increase [2].

Figure 3 shows the $\Delta R/R_0$ of fiber in cured epoxy upon tensile loading to a strain of ~ 0.3% and upon subsequent unloading. The $\Delta R/R_0$ decreased upon loading and

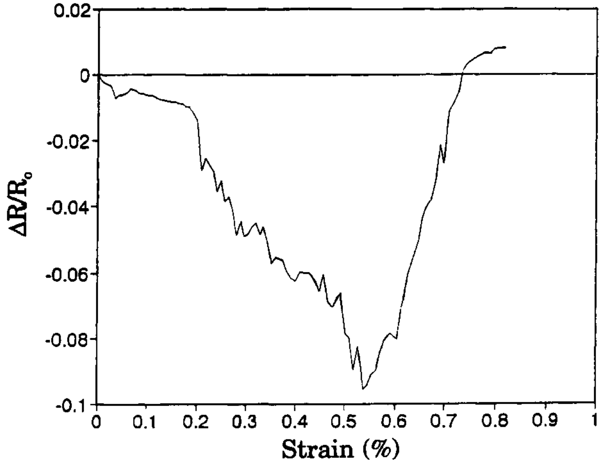

Figure 2. The fractional electrical resistance change of single carbon fiber in epoxy under tension.

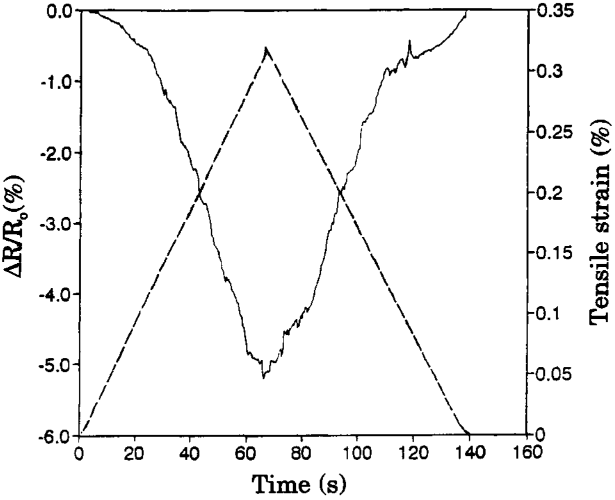

Figure 3. Plots of $\Delta R/R_0$ *vs* time and strain *vs* time during tensile loading and unloading for single carbon fiber embedded in epoxy. Solid curve: $\Delta R/R_0$ *vs* time. Dashed curve: tensile strain *vs* time.

increased back to the initial value upon unloading, indicating the reversibility of the electromechanical effect.

The $\Delta R/R_o$ per unit strain for the electromechanical effect of Fig. 3 is -17 (negative since $\Delta R/R_o$ is negative). In contrast, $\Delta R/R_o$ per unit strain for the electromechanical effect associated with a bare carbon fiber and due to dimensional changes is 2 (positive since $\Delta R/R_o$ is positive).

The technique described in this paper is most suitable for matrices that are electrically insulating. Data interpretation will be complicated by a matrix that is a little conducting, even if it is less conducting than the fiber.

4. CONCLUSION

The residual compressive stress in a single carbon fiber embedded in epoxy, caused by thermal contraction of epoxy after curing, was measured by applying tension to the exposed ends of the embedded fiber and noting the stress required to bring the resistivity of the fiber back down to its value prior to embedding. The residual stress along the fiber axis was thus found to be 1320 MPa for carbon fiber in epoxy. The associated fractional change in electrical resistance/resistivity was 10%. Excessive tensile stress caused the resistivity to increase, due to fiber damage.

REFERENCES

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