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Effect of moisture on the interlaminar interface of a carbon fiber polymer-matrix composite, studied by contact electrical resistivity measurement

SHOUKAI WANG and D. D. L. CHUNG

Composite Materials Research Laboratory, University at Buffalo, The State University of New York, Buffalo, NY 14260-4400, USA

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Abstract—Moisture was found to have a reversible effect on the interlaminar interface of a continuous carbon fiber epoxy—matrix composite. An increase in humidity increased the resistivity. The reversibility was essentially complete after the first cycle of humidity variation. The effect is attributed to expansion of the matrix at the interlaminar interface due to moisture uptake. It allows use of the composite for humidity sensing.

Keywords: Composite; interlaminar; moisture; humidity; polymer; epoxy; carbon fiber; electrical resistivity.

1. INTRODUCTION

Moisture is known to affect negatively numerous properties of polymers and their composites. This problem is of particular concern to advanced structural composites, since they are often used in demanding applications such as aircraft, helicopter rotor blades, fan blades and ocean platforms. Requirements on performance, durability and safety are strict for such applications.

Advanced structural composites are mainly polymer-matrix components containing continuous fibers such as carbon fibers, which are attractive for their high modulus, high strength, low density and thermal conductivity. Among the polymer matrices used for carbon fiber composites, epoxy (a thermoset) is most common.

Considerable attention has been given by numerous workers to address the effect of moisture on the mechanical behavior polymer-matrix composites, as the mechanical behavior is relevant to the effectiveness for structural applications. In the case of carbon fiber epoxy-matrix composites, the properties which are dominated by the matrix or the fiber-matrix interface are degraded by moisture absorption, whereas the properties that are dominated by the fibers are essentially

not affected [1]. In particular, the interfacial strength [2], the interlaminar tensile strength [3], the mode II critical strain-energy release rate [4], and the mode II interlaminar fracture toughness [5, 6] are degraded by moisture. The degradation is attributed to the weakening of the fiber-matrix bond [1, 7], the swelling action of the water [8], the softening of the matrix [1, 7] and the loss of shear strength of the matrix [6]. On the other hand, the curing residual stress is decreased by moisture [3] and the matrix can be plasticized by water [8], thereby increasing the fracture (delamination) toughness [8] or causing moisture to have little effect on the fracture properties [9] in some cases. The moisture effect is aggravated greatly by increasing the temperature [10-13], by using glass fiber in place of carbon fiber [14, 15] or by subjecting the composite to stress [16]. The composite material properties that are affected negatively by moisture include the stiffness [17, 18], the erosion resistance [19], the friction and wear properties [20], the creep compliance [21], the damping ratio [22], the maximum service temperature [23], and the resistance to curvature in case of non-symmetric laminates [24]. The problem can be alleviated by surface treatment of the carbon fiber [25-27]. The moisture absorption proceeds by diffusion and the absorption is at least partially reversible [28].

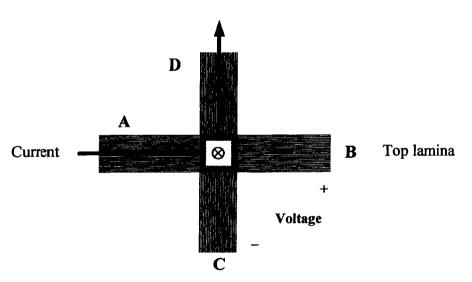
In contrast to prior work [1-28] this work uses electrical resistivity measurement to investigate the effect of moisture on carbon fiber epoxy-matrix composites. The quantity measured is the contact electrical resistivity of the interlaminar interface (i.e. interface between adjacent laminae in a composite). Because the interlaminar interface is a common site of damage in composites, it makes sense to focus on this interface in studying the effect of moisture. The contact electrical resistivity of this interface is affected by the interfacial structure, which changes with the temperature [29-33]. However, the effect of moisture on this resistivity has not been previously investigated.

2. EXPERIMENTAL METHODS

Two laminae of unidirectional carbon fiber epoxy-matrix prepregs (provided by Cape Composites Inc., San Diego, CA) (Table 1) in the form of strips crossing one another, with one strip on top of the other (Fig. 1), were fabricated into a composite at the overlapping region (3.7 mm \times 3.7 mm) of the two laminae by applying pressure (from 0 to 1.2 MPa) and heat to the overlapping region (without a mold). The pressure was provided by weights. A glass fiber epoxy-matrix composite spacer was placed between the weight and the junction (the overlapping area region of the two strips). The heat was provided by a Carver hot press. A Watlow model 981C-10CA-ARRR temperature controller was used to control the temperature and the ramping rate. Each of the specimens was put between the two heating platens of the hot press and heated linearly up to $121 \pm 2^{\circ}$ C at the rate of 2° C/min. Then it was cured at that temperature for 3 h and subsequently furnace cooled to room temperature.

Table 1.Carbon fiber and epoxy matrix properties (according to Cape Composite Inc., San Diego, CA)

Fortafil 555 continuous carbon fil	per
Diameter	$6.2~\mu\mathrm{m}$
Density	1.8 g/cm^3
Tensile modulus	231 GPa
Tensile strength	3.80 GPa
Cape C2002 epoxy	
Processing temperature	121°C
Flexural modulus	99.9 GPa
Flexural strength	1.17 GPa
$T_{\mathtt{g}}$	129°C
Density	1.15 g/cm^3



Bottom lamina

Figure 1. Composite configuration for measuring the contact electrical resistivity.

Humidity variation was conducted after curing and subsequent cooling of the composite by using water as the source of water vapor. All the time, the contact electrical resistance and the relative humidity were measured respectively by a Keithley (Keithley Instruments, Inc., Cleveland, OH) 2001 multimeter and a hygrometer with a digital output. Electrical contacts were made to the four ends of the two strips, so as to measure the contact electrical resistivity (resistance multiplied by contact area, which is the area of the overlapping region) between the two laminae in the composite, using the four-probe method (Fig. 1). The epoxy at the ends of each prepreg strip was burned out to expose the carbon fibers for the purpose of making electrical contacts. These exposed fibers were wrapped by pieces

of copper foil, with silver paint between the copper foil and the fibers. The electric current flowed from A to D, such that the dominant resistance was the contact resistance, as the volume resistance of the strips was negligible in comparison. The voltage between B and C is the voltage between the two laminae.

3. RESULTS AND DISCUSSION

Figure 2 shows the variation of the contact resistivity with time and of the relative humidity with time during cycling of the relative humidity for the composite made at a curing pressure of 0.21 MPa. The resistivity increased reversibly upon humidity increase. The reversibility was essentially complete after the first cycle of humidity variation. The behavior was similar for composites made at other curing pressures ranging from 0 to 1.2 MPa.

The observed trend is attributed to the distance between the fibers of adjacent laminae increasing as the epoxy matrix between the laminae expands during moisture uptake.

Moisture causes expansion of the epoxy matrix, as discussed above. On the other hand, an increase in temperature also causes expansion of the epoxy matrix, due to thermal expansion. In our previous study of the effect of temperature on the contact resistivity, we observed that an increase in temperature caused the resistivity to

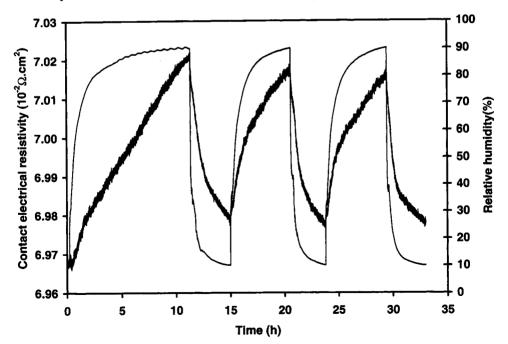


Figure 2. Variation of the contact electrical resistivity (thick curve) with time and of the relative humidity (thin curve) with time during humidity variation for composite made at a curing pressure of 0.21 MPa.

decrease, irrespective of the curing pressure [29]. This suggests that the expansion resulting from moisture uptake is not the same as that resulting from heating. The relief of residual stress upon heating is significant, whether the curing pressure is high or low.

4. CONCLUSION

Moisture was found to have a reversible effect on the interlaminar interface of a continuous crossply carbon fiber epoxy-matrix composite. An increase in humidity increased quite reversibly the resistivity. The effect is attributed to the distance between fibers of adjacent laminae increasing as the epoxy matrix expanded upon moisture uptake. The effect is potentially useful for humidity sensing.

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