THERMAL DAMAGE IN CARBON FIBER POLYMER-MATRIX COMPOSITES, SENSED BY ELECTRICAL RESISTIVITY MEASUREMENT

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Introduction

Polymer-matrix composites with continuous carbon fibers are widely used for lightweight structures, such as aircraft, rotating machinery and sporting goods. Although thermoset-matrix composites are dominant, partly due to their relatively long history of usage, thermoplastic-matrix composites are increasingly used due to their toughness and good hot/wet properties. Although toughness is associated with damage tolerance, the strategic applications associated with the use of advanced composites demand safety to a high degree. To avoid hazard due to damage, the monitoring of the damage (i.e., structural health monitoring) is necessary. Moreover, the damage mechanism needs to be investigated and the type of damage needs to be characterized, as such information is valuable for guiding the design and use of the composites.

Due to the difference in the coefficient of thermal expansion (CTE) between the fibers and the polymer matrix, variation in the thermal stress occurs in the composite upon thermal cycling, thus resulting in thermal fatigue [1,2]. Temperature excursions are encountered during the use of the composites. They may be due to the change in the temperature of the environment (e.g., the difference between the ground temperature and the flight temperature encountered by an aircraft). They may also be due to friction, erosion, fire, bullets, laser, de-icing, welding, machining, exhaust gas, engine operation, etc. Because of the importance of safety, especially in relation to aircraft, even a small extent of degradation is of concern. Degradation can occur after a number of thermal cycles, even when the extent of temperature excursion in each cycle is small. In contrast, without thermal cycling, degradation occurs at relatively high temperatures [3].

Because the extent of degradation during thermal damage can be small (but practically important), the technique used to monitor the degradation should be sensitive to minor degradation. Damage is conventionally detected by nondestructive methods such as ultrasonic and eddy current methods, which are carried out after the damage infliction rather than during the damage infliction. Observation after the damage infliction allows condition monitoring, whereas observation during the damage infliction enables better understanding of the cause, mechanism and nature of the damage. Moreover, observation after the damage infliction allows detection of irreversible effects, whereas observation during the damage infliction allows detection of both reversible and irreversible effects. A nondestructive method that is used during damage infliction is acoustic emission [4], but this technique does not give information on the reversibility of damage and tends to be sensitive to significant damage only. This paper uses a less common method, namely electrical resistance measurement, for observation during damage infliction [5-19].

Mechanical testing in the form of strength measurement [2] as well as microscopy are destructive. As a result, the thermal fatigue testing needs to be performed on numerous specimens, which are subjected to different numbers of thermal cycles. Because of the variation in the amount and distribution of flaws from specimen to specimen, the monitoring of the entire fatigue life of a single specimen gives results with much less data scatter than the use of different specimens for different data points. In order to monitor the entire fatigue life of a single specimen, the monitoring technique must be nondestructive.

Electrical resistance measurement has been previously used to sense strain and mechanical damage in carbon fiber thermoset (epoxy)-matrix composites. Tensile strain in the fiber (longitudinal) direction of the composite causes the electrical resistivity in the fiber direction to decrease and causes that in the through-thickness direction to increase, due to the increase in fiber alignment in the
fiber direction. Damage in the form of fiber breakage causes the electrical resistivity in the fiber direction to increase, whereas damage in the form of delamination causes the resistivity in the through-thickness direction to increase. Hence, electrical resistance measurement allows simultaneous strain and damage sensing.

Fiber damage is a more drastic kind of damage than matrix damage, as the fibers are much stronger than the matrix and damage tends to involve the matrix before involving the fibers. Thus, in practical structures, matrix damage is more common than fiber damage.

According to scanning electron microscopy (SEM) observation, the primary damage due to thermal fatigue is due to fiber-matrix debonding [20]. Such damage is expected to affect the through-thickness electrical resistivity of a laminate, as the damage affects the chance of contact between fibers of adjacent laminae. If this chance is increased, as in the case of the movement of the matrix after partial debonding, the resistivity is decreased. If this chance is decreased, as in the case of delamination, the resistivity is increased.

The through-thickness resistance of a laminate consists of the volume resistance of each lamina and the contact resistance of each interlaminar interface. Since the interlaminar interface is a location that is relatively prone to damage, the contact resistance is a quantity that is particularly relevant. The contact resistance multiplied by the contact area is the contact resistivity, which is a quantity that is independent of the contact area. Therefore, the contact resistivity of the interlaminar interface was measured in this work in order to monitor thermal damage nondestructively and in real time.

An activation energy is involved in the jump of an electron from one lamina to an adjacent one in the through-thickness direction [21]. As a result, the contact resistivity of an interlaminar interface decreases with increasing temperature. The effect is reversible and allows the contact resistivity to indicate the temperature. In other words, the interlaminar interface is a thermistor with a negative temperature coefficient. Therefore, measurement of the contact resistivity of an interlaminar interface provides both temperature and damage information simultaneously. During thermal cycling, temperature variation is indicated by reversible changes in the resistivity, whereas damage (as shown in this work) is indicated by anomalous and abrupt resistivity changes superimposed on the reversible resistivity variation due to the temperature variation.

Thermosets are more brittle than thermoplastics, so they are more prone to cracking, i.e., delamination in the composite. The molecules in a thermoplastic are more mobile than those in a thermoset and movement is enhanced by heat. Hence, thermoplastic-matrix composites are more prone to matrix molecular movement than thermoset-matrix composites. Due to these differences between thermoplastics and thermosets, the mechanism of thermal damage is expected to be different between thermoplastic-matrix composites and thermoset-matrix composites.

**Experimental methods**

The thermoplastic material used was polyphenylene sulfide (PPS), which had a glass transition temperature \( T_g \) of 90°C and a melting temperature \( T_m \) of 280°C. The material was in the form of continuous unidirectional carbon fiber prepreg, supplied by Quadrax Corp. (Portsmouth, Rhode Island; Product QLC4164). The thickness of the prepreg was 250 µm. The carbon fiber was AS-4C, from Hercules Advanced Materials and Systems Company (Magna, Utah), with a diameter of 8 µm. The fiber weight fraction in the prepreg was 64%.

The thermoset polymer was epoxy in the form of unidirectional carbon fiber epoxy-matrix prepregs (provided by Cape Composites Inc., San Diego, CA).

For thermal damage monitoring, specimens were in the form of unidirectional prepreg strips crossing one another, with one strip on top of the other (Fig. 1). The strips were fabricated into a composite at the overlapping region of the two laminae by applying pressure and heat to the overlapping region. The overlapping region was of the size 3.7 x 3.7 mm and 5 x 5 mm for epoxy and thermoplastic composites respectively. The composite fabrication temperature was 121 and 320°C for epoxy and thermoplastic composites respectively. The composite fabrication pressure was 0.33 and 0.21 MPa for epoxy and thermoplastic composites respectively. The time at temperature was 3 and 0.5 h for epoxy and thermoplastic composites respectively.

An electrical contact in the form of silver paint in conjunction with copper wire was applied to each of the four legs of the crossed prepreg strips.
Fig. 1 Composite configuration for testing contact resistivity as a function of temperature.

(Fig. 1). In the four-probe method of DC electrical resistance measurement, two of the electrical contacts (A and D in Fig. 1) were for passing current; the remaining two contacts (B and C) were for measuring voltage. The voltage divided by the current gave the contact resistance of the joint. The resistance multiplied by the contact area gave the contact resistivity. A Keithley 2001 multimeter was used.

For investigation of the effect of thermal fatigue, the contact resistivity was continuously measured while the temperature was cycled by using a small resistance heater for heating and using compressed air and a copper tubing with flowing water for cooling.

For investigation of the effect of the temperature on thermal damage, the contact resistivity was continuously measured while the temperature was cycled such that the maximum temperature of a cycle increased in steps and then decreased in steps. A group of cycles with increasing and then decreasing maximum temperature is referred to as a group.

**Results and discussion**

**Thermoset case**

The contact resistivity decreased upon heating in every cycle of every group. At the highest temperature (150°C) of a group, a spike of resistivity increase occurred, as shown in Fig. 2. This spike was observed similarly in other groups. It is attributed to damage at the interlaminar interface. In addition, the baseline resistivity (i.e., the top envelope) gradually and irreversibly shifted downward as cycling progressed. The baseline decrease is probably due to matrix damage within a lamina and the resulting decrease in modulus and hence decrease in residual stress; it is not due to thermal fatigue, since the damage was most significant in the early cycles and incremental damage diminished upon thermal cycling.

Fig. 3 shows similar results for a case of more severe damage occurring at the highest
temperature (170°C) of a group. The damage resulted in a large spike of resistivity increase at the highest temperature, in addition to a partially reversible upward shift of the baseline resistivity immediately after the spike. The extent of upward shift decreased rapidly from cycle to cycle during the two cycles immediately following the spike.

**Thermoplastic case**

Fig. 4 shows the fractional change in contact resistivity during initial thermal cycling. The resistivity decreased reversibly upon heating in every cycle. As cycling progressed, the baseline resistivity decreased continuously and then leveled off after about 135 cycles. The baseline decrease is probably of similar origin as that in the thermoset case (Fig. 2).

![Fig. 4](image)

**Fig. 4** Variation of the contact electrical resistivity (thick line) with Cycle No. during initial thermal cycling of thermoplastic-matrix composite.

Fig. 5 shows a result of thermal fatigue, which occurred later in the fatigue life. It involved an abrupt and irreversible decrease of the resistivity at Cycle 1,421. The decrease occurred almost at the peak temperature of a cycle. Such abrupt decreases were observed for multiple times (e.g., at Cycles 1,421, 1,489 and 1,557) during the course of thermal cycling. It is attributed to matrix molecular movement and the consequent increase in the chance for fibers of one lamina to touch those of an adjacent lamina.

![Fig. 5](image)

**Fig. 5** Variation of the contact electrical resistivity (thick line) with Cycle No. and of the temperature (thin line) with Cycle No. from 1,410 to 1,430 for thermoplastic-matrix composite. An abrupt and irreversible decrease of the contact electrical resistivity occurred at Cycle No. 1,421.

**Comparison of thermoset and thermoplastic cases**

The abrupt and irreversible resistivity decrease observed in the thermoplastic case is in contrast to the abrupt and partly reversible resistivity increase observed in the thermoset case. The contrast is due to the difference in mechanism. The matrix molecular movement, which is irreversible and is a consequence of fiber-matrix bond degradation, does not occur for the epoxy case, but occurs for the thermoplastic case. The reversibility in the epoxy case is probably due to the contact between fibers of adjacent laminae being lost locally and reversibly.

**Conclusion**

Thermal damage and temperature of continuous carbon fiber polymer-matrix composites were simultaneously monitored by measurement of the contact electrical resistivity of the interlaminar interface. A temperature increase caused the resistivity to decrease reversibly within each thermal cycle. In the case of a thermoset (epoxy) matrix, thermal damage caused the resistivity to increase abruptly and partly reversibly. In the case of a thermoplastic (PPS) matrix, thermal damage caused the resistivity to decrease abruptly and irreversibly, due to matrix molecular movement, and the consequent increase in the chance of fibers of one lamina touching those of another lamina. In contrast, matrix molecular movement could not occur in the thermoset case.
References