Thermal Fatigue in Carbon Fibre Polymer-Matrix Composites, Monitored in Real Time by Electrical Resistance Measurements

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SUMMARY

Thermal fatigue and temperature of a continuous carbon fiber epoxy-matrix composite 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, while thermal fatigue caused the resistivity to increase. The increase took the form of a spike increase at the maximum temperature of a cycle in an early stage of fatigue, and took the form of an abrupt increase of the baseline in a later stage of fatigue.

INTRODUCTION

Polymer-matrix composites containing continuous carbon fibres are widely used for lightweight structures, such as aircraft components and rotating machinery. These structures may encounter temperature excursions during use, whether due to exhaust gas, de-icing, ambient temperature variations or engine operation. Because of the large difference between the coefficient of thermal expansion (CTE) carbon fibres (typically around -0.6×10^{-6} °C^{-1} in the longitudinal direction and that of the polymer matrix (typically ranging from 81×10^{-6} to 117×10^{-6} °C^{-1} for epoxy), temperature excursions result in changes in the amount of thermal stress in the composite. The thermal stress is particularly significant when the laminae have fibres in different directions, as the CTE of each lamina is highly anisotropic. Such thermal stress variations may cause thermal fatigue, especially if a large number of cycles of temperature excursion have been encountered. The damage most commonly occurs at the interface between laminae (i.e., the interlaminar interface). Fibre fracture is a more drastic form of damage and is less commonly encountered. Damage degrades the modulus of the composite, thus enhancing vibrations and aggravating further damage.

Thermal fatigue is to be distinguished from mechanical fatigue, which is due to stress cycling and has received more attention[1-6]. The deformation during stress cycling causes temperature changes[5,6]. However, due to the high thermal conductivity of carbon fibres, the resulting amplitude of temperature change in mechanical fatigue is small, typically below 1°C. The amplitude of temperature change in thermal fatigue is much larger.

Thermal fatigue is conventionally studied by mechanical testing (usually destructive due to strength measurement) at room temperature after various numbers of cycles of temperature excursion[7-12]. Non-destructive mechanical testing (e.g., modulus measurement) at various temperatures during the temperature cycling is less common due to the greater complexity of the experimental set-up, but it is real-time monitoring and provides more precise information on the progress of thermal fatigue. Moreover, a specimen for mechanical testing (say by flexure) cannot be too small and a small specimen size is necessary for temperature variation at a reasonably high rate, in order for thermal fatigue testing of the specimen to be completed within a reasonable length of time. Therefore, these authors do not use mechanical testing for thermal fatigue monitoring, but rather use electrical resistance measurement, which can be conducted on small specimens. The technique is enabled by the high electrical conductivity of the carbon fibres compared to that of the polymer matrix. It involves measuring the contact electrical resistivity of the interlaminar interface. Degradation of this interface causes this resistivity to increase. This electrical method has been used previously to monitor thermal damage at different extents of temperature excursion[13], but has not been used previously to monitor thermal fatigue, i.e., the effects of large numbers of temperature cycles.

The through-thickness resistance of a fibre composite consists of the volume resistance of each lamina in the through-thickness direction and the contact resistance of each interlaminar interface. The through-thickness resistance has been previously used to monitor in real time the mechanical fatigue that causes delamination[1]. A change in the through-thickness resistance mainly reflects a change in the contact resistance of the interlaminar interface. The through-thickness resistance increases when delamination occurs. Thus, based on the through-thickness resistance, it was observed that delamination starts at 33% of the fatigue life. This paper uses the contact resistance rather than the through-thickness resistance to indicate damage, since the contact resistance gives more direct information on the interlaminar interface.

By using contact electrical resistivity measurement, this paper provides the first real-time monitoring of thermal fatigue of a carbon fibre polymer-matrix composite over a large number of temperature cycles. This monitoring entails that of both the damage and the temperature. Damage causes the resistivity to increase, whereas temperature increase causes the resistivity to decrease. The latter is because there is an activation energy for electron jumping between two laminae[14]. Simultaneous monitoring of temperature and thermal damage during thermal fatigue is valuable, since it provides information on exactly which point of which temperature cycle at which damage occurs. Damage can occur at the highest temperature point, the lowest temperature point, or any other point of a temperature cycle. This is akin to the simultaneous monitoring of strain and mechanical damage during mechanical fatigue by through-thickness resistance measurement[1]. In contrast, techniques such as acoustic emission can monitor damage, but cannot monitor temperature or strain.

A crossply configuration is used in this work for thermal fatigue monitoring, because the thermal stress and the sensitivity for temperature monitoring are higher for a crossply configuration than for a unidirectional configuration[14]. However, a unidirectional configuration could have been used instead.

The contact resistivity is given by the product of the contact resistance and the contact area. It is a quantity that is independent of the contact area. It reflects the structure of the interlaminar interface. The greater the number of contacts between fibres of adjacent laminae, the lower is the contact resistivity. Though the polymer matrix is electrically insulating, the contact resistivity is never zero. This is because there is always some contact between fibres of adjacent laminae, as resulting from the waviness of the fibres and the flow of the matrix (resin) during composite fabrication. Delamination or damage to the matrix between adjacent laminae decreases the number of contacts between fibres of adjacent laminae, thereby increasing the contact resistivity.

The contact resistance is measured in this work by using the four-probe method, in which the four electrical contacts are at the four ends of two crossply laminae that intersect like a cross (Figure 1). The junction of the two laminae is the interlaminar interface under study. Current is passed from contact A to contact D, while voltage is measured between contact B and contact C. The voltage divided by the current gives the contact resistance, as the volume resistance in the fibre direction within each lamina is negligible.

EXPERIMENTAL METHODS

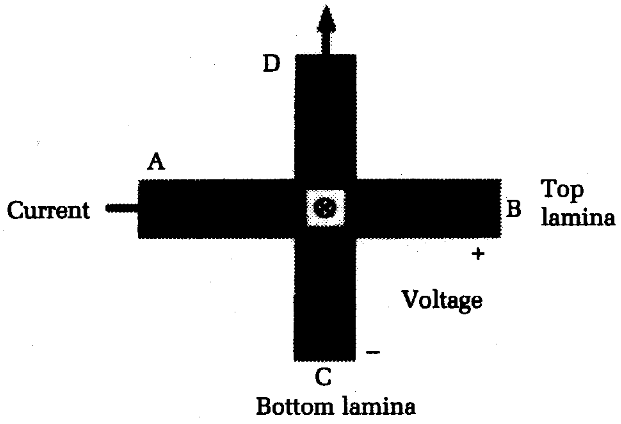
Two laminae of unidirectional carbon fibre epoxy-matrix prepregs (provided by Cape Composites Inc., San Diego, CA) (Table 1) in the form of strips crossing one another, with one strip on top of the other (Figure 1), were fabricated into a composite at the overlapping region (3.7 mm x 3.7 mm) of the two laminae by applying pressure (0.33 MPa) and heat to the overlapping region (without a mould). The pressure was provided by a weight. A glass fibre 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 samples was put between the two heating platens of the hot press and heated linearly up to 121 ± 2°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.

Thermal cycling was conducted after curing and subsequent cooling of the composite by using a small resistance heater for heating and using compressed

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

Fortafil 555 continuous carbon fibre	
Diameter	6.2 μm
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_g	129° C
Density	1.15 g/cm^3

Figure 1 Composite configuration for measuring contact electrical resistivity during thermal cycling



air and a copper tubing with flowing water for cooling (Figure 2). The temperature was varied from 28 to 118°C in each thermal cycle, such that the time of temperature increase was 13 s and the time of temperature decrease was 17 s. All the time, the contact electrical resistance and the temperature of the sample were measured respectively by a Keithley (Keithley Instruments, Inc., Cleveland, OH) 2001 multimeter and a T-type thermocouple, which was put just beside the junction. 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 (Figure 1). The epoxy at the ends of each prepreg strip was burned out to expose the carbon fibres for the purpose of making electrical contacts. These exposed fibres were wrapped by pieces of copper foil, with silver paint between the copper foil and the fibres. 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. Three specimens were subjected to thermal cycling testing, although the results for one specimen are given in the next section.

RESULTS AND DISCUSSION

Figure 3 shows the variation of the contact resistivity with temperature during initial thermal cycling. In each thermal cycle, the contact resistivity decreased with increasing temperature and increased with decreasing temperature, because increasing temperature increased the probability of electrons jumping from one lamina to the other[14]. It can also be noticed that the baseline of the contact resistivity decreased and gradually levelled off with increasing thermal cycle number. There are two possible reasons for the decrease. One reason is that the moisture content decreased with increasing thermal cycle number, due to the heating driving out moisture from the epoxy matrix. (Although the moisture could partly come back in the cooling part of each cycle, the moisture absorption was much slower than the moisture desorption during heating.) Moisture made

Figure 2 Experimental set-up for cyclic heating and cooling

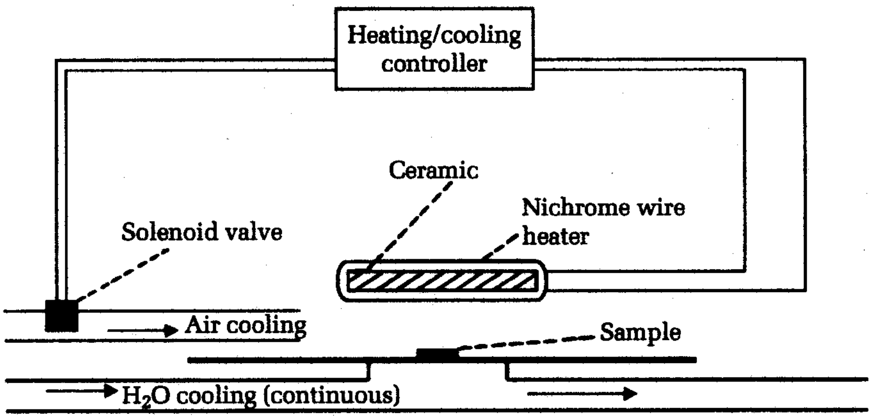
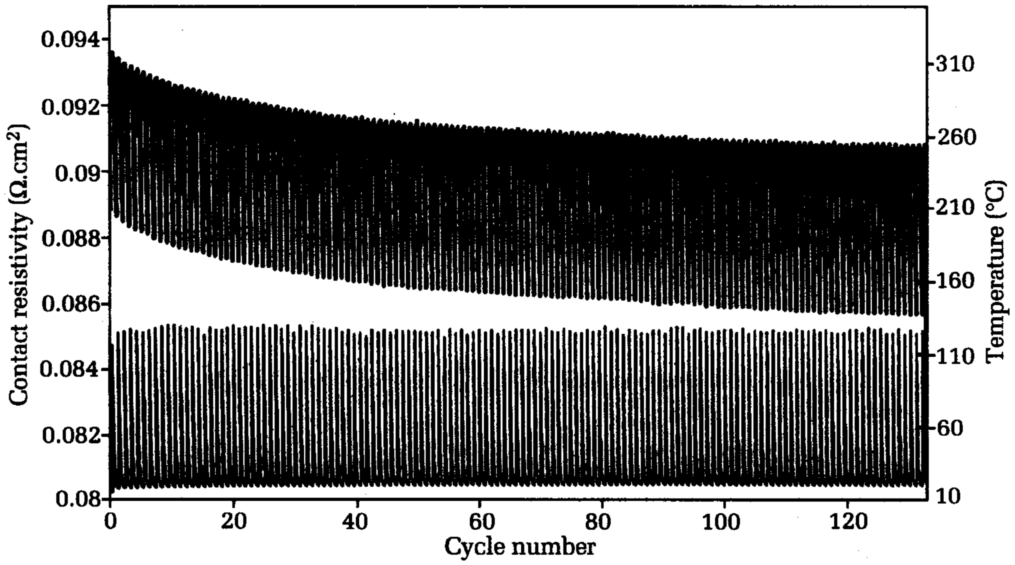


Figure 3 Variation of the contact electrical resistivity (thick line) with cycle number, and of the temperature (thin line) with cycle number during the first 133 thermal cycles



the epoxy expand. Therefore, the lower the moisture content, the higher the density of the epoxy, the higher the fibre volume fraction, the greater the number of fibre contacts between the two laminae, and the lower was the contact resistivity. The other possible reason is that the thermal cycling damaged the epoxy matrix, thereby decreasing the modulus of the laminae, lowering the thermal stress, and decreasing the contact resistivity. This damage is not really due to thermal fatigue, since the damage was most significant in the first cycle and incremental damage diminished upon thermal cycling.

Figure 4 shows that a small spike of contact resistivity increase occurred at the maximum temperature of a cycle from Cycle No 13,481 onward. The increase was partly reversible and is attributed to thermal fatigue damage of the interlaminar interface. The damage may be a form of delamination and increased the contact resistivity.

Figure 5 shows another result of thermal fatigue, which occurred later in the fatigue life. It involved an abrupt increase in the contact resistivity baseline. The abrupt increase, which occurred more than once,

Figure 4 Variation of the contact electrical resistivity (thick line) with cycle number, and of the temperature (thin line) with cycle number from Cycle No. 13,476 to Cycle No. 13,487. The spike started to appear at Cycle No. 13,481 and continued thereafter

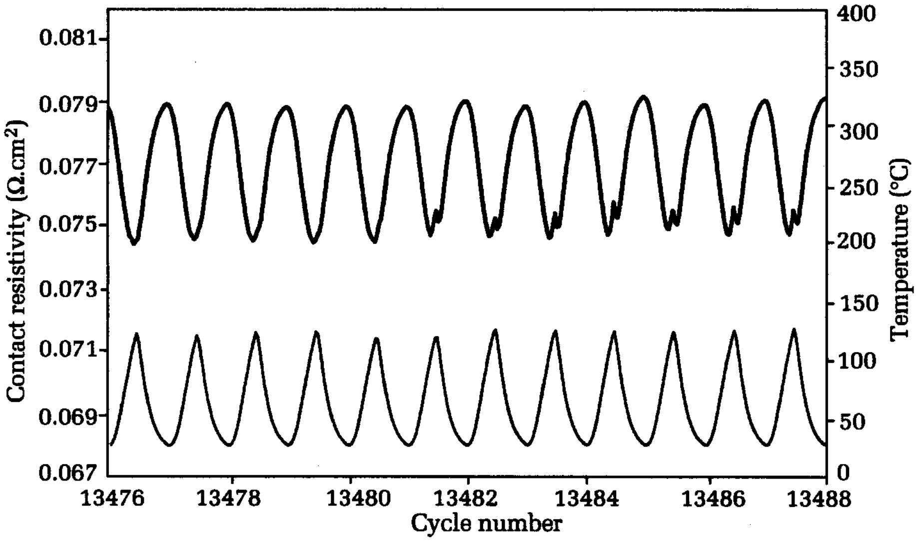
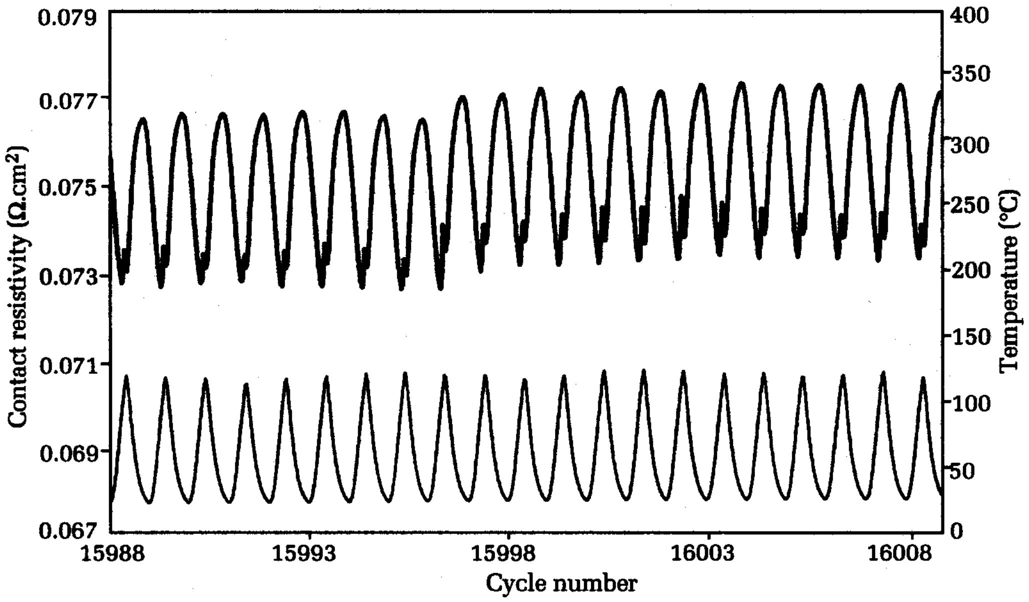


Figure 5 Variation of the contact electrical resistivity (thick line) with cycle number and of the temperature (thin line) with cycle number from Cycle No. 15,988 to Cycle No. 16,008. An abrupt increase in the baseline of the contact electrical resistivity occurred at cycle No. 15,996



also indicates damage to the interlaminar interface – perhaps more serious damage than that indicated by the spikes that occurred earlier in the fatigue life.

CONCLUSIONS

Thermal fatigue in a continuous carbon fibre epoxy-matrix composite was monitored in real time during thermal cycling between 28 and 118°C by measurement of the contact electrical resistivity of the interlaminar interface. Simultaneous to monitoring damage was the monitoring of the temperature by the same resistivity measurement, as the resistivity decreased reversibly upon heating in each thermal cycle, due to the energy barrier for electron jumping from one lamina to the other. The initial stage of damage occurred primarily during the first 100 thermal cycles, and was associated with decrease of the resistivity baseline, probably due to moisture desorption and/or matrix modulus decrease. The second stage of damage involved thermal fatigue damage in the form of interlaminar interface degradation (probably delamination), which was associated with a spike of resistivity increase at the maximum temperature of a thermal cycle. The third stage of damage involved thermal fatigue damage, also in the form of interlaminar interface degradation, which was associated with abrupt increases of the resistivity baseline.

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