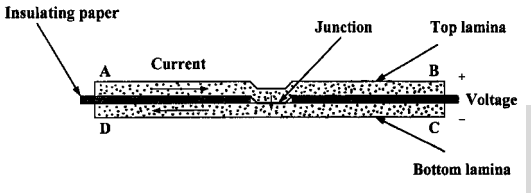
# Fibrous Composite Interfaces Studied by Electrical Resistance Measurement

By *Deborah D. L. Chung**




*Direct current (DC) electrical resistance measurement is effective for studying the interfaces in fibrous composite materials, particularly carbon fiber composites, which are electrically conducting. The measurement yields information on the fiber–matrix interface, residual stress, and interlaminar interface, and can be made in real time during debonding, residual stress reduction, temperature change, and interlaminar shear. This paper reviews the methods and applications in relation to polymer–matrix and cement–matrix composites.*

## 1. Introduction

Composite materials are materials which are made by artificially combining two or more components. Thus, interfaces are present in a composite material and they tend to govern the properties of a composite material.

The interface between the reinforcement (whether particulate or fibrous) and the matrix needs to the strong enough in order to allow load transfer to the reinforcement. As the composite may be fabricated at an elevated temperature and the reinforcement tends to have a lower coefficient of thermal expansion (CTE) than the matrix, the composite at room temperature tends to have residual stress, such that the reinforcement is under compression. The residual stress is particularly large when the reinforcement is in the form of fibers. The residual stress affects both the reinforcement and the interface between reinforcement and matrix.

In the case of a composite material with continuous fibers as the reinforcement, such that the fibers are in the form of layers (called laminae) stacked up to form a laminate, the interface between adjacent laminae (called the interlaminar interface) is usually the weak link where damage (e.g., delamination) occurs. The interlaminar interface involves a large number of fibers in each lamina. It includes fiber–matrix–fiber points where the matrix interlayer is as thin as a few Å, so that electrical contact occurs between the fibers. This is due to the flow of the matrix (or its precursor) during composite fabrication and also due to the fiber waviness. It also includes fiber–matrix–fiber points where the matrix interlayer is too thick to allow electrical contact between the fibers. In the case of a laminate in which the fibers of adjacent laminae are oriented in different directions, the anisotropy causes residual stress, which affects the interlaminar interface.

Understanding the science of composite interfaces is crucial to the development and improvement of composite materials. Microscopy and interfacial chemical analysis are widely used to study the structure of composite interfaces, though these techniques tend to be tedious and tend to give local rather than global information on the interfaces. In the case of at least one of the components in the composite being electrically conducting, electrical resistance measurements can be used to study composite interfaces. Resistance measurements are quick and involve relatively simple equipment. Moreover, they tend to give global information a composite interfaces. This paper reviews the methods and applications of electrical resistance measurements for composite interface studies.

Composites for study by electrical resistance measurement are preferably those containing a reinforcement which is more conducting electrically then the matrix. Otherwise, the matrix acts like a short circuit path and makes the electrical

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resistance measurement quite insensitive to the reinforcement. Due to the electrical conductivity of carbon fibers, carbon fiber polymer–matrix and cement–matrix composites are well suited to study by electrical resistance measurement. In contrast, metal–matrix composites tend to be not suitable, due to the high conductivity of the metal matrix.

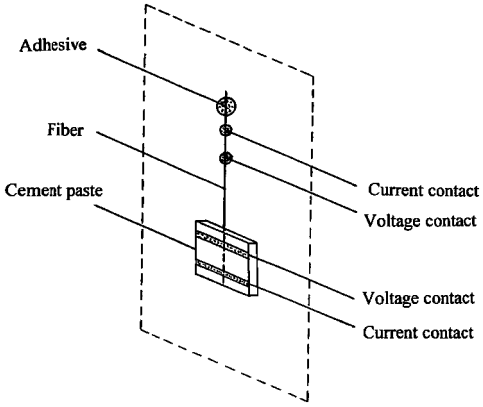


*Fig. 1. Sample configuration for measuring the contact electrical resistivity of the interface between a fiber and matrix.*

The information gained by electrical resistance measurement depends on the geometry and configurations of the measurement. The use of the four-probe method rather than the two-probe method is important in order to avoid the electrical resistance of the electrical contacts.

Measurement of the contact resistivity of the fiber–matrix interface provides information on this interface. This can be done by embedding a single fiber at one end in a matrix, putting a current contact and a voltage contact on the exposed fiber, putting a current contact and a voltage contact on the matrix embedding the fiber, and measuring the resistance between the two voltage contacts, which are the two inner contacts (Fig. 1).[1,2] This resistance is the sum of the contact resistance of the fiber–matrix interface, the volume resistance of the fiber and the volume resistance of the matrix in the direction from the fiber–matrix interface to the voltage contact on the outer surface of the matrix. Due to the high conductivity of the fiber, the volume resistance of the fiber is negligible. The volume resistance of the matrix may or may not be negligible, depending on the size of the matrix embedding the fiber. If it is not negligible, it can be obtained by separate measurement of the volume resistivity of the matrix and calculation of the volume resistance from the dimensions. After deducting this resistance from the measured resistance, the contact resistance of the interface is obtained. The product of the contact resistance and the area of the interface is the contact resistivity, which is independent of the contact area.

The contact resistivity tends to be higher when the interfacial void content is increased, as voids are insulating. It is also affected by the interfacial phases, which tend to have volume resistivities that are different from those of the fiber and matrix. Thus, the contact resistivity gives information on the interfacial structure. The method illustrated in Figure 1 requires that both the fiber and the matrix are conducting. This method has been applied to study the interface between steel (or carbon) fiber and cement matrix[1,2] and that between steel reinforcing bar (rebar) and concrete (with coarse and fine aggregates).[3]

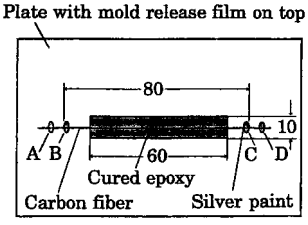


*Fig. 2. Sketch of the resistance measurement set-up for single fiber embedded in epoxy. A, B, C, and D are four probes. A and D are for passing current; B and C are for voltage measurement. Dimensions are in mm.*

Measurement of the volume electrical resistivity of a single fiber while it is embedded by a matrix, but exposed at both ends, gives information on the residual stress in the fiber, as the residual stress tends to increase the fiber resistivity. At each exposed end of the fiber, there are a current contact and a voltage contact (Fig. 2). The resistance between the two voltage contacts is measured. This method has been applied to study the interface between carbon fiber and an epoxy matrix.[4,5]

Measurement of the contact electrical resistivity of the interlaminar interface provides information on the structure of this interface. The greater is the number of fiber–fiber contacts (i.e., fiber–matrix–fiber points in which the matrix interlayer is less than a few Å in thickness), the lower is the contact re-

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sistivity of the interlaminar interface. The residual stress, which is particularly high when the laminae involve fibers in different directions, makes it more difficult (i.e., increasing the activation energy) for electrons to jump across the interlaminar interface.

The contact resistivity of the interlaminar interface can be measured by allowing two laminae to overlap to form a junction (i.e., the interlaminar interface) plus four electrical leads (which are the parts of the laminae outside the junction) (Fig. 3). Electrical current is passed from the top lamina to the bottom lamina across the junction by using one lead from the top lamina and one lead from the bottom lamina. Voltage across the junction is measured by using the other lead from the top lamina and the other lead from the bottom lamina. The voltage divided by the current gives the contact resistance of the junction, since the resistance of the leads is negligible compared to that of the junction. The contact resistance multiplied by the junction area gives the contact resistivity. This method has been applied to study the interlaminar interface in carbon fiber polymer–matrix composites.[6,7]

Due to its fast response and non-destructiveness, electrical resistance measurement can be conducted in real time during composite fabrication, during mechanical loading and unloading, and during heating and cooling, thereby providing much more information than static measurement. When the applied stress or heat is low, the effects on the composite interfaces can be reversible.[8,9] For example, the residual compressive stress in the fiber is reduced either by applying tensile stress on the fiber or by heating, and the effect is reversible. However, when the applied stress or heat is high, the effects can be irreversible, due to damage. The reversible and irreversible effects are usually in opposite directions. For example, residual stress reduction causes the volume resistivity to decrease reversibly, whereas damage causes the resistivity to increase irreversibly.

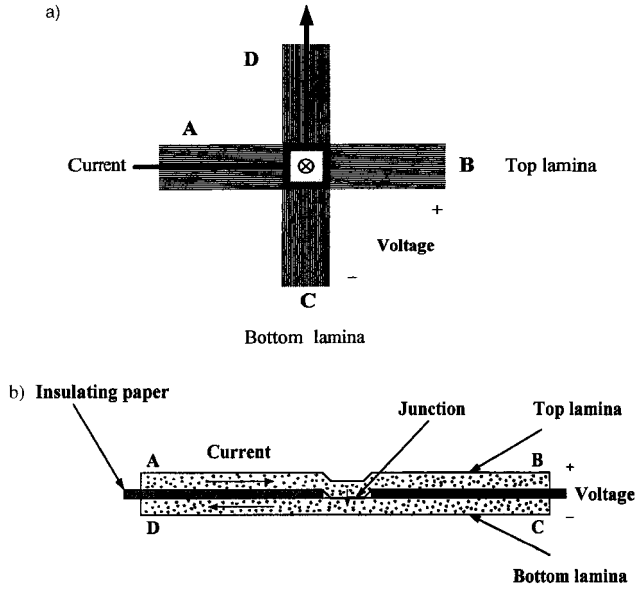


*Fig. 3. Composite configurations for testing contact resistivity as a function of temperature. a) Crossply. b) Unidirectional.*

Measurement of the volume resistivity of a composite in various directions gives information on the structure of the composite. For example, the volume resistance of a composite laminate in the through-thickness direction is the sum of the volume resistance of each lamina and the contact resistance of each interlaminar interface. Although the measured resistance is related to the contact resistance of the interlaminar interface, it is not a direct measure of the contact resistance. A direct measure of quantities that describe the interface is the goal of this paper. Hence, this review does not address electrical resistance measurements that are directed at studying the structure of a composite, as opposed to the structure of a composite interface.

## 2. Fiber–Matrix Interface

The shear bond strength between fiber and matrix is commonly determined by single fiber pull-out testing, which involves embedding one end of a single fiber in the matrix and pulling the fiber out of the matrix during the test.[10–16] A problem of this method is the large amount of scatter in the shear bond strength data obtained on different samples that are identically prepared, whether the embedment length is fixed or not. Due to the scatter, the standard deviation is large and the shear bond strength determination is limited in accuracy.[11,16] As a result, small but real differences in bond strength between samples that are not identically prepared (say, with different surface treatments of the fiber) are hard to measure. The origin of the data scatter had long been assumed to be experimental error associated with pull-out testing, though fracture mechanics analysis suggested that the data scatter is inherent in the specimens themselves.[16] By measuring the contact electrical resistivity between fiber and matrix on every sample that was subjected to pull-out testing, it was experimentally confirmed that the data scatter is not due to experimental error in pull-out testing, but rather due to real differences in the structure of the fiber–matrix interface among samples that are identically prepared. A strong correlation between the bond strength and the contact resistivity was observed. The curve describing this correlation changed (say, shifted) when either the fiber or the matrix was modified, so that even small differences in bond strength due to fiber or matrix modification were determined. This technique is called "single fiber electromechanical pull-out testing". This technique is valuable for measuring even small differences in bond strength and provides information on the origin of the bonding and on the structure of the interface. Moreover, contact resistivity measurement (without fiber pull-out), is a nondestructive method for measuring the bond strength, provided that the curve correlating the two quantities is given.

The method of single fiber electromechanical pull-out testing involves embedding one end of a single fiber in a matrix, as in conventional single fiber pull-out testing. In addition, four electrical contacts are applied—two on the fiber and two on the matrix surrounding the fiber, as illustrated in Figure 1.

The measurement of the fiber–matrix contact resistivity does not require any pull-out of the fiber, as it is non-destructive. However, measurement of this quantity during fiber pull-out (optional) can give further information on the interface. As in conventional single fiber pull-out testing, the shear bond strength is determined using the sample configuration of Figure 1. Thus, the configuration of Figure 1 allows measurement of bond strength and contact resistivity on the same sample. This combined measurement constitutes the heart of single fiber electromechanical pull-out testing.

The technique of single fiber electromechanical pull-out testing is illustrated below by using cement paste (slightly conducting) as the matrix and stainless steel as the fiber.[1,2]

The contact electrical resistivity between the fiber and the cement paste was measured at 1, 7, 14, and 28 days of curing using the four-probe method and silver paint as electrical contacts, as illustrated in Figure 1. One current contact and one voltage contact were on the fiber, while the other voltage and current contacts were on the cement paste embedding the fiber to a distance of 1 cm. The cement paste thickness was 1.5 mm on each side sandwiching the fiber. The fiber length was 5 cm. The resistance between the two voltage probes corresponds to the sum of the fiber volume resistance, the interface contact resistance and the cement paste volume resistance. The measured resistance turns out to be dominated by the contact resistance, to the extent that the two volume resistance terms can be neglected. The contact resistivity (in $\Omega\,cm^2$) is given by the product of the contact resistance (in $\Omega$) and the contact interface area (in $cm^2$).

Single fiber pull-out testing was conducted on the same interface samples and at the same time as the electrical resistance measurement. One end of the fiber was embedded in cement paste, as in Figure 1. The contact resistivity was taken as the value prior to pull-out testing. The bond strength was taken as the maximum shear stress during pull-out testing. Seven interface samples were tested for each combination of fiber surface treatment (as-received, acetone washed or acid washed) and curing time (1, 7 14, or 28 days).

Figures 4 and 5 give typical plots of shear stress vs. displacement and simultaneously obtained plots of contact electrical resistivity vs. displacement for as-received and acid washed stainless steel fibers respectively at 28 days of curing. In both cases, the contact resistivity abruptly increases when the shear stress reaches its maximum, i.e., when fiber–matrix debonding is complete. For the as-received fibers (Fig. 4), the contact resistivity does not change before the abrupt increase when the shear stress has reached its maximum. For the acid washed fibers (Fig. 5), the contact resistivity gradually increases prior to the abrupt increase when the shear stress has reached its maximum.

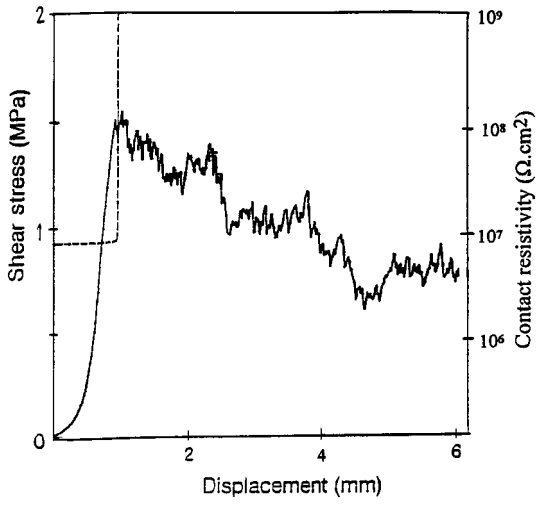


*Fig. 4. Plots of shear stress vs. displacement (solid curve) and of contact electrical resistivity vs. displacement (dashed curve) simultaneously obtained during pull-out testing of as-received stainless steel fiber from cement paste at 28 days of curing.*

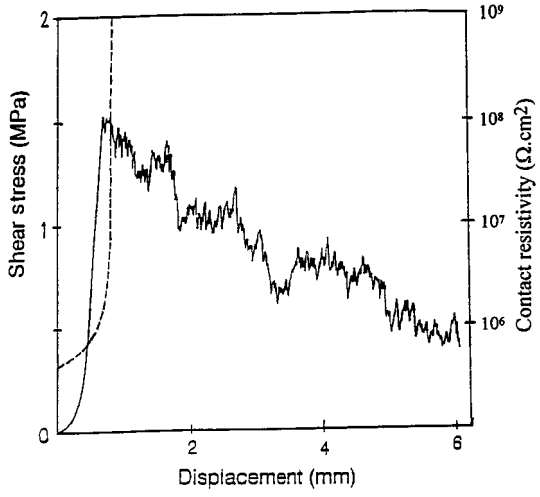


*Fig. 5. Plots of shear stress vs. displacement (solid curve) and of contact electrical resistivity vs. displacement (dashed curve) simultaneously obtained during pull-out testing of acid washed stainless steel fiber from cement paste at 28 days of curing.*

Figure 6 shows the correlation of the contact resistivity with the bond strength at 28 days for the as-received, acetone washed and acid washed fibers. For each type of surface treatment, the bond strength as well as contact resistivity vary among the seven samples (identically prepared) tested. Nevertheless, the contact resistivity correlates strongly with the bond strength among the data for each type of surface treatment. The increase is roughly linear, except for the negative deviation from linearity in the high bond strength regime. For the as-received and acetone washed fibers, the contact resistivity increases with increasing bond strength. The increase is roughly linear, except for the negative deviation from linearity in the high bond strength regime. For the case of acid washed fibers, the contact resistivity decreases with increasing bond strength. The range of bond strength is simi-

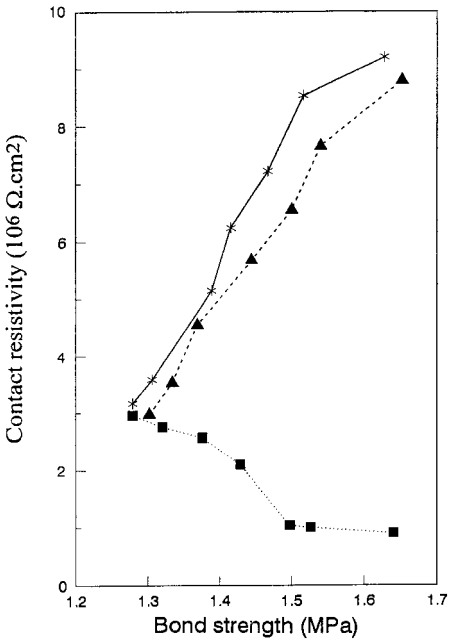


*Fig. 6. Variation of contact electrical resistivity with bond strength at 28 days of curing for as-received (stars), acetone washed (triangles), and acid washed (squares) stainless steel fibers.*

lar for the three types of surface treatment, but the range of contact resistivity is lower for the acid washed case than the as-received and acetone washed cases.

Figure 6 shows that the contact resistivity increases with increasing bond strength for the as-received and acetone washed fibers but decreases with increasing bond strength for the acid washed fibers. This means that high resistivity phase(s) at the steel–cement interface (higher in resistivity than the cement paste) dominates the mechanism for enhancing the bonding for the as-received and acetone washed fiber, whereas decrease in the amount of interfacial voids (which are high in resistivity) dominates the mechanism for enhancing the bonding for the acid washed fibers. This interpretation is consistent with the fact that the range of contact resistivity exhibited by the acid washed fibers is lower than that exhibited by the as-received or acetone washed fibers (Fig. 6). The acid washing removes some phase(s) (probably metal oxides and other compounds) from the surface of the fibers, as suggested by the roughening of the surface and the 20 % weight loss. The removal of the phase(s) by acid washing apparently makes it impossible for the high resistivity phase(s) that enhance the bonding to form when the fiber subsequently encounters the cement paste. Due to the presence of the high resistivity phase(s) that enhance bonding in the as-received and acetone washed cases, the interfacial voids (which are also high in resistivity) cannot be distinguished electrically, leading to no change in contact resistivity during debonding (Fig. 4). On the other hand, for the acid washed case, the interfacial voids govern the bond strength, so the contact resistivity increases as the interfacial void content increases during debonding (Fig. 5). In all three cases, the contact resistivity shoots up by orders of magnitude at the completion of debonding and the start of fiber pull-out. Thus, the contact resistivity measurement provides information on the structure of the fiber–cement interface. In the case of the acid washed fibers, contact resistivity measurement also provides a means of monitoring the progress of debonding (Fig. 5).

Acetone washing increases the bond strength and decreases the contact resistivity (Fig. 6). This is partly because of the removal of organic material by the acetone washing (consistent with the 3 % weight loss after washing), the electrically insulating character of the organic material and the detrimental effect of the organic interfacial layer on the bond strength. Acetone washing slightly roughens the fiber surface. The surface roughening increases the true interfacial area, so it also plays a role in increasing the apparent bond strength and decreasing the apparent contact resistivity.

Figure 7 shows the dependence of the bond strength and contact resistivity on the curing age for as-received stainless steel fiber. The bond strength decreases while the contact resistivity increases with curing age; at each curing age, the contact resistivity increases roughly linearly with increasing bond strength, such that negative deviation from linearity occurs in the high bond strength regime.

Figure 7 shows that the bond strength decreases while the contact resistivity increases with curing age from 1 to 28 days. These effects suggest that, as curing occurs, the interfacial void content increases.

The negative deviation from linearity in the high bond strength regime (Fig. 7) is attributed to the need to have a low interfacial void content in order to attain a high bond strength and the decrease of the contact resistivity when the interfacial

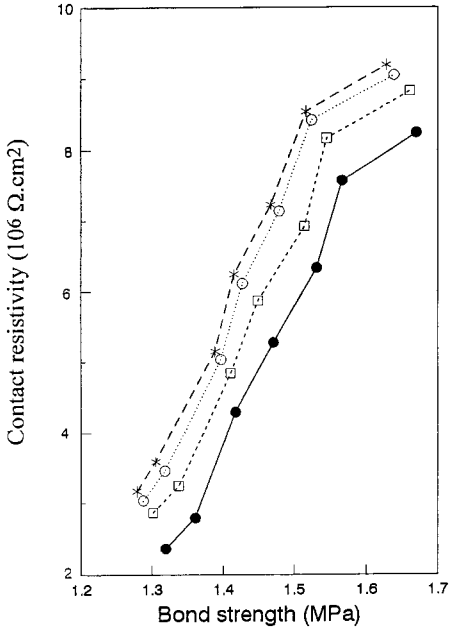


*Fig. 7. Variation of contact electrical resistivity with bond strength for as-received stainless steel fibers at 1 (solid circles), 7 (squares), 14 (open circles), and 28 (stars) days of curing.*

void content is decreased. In other words, both the high resistivity interfacial phase that helps bonding and a low interfacial void content are needed in order to attain a high bond strength.

## 3. Residual Stress in Fiber Embedded in Matrix

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.[17] 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 (XRD), 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, a method which involves simultaneous electrical and mechanical measurements on the same sample under load has been developed. This method is in contrast to the separate electrical and mechanical measurements. This electromechanical testing provides a simple and effective method for measuring the fiber residual stress along the fiber direction, as illustrated below for the case of carbon fiber in epoxy.[4] Carbon fiber epoxy–matrix composites are the most widely used form of carbon fiber composites due to the good adhesion between fiber and 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 Figure 6. 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. 2) 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. The resistivity of fiber increases by ~10 % after curing and subsequent cooling. The fractional resistance increase is 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 only the residual stress along the fiber direction (one dimension) is considered. 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, $\sigma_f$ is the longitudinal residual stress built up in the fiber, $\sigma_m$ is the residual stress built up in the matrix, $E_f$ is the modulus of fiber, $E_m$ is the modulus of matrix, $\alpha_f$ is the coefficient of thermal expansion of fiber, $\alpha_m$ is the coefficient of thermal expansion of matrix, and $\Delta T$ 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 case, $E_f$ = 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 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.

Figure 8 shows the fractional change in resistance ($\Delta R/R_0$) of fiber in cured epoxy upon static tension up to fiber fracture. Due to the small strains involved, $\Delta R/R_0$ is essentially equal to the fractional change in resistivity. The $\Delta R/R_0$ decreases by up to ~10 % upon tension to a strain of ~0.5 % (a stress of 1320 MPa) and then increases 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 is 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 Figure 8 is attributed to the reduction of the residual compressive stress in the fiber. The later increase in $\Delta R/R_0$ in Figure 8 is attributed to damage in the fiber. Electromechanical testing of a bare carbon fiber of the same type has shown that damage causes the resistivity of the fiber to increase.[18]

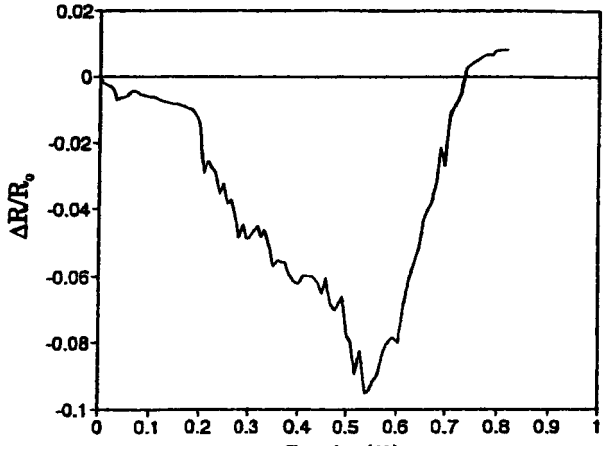


*Fig. 8. The fractional electrical resistance change of single carbon fiber in epoxy under tension.*

Figure 9 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 $\Delta R/R_0$ decreases upon loading and increases back to the initial value upon unloading, indicating the reversibility of the electromechanical effect.

The $\Delta R/R_0$ per unit strain for the electromechanical effect of Figure 9 is –17 (negative since $\Delta R/R_0$ is negative). In contrast, $\Delta R/R_0$ per unit strain for the electromechanical effect associated with a bare carbon fiber and due to dimensional changes is 2 (positive since $\Delta R/R_0$ is positive).

## 4. Interlaminar Interface

The study of the interlaminar interface is commonly performed by measuring the interlaminar shear strength (ILSS) by techniques such as the short-beam method,[19] the Iospiescu method,[20] and other methods.[21] Although ILSS is a valuable quantity that describes the mechanical property of the joint between laminae, it gives little information on the interfacial structure, such as the extent of direct contact (with essentially no polymer matrix in between) between fibers of adjacent laminae and the residual interlaminar stress resulting from the anisotropy between adjacent laminae. The anisotropy is severe when the fibers in the adjacent laminae are in different directions, since the fibers and polymer matrix differ greatly in modulus and thermal expansion coefficient. Direct contact between fibers of adjacent laminae occurs due to the flow of the matrix during composite fabrication and the waviness of the fibers. Direct contact means that the thickness of the matrix between the adjacent fibers is so small (say, a few Å) that electrons can tunnel or hop from one fiber to the other. The presence of direct contact has been shown by the fact that the volume electrical resistivity of carbon fiber epoxy–matrix composites in the through-thickness direction is finite, even though the epoxy matrix is electrically insulating.[22]

The contact electrical resistivity of the interlaminar interface can be used as a quantity to describe the structure of this interface.[6,7] Figure 10 shows the variation of the contact resistivity $\rho_c$ with temperature during reheating and subsequent cooling, both at 0.15 °C/min, for samples cured at 0 and 0.33 MPa. The corresponding Arrhenius plots of log contact conductivity (inverse of contact resistivity) versus inverse absolute temperature during heating are shown in Figure 11. From the slope (negative) of the Arrhenius plot, which is quite linear the activation energy can be calculated by using the equation

$$\text{slope} = -\frac{E}{2.3k} \tag{4}$$

where $k$ is the Boltzmann's constant, $T$ is the absolute temperature (in K), and $E$ is the activation energy. The linearity of the Arrhenius plot means that the activation energy does not change throughout the temperature variation. This activation energy is the energy for electron jumping from one lamina to the other. Electronic excitation across this energy enables conduction in the through-thickness direction. This activation phenomenon is common in the electrical conduc-

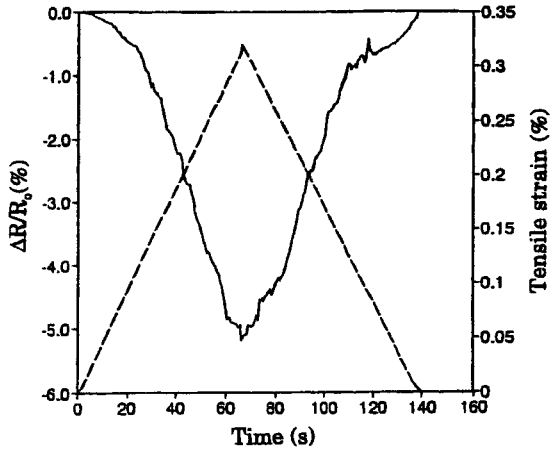


*Fig. 9. Plots of $\Delta R/R_0$ vs. time and strain vs. time during tensile loading and unloading for single carbon fiber embedded in epoxy. Solid curve: $\Delta R/R_0$ vs. time. Dashed curve: tensile strain vs. time.*

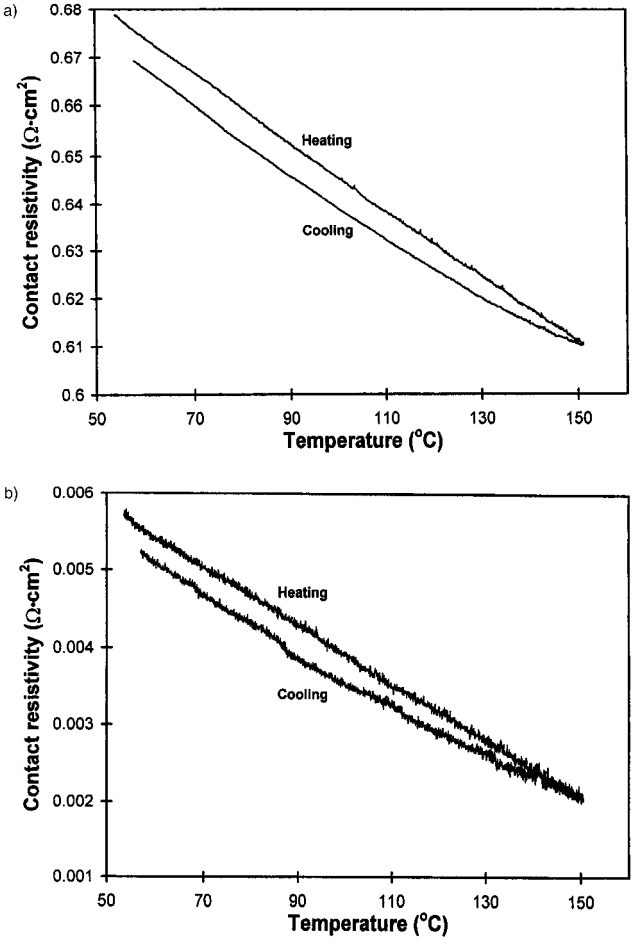


*Fig. 10. Variation of contact electrical resistivity with temperature during heating and cooling at 0.15 °C/min a) for sample made without any curing pressure and b) for sample made with a curing pressure 0.33 MPa.*

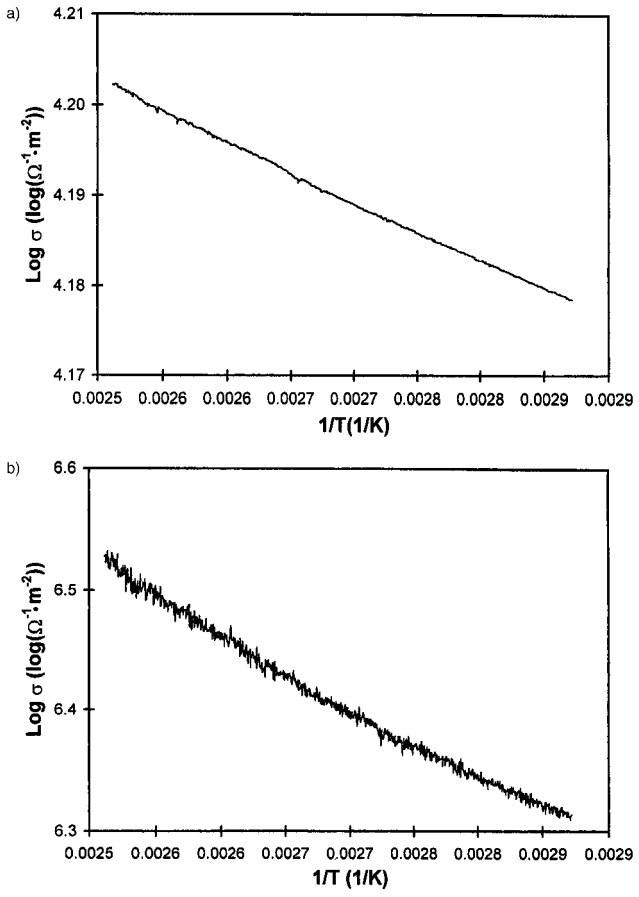


*Fig. 11. Arrhenius plot of log contact conductivity vs. inverse absolute temperature during heating at 0.15 °C/min a) for sample made without any curing pressure and b) for sample made with curing pressure 0.33 MPa.*

tion of composite materials with an insulating matrix and an electrically conducting filler (whether particles or fibers). Based on volume resistivity measurement, an activation energy in the range from 0.060 to 0.069 eV has been previously reported for short carbon fiber polymer–matrix composites.[23] Direct measurement of the contact resistivity is impossible for the short fiber composites.

The activation energies, thicknesses and room temperature contact resistivities for samples made at different curing pressures and composite configurations are shown in Table 1. For the same composite configuration (crossply), the higher is the curing pressure, the smaller is the composite thickness (because of more epoxy being squeezed out), the lower is the contact resisitivity, and the higher is the activation energy. A smaller composite thickness corresponds to a higher fiber volume fraction in the composite. During curing and subsequent cooling, the matrix shrinks while the carbon fibers essentially do not, so a longitudinal compressive stress will develop in the fibers. For carbon fibers, the modulus in the longitudinal direction is much higher than that in the transverse direction. Thus, the overall shrinkage in the longitudinal direction tends to be less than that in the transverse direction. Therefore, there will be a residual interlaminar stress in the two crossply

*Table 1. Activation energy for various composites. The standard deviations are shown in parentheses.*

| Composite configuration | Curing pressure [MPa] | Composite thickness [mm] | Contact resistivity $\rho_{co}$ [$\Omega.cm^2$] | Activation energy [eV] | | |
|---|---|---|---|---|---|---|
| | | | | Heating at 0.15°C/min | Heating at 1°C/min | Cooling at 0.15°/min |
| Crossply | 0 | 0.36 | 0.73 | 0.0131 ($2 \times 10^{-5}$) | 0.0129 ($3 \times 10^{-5}$) | 0.0125 ($8 \times 10^{-6}$) |
| | 0.062 | 0.32 | 0.14 | 0.0131 ($4 \times 10^{-5}$) | 0.0127 ($7 \times 10^{-5}$) | 0.0127 ($4 \times 10^{-5}$) |
| | 0.013 | 0.31 | 0.18 | 0.0168 ($3 \times 10^{-5}$) | 0.0163 ($4 \times 10^{-5}$) | 0.0161 ($2 \times 10^{-5}$) |
| | 0.19 | 0.29 | 0.054 | 0.0222 ($3 \times 10^{-5}$) | 0.0223 ($3 \times 10^{-5}$) | 0.0221 ($1 \times 10^{-5}$) |
| | 0.33 | 0.26 | 0.0040 | 0.118 ($4 \times 10^{-4}$) | 0.129 ($8 \times 10^{-4}$) | 0.117 ($3 \times 10^{-4}$) |
| Unidirectional | 0.42 | 0.23 | 0.29 | 0.0106 ($3 \times 10^{-5}$) | 0.0085 ($4 \times 10^{-5}$) | .0081 ($2 \times 10^{-5}$) |

layers in a given direction. This stress accentuates the barrier for the electrons to jump from one lamina to the other. The greater the residual interlaminar stress, the higher the barrier, which is the activation energy. After curing and subsequent cooling, heating will decrease the thermal stress, due to the CTE mismatch between fibers and matrix. Both the thermal stress and the curing stress contribute to the residual interlaminar stress. Therefore, the higher the curing pressure, the larger the fiber volume fraction, the greater the residual interlaminar stress, and the higher the activation energy, as shown in Table 1.

The curing pressure for the sample in the unidirectional composite configuration is higher than that of any of the crossply samples (Table 1). Consequently, the thickness is the lowest. As a result, the fiber volume fraction is the highest. However, the contact resistivity of the unidirectional sample is the second highest rather than being the lowest, and its activation energy is the lowest rather than the highest. The low activation energy is consistent with the fact that there is no CTE or curing shrinkage mismatch between the two unidirectional laminae and, as a result, no interlaminar stress between the laminae. This low value supports the notion that the interlaminar stress is important in affecting the activation energy. The high contact resistivity for the unidirectional case can be explained in the following way. In the crossply samples, the pressure during curing forces the fibers of the two laminae to press on to one another and hence contact tightly. In the unidirectional sample, the fibers of one of the laminae just sink into the other lamina at the junction, so pressure helps relatively little in the contact between fibers of adjacent laminae. Moreover, in the crossply situation, every fiber at the lamina–lamina interface contacts many fibers of the other lamina, while, in the unidirectional situation, every fiber has little chance to contact the fibers of the other lamina. Therefore, the number of contact points between the two laminae is less for the unidirectional sample than the crossply samples.

By measuring the contact electrical resistance of the interlaminar interface of a unidirectional continuous carbon fiber epoxy–matrix composite during shear, the interlaminar shear process can be monitored in real time.[7] The resistance increases throughout the shear process for a low curing pressure, but decreases in the initial stage of shear for a high curing pressure. The resistance increase is due to delamination and strain in the interface region during shear. The resistance decrease observed for a high curing pressure is believed to be due to interlaminar rubbing and slight damage of the matrix between the fiber layers, and the consequent increase in the number of contacts between fibers of the adjacent laminae. The interlaminar displacement is negligible prior to shear failure.

The contact electrical resistivity of the interlaminar interface can be used to monitor thermal damage in a continuous carbon fiber epoxy–matrix composite in real time during thermal cycling.[24] The resistivity increases in spikes and its baseline shifts due to thermal damage.

## 5. Conclusion

The use of electrical resistance measurement to study fibrous composite interfaces provides information on the fiber–matrix interface, the residual stress in the fiber embedded in the matrix, and the interlaminar interface. Measurements relate to the contact resistance of the interface and the volume resistance of the fiber, and can be made in real time during debonding, residual stress reduction, temperature change, and interlaminar shear. The methods are demonstrated by using carbon fiber polymer–matrix and cement–matrix composites, due to the conductivity of carbon fibers.

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