

Interlaminar shear in carbon fiber polymer-matrix composites, studied by measuring the contact electrical resistance of the interlaminar interface during shear

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Received 11 June 1998; accepted 5 January 1999

Abstract—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 was monitored in real time. The resistance increased throughout the entire shear process for a low curing pressure, but decreased in the initial stage of shear for a high curing pressure. The resistance increase was 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 was negligible prior to shear failure.

Keywords: Polymer-matrix composites; carbon fiber; interlaminar; electrical resistance; contact resistance; interface; shear; epoxy.

1. INTRODUCTION

Interlaminar shear is one of the dominant modes of failure of continuous fiber composites, especially those that are not woven in the direction perpendicular to the laminae (fiber layers). The study of the interlaminar shear process has been focused on measurement of the interlaminar shear strength (ILSS) by techniques such as the short-beam method [1], the Iosipescu method [2] and related methods [3]. Much less attention has been given to the observation of the interlaminar shear process prior to shear failure, due to the limited techniques available, such as the photo-stress method [3]. However, observation of the interlaminar shear process in real time is valuable for understanding the mechanism of interlaminar shear. This

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paper provides a new method for observing the interlaminar shear process. This method involves measurement of the contact electrical resistance of the interlaminar interface during shear. We use the term 'interlaminar interface' to indicate the interface between the fibers of adjacent laminae and to differentiate from the interface between the fibers within a lamina. The method was applied to carbon fiber polymer-matrix composites, which are widely used for lightweight structures. The electrical conductivity of the carbon fibers is important for the applicability of this method.

Although the polymer matrix (epoxy) is electrically insulating, a carbon fiber polymer-matrix composite is electrically conducting in the through-thickness direction. This is because of contacts between fibers of adjacent laminae. The contacts are due to non-uniformity in the resin flow during composite fabrication and due to fiber waviness. Thus, during delamination, the volume electrical resistivity of the composite in the through-thickness direction increases [4]. Instead of measuring the volume electrical resistivity of the composite, we measured the contact electrical resistivity of the interlaminar interface, as the latter quantity directly describes the state of the particular interface under observation, whereas the former quantity describes the overall state of many interfaces that are all perpendicular to the through-thickness direction, which is the direction of resistivity measurement. Furthermore, the volume resistivity is affected both by the interlaminar interface resistance as well as the volume resistance within a lamina in the through-thickness direction. The contact resistivity of the interlaminar interface has previously been measured as a function of temperature [5], but has not been previously measured during interlaminar shear.

The contact electrical resistivity ρ of an area A is

$$\rho = RA, \quad (1)$$

where R is the contact resistance. R depends on geometry, but ρ does not. In other words, ρ describes the quality of that contact. Both A and ρ affect R , which is the quantity measured. During interlaminar shear, A may decrease (due to interfacial shear displacement) and ρ may increase (due to interface degradation). In order to distinguish between these two contributions, the interfacial shear displacement was measured in this work by subtracting from the extension of an extensometer attached to the specimen across the interface area (parallel to the interface) the contribution by the tensile strain of the laminae undergoing interlaminar shear. After the subtraction, we found that the interfacial shear displacement is negligible. Hence, A essentially does not change during interlaminar shear prior to failure. As a result, increase in ρ is the cause of the measured increase in R during interlaminar shear. Thus, by monitoring R , the change in the quality of the interlaminar interface during interlaminar shear was studied in this work.

The pressure applied on the composite in the through-thickness direction during composite fabrication (during curing of the epoxy) must affect the structure of the interlaminar interface, thereby affecting the interlaminar shear process. Although

much work has been done on studying the effects of isothermal aging [6], fiber surface treatment [7–9] and void content [10] on ILSS, little work, if any, has been done on studying the effect of curing pressure. This paper is also aimed at studying the effect of curing pressure on the interlaminar shear process by contact resistance measurement during interlaminar shear.

Specific questions addressed in this paper are the following. Firstly, how does the quality of the interlaminar interface change during interlaminar shear? Secondly, what is the extent of interlaminar shear displacement needed for interlaminar shear failure? Thirdly, how does the pressure during composite fabrication affect the interlaminar shear process?

The experimental method used in this work for simultaneous measurement of contact electrical resistance and load during interlaminar shear is an extension of the technique of electromechanical pull-out testing [11], which is a method for simultaneous measurement of the contact resistance, shear load and displacement during the pull-out of a single, electrically conducting fiber embedded at one end in an electrically conducting matrix, such as cement. In contrast to electromechanical pull-out testing, in which shear occurs between fiber and matrix, this work involves shear between one lamina and another in the same composite. Because of the electrical conductivity of the carbon fibers, every lamina is conducting. Due to the need for simultaneous electrical and mechanical measurements, the specimen geometry for interlaminar shear testing in this work is different from those of other methods for interlaminar shear testing [1–3].

2. EXPERIMENTAL

Samples were made from unidirectional carbon fiber prepreg tapes manufactured by ICI Fiberite (Tempe, AZ). The product used was Hy-E1076E, which consisted of a 976 epoxy matrix and 10E carbon fibers (10E-Torayca T-300 (6K) untwisted, UC-309 sized). Each sample was made from four strips (A1, A2, C and B) of the prepreg tapes, as shown in Fig. 1a. Strips A1 and A2 formed a 'socket'; strip B formed a 'plug'. When strip B was pulled out of the socket formed by strips A1 and A2, interlaminar shear occurred at the interface between A1 and B and that between A2 and B. Strip C and two strips of releasing paper (R1 and R2) were present to make the curing pressure on the laminate more uniform and the sample configuration more symmetrical (for avoiding bending moment). Each strip was 6 mm in width (dimension perpendicular to the plane of Fig. 1a and 50 mm in length, except that strip C was 47 mm long. The interfaces at which interlaminar shear occurred was 2 mm long. The area undergoing shear was the combined area of these two symmetrically positioned interfaces.

After the strips of prepreg and releasing paper had been arranged as in Fig. 1a, they were put into a steel mold and placed between the two heating platens of a Carver hot press, where it was cured at $175 \pm 2^\circ\text{C}$ for 10 h. The heating rate was $2.5^\circ\text{C}/\text{min}$. The pressure (0, 0.042, 0.87 or 3.5 MPa) on the laminate during curing

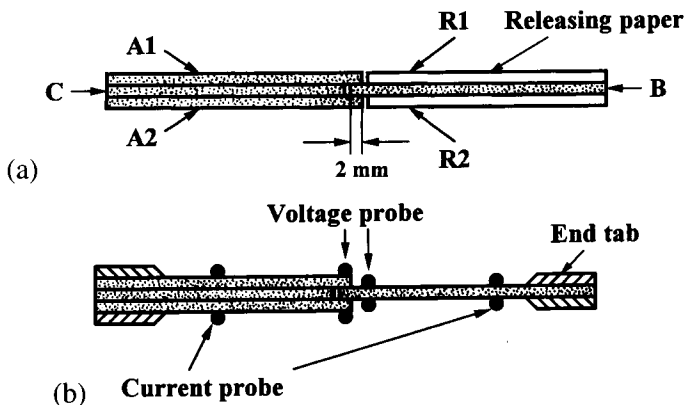


Figure 1. Sample configuration (side view) (a) during curing (A1, A2, B and C are strips of prepreg, R1 and R2 are strips of releasing paper), (b) for mechanical testing and electrical resistance measurement.

was provided by a steel weight or a hydraulic press (for 3.5 MPa only). The volume fractions of fibers for the curing pressures of 0, 0.042, 0.87 and 3.5 MPa are 0.523, 0.550, 0.618 and 0.619, respectively. The pressures of 0 and 0.042 MPa were below typical conventional curing pressures; the pressure of 0.87 MPa was close to typical curing pressures (~ 0.6 MPa); the pressure of 3.5 MPa was above typical curing pressures. After curing, the samples were cooled to room temperature at a rate of $0.18^{\circ}\text{C}/\text{min}$. After curing and subsequent cooling, the pieces of releasing paper were removed, and end tabs and electrical contacts were attached to each sample, as shown in Fig. 1b.

The pull-out test was conducted by using a screw-action mechanical testing system (Sintech 2/D). The displacement rate was 0.30 mm/min. A one-inch extensometer was attached to the specimen across the interlaminar interface area (parallel to the interface) to measure the extension of the center part of the specimen (Fig. 1b). In order to obtain the elongations of both the one-lamina part and the three-lamina part of each specimen (Fig. 1b) without disturbing the pull-out testing, strain gages were attached to ‘dummy’ samples, which, unlike Fig. 1b, had either one lamina or three laminae *throughout* the length of the sample; these samples were prepared and tested in the same way as the specimens of Fig. 1b. The elongations of both the one-lamina part and the three-lamina part of the samples of Fig. 1b were calculated from the strains of the corresponding dummy samples under the same load and from their original lengths governed by the range of the extensometer. Simultaneous to mechanical testing, DC electrical resistance was measured using the four-probe method with a Keithley 2001 multimeter. Silver electrically conducting paint was used for all electrical contacts. The two voltage probes were so close to each other (at a distance of 2 mm) that the contribution of the volume electrical resistance to the measured resistance between the voltage probes was negligible compared to the contribution of the contact resistance at the interfaces undergoing interlaminar shear.

Fracture surfaces (obtained after pull-out) of both 'plug' and 'socket' of each sample were examined using scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSION

Figure 2 shows an SEM photograph of a plug after it has been pulled out in the direction from left to right in the figure. The left half of the photograph is the interlaminar interface, while the right half is the exposed part of the plug. The left half shows matrix shear cusps, which indicate shear fracture [12].

Figure 3 shows the variation of the fractional change in contact electrical resistance ($\Delta R/R_0$) with the interlaminar shear stress. Generally, with the interlaminar shear stress increasing, the contact resistance increases. At the point of shear failure, the resistance increases to infinity. The difference between the extension measured by the extensometer and the elongation calculated from the strain of the dummy samples is less than 0.05 mm — negligible compared to the length (2 mm) of the interface. Thus, there is almost no displacement at the interlaminar interface before shear failure. Moreover, observation of the entire fracture surface of each sample showed no distinct regions. This negative observation supports the absence of displacement before shear failure. Therefore, the increase in contact resistance is basically because of the delamination and shear strain at the interface. Both de-

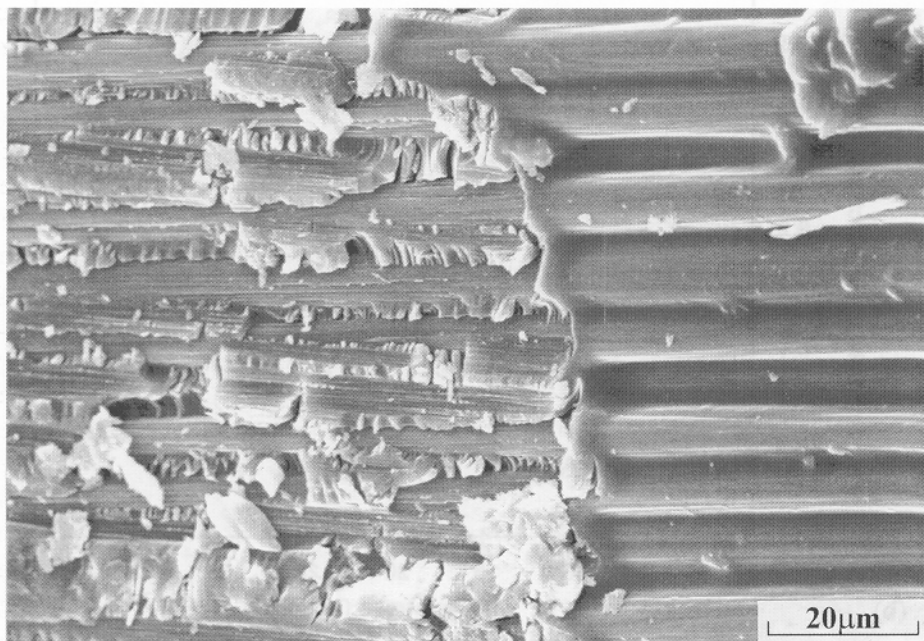


Figure 2. SEM photograph of a typical fracture surface of a plug after pull-out from left to right. The right half is the free surface adjacent to the interlaminar interface. The left half is the interlaminar interface, which indicates shear fracture.

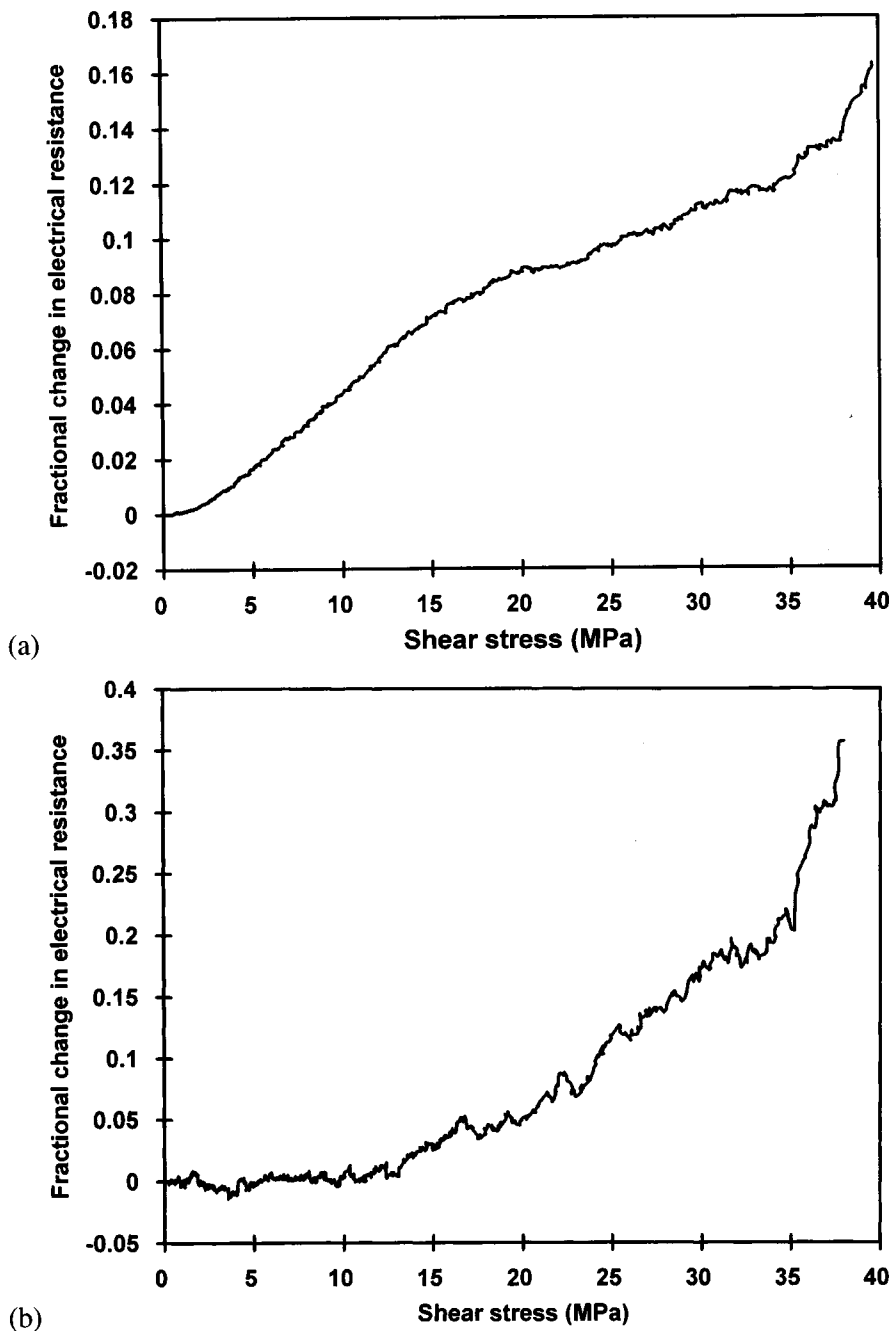


Figure 3. Variation of stress fractional change in electrical resistance with interlaminar shear stress for samples with curing pressures 0 MPa (a), 0.042 MPa (b), 0.87 MPa (c), and 3.5 MPa (d), respectively.

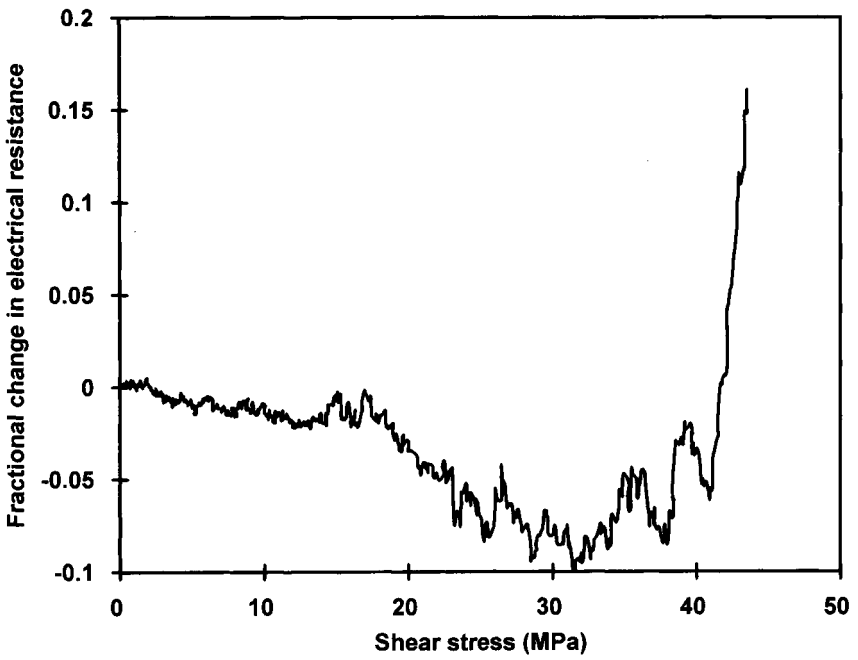
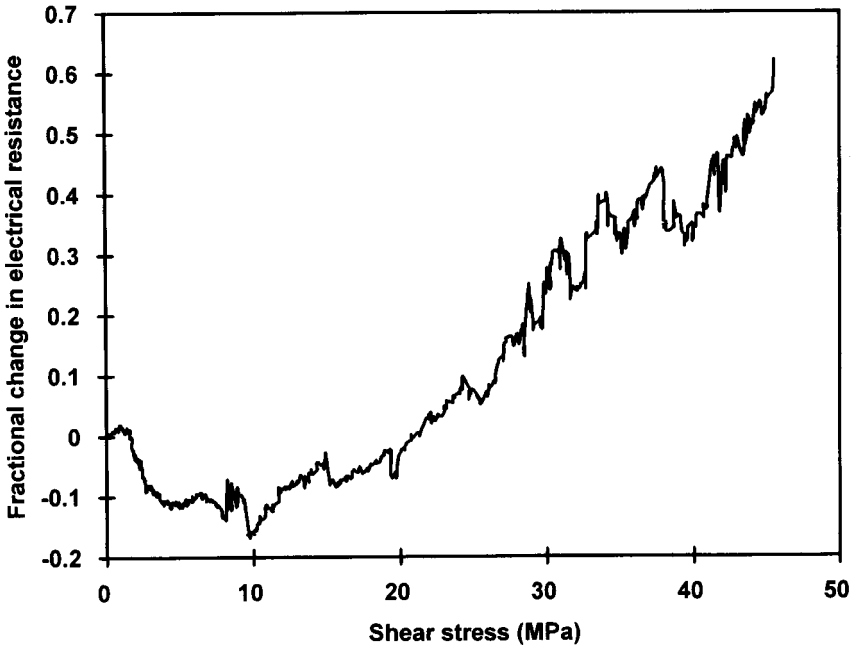


Figure 3. (Continued).

lamination and strain decrease the interlaminar fiber–fiber contact, and increase the interlaminar contact resistivity. Table 1 shows the load per unit original interface area (F/A_0) and $\Delta R/R_0$ for different curing pressures right before failure. At curing pressures below 3.5 MPa, a higher curing pressure is associated with a higher $\Delta R/R_0$ before failure. This is reasonable, since a higher curing pressure tends to improve the interlaminar bond.

Comparing the curves in Fig. 3a, b, c and d, it can be noticed that these curves are quite different. In Fig. 3a, with the lowest curing pressure of 0 MPa, the resistance starts to increase at a small shear stress. In Fig. 3b, with the curing pressure of 0.042 MPa, the resistance starts to increase at a much higher shear stress. In Fig. 3c, with the curing pressure of 0.87 MPa, the resistance even decreases at small shear stresses prior to increasing at larger shear stresses. In Fig. 3d, with the highest curing pressure of 3.5 MPa, the resistance change remains negative over almost the entire shear process; the resistance increases abruptly just near the end of the process.

Since delamination and strain must increase the contact resistance, there must exist another mechanism, which decreases the resistance as shear occurs. During cure, the pressure can squeeze some fibers of a lamina into the adjacent lamina. It is possible that during shear, the shear strain, though small, would make these fibers rub and interlock with the fibers of the adjacent lamina. The rubbing and interlocking among the fibers, combined with the damage of the matrix between the adjacent fiber layers, would increase the chance of interlaminar fiber–fiber contact and hence decrease the interlaminar electrical resistance. (SEM observation failed to show discernible differences in fracture surfaces for different curing pressures and $\Delta R/R_0$.) This mechanism will weaken the opposite effect of delamination and strain. When the curing pressure is low (0 and 0.042 MPa), there are relatively few fibers of one lamina squeezed into the adjacent lamina, and the matrix between the laminae is relatively thick, so the rubbing and interlocking as well as matrix damage have relatively little effect on the interlaminar fiber–fiber contact. Hence, the mechanism that decreases the interlaminar resistance is nearly absent and the resistance increases with shear stress without initially decreasing. When the curing pressure is high (0.87 and 3.5 MPa), the fibers of one lamina that are squeezed

Table 1.
Effect of curing pressure on interlaminar shear

Curing pressure (MPa)	Load/original interface area right before failure* (MPa)	Fractional change in electrical resistance right before failure
0	39.7	0.169
0.042	38.2	0.361
0.87	45.5	0.646
3.5	43.8	0.186

* Same as the interlaminar shear strength.

into the adjacent lamina are more plentiful and they penetrate more deeply into the adjacent lamina. In addition, the matrix between the laminae is thinner. Therefore, rubbing and interlocking occur between the laminae. The effect of rubbing, interlocking and matrix damage is so strong that it overshadows the opposite effect of delamination and strain, so the change in the resistance even becomes negative, at least in the initial stage of shear. It is this decrease in resistance that causes the fractional change in resistance right before failure to be particularly low for the curing pressure of 3.5 MPa.

Figure 4 shows the fracture surface of the plug of the sample made at the maximum curing pressure (3.5 MPa). It shows ditches in the shear direction. One of the ditches is indicated by an arrow in Fig. 4. At the left end of a ditch is a clump of matrix, which appears to have been squeezed or rubbed out from the ditch during pull-out in the direction from left to right. The ditches are probably due to the fibers of the adjacent lamina having got into this lamina and subsequently being pulled out during the final fracture. These ditches were not found in the fracture surfaces of the samples made at lower curing pressures.

$\Delta R/R_0$ depends on the quantity and quality of the interlaminar fiber-fiber contacts, which, for a sample made at a certain curing pressure, is a function of the extent of delamination, strain, matrix damage, and the rubbing and interlocking among the fibers, etc. All these factors relate to the interlaminar shear stress.

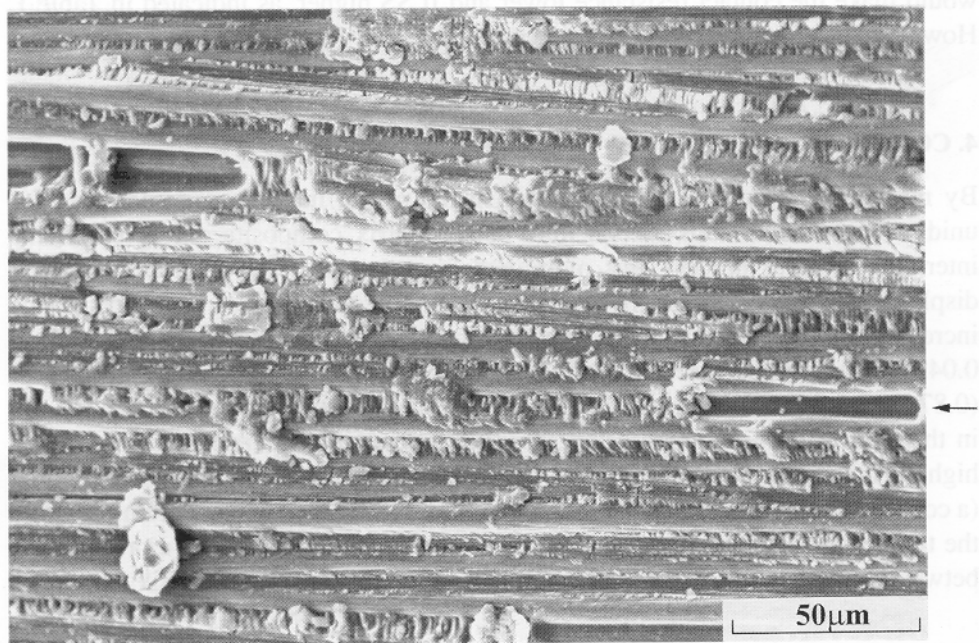


Figure 4. SEM photograph of the fracture surface of the plug of the sample made at the curing pressure of 3.5 MPa. The ditch, indicated by the arrow, suggests that fibers in the adjacent lamina had got into this lamina and were pulled out during the final failure.

The relationship between these factors and $\Delta R/R_0$ is complicated and difficult to quantify. $\Delta R/R_0$ is a function of interlaminar shear stress, such that, generally speaking, there exist two stages and a critical interlaminar shear stress between them. During the first stage, $\Delta R/R_0$ decreases with increasing interlaminar shear stress because the rubbing and interlocking are dominant when the interlaminar shear stress is low. During the second stage, $\Delta R/R_0$ increases with increasing interlaminar shear stress because strain and delamination are dominant in this stage. For a composite with a low curing pressure, the first stage nearly vanishes and the critical shear stress is zero, since the fibers of adjacent laminae are not close enough to provide a significant chance of rubbing and interlocking. However, when the curing pressure increases, the first stage becomes more extended and the critical interlaminar shear stress becomes higher.

For a sample made with a certain curing pressure, $\Delta R/R_0$ provides an indication of what occurs in the sample during interlaminar shear. During the initial stage, in which $\Delta R/R_0$ decreases with increasing interlaminar shear stress, the rubbing and interlocking are dominant; this phenomenon serves to indicate that the sample is not near fracture. In the final stage, $\Delta R/R_0$ increases rapidly with increasing shear stress; this phenomenon serves to indicate that the sample is about to break.

The impact of curing pressure on the extent of cure of the resin should not be significant, for the curing time is long enough (10 h) for every sample. With a higher curing pressure, the void content in the composite should be lower, which would make the contact resistance lower and ILSS higher, as indicated in Table 1. However, the effect of the lower void content on $\Delta R/R_0$ is not clear.

4. CONCLUSION

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 was monitored in real time. The interlaminar shear displacement was found to be negligible prior to shear failure. The resistance increased throughout the entire shear process for a low curing pressure (0 or 0.042 MPa), but decreased in the initial stage of shear for a high curing pressure (0.87 or 3.5 MPa). The resistance increase was due to delamination and strain in the interface region area during shear. The resistance decrease observed for a high curing pressure is believed to be due to the increase in interlaminar rubbing (a consequence of increased interlocking among fibers of the adjacent laminae), and the thinner matrix layer between the laminae, which together led to more contacts between fibers of the adjacent laminae upon slight damage of the matrix.

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