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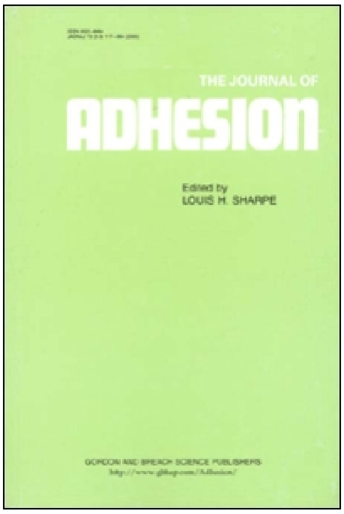

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Effects of External Loading of Fiber on Fiber/Matrix Interfacial Shear Strength*

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The effect of external loading of fiber on fiber/resin interfacial adhesion has been studied using the microbond technique. The results show that the applied load on the fiber weakened the fiber/matrix bonding at the interface. The effect of cyclic loading of fiber on fiber/matrix interfacial adhesion was also investigated by loading and unloading the fiber by means of a moving platform oscillating vertically at a specific frequency. After the cyclic loading process, residual bond strengths were measured using the TRI microbond pullout procedure.

Fiber/matrix combinations of Kevlar 49®/Epon 828® and Kevlar 49/polycarbonate have been investigated using this technique. Significant bond strength reduction is observed when load is applied and after cyclic loading. In the latter case, most of the loss occurs after relatively few cycles. Possible bond weakening mechanisms are proposed to explain the experimental results.

KEYWORDS: interface; composite; adhesion; external loading; cyclic loading; microbond

INTRODUCTION

Composite materials are usually exposed to conditions of extended fatigue rather than a single loading that approaches or exceeds the ultimate strength of the composite structure. Fatigue resistance is, therefore, an important long term property of composite materials. Studies have been conducted to investigate the fatigue life of various types of fiber reinforced composites[1–3] as well as the effects of fatigue on other properties, such as tensile strength,[4] interlaminar shear strength,[5] stiffness,[6] and dynamic visco-elastic properties.[7] A number of review papers have also presented analyses of damage mechanisms in composite fatigue.[8–12]

Although there is a large data base, the role of the interphase in the fatigue processes is still not well understood. This is due to the complex nature of the composite system; the structure can be weakened by failure of fibers or matrix or cracks at the interface. Most fatigue studies reported in the literature are performed on actual composite specimens, and the complexities of specimen failure make it difficult to isolate the effects of fatiguing on the interfacial bond.

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*One of a Collection of papers honoring Lawrence T. Drzal, the recipient in February 1994 of *The Adhesion Society Award for Excellence in Adhesion Science, Sponsored by 3M.*

Monofilament composites have been used to isolate the effects of fatigue on the fiber/resin interphase. The first "interfacial bond fatigue test" was performed on a macroscopic model (1 cm diameter steel rod embedded in transparent epoxy), which showed a slow crack debonding process.[13] This suggested that interfacial debonding in fiber reinforced composites might follow a cumulative fracture or fatigue failure process. Full-scale interfacial fatigue behavior in real engineering fiber/resin systems was subsequently observed by fatiguing single filament microcomposites. Dibenedetto *et al.*[14] reported the interfacial bond fatigue behavior of single carbon fiber embedded in epoxy resin. An oscillating load was applied to a fragmentation coupon sample. They noticed that the number and size of local zones of matrix yielding taking place along the interface increased with increasing fatigue cycles. This qualitative observation was in agreement with the macroscopic model, indicating a cumulative fracture process. Schadler *et al.*[15] studied the fatigue behavior of a carbon fiber/polycarbonate system using the same fragmentation technique. In this study, fatigue loading with varying frequency and amplitude was applied to the coupon in either the axial or transverse direction, and the residual stress transfer efficiency of each fatigued specimen was determined. They concluded that fatigue behavior at the interphase is matrix dominated in the transverse direction and dependent on the fiber strength in the axial direction. Latour *et al.*[16, 17] studied the fiber/resin interfacial fatigue behavior using the microbond single fiber pull-out technique developed at TRI.[18] A sinusoidal pull-out load was applied to the microdroplet on the fiber until fiber/matrix debonding occurred and the numbers of fatigue cycles to failure were recorded as the fatigue characteristics of the studied systems. They observed that fatigue behavior was related to the ultimate interfacial bond strength.

The common feature of all of these fatigue studies on monofilament composites is that the resin phase was always constrained by some means. In the fragmentation test, the fatiguing stress is applied directly to the resin coupon with the fiber embedded in it. In the microbond test, shearing plates apply stress to the resin droplet against the pull-out load applied to the single filament. These conditions may cause crack initiation and propagation in the resin phase, thus complicating the fatiguing process.

In this study, we have studied the micro-composite systems using a different technique, *i.e.*, applying load on the fiber. The cyclic and static loads were applied to the fiber without any external restraining contact with the resin droplet. An external tensile load is applied to the free fiber ends of a microbond specimen. This avoids crack initiation and propagation in the resin phase. The effects of loading, both static and cyclic, on the interfacial bonding are quantified by measuring the bond strength of the specimens with the applied load on the fiber or after cyclic loading. With the high modulus of Kevlar fiber, it might be expected that the load applied to the fiber will induce little strain and, consequently, result in limited effect at the interface; however, as we report, the experimental data show that applied external loading did weaken the fiber/matrix interfacial adhesion.

EXPERIMENTAL

The microbond technique for determining fiber/matrix bond strength involves depositing and curing a resin microdroplet on a single fiber. The fiber is then pulled out from the resin and the interfacial shear strength determined from the pull-out load and the contact area between the two phases. The details of this procedure are given in References 18 and 19.

To study the effect of external static loading of the fiber on the microbond measurement, we conducted modified microbond experiments on Kevlar 49/Epon 828 specimens, which were processed and tested under different tensioning conditions. Besides the control sample (regular run), three series of experiments were designed and conducted as diagrammed in Figure 1. In Series I, three different weights (5, 10, and 16.7 grams) were used to investigate the effect of different levels of fiber tension applied after

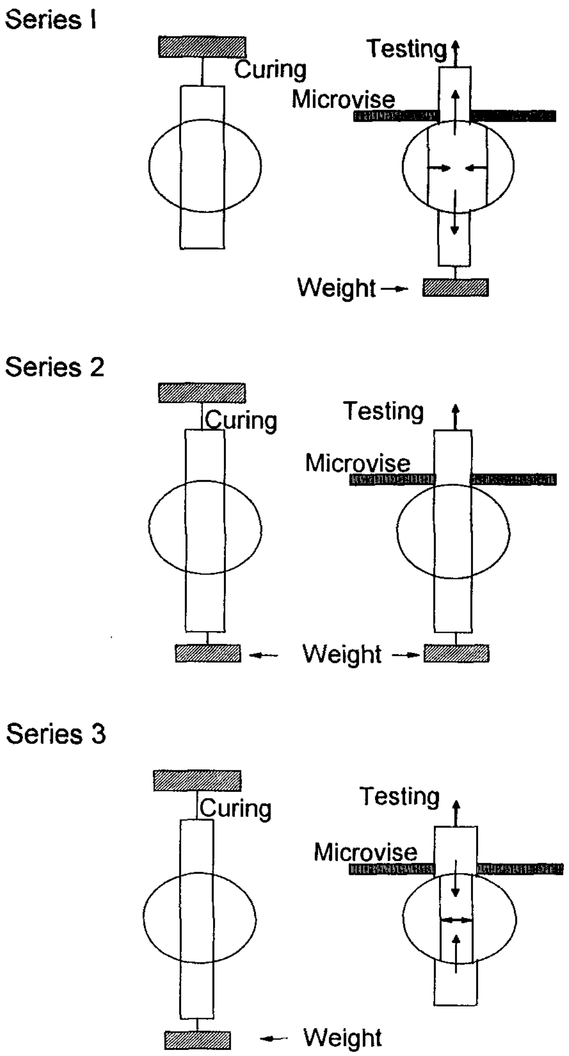


FIGURE 1 Experimental design.

curing, that is, during the microbond measurements. In Series II and III, a 5-gram weight was attached to the fiber before curing. In Series II, the weight was left on during microbond measurements, in Series III the weight was taken off before the measurements.

The cyclic loading study was conducted on two systems: Kevlar 49/epoxy (Epon 828 from Shell) and Kevlar 49/PC (polycarbonate from Coburn). Epon 828 was cured with 4:1 w/w methylene dianiline (Tonox from Miller-Stephenson) at 80°C for 2 hours followed by 3 hours postcure at 150°C. PC droplets were formed by melting thin PC film on single fibers at 275°C for 30 minutes, as described elsewhere.[19] Specimens prepared with fibers free of tension were then suspended from a stationary platen, and each fiber loaded with a 20 gram weight. The ultimate tensile strength (UTS) of Kevlar 49 fiber is about 3.8 GPa,[20] which is equivalent to a load of about 40 grams. Cyclic loading of fibers was accomplished by loading and unloading the weights by means of a moving platform oscillating vertically at a frequency of 30 cycles/minute, as shown in Figure 2. After cyclic loading, the residual bond strengths were measured using the microbond pullout procedure.

Some filaments broke almost immediately after load was applied, and 90% ruptured within the first 24 hours. At this point the experiment was terminated, and all the specimens (broken and unbroken) were evaluated using the microbond technique. For those fibers that had ruptured during the cyclic loading process, the break was usually far enough from the microdroplet that a bond strength measurement could still be obtained.

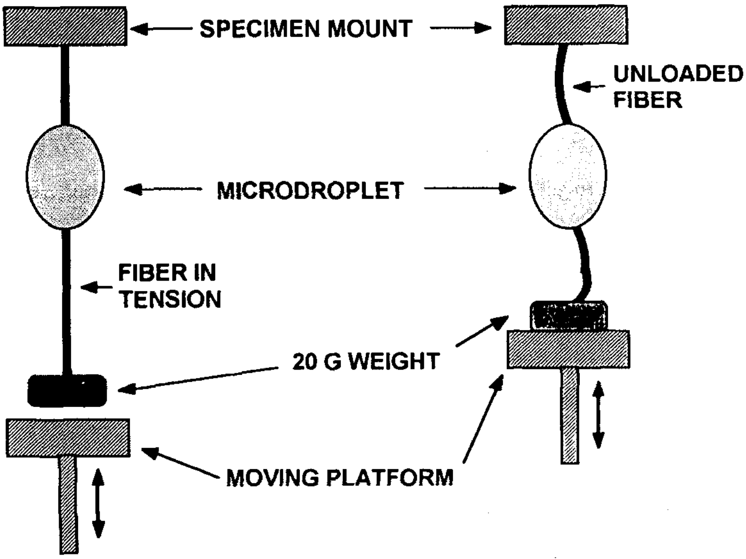


FIGURE 2 Schematic of TRI fatigue setup for imposing cyclic stress on a fiber bearing a resin microdroplet.

RESULTS AND DISCUSSION

1 Effects of Static Loading on Fiber

The average measured bond strengths of microbond specimens tested with fibers under tension, normalized with respect to the control values, are shown in Figure 3. In each case the fiber/resin bond strength has been reduced to about 69–75% of the original value.

It is proposed that the lowered interfacial bond strengths of the specimens with fibers under tension are due to the stresses generated at the interface by loading the ends of fibers. A fiber under tension can undergo axial extension and, due to the Poisson's ratio effect, radial shrinkage. However, the portion of the fiber that is embedded in the resin is constrained from deformation by adherence to the resin. This generates both shearing and axial (shrinkage from Poisson's ratio) stresses at the interface.

Because of the high modulus of the Kevlar fiber, the stresses generated on the fiber from the external loading ought to be small, however, the experimental results showed that a stress equivalent to at least 25% of the original bond strength was generated, indicating that small stresses present at the interface would have a significant effect on the interfacial adhesion. As mentioned earlier, there should be two stresses, shearing and axial, present at the interface. At this point, we are not able to determine the relative contributions of the shearing and shrinkage stress effects on the microbond measurement. However, it has been reported that in a fiber pull-out test the debonding load seems to be more sensitive to the change of axial stress at the interface.[21] This series of experiments provides a quantitative measure of the effective shear stress on the interface as a result of loading both fiber ends of a microbond specimen. However, it is not apparent why the reduction in bond strength is independent of the amount of loading.

The hypothesis that the stresses are generated at the interface from loading the fiber is further supported by the experimental results obtained from the Series II experiment. In this case, loading was applied to the fibers before curing the resin droplets, that is,

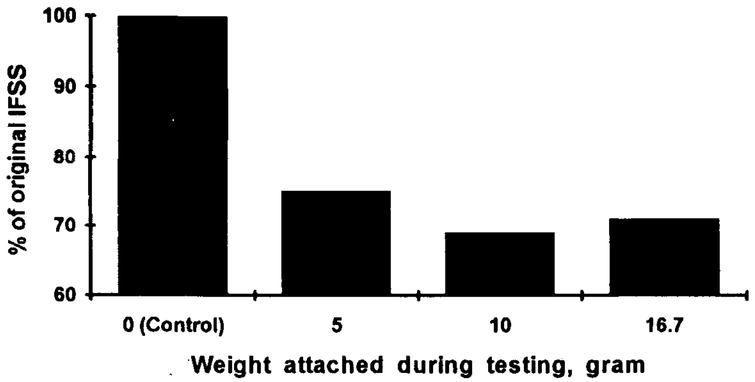


FIGURE 3 Normalized bond strengths of Kevlar 49/Epopn 828 with fiber under tension.

predeforming the fibers by applying tensile load and then subsequently depositing the liquid resin microdroplet on deformed filaments. The bond strengths were measured on these specimens while they were still under the same tensile load. Comparison of the measured bond strengths with the control values showed *no* change in bond strength as a result of deforming the fiber prior to resin deposition. This supports the hypothesis that it is the post-cure loading of the fiber that generates stresses at the interface.

For Series III, another set of microbond specimens was prepared and cured with a weight attached to the fiber, but the specimens were unloaded before the bond strength measurements were made. Upon unloading a specimen, the portion of the fiber which was embedded in the cured resin might be expected to try to return to its original undeformed dimensions, *i.e.*, shrink in the axial direction and expand in the radial direction. The fiber axial shrinkage should generate a reverse shear stress at the interface, while the fiber radial expansion should induce a compressive stress at the interface. Both stresses would be expected to increase the bond strength.

The experimental results showed, however, that there was *no* change in the measured bond strength in this series of specimens, implying that no stresses were generated at the interface with the pre-extended embedded fiber. Although Kevlar 49 is well known for its high creep resistance, it has been reported that its creep increases with increasing temperature.[22] Therefore, it is possible that the fiber has undergone permanent inelastic deformation as a result of tensile stress during high temperature curing (150°C for 3 hours) and, consequently, was not able to retract to its original dimensions when the specimen was unloaded. Scherf and Wagner[23] observed that, in a carbon fiber/epoxy system, a relatively high level of pre-tension on the fiber is required to affect the IFSS measurement. Therefore, it is also possible that the 5 gram weight attached to the fiber before resin curing was not great enough to cause any noticeable change in the microbond measurement.

2 Cyclic Loading Studies

Cyclic loading was applied to microbond specimens. In a typical experiment, 40–50 specimens were tested and some filaments failed during the process. A typical distribution of filament failure times is presented in Figure 4. Specimens that broke immediately and those that broke after 7 to 23 hours (overnight failures) were not evaluated, since their exact cyclic loading times could not be established.

Because premature fiber failure occurred at widely varying times, specimens with a broad distribution of cyclic loading times were available to study the changes in bond strength as a function of time. Bond strength as a function of loading time for Kevlar 49/Epon 828 and Kevlar 49/PC systems are shown in Figures 5 and 6, respectively. The average value of IFSS shows a sharp drop at the onset of cyclic loading (within 15 minutes), followed by a small increase with additional fatiguing in the case of the Kevlar 49/Epon 828 system. A similar trend of time-dependent bond strength reduction and partial reversal of the effect upon extended cyclic loading was also observed for the Kevlar 49/PC system.

The data were divided into three subgroups on the basis of cyclic loading time. A Student *t* test was performed to distinguish any statistically-significant differences between the data sets. The average bond strengths with their standard deviations for

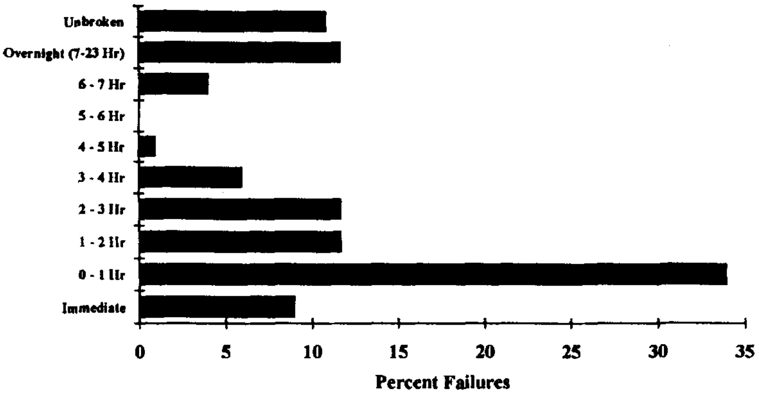


FIGURE 4 Distribution of times to fiber failure in the cyclic microbond specimen fatigue experiment.

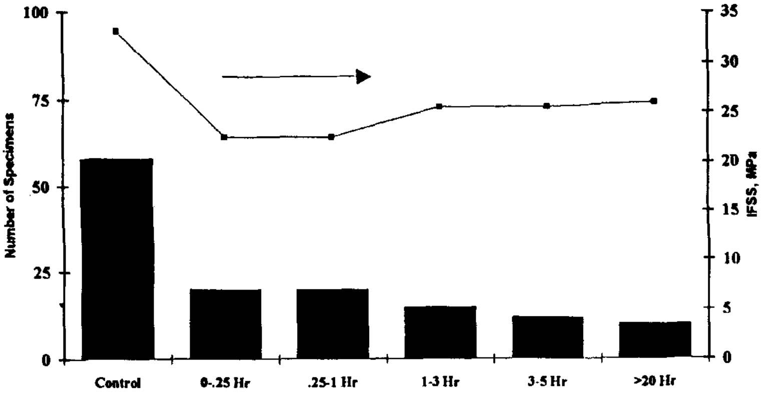


FIGURE 5 Distribution of fatigue times and interfacial shear strengths with fatigue time for cyclic fatiguing of Kevlar 49/Epon 828 microbond specimens.

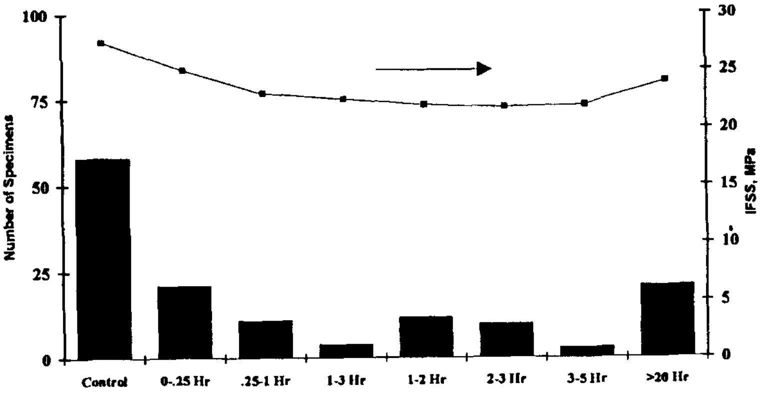


FIGURE 6 Distribution of fatigue times and interfacial shear strengths with fatigue time for cyclic fatiguing of Kevlar 49/PC microbond specimens.

these groups are tabulated in Table I, along with the number of specimens in each group, the average embedment length, and the computed t values compared with the control sample. The criterion for being statistically different at a 95% confidence level between two populations with this sample size is $t > 2.00$. The data show that there are statistically-significant reductions of the IFSS's for the Kevlar 49/Epon 828 and Kevlar 49/PC systems, about 30% and 13% of their original values, respectively. The higher bond strength reduction of Kevlar 49/Epon 828 suggests that this system was under a larger stress than the Kevlar 49/PC system. The literature values of the tensile modulus at room temperature for Epon 828 resin cured with aromatic curing agent and PC are 2.8 GPa[24] and 2.3 GPa,[25] respectively. This is in agreement with the idea that, for the same extent of fiber deformation, a higher resin modulus is expected to induce more generated stress at the interface.

The leveling off of bond strength observed for both systems beyond one hour led us to reorganize the collections into two groups with all specimens that had been loaded for more than an hour pooled together. We gain further insight into the mechanism of bond weakening by comparing the distributions of bond strengths for each group with the distribution for the control collection. These comparisons are shown in Figure 7 for the two systems. The distributions for the loaded specimens are wider than those for the control specimens, and show an emergence of a population with low values of bond strength after one hour of cyclic loading. For each collection of loaded specimens, there are several specimens for which the bond strength values are equivalent to high values in the control distribution. This implies that there are some specimens with high original bond strength values that are apparently unchanged upon loading. It implies, further, that it is the weaker bonds that are more prone to bond strength reduction as a consequence of cyclic loading, resulting in a bimodal distribution.

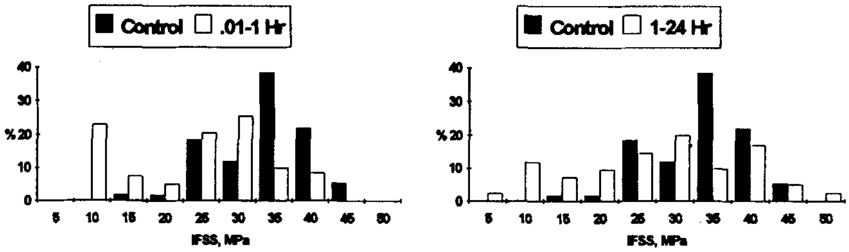
Two characteristics of this cyclic loading process for both systems are that the bond strength drops within one hour of loading time and then levels off, with a possible tendency of increase at long cyclic loading time, and that the weaker bonds seem to be weakened preferentially. We first thought the leveling off of bond strength might be due

TABLE I
Average Bond Strengths as a Function of Fatigue Time

	# of specimens	Av. Embedment length (μm)	Av. IFSS ± STD* (MPa)	t-value
Kevlar 49/Epon 828				
All controls	57	97.9	31.3 ± 5.99	–
Fatigued for 0.01-1 hr	39	98	21.9 ± 9.55	5.93
1–6 hrs	27	96.2	25.4 ± 10.7	3.24
> 20 hrs	11	106.9	26.3 ± 7.6	2.10
Kevlar 49/PC				
All controls	57	116.1	27.0 ± 4.86	–
Fatigued for 0.01-1 hr	40	104.5	23.6 ± 7.49	2.71
1–6 hrs	30	103.7	21.6 ± 5.92	4.56
> 20 hrs	23	106.2	23.2 ± 6.57	2.85

* Standard deviation

Kevlar 49/ Epon 828



Kevlar 49/ Polycarbonate

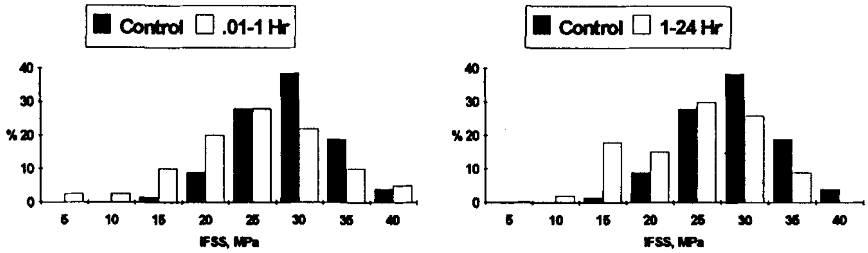


FIGURE 7 Bond strength distribution for Kevlar 49/Epon 828 (top) and Kevlar 49/PC (bottom) microbond specimens fatigued less than an hour and for 1–24 hours, compared with unfatigued specimens.

to creep of the fiber. Inelastic deformation of the fiber during the cyclic fatiguing procedure will limit the stress exerted at the interface. Consequently, the fiber deforms permanently and the crack stops propagating after some loading time, causing bond strength reduction to cease. However, Kevlar fiber exhibits a low creep rate at room temperature due to its liquid crystal structure.[20] This leads us to believe that the leveling off of the bond strength reduction must result from some kind of weakening mechanism.

There are two possible bond strength weakening mechanisms for the systems studied: (1) a general bond weakening (dispersed microcracking) process that occurs uniformly along the interface, and (2) a crack propagation (macro-scale fiber/matrix separation) process at the interface.

A general bond weakening process would lead to a decreasing bond strength with increasing cyclic loading time. However, as the interface gets weaker, the stress transfer efficiency becomes lower. For an interface exhibiting lower stress transfer efficiency, the stress induced by fiber deformation is expected to be lower. Consequently, the stress becomes smaller with cyclic loading time and eventually has little impact on the bond strength. However, this mechanism predicts a lower bond weakening effect for a weaker interface, which conflicts with the observation that the effect is more pronounced on a weaker interface. This leads us to the crack propagation mechanism which was observed or proposed for other systems.[13-16] As the crack propagates, the effective contact area between the fiber and matrix becomes smaller, which results in a reduced required debonding load for the specimen, although the bond may still have its original bond strength. Since the apparent contact area (*i.e.*, the full embedded length) is used for the IFSS computation, a lower IFSS value should be obtained. However, at any debonded area (from crack propagation), a frictional force will be present between fiber and resin. Consequently, additional load is required to pull the fiber out of the matrix because of this frictional force. The combination of decreasing bonded area and presence of frictional force at debonded locations results in a complicated relationship between the apparent IFSS and the size of crack at the interface. Many models have been developed to interpret the experimental data of microbond measurements by considering a non-linear stress distribution as well as the frictional force at the interface.[26-29] For example, the analysis of Palley and Stevans[27] predicted that, depending on the magnitude of the frictional force, as shown in Figure 8, as the crack length increases the pull-out load first drops rapidly, then it either stabilizes or starts growing after reaching a certain minimum value.

In our experimental data, the average values of IFSS do show a trend of an initial reduction followed by a small increase. This observation is qualitatively consistent with Figure 8, with a friction factor in the range of 30% to 50%. Furthermore, it has been shown that, during the microbond test, an extra compressive stress may be applied to the resin microdroplet from the shearing plates.[30] This compressive stress can increase the frictional force at the debonded interface resulting in an even higher apparent IFSS value. Since it is more difficult to initiate the crack propagation process at a stronger interface, this mechanism also explains the bimodal distribution of the bond strengths of the tested specimens.

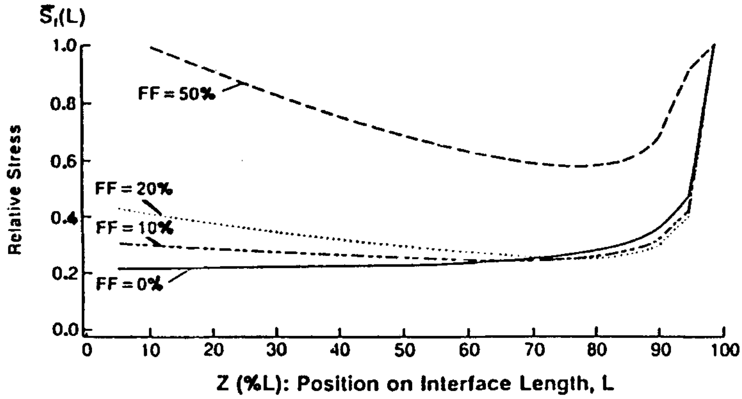


FIGURE 8 Pull-out stress as a function of the crack position (Ref. 27) Source: I. Palley and D. Stevans, "A Fracture Mechanics Approach to the Single Fiber Pull-Out Problem as Applied to the Evaluation of the Adhesion Strength Between the Fiber and the Matrix," J. Adhes. Sci. Technol., 3, 141 (1989).

CONCLUSIONS

The microbond technique was used to study the effect of external loading of fiber on the fiber/resin interface. Although Kevlar fiber has a relatively high modulus, the experimental data show that a stress equivalent to ~ 25% of the ultimate IFSS can be applied to the interface by loading the fiber ends of microbond specimens. Statistically-significant bond strength reduction was observed after cyclically loading the interface using this technique. It is likely that the bond strength reduction process is a crack propagation mechanism along the interface. For the fatiguing conditions investigated, most of the reductions are apparent within one hour, and bond strength levels off after this initial drop. The plateauing of the residual shear strength as a function of cyclic loading time may be attributed to the additional friction force at the debonded area at the interface. Since it is more difficult to initiate the crack propagation process at a stronger interface, cyclic loading seemed to affect the weaker bonds more than the stronger bonds.

It is worth pointing out that the stress developed at the interface by the cyclic loading method used in this study has a different directionality for the two halves of the microbond specimen. At the end that is subsequently placed in contact with the microvise for the measurement of residual bond strength, the stress is in the same direction as that produced during the pull-out test and would aid the debonding process. At the other end, the stress would be in the opposite direction and would act against the debonding. If both ends of the bond were equally involved in the bond shearing process, we would expect no net loss of bond strength from the external loading. The fact that we do observe a significant loss supports the generally-held idea that it is the interfacial region close to the shearing plates that debonds preferentially (*i.e.*, localized crack propagation).

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