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STRAIN RATE EFFECTS IN THE SINGLE FIBER FRAGMENTATION TEST FOR FIBER-MATRIX ADHESION

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Introduction

The development of a reliable microtest method that measures fiber-matrix interface properties in composite materials has been the focus of numerous research efforts.[1] This research has been driven by the recognition that the interface or interphase has a profound effect on composite properties, particularly the response to loads that are perpendicular to the fiber direction in unidirectional composites or to the laminate plane in laminated materials.[2] A wide variety of microtests have been studied, but the most popular is the single fiber fragmentation test (SFFT).[1] It is also a good choice for durability experiments since the fiber is completely embedded in the resin (similar to fibers in full-scale composites).[3]

In the SFFT, a dog bone is made with a high extension-to-failure resin and a single fiber embedded along the axis of the dog bone. The sample is pulled in tension, and some of the load is transferred through the fiber-matrix interface into the fiber. Since the fiber has a lower strain-to-failure than the resin, at some load, the fiber breaks at its weakest flaw. If the load is increased further, additional breaks occur in the fiber. This fragmentation process continues until all of the resulting fiber fragments are shorter than the critical transfer length. The critical transfer length is defined as the length below which the fragments are too short for sufficient load to be transmitted into them to cause additional failure. This point in the experiment is termed saturation. The lengths of the fragments at saturation are measured, and a micromechanics model (e.g., Cox, Kelly-Tyson)[4,5] is used to convert the average fragment length into a parameter that characterizes the interface quality. This parameter is often called the interface shear strength but might be described more accurately as a stress transfer coefficient which measures the efficiency of the interface in transferring stress between the matrix and fiber.

It should be noted that the current micromechanics models involve a number of important assumptions. One of the most critical is that the resin behaves as a linear elastic or elastic perfectly plastic solid. A number of recent papers have shown the shortcomings of these assumptions.[1] For example, recent work in our laboratory[6,7] on a model epoxy system often used in SFFT experiments demonstrated that the resin exhibits nonlinear viscoelastic behavior at strains above 1 %. Since all of the fiber breaks occur at strains between 1 % and 5 % for most carbon or glass fibers, the applicability of current models is suspect. One of the most important consequences of non-linear viscoelastic behavior is the sample response depends not only on the load but also on the loading history. This could affect the fragmentation test, so the purpose of the work described here is to study this possibility by conducting experiments using three different loading histories.

Experimental Section

Fragmentation samples were prepared with E-glass fibers and two different resin systems. The first is a standard epoxy often used in such tests: diglycidyl ether of bisphenol-A cured with a stoichiometric amount of meta-phenylenediamine. The second is an isocyanurate. Details of the sample preparation and resin systems are described elsewhere.[6,8] The tests were conducted on an apparatus, which allows measurement of both strain and load. A complete description of the equipment has been published previously.[7,9] The relative combined standard uncertainty in measurements of global strain is 0.023 %, while the combined standard uncertainty in the measurement of fragment lengths is 1.6 μm. The standard relative uncertainty in the load and stress measurements is 3 % .

Experimental Protocol. The tests were conducted by loading the sample in a series of strain steps. The application time of each step was (1.10 ± 0.17) s and the deformation was (14.45 ± 3.11) μm, where the ranges represent the standard uncertainty. Each step was followed by a hold time, and near the end of the hold time, the sample was scanned with a microscope to detect any fiber breaks that were present. The first break in the fiber generally occurred at strains between 1 % and 2 %. The experiment continued until the number of breaks found after three successive strain steps was unchanged. At this point, the distribution of fragment lengths was measured.

The loading history was changed by using different hold times between strain steps. Three different histories were tested. In the first, all hold times were 10 min. In the second, all hold times were 1 h. In the third, the hold time was varied. For the initial steps it was 10 min. Once fiber breaks occurred, the experimental protocol was changed to include both counting of fiber breaks and measuring the length for each individual fragment. The time required to do this increased as the number of breaks increased. As a result, the hold times became longer. At saturation, the hold times were approximately 1 h. The three testing protocols produced different effective overall strain rates. These will be designated fast, slow, and intermediate, respectively. Figure 1 shows typical loading plots for the three protocols. The turn over in the curves for fast and slow protocols indicates the onset of non-linearity. The relaxation during the hold times shows the viscoelasticity.

With the epoxy resin, four different samples were tested at the intermediate and slow strain rates, and one sample was tested at the fast rate. With the isocyanurate, one sample was tested at each rate. Comparisons were made in terms of the average fragment lengths and the distribution of fragment lengths at saturation for each resin and test protocol.

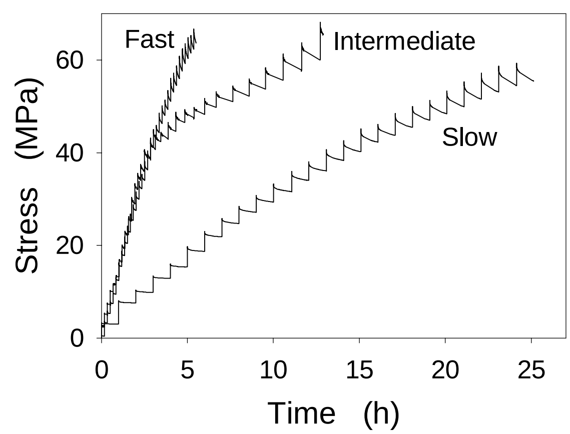

Figure 1: Stress vs time curves for the three loading histories.

Results and Discussion

Epoxy Data. Figure 2 shows the results for the epoxy samples measured with the three loading histories. The average fragment lengths are indistinguishable at the fast and intermediate rates, but the slow rate produces an average fragment length that is clearly less than that for the other test conditions. This is also seen in Figure 3 which shows a plot of the fragment distribution for three typical samples, one for each test condition. The slow rate produces more short fragments while the intermediate and fast rates give longer fragments not found in the slow test. A detailed statistical analysis of the data will be published elsewhere.[9]

It is interesting to speculate about the factors that might be involved in this effect. For example, if the loading curves are examined (Figure 1), it can be seen that the average load in the sample at saturation is significantly higher when the loading rate is faster. This might be expected to produce shorter fragments, but the observed trend is in the opposite direction. A second possible factor is a rate dependence of the interface strength. Shorter fragments should correspond to higher interface strength. If the failure mechanism involves yielding as some people have suggested, high loading rates might be expected to produce high interface strengths since the yield stress of the resin increases as the loading rate becomes faster. Again, this trend is in the wrong direction. On the other hand, if the interface failure involves fracture behavior, a logical expectation would be that higher rates produce a weaker interface since the resin fracture energy of the resin itself

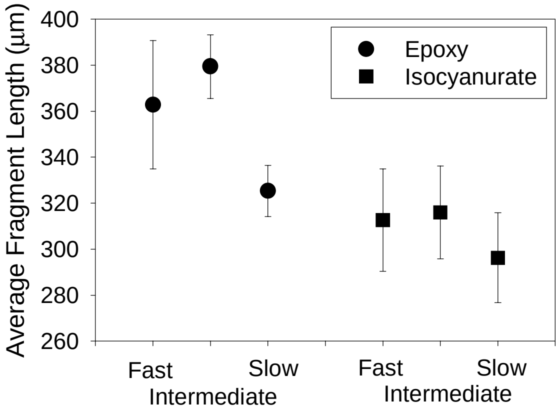

Figure 2: Average fragment length at saturation for the six sample-protocol combinations.

should go down with increasing loading rate. This is the correct trend, but for samples like these where the test conditions are more than 100 ^{o}C below the glass transition temperature of the resin, the rate dependence of fracture energy should be small.

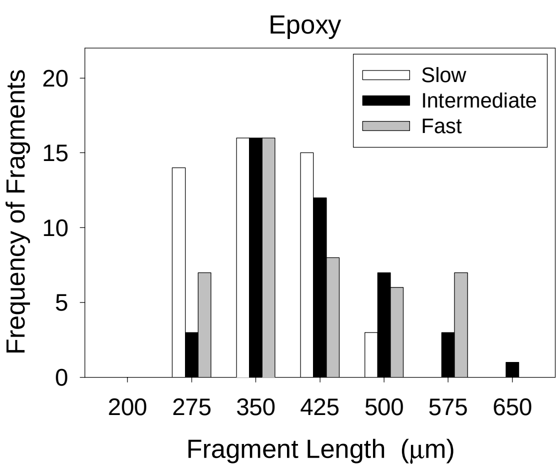

Figure 3: Distribution of fragment lengths for typical epoxy samples tested using the three protocols.

Isocyanurate Data. To get additional information about the effect of rate, experiments were conducted on a second resin system. The results for average fragment length from test on the three isocyanurate samples are shown in Figure 2 along with the epoxy data. For this second resin system, the average fragment lengths are virtually independent of rate. The small differences that are found fall within the experimental uncertainty. The fragment distributions for these samples are shown in Figure 4, and the similarities are very evident.

One difference in the results for the two resin systems is that the isocyanurate fragments are somewhat shorter than the epoxy fragments. This can be seen in the average values given in Figure 2 or by comparing Figures 3 and 4 which are plotted with the same axes to facilitate comparisons. Shorter fragments would suggest that the interface bonding is slightly stronger for isocyanurate. If so, one could speculate on how this might affect the behavior. When a fiber break occurs, high stresses are generated near the fiber break due to stress concentrations. This can cause local debonding at the interface. If the sample is loaded more slowly, yielding of the resin will decrease the local stress concentrations and perhaps reduce the debonding. If debonding decreases the ability of the interface to transfer stress, the result could be longer fragments. On the other hand, if the interface itself is stronger, the debonding and its effect on fragment length might be less. Such a hypothesis would be consistent with the observed results, but clearly more experimental work is needed before any of these ideas can be treated as more than speculation.

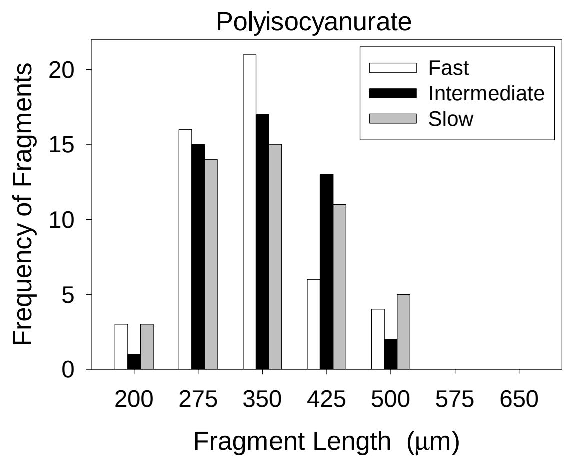

Figure 4: Distribution of fragment lengths in the isocyanurate samples tested with the three protocols.

Conclusions

Experiments on model resin systems show that the results in a single fiber fragmentation test can depend on loading history. Although a number of possible explanations can be proposed, more work is needed to determine the correct explanation. An understanding of this effect is required before the full meaning of the test results can be obtained. One point is clear, however. A fixed (standardized) test protocol is required if reproducible results are to be obtained within a given laboratory or valid comparisons are to be made between different laboratories.

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