# Multiple Fiber Technique for the Single Fiber Fragmentation Test

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*Received 17 March 1997; accepted 27 August 1997*

**ABSTRACT:** The single fiber fragmentation test has been modified by embedding multiple fibers into matrix resin. During testing, we examined the interfacial shear strengths between the fibers and the matrix. In addition, the time-dependent nature of the fragmentation process was considered. In the fragmentation test, we examined the failure process of two fibers placed far from each other, and we found that the failure profile of the two fibers were similar to the failure profiles from tests done on single fibers. When we examined three fibers, we found that the measured interfacial shear strength values were much greater than the shear strength values from either the single or two fiber tests. However, when we used three fibers, we found it difficult to control the interfiber spacing. Consequently, whenever the interfiber spacing was too small, breaks in one fiber caused breaks in the adjacent fiber. In conclusion, using multiple fibers in a fragmentation test has many merits, such as saving time in testing, ease of comparing the effects of fiber surface treatment, and testing different fibers in the same matrix exposed to the same processing conditions. © 1998 John Wiley & Sons, Inc. J Appl Polym Sci **67:** 1701–1709, 1998

## INTRODUCTION

The interface between a reinforcing fiber and a polymeric matrix plays a very important role in determining the final performance of a composite. Therefore, the ability to evaluate the interfacial shear strength accurately is becoming more important. Unfortunately, it is difficult to get unambiguous results when testing bulk composites. Consequently, several techniques such as the pullout,[1–4] microbond,[5–9] fragmentation,[10–16] and indentation[17] test methods have been developed to try to measure the interfacial shear strength correctly.

Among the number of techniques mentioned above, one popular technique is the single fiber fragmentation test.[10–16] The merits of this test method are that much data are generated from one sample test, and the test geometry lends itself well to studying durability issues. In the fragmentation test, a single fiber is encapsulated in a mass of resin in the shape of a dog bone. The sample is loaded axially in tension, with the stress being transferred from the resin to the fiber via interphase region. Upon loading, eventually, the breaking strength of the weakest flaw is reached and the fiber breaks. Upon further loading, this breaking process will continue until all of the remaining fragment lengths fall below the length necessary to transfer sufficient stress to cause further breakage of the fiber.[18] This length is the critical length. To calculate the interfacial shear strength in the single fiber composite after testing, we need to know the tensile strength of the fiber. Kelly and Tyson[18] used this method on tungsten and molybdenum wires embedded in copper matrices, and they developed an equation for

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Journal of Applied Polymer Science, Vol. 67, 1701–1709 (1998)
 CCC 0021-8995/98/101701-09

**Table I The Effect of Molding Pressure on the Interfacial Shear Strength in Single E-Glass Fiber–Polyisocyanurate Resin Fragmentation Test**

| Pressure (MPa) | $\tau$ (MPa) |
|---|---|
| 0.20 | 40.06 ± 2.76 |
| 0.55 | 40.47 ± 1.84 |
| 0.69 | 41.08 ± 2.42 |

specimens that had plastic deformation of the resin and elastic deformation of the fiber. The equation is as follows.

$$\tau = \sigma_f D_f / 2L_c \quad (1)$$

where $\tau$ is the interfacial shear strength, $\sigma_f$ is tensile strength of the fiber at a length equal to $L_c$, $D_f$ is the fiber diameter, and $L_c$ is the critical length of the fiber. Typically, the fiber strength is estimated by conducting many tensile tests on individual fibers at various lengths and then doing a statistical analysis on the data. Since $\sigma_f$ depends on the fiber length, an extrapolation is used to determine the value at $L_c$. This method of estimating the tensile strength of the fiber becomes problematic when we want to study durability issues. Schutte et al.[19] has shown that E-glass fiber is attacked by distilled water, but we cannot measure directly the tensile strength of the fiber using fragmentation test samples. Therefore, we need to be able to estimate the degraded strength of the fiber after exposure. So Wagner and Eitan[20] and Shioya et al.[21] proposed new methods to estimate the tensile strength of a fiber from a single fiber fragmentation test. These methods were used the relationship between each specific strain and the number of fiber breaks that occurred at that strain level.

By being able to estimate the tensile strength of the glass fiber in the fragmentation samples as a function of moisture, it now becomes possible to estimate the extent of interfacial degradation correctly. However, they did not consider the viscoelastic nature of the polymeric matrix. In the single fiber fragmentation test, we found that the number of fiber breaks at each specific strain depended on time. Eventually, at interphase, it needs some time for stress to transfer from the matrix to the fiber due to the viscoelastic nature of polymeric matrix. In a fragmentation test, if time dependence is considered, its test must take much longer time than the present method.

Other works have been done exploring the effects of testing multiple fibers in a fragmentation test. Three-fiber and multifiber techniques were used by Phoenix and coworkers[22] and Wagner and Steenbakkers,[23] respectively. Phoenix showed photographs to indicate the effects of fiber–fiber interactions on fiber fracture behavior in a matrix. Wagner calculated the interfacial shear strength by measuring pullout length of fiber when multifiber composites were fractured.

The purpose of this study is to investigate quantitatively if using multiple fibers in the fragmentation test will be useful in gathering more information at a faster rate. Currently, it can take around four hours to test a single sample. Being able to test multiple fibers would give us the opportunity to greatly increase the output of this test. In addition, we would also be able to test the samples having two different fibers in the same sample. Finally, we investigated the time-dependent nature of the number of fiber breaks in a multiple fiber composite at specific strains during a fragmentation test.

## EXPERIMENTAL

### Materials

The materials used in this study are as follows. The fibers were unsized E-glass fiber (Owens-Corning) and unsized carbon fiber (Hecules AU-4). The average fiber strengths were 2.1 and 2.71 GPa, respectively. The gauge length of both samples was 20 mm; and before testing, the diameter of every samples was measured using an optical microscope. The average fiber strength was determined by single fiber tensile testing about 30 samples.

The matrix polymer was a polyisocyanurate (Spectrim 364 from Dow Chemical), which was

**Table II The Effect of Fiber Number on the Interfacial Shear Strength in E-Glass Fiber–Polyisocyanurate Fragmentation Test**

| No. of Fibers | $\tau$ (MPa) |
|---|---|
| 1 | 41.33 ± 3.84 |
| 2 | 40.50 ± 2.18 |
| 3 | 56.69 ± 7.21 |

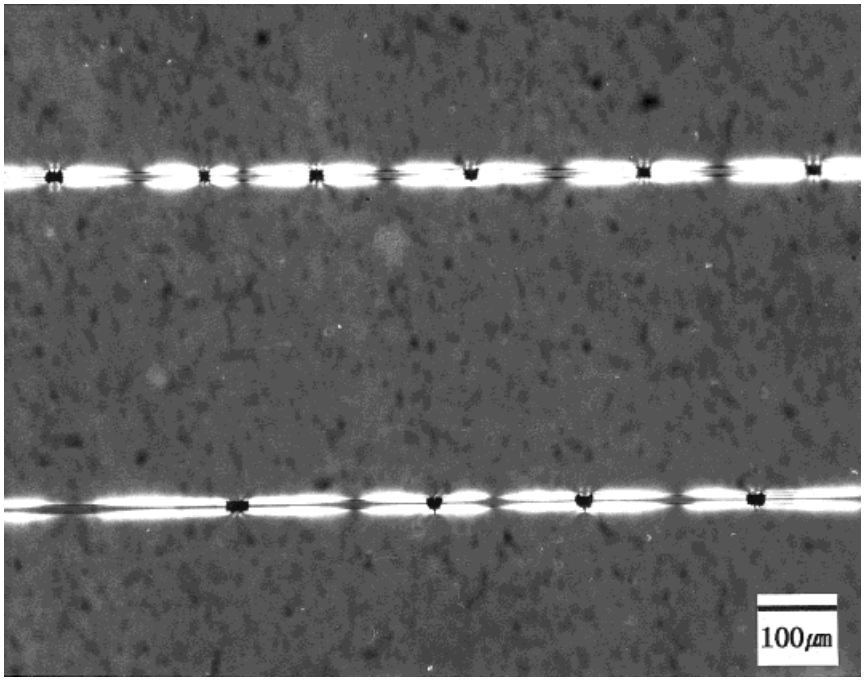


**Figure 1** Polarized transmitted light micrograph of the two E-glass fiber–polyisocyanurate fragmentation test at saturation.

made by mixing polyol, isocyanate, and catalyst. The amount of isocyanate was 2.4 times of polyol, and the catalyst was 0.3% of the polyol by weight.

### Preparation of Fragmentation Test Samples

The sample preparation for single and multifiber testing were similar to that described by Drzal et al.,[10] and a brief description is as follows. The silicone (GE silicone RTV-664) mold with eight dogbone-shaped cavities was used for the preparation of fragmentation test samples. Each cavity in the mold has sprue slots in the center of each end to aid in aligning the fiber in the center of the cavity. We placed single and multiple fibers of E-glass and carbon fibers through the sprue slots of a silicone mold and fixed them in place by putting a small drop of five-minute epoxy (Hardman Adhesives) at the far end of each sprue slot.

Once the epoxy droplets had hardened, we were ready to prepare the matrix resin. And then we filled two syringes and slowly injected the resin into the mold cavities. After pouring the resin into mold cavities, we transferred the mold to an autoclave (United McGiLL). The autoclave was held at 93°C and 0.55 MPa for 30 min. Then the pressure was reduced quickly to atmospheric pressure, and the temperature was quickly reduced to 66°C. After this, the mold was cooled slowly overnight to room temperature. Subsequently, the samples were postcured in an oven (Blue M) for 1 h at 150°C and then allowed to cool slowly to room temperature. Finally, we removed the samples from the mold and examined them. Valid samples were regarded as those that had no air bubbles and whose fibers remained straight. Some samples contained a single glass fiber; others contained multiple filaments of glass. The same was done for the carbon fibers. In addition, we made some samples that contained both glass and carbon fibers.

### Fragmentation Test

The fiber fragmentation tests were carried out on a small, hand-operated testing machine such as that described by Drzal and coworkers.[10–12]

Prior to testing a new sample, we marked the dogbone with two ink marks, spaced approximately 10 mm apart, to measure the strain after loading manually.

We used three test methods to investigate the effect of loading mode on the interfacial shear strength and the time-dependent nature of the fragmentation process.

The first method was that the strain was in-

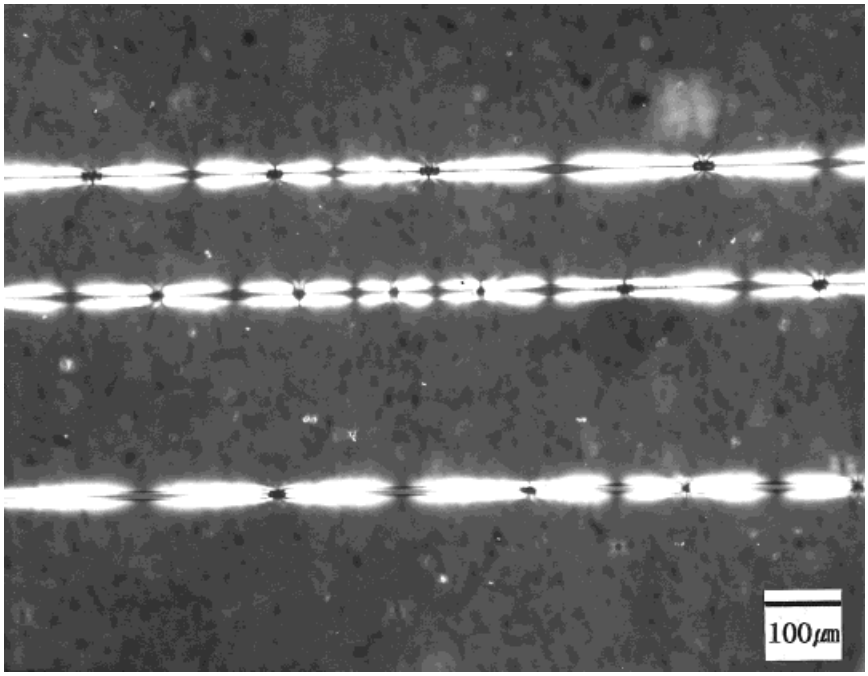


**Figure 2** Polarized transmitted light micrograph of the three E-glass fiber–polyisocyanurate fragmentation test at saturation.

creased stepwisely in unit of approximately 0.3% (five units of dial gauge). In each step, the number of fiber breaks was counted. We continued this process until the number of fiber breaks within gauge length reached saturation.

The second method was similar to the first method. However, the strain was kept constant for 10 min, and then the number of fiber breaks was counted in each step. This process was also continued until the number of fiber breaks reached saturation.

In the third method, the sample was under constant strain until no further fiber breaks were observed at each step, and then the number of fiber breaks was counted. We repeated this process until the total number of fiber breaks reached saturation.

After the number of fiber breaks reached saturation, the test was stopped, and the individual fiber fragment lengths were measured to calculate the interfacial shear strength.

We mainly used the second method, unless otherwise stated, throughout the study for the effective comparison among the data. We calculated the interfacial shear strength using the following eq. (2). The distribution of fragment lengths has been determined to be satisfactorily described by a two-parameter Weibull analysis, causing the expression for the interfacial shear strength[10] $\tau$, to become

$$\tau = \sigma_f \Gamma\left(1 - \frac{1}{\alpha}\right)/2\beta \qquad (2)$$

where $\alpha$ and $\beta$ are the shape and scale parameters, respectively.

$\Gamma$ is the Gamma function. In eq. (1), $\sigma_f$ is the average fiber tensile strength at the critical fiber length needed to calculate interfacial shear strength. However, in this equation, we used the single fiber tensile strength at 20 mm of gauge length. One goal of this study is to discuss whether a fragmentation test of multifiber is useful or not.

## RESULTS AND DISCUSSION

Table I shows the effect of molding pressure on the interfacial shear strength in single E-glass fiber–polyisocyanurate resin fragmentation test. We can see that interfacial shear strengths were independent of molding pressure in this pressure region. When samples were made at atmospheric pressure, they had many voids throughout. Even

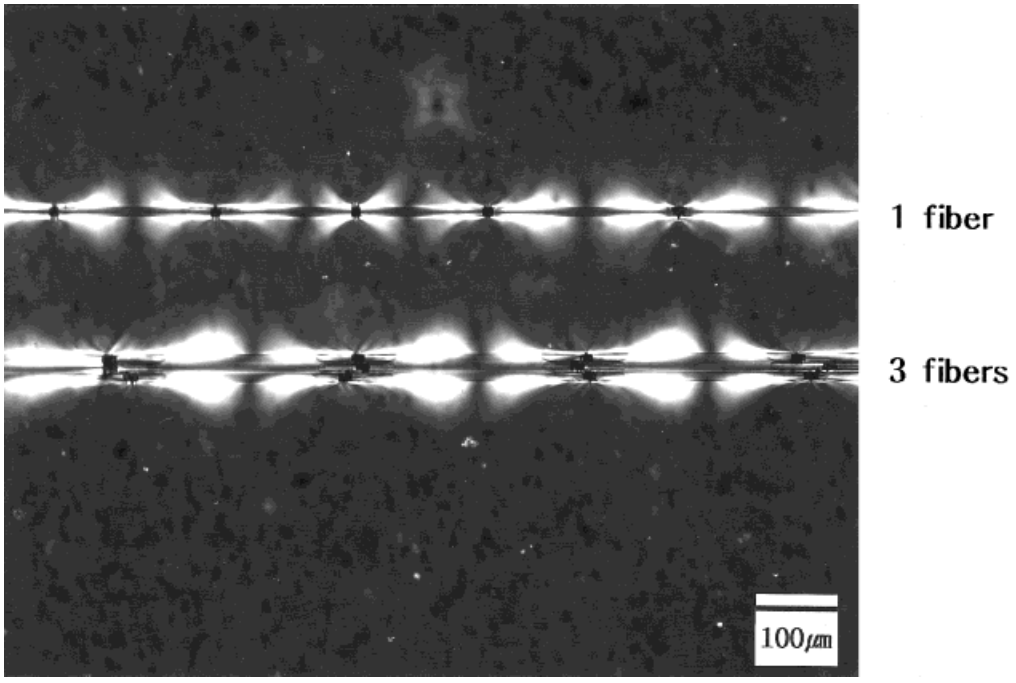


**Figure 3** Polarized transmitted light micrograph of the four E-glass fiber–polyisocyanurate fragmentation test at saturation.

when the molding pressure was 0.2 MPa, samples still had a few voids on the top surface. But when the molding pressure was 0.55 and 0.69 MPa, samples did not have any voids that were visible. Consequently, we decided to use molding pressure of 0.55 MPa as standard molding pressure for this study.

Table II shows the effect of the number of fibers per sample on the interfacial shear strength in E-glass fiber–polyisocyanurate resin fragmentation test. As described previously, the silicone mold had sprue slots to align the fibers. The width of each sprue slot was about 400 $\mu$m. When making two fiber samples, as well as single fiber samples, we placed the fiber into silicone mold cavities using the slot width. That is, the first fiber was placed on one side of the slot, and the second fiber was placed on the other side of the slot. In doing so, we could ensure the interfiber spacing was about 400 $\mu$m in the two-fiber samples. We could also make one and two fiber samples consistently, but we were not able to correctly control the interfiber spacing consistently in the three fiber samples. In Table II, the results for one- and two-fiber samples were similar; but in the case of three fibers, the value was much larger than the value of one or two fiber samples.

Figure 1 shows the polarized photograph of a fragmentation test sample with two fibers after the number of fiber breaks was saturated. The interfiber spacing was approximately 400 $\mu$m, and we can see that there were no fiber–fiber interactions. In this study, unless otherwise stated, the interfiber spacing of all two fiber samples was about 400 $\mu$m.

Figure 2 shows the polarized typical photograph of the fragmentation test sample of three fibers. Even though we can see that the breaks in the neighboring fibers were not at the same location, the average fragment length was shorter than the samples of one and two fibers. Therefore, the average interfacial shear strength was greater for these samples than for samples containing one and two fibers, as seen in Table II. In fact, the interfiber spacing between the first (upper) and the second fiber (center) was approximately 150 $\mu$m, and the interfiber spacing between the second and third fiber (down) was about 250 $\mu$m. The interfacial shear strength of the first and second fiber was much greater than that of the third fiber. The value of the third fiber was similar to the results of samples containing one and two fibers. Consequently, we believe that there exists some interaction between fibers if the interfiber spacing is small.

Figure 3 shows the polarized photograph of the fragmentation test sample. In this instance, we see four fibers: three fibers were next to each

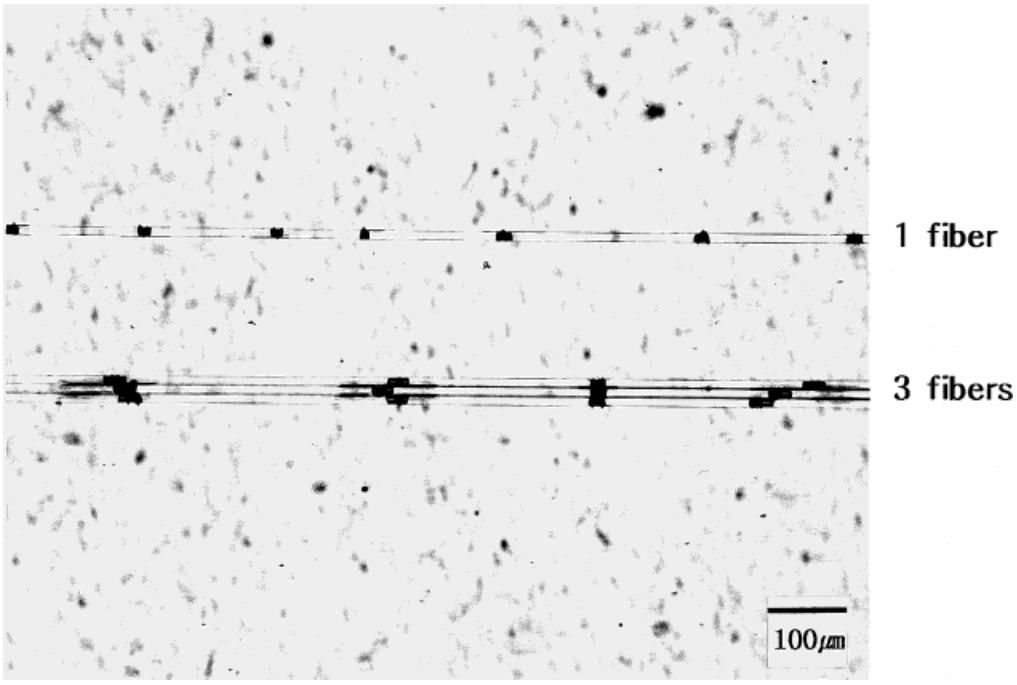


**Figure 4** Micrograph of the four E-glass fiber–polyisocyanurate fragmentation test at saturation.

other, with the fourth one separated from this group. The interfiber spacing of the three fibers was approximately 1–2 $\mu$m, and the interfacial shear strength was lower than that of single fiber samples. The distance between these and the fourth fiber was approximately 200 $\mu$m, and the interfacial shear strength was similar to that of single fiber samples. From this photo, we can see the following: if the interfiber spacing is large ($>$200 $\mu$m), even three fiber breaks cannot affect the other one fiber break. If the interfiber spacing is too close, as seen in Figure 4, one fiber breaks can affect the other fiber breaks. In addition, the breaks in the adjacent fibers are at the same location. It looks just like a single fiber fragment with a large diameter. This is a similar result as that of the bigger fiber diameter, which had the smaller interfacial shear strength between fiber and epoxy resin.[8] We can guess from this that interfacial shear strength in real composites is much smaller than that of single fiber fragmentation samples.

Furthermore, we can see that even using multiple fibers in a sample, if interfiber distance is large enough, over 200 $\mu$m in this study, the results are similar to when a single embedded fiber in tested.

Figure 4 shows the microscopic photograph of a multifiber fragmentation test after the number of fiber breaks was saturated. It was the same kind of sample as Figure 3. Through this, we can see clearly the effect when interfiber spacing is different.

Table III shows the comparison of interfacial shear strength in a one- and two-carbon fiber–polyisocyanurate fragmentation test. In a carbon fiber fragmentation test, we can see that the results were similar whether we used one or two fibers in one sample.

In the case of two fiber samples, the interfiber spacing was about 400 $\mu$m.

**Table III The Comparison of Interfacial Shear Strength in One- and Two-Carbon Fiber–Polyisocyanurate Resin Fragmentation Test**

| Fiber No. | 1 | 2 |
|---|---|---|
| $\tau$ (MPa) | 29.90 ± 3.42 | 30.81 ± 3.38 |

**Table IV The Comparison of Interfacial Shear Strength in Carbon and E-Glass Fiber–Polyisocyanurate Fragmentation Test**

| Fiber | Carbon | E-glass |
|---|---|---|
| $\tau$ (MPa) | 30.75 ± 1.74 | 40.97 ± 3.26 |

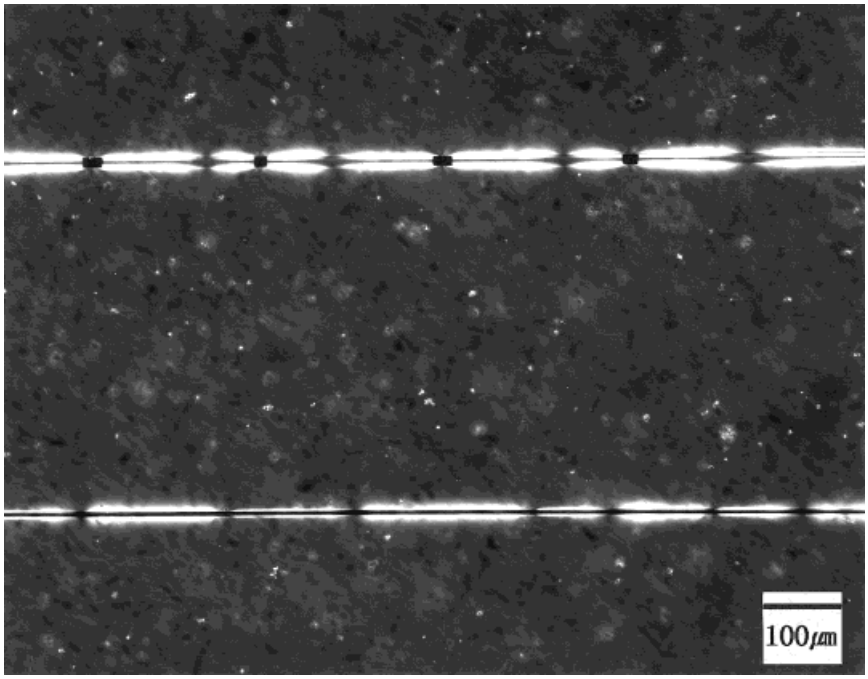


**Figure 5** Polarized transmitted light micrograph of the E-glass and carbon fiber–polyisocyanurate fragmentation test at saturation: (a) upper, E-glass fiber; (b) down, carbon fiber.

Table IV shows the comparison of interfacial shear strength in carbon and E-glass fiber–polyisocyanurate fragmentation test. We found that the result of E-glass fiber was greater than the interfacial shear strength of carbon fiber.

Figure 5 shows the polarized photo of an E-glass (upper) and carbon (down) fiber–polyisocyanurate fragmentation test. Its interfiber distance was about 400 $\mu$m. We found that there was no fiber–fiber interactions. So we can see that it is possible to estimate the interfacial shear strength of different fibers in the same sample.

Figure 6 shows the normalized number of fiber breaks as a function of applied strain in one and two E-glass fiber–polyisocyanurate resin fragmentation test. We can see that the results were similar whether we used one or two fibers in the same sample.

Figure 7 shows the normalized number of fiber breaks as a function of applied strain in one- and two-carbon fiber–polyisocyanurate resin fragmentation test. We can see that the results of one- and two-fiber tests were almost the same.

Figure 8 shows the normalized number of fiber breaks as a function of applied strain in carbon and E-glass fiber–polyisocyanurate fragmentation test. We can see that after the number of carbon fiber breaks was saturated, the number of E-glass fiber breaks saturated. Also, we can see that the extent of carbon fiber strain was smaller than that of E-glass fiber.

Figure 9 shows the relationship between the fiber break number and the time at each specific

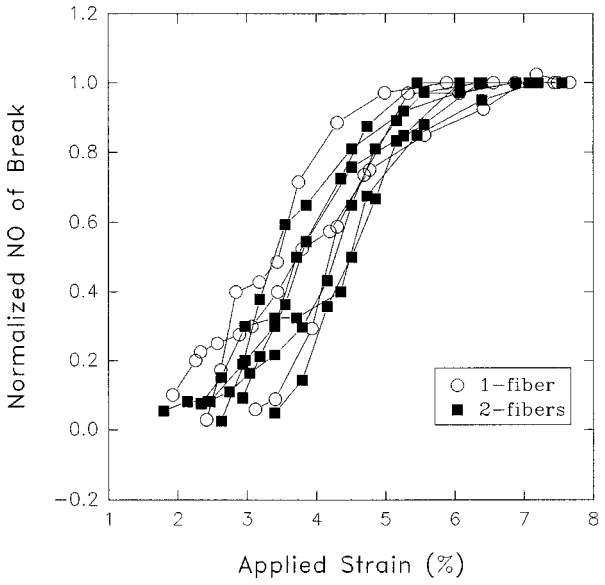


**Figure 6** Plot of the normalized number of fragments as a function of applied strain in one and two E-glass fiber–polyisocyanurate resin fragmentation test.

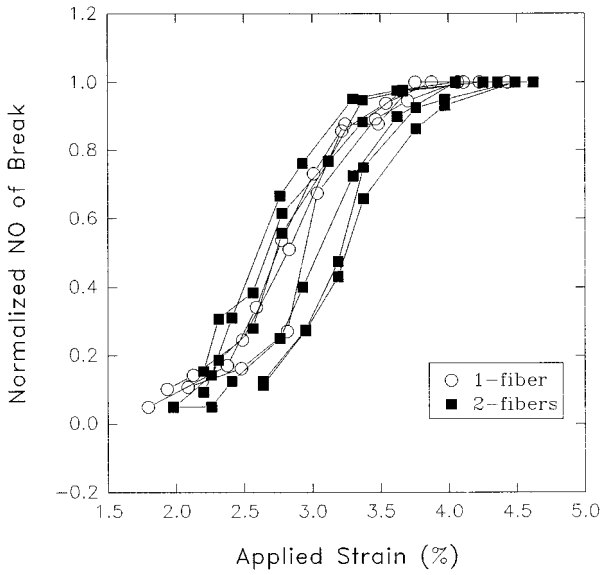


**Figure 7** Plot of the normalized number of fragments as a function of applied strain in one and two carbon fiber–polyisocyanurate resin fragmentation test.

strain. We can see that the number of fiber breaks at each specific strain increased with time and then saturated. The trend of one- and two-fiber samples was the same. But we can see that the beginning number of fiber breaks is large. These two samples were increased stepwisely in unit of about 0.3% strain, and the number of fiber breaks was counted from after loading of indicated total

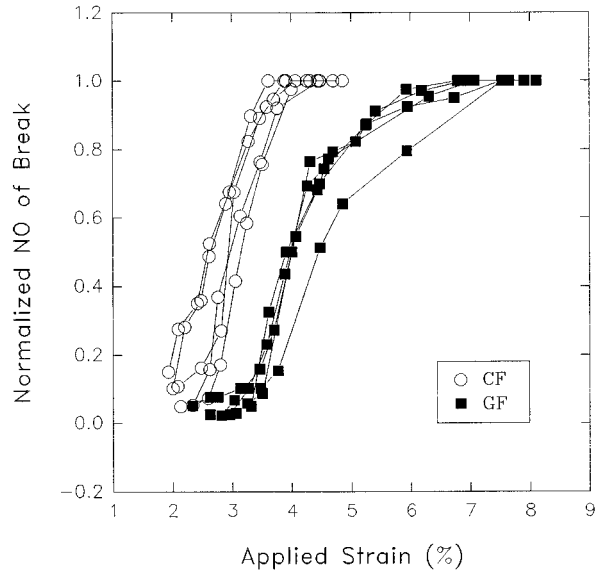


**Figure 8** Plot of the normalized number of fragments as a function of applied strain in carbon and E-glass fiber–polyisocyanurate resin fragmentation test.

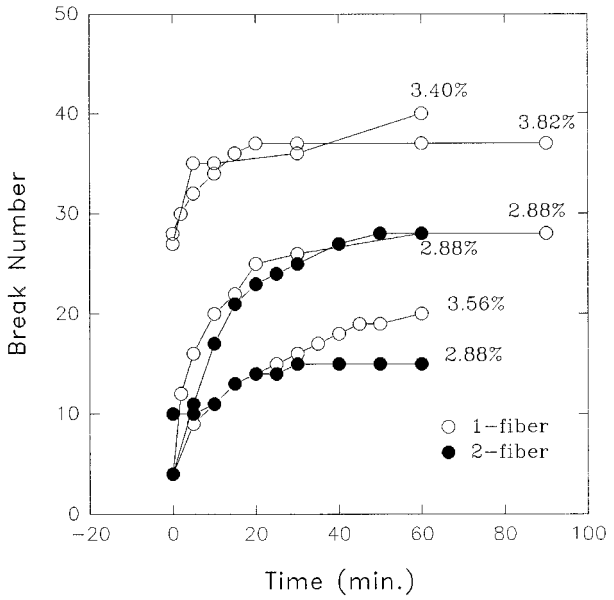


**Figure 9** Plot of the fiber breaks number as a function of time at each strain in one and two E-glass fiber–polyisocyanurate resin fragmentation test.

strain. And we can also see that the results are not similar, even using the same sample and applied strain. Through this, we can guess, at each specific strain, the relationship of between the number of fiber breaks and the time may be affected by various factors, such as each fiber strength and modulus, interfacial shear strength, viscoelastic extent of polymeric matrix, amount of strain and increasing strain at each step, and the total applied strain value. From Figure 9, we can see that, in order to count the number of fiber breaks accurately under each specific strain in the fragmentation test, time dependence has to be considered. So, in order to count the number of fiber breaks correctly, we propose the third test method, as described previously. That is, after the number of fiber breaks is saturated at each strain, the next step strain must be loaded. Therefore, at each specific strain, we can count the correct number of fiber breaks in the fragmentation test process.

Table V shows the effect of loading mode on the interfacial shear strength in two E-glass fiber–

**Table V The Effect of Loading Mode on the Interfacial Shear Strength in Two E-Glass Fiber–Polyisocyanurate Resin Fragmentation Test**

| Loading Mode | Slow | Fast |
|---|---|---|
| $\tau$ (MPa) | 39.65 ± 2.99 | 40.07 ± 2.11 |

polyisocyanurate resin fragmentation test. In Table V, the fast of loading mode means the first test method, as mentioned above. The slow loading mode means the third test method. In this loading mode extent, we can see that the results were almost the same. Finally, the interfacial shear strength was independent of the loading mode.

## CONCLUSION

The conclusions drawn from this study on a multifiber technique and the time dependence correlating with the number of fiber breaks in the fragmentation test were as follows.

1. In the case that the interfiber spacing was too close in the multiple fiber fragmentation test, one fiber breaks, affecting the other fiber breaks; but in the case that interfiber spacing was large enough, there was no fiber–fiber interaction. Therefore, the multiple fibers technique in the fragmentation test is useful if interfiber spacing is large enough ($>200$ $\mu$m).
2. Using multiple fibers in a fragmentation test had many merits, such as saving time in testing, ease of comparing the effects of fiber surface treatment, and testing different fibers in the same matrix exposed to the same processing conditions.
3. The number of fiber breaks at the specific strain in the fragmentation test was affected by the viscoelastic nature of the polymeric matrix. Therefore, in order to count the number of fiber breaks accurately under a specific strain, time dependence has to be considered.
4. The interfacial shear strength values were independent of the loading mode.

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