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The Influence of the Matrix Modulus on the Interfacial Shear Strength Parameter

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INTRODUCTION

As noted by Piggott [1], the single fiber fragmentation test (SFFT) is the most realistic of the interface tests from the point of view of accounting for the impact of interfacial pressure on the determined interfacial shear strength parameter. Since the fiber is neither pushed nor pulled directly, fiber Poisson effects are similar to those occurring in a fiber composite. In the SFFT, a dogbone is made with a resin having a high extension to failure and a single fiber embedded along the axis of the dogbone. The sample is pulled in tension, and stress is transmitted into the fiber through the fiber-matrix interface. Since the fiber has a lower strain to failure than the resin, the fiber breaks at the weakest flaw as the strain is increased. This process continues until the remaining fiber fragments are all less than a critical transfer length, l_c. The critical transfer length is the length below which the fragments are too short for sufficient load to be transmitted into them to cause failure. This point is termed saturation. The fragment lengths at saturation are measured and a micro mechanics model is used to convert the average fragment length into a measure of the interface strength or stress transfer efficiency.

Adhesion at the fiber-matrix interface region is a critical factor in assessing the performance of composites in structural applications. Therefore, tests which accurately determine fiber-matrix adhesion will facilitate the use of composites in structural applications. One method of determining fiber-matrix adhesion is from SFFT experiments. In this test fiber-matrix adhesion is indirectly determined from experimental data and theoretical models. These models make varying assumptions about the fiber, matrix, and fragmentation process. Thus, the success of this approach is dependent on how accurately the model captures the reality of fiber fragmentation in the tested system.

The two most common models used for determining the interfacial shear strength parameter are the Kelly-Tyson model [2] and the Cox model [3]. The Kelly-Tyson model assumes the matrix to be elastic-perfectly plastic. In the original development the copper matrix was shown to have an elastic strain limit of only 0.03 %. Consequently, its contribution to the stress-transfer process could be ignored. Hence, the plastically deformed copper matrix affords a constant shear stress condition across the fiber matrix interface (see eq. 1). the Cox model (see eq. 2) assumes the following: (1) the matrix material is linear elastic, (2) a perfect bond exists between the fiber and the matrix, (3) the radius of the matrix, r_m, is unknown and typically assumed to be 1/2 the thickness of the test specimen, and (4) yielding of the matrix is not considered. In addition, as noted by Hunston, Shioya, et al., these models make no assumption about the failure process occurring at the interface [4].

$$\tau\{l_c\}_{interface} = \frac{d_f}{2l_c}\sigma_f\{l_c\} \tag{1}$$

$$\tau\{l_c\}_{interface} = \frac{d_f\beta}{4}\frac{\sinh\beta(l_c/2)}{\cosh\beta(l_c/2)-1}\sigma_f\{l_c\} \tag{2}$$

$$\beta = \frac{2}{d_f}\left[\frac{E_m}{(1+\nu_m)(E_f-E_m)\ln(2r_m/d_f)}\right]^{1/2} \tag{3}$$

where

E_m, ν_m are the matrix modulus and Poisson's ratio, respectively

E_f, d_f are the fiber modulus and fiber diameter, respectively

It is known that results from interface testing methods can be misleading . In the case of the single fiber fragmentation test, Piggott [5] calls attention to the determination of the fiber strength, $\sigma_f\{l_c\}$, by unreliable methods and the inability of the Kelly-Tyson model to accurately predict the shear stress profile in carbon fiber/epoxy SFFT specimens. The latter observation suggests that the interfacial shear strength at the fiber epoxy interface is higher than the value determined by the Kelly-Tyson model. Research by Galiotis *et al.*[6] on polydiacetylene fiber embedded in an epoxy matrix has demonstrated that the fiber stress distribution in a SFFT specimen can be approximated by the Cox model. Galiotis [7] also notes that the constant shear stress condition across the fiber matrix interface is seldom achieved in polymer matrix composites. Hence, the Kelly-Tyson model for determining interfacial shear strength is seldom applicable to polymer matrix composites. Although the results by Galiotis and Young support the fiber stress profile predicted by the Cox model in the bonded

fiber-matrix interface region, the results of Feillard et al. [8] call into question the appropriateness of the linear elastic Cox model. Their numerical simulations indicate that utilization of the linear elastic matrix modulus in the Cox equation over predicts the number of fragments observed experimentally. These authors found better agreement by using a secant modulus in the Cox equation. Hence, the impact of material properties on the determined interfacial shear strength parameter is an issue that should be addressed. In our investigation of this issue, our focus will be restricted to the Cox model, since the fiber stress profile predicted by this model is supported by experimental results, and this model includes material parameters.

EXPERIMENTAL

Fiber and Mold Preparation

To make single fiber fragmentation specimens, eight-cavity silicone molds were made following the procedure described by Drzal [9]. All molds were post cured at 150 ^{o}C and rinsed with acetone prior to use. A tow 12" long was cut from a spool of bare E-glass fibers. The tow was washed with distilled acetone in glass and vacuum dried at 100 ^{o}C overnight and cooled prior to use. Single filaments of E-glass fiber were separated from the 12" tow using flexible gloves, while being careful to touch only the ends of the fiber. The fibers were aligned in the mold cavity via the sprue slots in the center of each cavity. The fibers were temporarily fixed in place by pressing them onto double-stick tape. Small strips of double-stick tape were put over each fiber end to hold them in place until each fiber was permanently mounted with 5-minute epoxy.

Embedding Procedure

One hundred grams of diglycidyl ether of bisphenol-A (DGEBA) and 14.5 grams of meta-phenylene diamine (m-PDA), were weighed out in separate beakers. To lower the viscosity of the resin and melt the m-PDA crystals, both beakers are placed in a vacuum oven set at 75 ^{o}C. After the m-PDA crystals are completely melted, the silicone molds containing the fibers are placed into another oven that is preheated to 100 ^{o}C. With the preheated oven turned off, the silicone molds are placed in the oven for approximately 20 minutes. This last procedure drys the molds and minimizes the formation of air bubbles during the curing process.

At approximately 9 minutes before the preheated molds are removed from the oven, the m-PDA is poured into the DGEBA and mixed thoroughly. The mixture is placed into the vacuum oven and degassed for about 7 minutes. After 20 minutes, the preheated molds are removed from the oven and are filled with the DGEBA/m-PDA resin mixture using 10 cc disposable syringes. The filled molds are then placed into a programmable oven. A cure cycle of two hours at 75 ^{o}C followed by two hours at 125 ^{o}C is used.

Fragmentation Test

The fiber fragmentation tests are carried out on a small hand operated loading frame mounted on a polarizing microscope. The loading frame is similar to the one described by Drzal [9]. The image is viewed using a video camera and monitor. Before the test, the fiber diameter is measured with an optical micrometer attached to the video system. The sample is scanned by translating the loading frame under the microscope using a micrometer. The position of the load frame is monitored by an LVDT connected to an A-to-D printed circuit board in a computer. To measure fragment lengths or other points of interest in the sample, the location is aligned with a cross hair in the microscope as seen on the video monitor, and the position of the LVDT is digitized into the computer. The load is also monitored during the experiment using a 2,224 N (500 pound) load cell connected to a bridge. The bridge is attached to the same computer via a serial connection. The relative standard uncertainty of the load measurements is 3 % of the load. A custom program was developed to record continuously the load and any LVDT measurements that are made.

RESULTS AND DISCUSSION

A typical load vs. time curve for a DGEBA/m-PDA epoxy resin SFFT specimen is shown in Figure 1. Readily visible in this loading curve is the relaxation of the load after each incremental increase in strain. The relaxation of the stress with time is consistent with viscoelastic constitutive laws that govern the response of polymeric materials to step-strain inputs. In addition, initial fragmentation in the fiber occurs at approximately 2.5 h and the strain at the initial break is (1.82 $\pm$ 0.02) %. Stress-strain plots [10] reveal that initial fragmentation occurs when the matrix is undergoing nonlinear viscoelastic behavior. In addition, these plots show that the epoxy matrix undergoes strain-hardening after the onset of yield. Therefore, the linear elastic matrix assumption in the Cox model [3] is violated during the fragmentation process. Furthermore, the strain-hardening after yield indicates that the matrix does not behave in a manner consistent with the Kelly-Tyson model [2], i.e., elastic perfectly-plastic behavior.

Linear viscoelastic behavior is readily incorporated in the Cox model by the use of *Schapery's Correspondence Principle*. However, extension of the linear viscoelastic Cox model to the nonlinear viscoelastic regime requires a more rigorous theo-

retical development. As a first approximation the nonlinear viscoelastic behavior of the matrix has been estimated by using a strain-dependent *secant modulus* in the linear viscoelastic Cox model. This approach has been successful in numerical simulations designed to predict the number of fragments occurring in actual fragmentation experiments. However, the wide spread applicability of this approach has not been determined. Recently, a nonseparable nonlinear constitutive equation was developed for the DGEBA/m-PDA epoxy matrix from single and multi-step stress relaxation data [11]. This equation supports the softening of the epoxy resin matrix at higher strains via nonaffine motion and nonlinearity in the viscoelastic relaxation process. For the sample shown in Figure 1, the change in the *secant modulus* with increasing strain is shown in Figure 2. During the course of the experiment, the stiffness of the matrix, *pseudo-elastic* modulus, is only 64 % of its initial value at small strains. If the stiffness change is taken into account, the critical transfer length, l_c, is approximately 25 % higher than it would be if the modulus change were not considered, since "β" is related to l_c, and "β" depends on the matrix modulus. Details of the critical transfer length calculations can be found elsewhere [10].

To demonstrate the effect of matrix assumptions on the calculated interfacial shear strength, experimental data from three samples were used and the fiber strength was assumed to be approximately (2.5 to 2.8) GPa [10]. To assess the impact of nonlinear viscoelastic behavior on the interfacial shear strength, the *secant modulus* at saturation for each of the samples was used. These interfacial shear strength values are shown in Table 1. The interfacial shear strength value calculated using the secant modulus is only 85 % of what it would be if the modulus change were ignored and the initial *pseudo-elastic* modulus were used. This result is consistent with numerical simulation results mentioned previously. In addition, assuming elastic perfectly plastic behavior for the modulus, i.e., the Kelly-Tyson model, the interfacial shear strength is 33 % of the value obtained using the secant modulus. Recall that Piggott noted the inability of the Kelly-Tyson model to accurately predict the carbon fiber shear stress profile determined experimentally by Raman spectroscopy. Although each approach ranks the interfacial shear strengths in the same order (see Table 1), these calculations show that assumptions concerning the matrix modulus can have a profound effect on the determined interfacial shear strength parameter. This becomes particularly important in durability studies where moisture alters the matrix properties through plasticization. Hence, to assess the durability of the interface, the reduction in interfacial shear strength due to matrix plasticization must be decoupled from decreases in the interface due to chemical degradation.

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Table 1

Calculated Interfacial Shear Strength Values for 3 Bare Fiber SFFT Samples

Analysis Method	Interfacial Shear Strength Values		
	Bare Fiber No. 1	Bare Fiber No. 2	Bare Fiber No. 3
Elastic Cox Model	116-130 MPa	156-175 MPa	129-144 MPa
Secant-Cox Model	93-104 MPa	126-142 MPa	105-117 Mpa
Kelly-Tyson Model	30-34 MPa	42-48 MPa	34-38 MPa

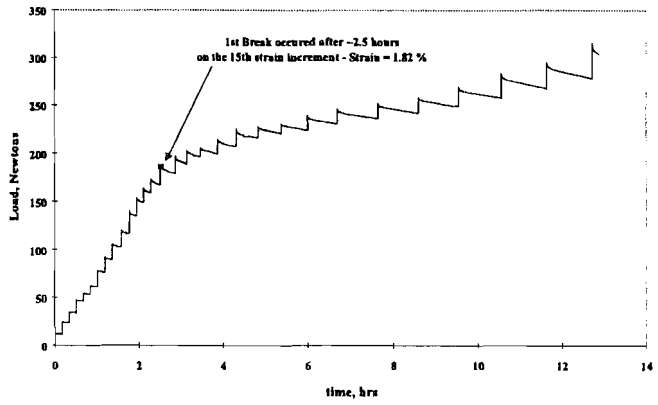

Fig. 1. Load-Time Curve of Bare E-Glass Fiber Specimen.

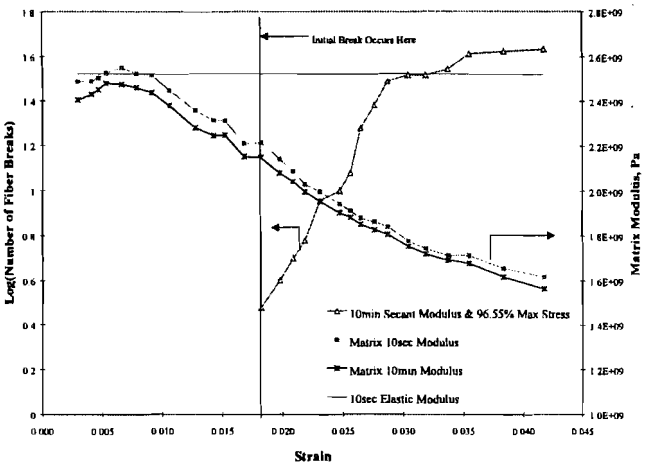

Fig. 2. Variation of Secant Modulus and Number of Fiber Breaks with Strain.

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