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Measuring Interface Strength in Single Fiber Composites: The Effect of Stress Concentrations

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ABSTRACT

Fiber-matrix interface strength is known to be a critical factor in controlling the long-term performance of structural composites. This parameter is often obtained by using the average fragment length data generated from the single-fiber fragmentation test (SFFT). The interfacial shear strength is then obtained by using this data in a micro-mechanics model that describes the shear-stress transfer process between the matrix and the fiber. Recently, a non-linear viscoelastic micro-mechanics model was developed to more accurately account for the matrix material properties. This new model indicates that the interface strength is dependent on the testing rate. Experimentally, it has been shown that the final fragment length distribution in some systems is dependent on the testing rate. However, data analysis using the new model indicates that the distribution change with testing rate is promoted by the presence of high stress concentrations at the end of the fiber fragments. From the model, these stress concentrations were found to exist at very low strain values. Experimentally, the fragment distributions obtained from specimens tested by different testing rates were found to be significantly different at strain values well below the strain values required to complete the test. These results are consistent with the research of Jahankhani and Galiotis and finite element calculations performed by Carrara and McGarry. These authors concluded that stress concentrations can promote failure of the fiber-matrix interface on the molecular level. Our results support this conclusion. In addition, our research results suggest that altering the SFFT testing rate can lower the magnitude of these stress concentrations and minimize failure of the fiber-matrix interface.

INTRODUCTION

It is well documented that the interface strength is a critical parameter for determining the strength and failure characteristics of composite structures. Although the single fiber fragmentation test (SFFT) is widely used to determine this parameter, interpreting the results from this test has been the subject of controversy. In 1993, results from a round robin testing program conducted under the auspices of the Versailles Project on Advanced Materials & Standards (VAMAS) revealed that the determined interface strength could vary by as much as 50 % between laboratories.[1] Intra-laboratory variation ranged from (9 to 36) %. Some of the inter-laboratory variation was attributed to differences in data reduction schemes. However, evaluation of the mean fragment lengths at the end of the test revealed that the inter-laboratory variation of the fragment lengths could be as much as 40 %. Since the mean fragment lengths are the experimental data that is used in the micro-mechanics models to determine the interface strength, these values should be independent of the data reduction methods. The variation in

the mean fragment lengths suggests that some of the differences in interface shear strength values obtained between laboratories may be due to differences in testing protocols.

Micro-mechanics models of the SFFT generally assume that the matrix material is linear elastic (Cox-type models) or elastic-perfectly plastic (Kelly-Tyson model). Since the embedded fiber (e.g., carbon or glass) is linear elastic, these models preclude the existence of test rate effects in their formulation. However, experimental data has shown that polymer matrices typically used in composite materials exhibit non-linear viscoelastic behavior during the SFFT. It was also found that these matrices, upon relieving the applied deformation at the end of the test (≈ 5.0 %), exhibited almost complete strain recovery (≈ 0.2 % permanent strain). Hence, the virtual absence of permanent set deformation after the test in the unstressed specimen precludes the existence of significant amounts of plastic deformation in the matrix. In addition, the delayed fragmentation of fiber fragments at times much longer than the application time of a step-strain suggests rate dependent effects may be involved. In response to these observations, the Cox micro-mechanics model was extended to the nonlinear viscoelastic regime by the use of the *Elastic-Viscoelastic Correspondence Principle* and *Schapery's Correspondence Principle*. The derived non-linear viscoelastic (n-LVE) micro-mechanics model has the following form for predicting the stress profile for an embedded fiber fragment:[2]

$$s_f\{z,e,t\} = \left(E_f - E_m\{e,t\}\right)e\left[1 - \frac{\cosh b\{e,t\}\left(\frac{l}{2} - z\right)}{\cosh b\{e,t\}\frac{l}{2}}\right] \tag{1}$$

where

$$b\{e,t\} = \frac{2}{d_f}\left[\frac{E_m\{e,t\}}{(1+n_m)\left(E_f - E_m\{e,t\}\right)\ln\left(2r_m/d_f\right)}\right]^{1/2}$$

$\sigma_f\{z, \varepsilon, t\}$	is the stress profile in the fiber
ε	is the applied strain
E_f	is the fiber modulus
$E_m\{\varepsilon, t\}$	is the non-linear viscoelastic relaxation modulus of the matrix
ν_m	is the matrix Poisson's ratio
d_f	is the fiber diameter
r_m	is the radius of matrix parameter
l	is the length of the fiber fragment
z	is the distance from the end of the fiber fragment

This equation is based on several additional assumptions that are typically used to derive Cox-type micro-mechanics models. These are: (1) deformation in the matrix material occurs along straight lines,

(2) perfect bonding exists at the fiber-matrix interface, and (3) interface failure behavior can be ignored. For a viscoelastic material subjected to a constant strain deformation, the stress in the matrix, and hence the matrix modulus, decreases with time. Therefore, equation 1 indicates that as the stress in the matrix relaxes in a given strain step with time, the stress at the center of the fiber increases with time. Although this increase in stress is consistent with the occurrence of delayed fracture events during the test, theoretical calculations suggest that the increase is only about 5 % of the initial maximum fiber stress. Thus, the increase in stress at the center of the fiber due to viscoelastic relaxation may not account for all of the delayed fracture events.

Consistent with viscoelastic materials behavior, the model also suggests that the size of the fragments at saturation should increase if the time between strain increments is increased. The magnitude of the increase, however, depends on the nebulous r_m parameter. The model suggests that a small value of the r_m parameter will result in only modest changes in the average fragment's length at the end of the test. Research in this laboratory has shown for a bare E-glass fiber embedded in a diglycidyl ether of bisphenol-A (DGEBA) resin cured with meta-phenylenediamine (m-PDA) that the average fragment lengths at the end of the test decreases when the time between strain increments during the SFFT is increased (see Figure 1).[3]

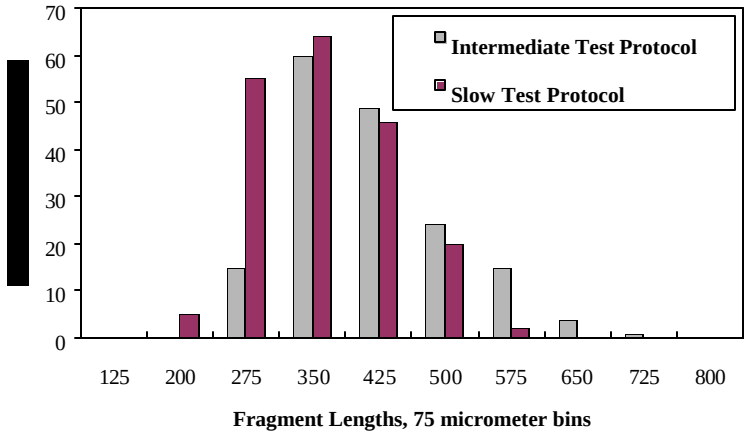

Figure 1. Histograms of Intermediate Test Protocol Specimens and Slow Test Protocol Specimens.

In Figure 1, the intermediate test protocol specimens were tested at an average effective strain rate of 0.000050 min^{-1} while the slow test protocol specimens were tested at an average effective strain rate of 0.000025 min^{-1}.

Since changes in the matrix behavior with strain are captured in the new micro-mechanics model, these results suggests additional mechanisms occurring at the fiber-matrix interface that change the size of the fragment lengths when the time between strain increments is increased. In this paper, the unexpected change in the size of the fragment lengths at saturation with increasing time is analyzed for the E-glass/DGEBA/m-PDA system using the new micro-mechanics model.

EXPERIMENTAL

The details of the experimental procedure and measurement uncertainties can be found elsewhere.[2,3]

RESULTS AND DISCUSSION

Using the n-LVE equation as a basis, one approach to understanding the change in fragment distribution with testing rate is to monitor the variation in the r_m parameter with increasing strain. For a linear elastic fiber, the strain profile in the embedded fiber is readily written from equation 1 as:

$$e_f\{z,e,t\} = \frac{\left(E_f - E_m\{e,t\}\right)}{E_f} e \left[1 - \frac{\cosh b\{e,t\}\left(\frac{l}{2} - z\right)}{\cosh b\{e,t\}\frac{l}{2}} \right] \tag{2}$$

Experimentally, the average strain in a fiber fragment can be determined at each strain increment by measuring the length of the fragment at a given strain increment and comparing it to the length of the fiber fragment in the unstressed state. The average measured strain can be equated to the strain predicted by equation 2 using the following expression:

$$\left\langle e_f\{z,e,t\}\right\rangle_{measured} = \frac{E^* e}{N} \sum_{i=1}^{N} \left[1 - \frac{\cosh b\{e,t\}\left(\frac{l}{2} - z\right)}{\cosh b\{e,t\}\frac{l}{2}} \right] \tag{3}$$

where

N denotes the number of theoretical strain calculations made along the fragment length

$$E^* = \frac{\left(E_f - E_m\{e,t\}\right)}{E_f}$$

Since the fiber diameter can be measured, there are four parameters that must be determined in the above equation. The Poisson's ratio of the matrix is taken to be 0.35. In lieu of a nonlinear constitutive law for the matrix, the strain dependent secant modulus, $\left\langle E_m\{e,t\}\right\rangle_{secant}$, has been used.[2]

Theoretically, the average measured strain in an embedded fiber fragment should scale with the applied strain, ε. For this case an estimate of the r_m parameter is obtained by adjusting this parameter until the right side of equation 2 matches the average measured strain. The dynamics of the fragmentation process and the redistribution of stress along the fiber in a viscoelastic matrix indicate that the applied strain will not immediately produce average measured strains in the fiber fragments that scale

at a given strain increment. Thus, if one assumes that at each strain increment the average measured strain in a fiber fragment scales with the applied strain, deviations from this assumption will result in an apparent increase in the r_m parameter for a given strain increment. This increase should be followed by a decrease in r_m as the stress in the viscoelastic matrix is redistributed. Since r_m determined in the manner will fluctuate about a given mean, a significant change in r_m can only be detected by a consistent increase of r_m over several strain increments.

In Figures 2 and 3, the variation of r_m with increasing strain are plotted for several fragments formed in specimens tested by the slow and intermediate test protocols, respectively. In Figure 2 none of the fragments plotted are longer than 500 µm. Plots of two of the three fragments (# 21 and 42) end before 4.3 % strain. This indicates that these fragments fractured into smaller fragments. In addition, r_m is relatively constant with increasing strain throughout the test. The fragments generated from the intermediate test protocol specimens are shown in Figure 3. Two of the three fragments are greater than 500 µm in length. None of these fragments fractured into smaller fragments. In contrast to the r_m values plotted in Figure 2, these r_m values steadily increase in value after 2.6 % strain. Although it is not readily apparent from the two figures, the average value of r_m at strain values below 2.6 % strain is consistently lower in the intermediate test protocol specimen.

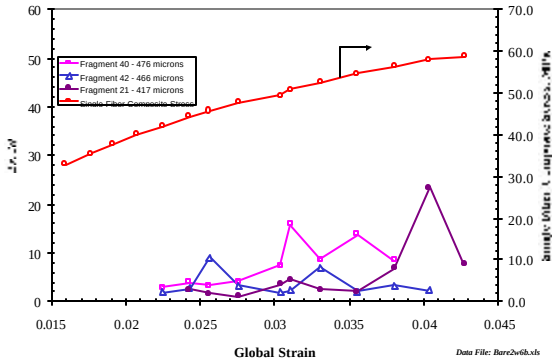

Figure 2. Variation of r_m with increasing strain in fragments obtained from specimens tested by the Slow Test Protocol.

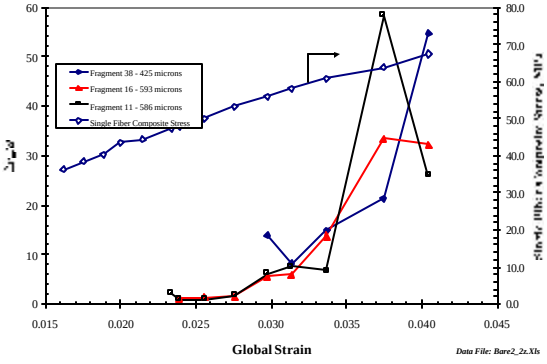

Figure 3. Variation of r_m with increasing strain in fragments obtained from specimens tested by the Intermediate Test Protocol.

From the r_m fits obtained above, the maximum shear stress in the fiber-matrix interface at the end of a fiber fragment can be obtained using the fiber-matrix interface shear equation (not shown - see reference 2). Carrara and McGarry [4] defined the stress concentration factor (SCF) as the ratio of the maximum shear stress in the fiber-matrix interface over the applied composite stress. From their finite element calculations, Carrara and McGarry noted that Cox-type models under-predict the maximum shear stress in the fiber-matrix interface at the end of a fiber fragment by a factor of 3 to 4. These authors reasoned that in the development of Cox-type micro-mechanics models the straight line deformation assumption that is used to effect a closed form solution to the problem precludes the existence of stress concentrations at the fiber-matrix interface. Jahankhani and Galiotis [5] found that if r_m is used as a fitting parameter in the Cox-type micro-mechanics model the Cox type model can be made to fit the stress profile in a fiber fragment. These authors reasoned that very low values of the r_m parameter were indicative of high stress concentrations at the fiber-matrix interface. For the data presented here, this factor is plotted in Figure 4 relative to the applied strain for typical fragments obtained from the slow test protocol specimens (fragment 42) and the intermediate test protocol specimens (fragment 11). At low strains, the stress concentration factor in fragment 11 is high and drops below the stress concentration factor obtained for Fragment 42 above 2.8 % strain. This drop is the SCF is consistent with a reduction in the interface strength in fragment 11 by breaking some of the highly stressed bonds at the fiber matrix interface. This reduction in interface strength will also reduce the efficiency in which the stress is transferred from the matrix to the fiber fragment. This in turn will result in the larger fragments at saturation when tested by the intermediate test protocol. The lower stress concentration in the slow test protocol specimens is associated with the increase time between strain increments. Since the matrix is viscoelastic, the increase time between each strain increment results in a decrease in the maximum shear stress in the fiber-matrix interface at the end of the fiber fragments.

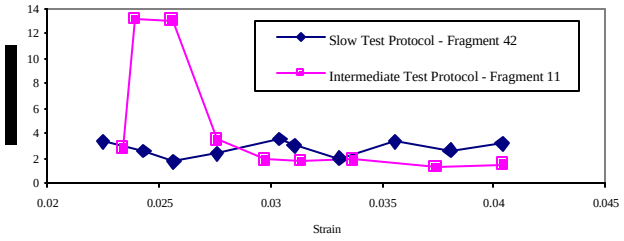

Figure 4. Theoretical Prediction of Stress Concentrations at the Fiber Ends of Specimens Tested by Intermediate and Slow Test Protocols.

Analysis of the fragmentation data indicates that the fragment distributions obtained by the intermediate test protocol are distinguishable from the slow test protocol fragment distributions at strain values lower that 2.5 %. This experimental data is consistent with the existence of high stress concentrations at the fiber-matrix interface of specimens tested by the intermediate test protocol. Research results by Jahankhani and Galiotis [5] on carbon fiber epoxy specimens indicates that stress concentrations can cause interface failure at the molecular level.

CONCLUSIONS

Data analysis using the n-LVE micro-mechanics model indicates that the change in the average fragment length of E-glass/DGEBA/m-PDA SFFT specimens when the testing rate is changed, results from the presence of stress concentrations in the fiber-matrix interface region at the end of the broken fiber fragments. When tested slowly, the magnitude of these stress concentrations are minimized due to increased viscoelastic relaxation of the matrix. As a result, additional failure in the fiber-matrix interface region is minimized and the average fragment length become shorter at slower testing rates. This interpretation was found to be consistent with the research results of Carrara and McGarry [4] and Jahankhani and Galiotis [5].

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