How Nonlinear Viscoelastic Matrix Behavior Influences Interfacial Shear Strength Measurements

G. A. Holmes, R. C. Peterson, D. L. Hunston, and W. G. McDonough

National Institute of Standards and Technology, Polymers Division,

Gaithersburg, Maryland 20899-8543

Abstract

Precise measurements of fiber break regions formed during single fiber fragmentation tests indicate that the calculated interfacial shear strength (IFSS) or the interfacial shear stress transfer coefficient (I-STC) is dependent on the testing protocol. E-glass/diglycidyl ether of bisphenol-A (DGEBA)/meta-phenylenediamine (m-PDA) single fiber fragmentation test (SFFT) specimens were found to be more sensitive than E-glass/polyisocyanurate SFFT specimens. For E-glass/DGEBA/m-PDA SFFT specimens, the change in fiber fragment distribution with testing protocol was found to be inconsistent with the effect expected when one only considers the nonlinear viscoelastic behavior of the matrix. These results are interpreted in terms of failure of the fiber matrix interface during the testing procedure.

Introduction

The development of a reliable micro-test method that measures the strength of the fiber-matrix interface in composite materials has been the focus of numerous research efforts. This research has been driven by the recognition that the interface or interphase in composites has a profound effect on the onset of failure of many composite materials (e.g., off-axis properties in unidirectional composites and the shear strength of composite laminates). In composites, internal materials failure generally precedes macroscopic failure. The internal failure modes of composite materials that may be observed are (1) fiber fracture, (2) micro-cracking of the matrix, (3) fiber-matrix de-bonding, and (4) delamination. The strength and toughness (damage tolerance) of the interface/interphase are important considerations in all these failure modes.

The interfacial stress transfer coefficient (I-STC) or interfacial shear strength (IFSS) is typically determined by using the single fiber fragmentation test (SFFT) procedure. In the SFFT, a dog bone is made with a high extension to failure resin and a single fiber is embedded along the axis of the dog bone. The sample is pulled in tension by the application of sequential step-strains of approximately 1.1 s in duration and 15 μm in length. Changing the dwell time between step-strains alters the test time and the effective strain rate of the test. By straining the matrix, the 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 lengths of the fragments at saturation are measured, and a micro-mechanics model is used to convert the average fragment length into a measure of the I-STC. Therefore, the success of this approach depends on how realistically the micro-mechanics model captures the actual behavior of the test specimen.

Researchers have observed that IFSS or I-STC values often exceed the yield strength of the matrix by several orders of magnitude. This is partly due to the simplifying assumptions made about the matrix during the development of micro-mechanics models. At a recent workshop on Micro-Mechanics Measurement Technologies for Fiber-Polymer Interfaces, there was a universal call for the development of more realistic micro-mechanics models (McDONOUGH *et al.* [1997]). It has been suggested that the final fragment distribution at saturation is dependent on the test protocol. Since the average fragment length at saturation is central to the determination of the I-STC, significant changes in the final fragment distribution are important. In addition, fragmentation has been shown to occur at times much greater than the application of a step-strain (MOON and McDONOUGH [1998]). These observations are inconsistent with the elastic or elastic plastic assumptions typically made about the matrix in the development of micro-mechanics models. Indeed, these time-dependent observations suggest viscoelastic matrix behavior. This paper looks at the behavior of the matrix during fiber fracture and its effect on the fragment distribution at saturation.

Experimental

Details of the experimental procedures used in obtaining the experimental data are described elsewhere (HOLMES *et al.* [1999]).

Results and Discussions

Figure 1 shows the load-time curve for a diglycidyl ether of bisphenol-A (DGEBA) meta-phylenediamine (m-PDA) matrix with an embedded E-glass fiber. This matrix, which is typically used to assess the I-STC between epoxy resin and glass fibers, exhibits nonlinear viscoelastic behavior above 1 % strain (10^{th} strain increment in Figure 1). Initial fragmentation of the embedded E-glass fiber also occurs when the matrix is exhibiting nonlinear viscoelastic behavior. Hence, determination of the I-STC from current micro-mechanics models becomes problematic, since most models (e.g., Cox, Kelly-Tyson) assume linear elastic and elastic perfectly plastic matrix behavior. At saturation, the actual stress in the DGEBA/m-PDA matrix is intermediate between the response predicted by the linear elastic modulus used in the Cox model and the elastic perfectly plastic assumption used in the Kelly-Tyson model (see Figure 1). Thus, a new model was developed to account for the nonlinear viscoelastic behavior of the matrix (HOLMES *et al.* [1999]). I-STC values obtained from the new model are generally 20 % lower than values obtained from the Cox model.

Consistent with viscoelastic behavior, the new model predicts that the critical transfer length, l_c, should increase with matrix stress relaxation. By increasing the time between strain increments, additional matrix relaxation should increase the average size of the fragments and result in a decrease in the I-STC value. This prediction was investigated by changing the dwell time between strain increments (see Figure 2). In contrast to the behavior predicted by the model, the fragment lengths became shorter with increasing dwell time between strain increments (see Figure 3). From these experiments, two sets of data exhibited similar stress-strain behavior. Each set consisted of two specimens, with one tested by the intermediate protocol and one tested by the slow protocol. In each data set the intermediate specimen yielded the largest fragments. These results suggest that the localized stress-strain behavior at the fiber matrix interface may be different. To further understand these results, E-glass fibers embedded in a second resin system (polyisocyanurate) were tested. Fragment distributions from these specimens were virtually unaffected by changes in the testing protocol (see Figure 4). This difference in behavior was observed despite the similar stress-strain behavior of the DGEBA/m-PDA matrix and the polyisocyanurate matrix. Both matrices exhibit nonlinear stress-strain behavior when fiber fracture occurs.

At the current time, no definitive explanation has been advanced to explain the above behavior. However, two avenues are being pursued which may be different aspects of the same mechanism. The first assumes that differences in the post yield behavior of the matrix may be different by changes in the dwell time between step-strains. A second approach assumes that fiber-matrix interface failure may be occurring during the test. Support for the latter assumption can be found in the work of Carrara and McGarry [1968]. These researchers found that Cox type models underpredict the maximum shear stress at the fiber ends by approximately 50 % when compared to results obtained from finite element analysis. Carrara and McGarry showed that the Cox approximation is reasonable at regions far from the fiber end, but the radial deformation lines bend sharply near the fiber ends and can only be crudely represented by straight lines. Hence, Cox type models effectively disregard stress concentration effects at the fiber ends. Recent investigations by Jahankhani and Galiotis [1991] using laser Raman spectroscopy revealed that stress concentrations at the fiber ends can result in premature failure of the fiber-matrix interface and a reduction in the IFSS. He also found, by reducing a parameter in the Cox model by a factor of three, that the shear profile at the fiber ends could be fitted at the fiber ends using the Cox model.

Applying the above results to the current research, increasing the dwell time between strain increments allow the high stresses at the fiber-matrix interface more time to relax. This may forestall failure of the fiber-matrix interface at the fiber ends during the test. Hence, by reducing interface failure in the slow protocol test specimens, the final fragment distributions become shorter with increasing dwell time between strain increments. Support for this interpretation can be found in the tangent moduli exhibited by specimens tested by the intermediate and slow test protocols with increasing strain. Specimens tested by the intermediate test protocol exhibit a tangent modulus of 1.2 GPa at saturation. In contrast, the tangent modulus at saturation of specimens tested by the slow protocol is approximately 0.8 GPa. This 33 % drop in stiffness is due to the increased dwell time and additional matrix stress relaxation between strain increments.

Conclusions

For the E-glass/DGEBA/m-PDA SFFT specimens investigated here, increasing the time between strain increments results in smaller fragments and a higher I-STC. This result is in contrast to what is predicted based solely on viscoelastic considerations and maybe associated with failure of the interface during the fragmentation test. Increasing the dwell time between strain increments allows additional time for stress at the fiber-matrix interface to relax and keep the interface stresses below the value necessary to initiate failure. The behavior exhibited by the E-glass/DGEBA/m-PDA SFFT specimens is not universal. Fragment distributions from E-glass/polyisocyanurate SFFT specimens are virtually insensitive to the testing protocol. Hence, variation of fragment distributions with testing protocol may reflect the damage tolerance of the fiber-matrix interface. These results, however, indicate that test protocols should be specified when reporting I-STC values.

Bibliography

1995 Wagner, H. D., Nairn, J. A., and Detassis, M., "Toughness of Interfaces from Initital Fiber-Matrix Debonding in a Single Fiber Composite Fragmentation Test," Applied Composite Materials, **2,** 107-117.

1997 McDonough, W. G., Holmes, G. A., Hunston, D. L., and Parnas, R. S., "Workshop on Micro-Mechanics Measurement Technologies for Fiber-Polymer Interfaces," **NISTIR 6102.**

1998 Moon, C. K. and McDonough, W. G., "Multiple Fiber Technique for the Single Fiber Fragmentation Test," Appl. Polym. Sci., **67**, 1701-1709.

1999 Holmes, G. A., Peterson, R. C., Hunston, D. L., and McDonough, W. G., "The Effect of Nonlinear Viscoelasticity on Interfacial Shear Strength Measurements," **ASTM STP 1357** (*in press*)

1968 Carrara, A. S. and McGarry, F. J., "Matrix and Interface Stresses in a Discontinuous Fiber Composite Model," J. Composite Materials, **2,** 222-243.

1991 Jahankhani, H. and Galiotis, C., "Interfacial Shear Stress Distribution in Model Composites, Part 1: A Kevlar 49 Fibre in an Epoxy Resin," J. Composite Materials, **25,** 609-631.

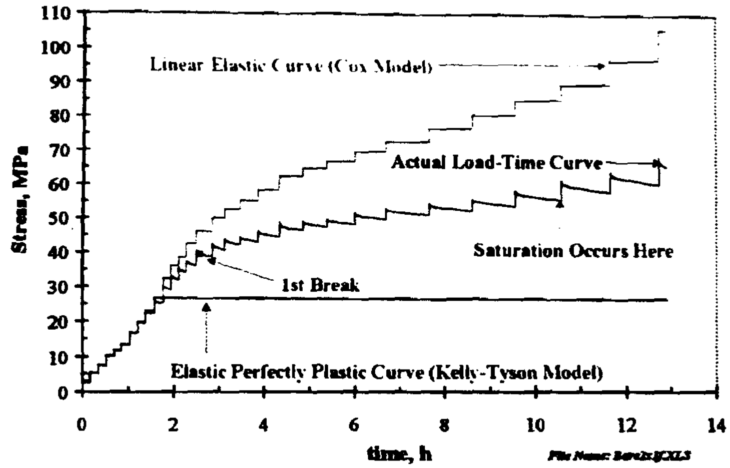

Figure 1. Typical Stress-Time Curve for E-Glass/DGEBA/m-PDA Single Fiber Composite

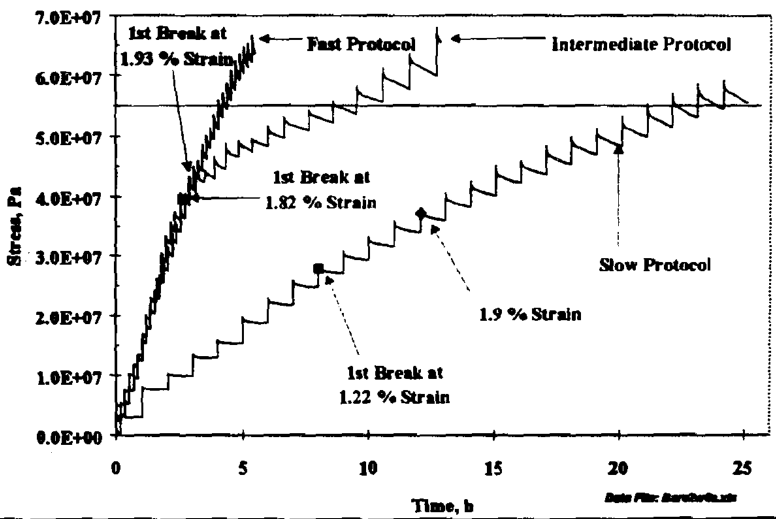

Figure 2. Test Protocols for Single Fiber Fragmentation Test Specimens.

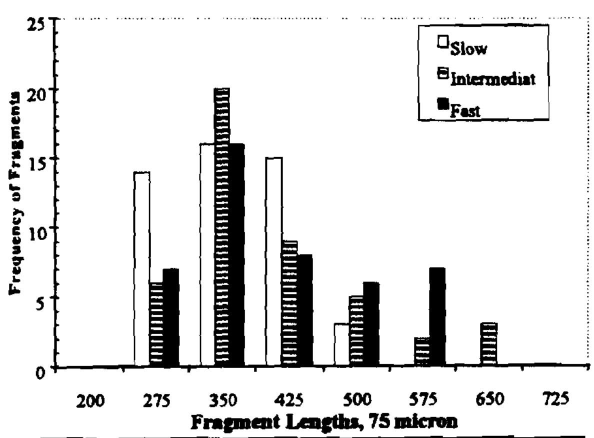

Figure 3. Effect of Test Protocol on bare E-Glass / DGEBA / m-PDA SFFT Specimens.

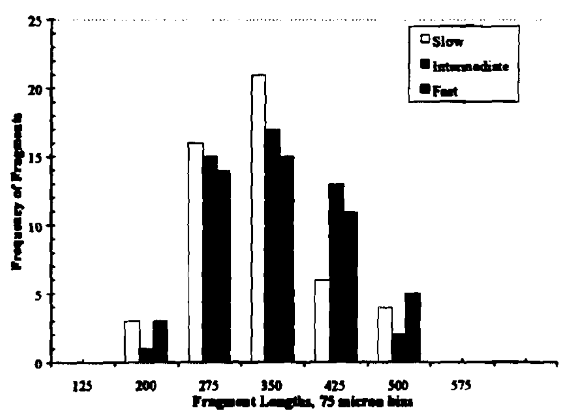

Figure 4. Effect of Test Protocol on bare E-Glass / Polyisocyanurate SFFT Specimens

Fourth International Conference on Constitutive Laws for Engineering Materials
July 27-30, 1999, Troy, New York

CONFERENCE PAPERS

Editors: R. C. Picu and E. Krempl

Chairman: E. Krempl, Rensselaer Polytechnic Institute
(Department of Mechanical Engineering, Aeronautical Engineering and Mechanics)

Co-Chairman: C. S. Desai, University of Arizona
(Department of Civil Engineering and Engineering Mechanics)

Local Organizing Committee:

Rensselaer Polytechnic Institute:

A. M. Maniatty, R. C. Picu, M. Szolwinski
(Department of Mechanical Engineering, Aeronautical Engineering and Mechanics)

J. Fish
(Department of Civil Engineering)

L. S. Schadler
(Department of Materials Science and Engineering)

Knolls Atomic Power Laboratory, Inc.:

T. L. Sham

U. S. Army Benet Laboratory:

P. C. T. Chen, J. Vasilakis

General Electric Corporation, Power Systems:

V. V. Kallianpur

Conference Secretary: D. A. Burgess
(Department of Mechanical Engineering, Aeronautical Engineering and Mechanics)

ISBN: 0-9609992-1-3