Design and Application of a Fiber Pullout Test for Examining Controlled Interfaces in Fiber Reinforced Polymers

Brian Goodfellow, Materials Science and Engineering, Cornell University

NNIN REU Site: Microelectronics Research Center, University of Texas at Austin
Principal Investigator & Mentor: Dr. Kenneth M. Liechti, Aerospace Engineering and Engineering Mechanics, University of Texas at Austin
Contact: bwg6@cornell.edu, kml@mail.utexas.edu

Abstract:

A fiber pullout test for examining controlled interfaces in fiber reinforced polymers has been designed and demonstrated. This test has been utilized in determining interfacial fracture toughness for sapphire/epoxy single fiber composite specimens. Some of these specimens incorporated a self-assembled monolayer at the sapphire/epoxy interface as a means of adhesion control—thus altering its interfacial fracture toughness. The application of self-assembled monolayers as controlled interfaces has a wide variety of applications in composite materials—predominantly in MEMS applications where near-frictionless interfaces are desired. The understanding and characterization of these self-assembled monolayers as fiber/polymer interface adhesion control is crucial to their potential in future applications.

Introduction:

Between components of a composite material, there exists an interface region on the order of several nanometers thick. The properties of this interface region can differ greatly from those of the bulk materials. Furthermore, the performance of the interface can significantly affect the overall performance of the materials system. Thus, by controlling the interface, one should be able to control the performance of the composite material.

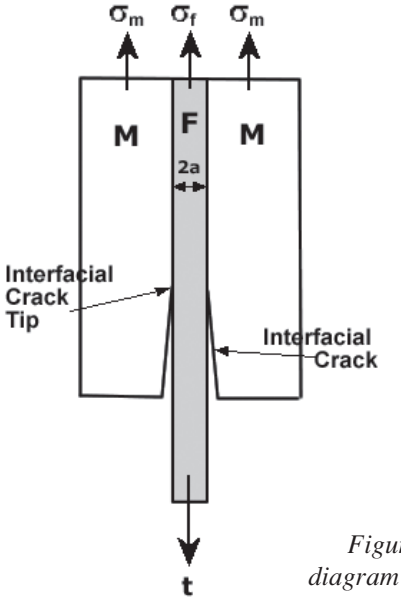

Figure 1: Schematic diagram of fiber composite.

The use of self-assembled monolayers (SAMs) as a means of controlling interface fracture has been proposed [1]. Potential uses of this idea include increasing or decreasing the interface fracture toughness as well as controlling the way in which the material fails. Examples of useful SAMs for this application are octadecyltrichlorosilane (OTS), BrUTS, and DTS. In theory, specifically chosen SAMs applied to an interface can lead to control over various materials fracture properties.

This research is devoted to the design and implementation of a fiber pullout test for the examination and characterization of these SAM interfaces. The theory for the test comes from fracture mechanics—specifically the calculation of the strain energy release rate G. From Figure 1, it is straight-forward to derive the strain energy release rate as

$$G = \frac{at^2}{4E_f}\left[\frac{\Sigma}{\Sigma + \frac{f}{1-f}}\right]$$

where a is the fiber radius, $\sum = E_m/E_f$ (ratio of Young's moduli), and *f* is the fiber volume fraction. Further manipulation of this equation leads to an expression for the critical load, P_c, at which point interface crack propagation begins:

$$P_c = 2\pi\sqrt{a^3 G_c E_f \left[1 + \frac{f}{\Sigma(1-f)}\right]}$$

where G_c is the critical strain energy release rate and the object of this test. Thus, when a single fiber composite test specimen is put in uniaxial tension, crack propagation should occur at P_c. From this data, G_c can be obtained.

Experimental Procedures:

The first stage of the test design was the design of the specimen. A sapphire fiber (~125 μm diameter) and an araldite epoxy matrix were chosen. To introduce a crack into the fiber/matrix interface, a small non-masked section of the fiber was coated with a thin coat of Teflon® before being embedded in the matrix. A dogbone shape was chosen as the specimen geometry because molds for this shape were available from previous experiments and only needed a few modifications. One side of the specimen was left

unchanged while the other was modified in order to effectively grip the sapphire fiber in the tensile machine. It is noteworthy to point out that the cross-section for this specimen is rectangular. While the equations mentioned earlier are derived for circular cross-sectioned specimens, this difference is essentially negligible for the interested range of specimen dimensions. The epoxy was cured for 2 hours at 60°C and allowed to cool overnight in order to lower residual stresses on the interface.

The second stage of the test design was the testing setup. Basically, a standard uniaxial tension test with a constant rate of displacement was used. The tension machine consisted of a stepper motor, a set of linear bearings positioned on vertical rods, and a 100 lb load cell. Also, a microscope and camera were mounted in order to observe and record the propagation of the interface crack. The specimen was to be pulled in uniaxial tension through several propagations until the fiber became totally debonded.

Lastly, some of the fibers were to be coated with OTS. The outline for the procedure used for this is as follows: 1) organic cleaning, 2) hydroxylate the surface, 3) remove excess water from the surface using oven, 4) deposit OTS (in dry box), 5) organic cleaning. These specimens were made and tested in the same ways as above with the exception that no Teflon® was used to introduce an interface crack.

Results and Discussion:

After much development, this experiment succeeded in obtaining a G_c value for the specimens. However, there were unexpected characteristics in the data which will be discussed later. Various stages of the crack propagation can be seen in Figure 2. For the specimens without the SAM interface, an average value of 147 J/m^2 for G_c was obtained from the data (SD: 77). This is a reasonable value for this materials system and thus was the first success of this research. The specimens with an OTS interface had an average value of 75 J/m^2 for G_c (SD: 41). This is almost half as large as the specimens without an OTS interface.

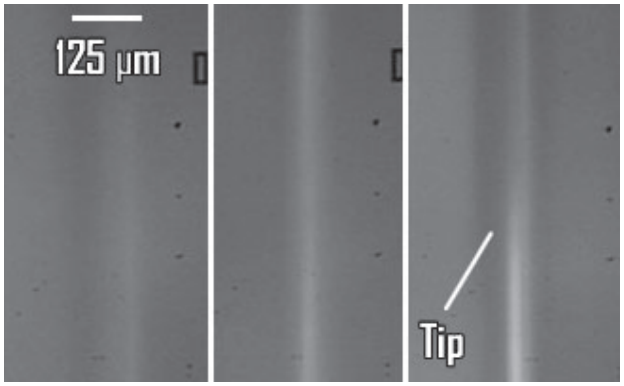

Figure 2: Region of fiber before crack propagation (left), after (middle), and crack tip (right).

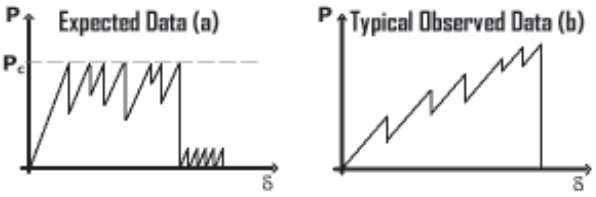

Figure 3: Discrepancy between (a) expected and (b) observed data.

Since it was expected that the OTS would lower the fracture toughness of the materials system, this is taken as the second success of this research.

Figure 3 shows the discrepancies between the expected and observed data. Specifically, in the observed data, the crack propagation peaks increase in height and there is fiber fracture instead of total debonding. Possible reasons for the increase in crack propagation peaks are; 1) residual stresses were present due to the curing process, and 2) crack initiation via Teflon® spray produced a non-uniform interface. To correct this, future research could involve curing the epoxy at room temperature for one week to greatly reduce residual stresses and finding a better way to initiate a crack at the fiber/epoxy interface to eliminate non-uniform effects of Teflon® spray.

Using fracture mechanics, it was found that, given the stress state, dimensions, and properties of the fiber, the fiber should have fractured when it did. To correct this, future research could involve increasing the fiber diameter or by coating the exposed fiber with silicone or a similar material.

Acknowledgments:

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References:

[1] Mello, A. W. and Liechti, K. M. (2002), "Controlling mixed-mode interfacial fracture toughness with self-assembled monolayers". Journal of Applied Mechanics, to appear.