IMAGING OF MULTI-FIBER, MICRO-MECHANICAL TESTING SPECIMENS USING OPTICAL COHERENCE TOMOGRAPHY

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INTRODUCTION

Predicting the strength and failure behavior of structural composites has been the subject of intense research for many years. It is known that the fiber-matrix interface strength plays an important role in the failure of these composites. However, correlating this knowledge with micro-mechanics tests, that are often used to assess this parameter, has proven elusive. This has forced designers of composite structures to use a "make and break" approach in determining the failure behavior of structural composites. This "trial and error" process is expensive, provides only limited data about the "failure envelope" of composite structures, and provides no fundamental understanding about the failure behavior of a composite in other structural applications, where the load profile may be different.

The lack of correlation between micro-mechanics test results and full-scale composite behavior has often been associated with the inability of micro-mechanics tests to effectively account for fiber-fiber interactions in the failure process. A research program at NIST has been initiated to investigate this premise and determine if research results from model multi-fiber composites will provide a bridge between micro-mechanics test results and full-scale composite behavior. Central to the success of such a program is the construction of model multi-planar, multi-fiber composites with controlled architectures and the development of technology to efficiently monitor and record the fragmentation behavior of several fibers within the test specimen. A schematic of the model composite is shown in Figure 1.

A critical part of this work is the need to acquire image data on the multi-fiber samples at different image planes throughout the sample. Conventional microscopy used for single fiber micro-mechanics testing has neither the thickness resolution nor the sensitivity to fulfill the requirements for these samples. For these samples, we will use a technique called optical coherence tomography.

Optical coherence tomography (OCT) is a non-invasive, non-contact optical imaging technique that allows the visualization of features within scattering media with precise knowledge of the location of these features. [1,2,3] OCT has the high sensitivity (>100 dB) and resolution (5 to 10) μm required to image small fibers (about (10 to 20) μm in diameter with a spacing of a single fiber diameter) in a multi-planar, multi-fiber micro-mechanical specimen. In addition, OCT is able to simultaneously capture images of fibers in different planes of the sample.

In this initial work, we interfaced a micro-mechanical testing stage with the OCT. We then demonstrated the feasibility of this approach by comparing OCT images of single and multi-fiber samples to optical microscopy images for resolution and sampling repeatability evaluation. Then, we collected a volumetric image set of a single fiber sample at 0% and 2.5% strain and compared the fiber break image data to data collected on the same sample with conventional video microscopy.

EXPERIMENTAL

Dogbone Preparation

The preparation of dogbone specimens was described in detail elsewhere. [4]

Sample Testing

After the samples had been sanded, two marks were placed on the specimen surface approximately 1 cm apart and perpendicular to the long axis of the specimen. These marks were used subsequently to measure the strain in the specimen during the test. Most of the single fiber fragmentation tests were carried out on a hand operated testing apparatus similar to the one described by Drzal and Herrera-Franco. [5] This apparatus was attached to a polarizing microscope (a Nikon Optiphot-Pol). [6] The stationary grip of this apparatus was attached through a load jig to a 1.1 kN load cell (Cooper Instruments, LPM 530). With the sample mounted in the apparatus, fiber diameters were measured at 3 different locations along the fiber, and the measurements were made using the microscope, video camera (Optronics LX-450A RGB Remote-Head microscope camera), video calipers (Boeckeler VIA-100, Boeckeler Instruments) and a monitor (Sony Corporation, PVM-1344Q). Additionally, the initial distance between the two strain marks was measured with the aid of a transducer (Trans-Tek, Inc. model 1002-0012). During the test, a small step strain was applied manually by turning a knob attached to the movable grip of the apparatus. The strain increments are on the order of 0.1% strain. After the strain increment, there was a delay of several minutes before the next step-

strain. After 2.5% strain was reached, the sample remained under strain overnight to reach saturation. This was done to ensure the number of breaks in the video images and in the OCT images would be identical.

Optical Microscopy (OM)

The 10X objective was used to collect images of interest from the 0% and 2.5% strain samples. The total magnification is 30X when a 3X optic inside the microscope is considered. Fifteen images were collected, each 375 μm in length. Each individual image was saved using the Snappy image capture package and then collaged in MS Word to create a composite image that was 5.3 mm long.

Optical Coherence Tomography (OCT)

OCT uses light in a manner analogous to the way ultrasound imaging uses sound, providing significantly higher spatial resolution (10 to 20) μm albeit with shallower penetration depth. OCT is based upon low-coherence optical ranging techniques where the optical distance to individual sites within the sample is determined by the difference in time, relative to a reference light beam, for an incident light beam to penetrate and backscatter within the sample. This temporal delay is probed using a fiber optic interferometer and a broadband laser light source. The fiber optic interferometer consists of a single-mode optical fiber coupled with a 50/50 fiber optic splitter that illuminates both the sample and a linearly translating reference mirror. Light reflected from the reference mirror recombines with light back-scattered and reflected from the sample at the 50/50 splitter to create a temporal interference pattern which is measured with a photodiode detector. The resulting interference patterns are present only when the optical path difference of the reference arm matches that of the sample arm to within the coherence length of the source. The incident light beam is scanned and repeated measurements are performed at different transverse positions to generate a two dimensional array which represents the backscattering or back reflection of a cross sectional plane of the material. This data can be displayed as a gray scale or false color image.

In this work, the image resolution is 11 μm along the x axis, 15 μm along the z axis, and 11 μm along the y axis. The average fiber diameter is 15 μm. In order to compensate for the barely adequate spatial resolution, the images were oversampled at 2 μm in the x and y directions (see Figure 1 for axes), 3 μm in the z direction.

RESULTS AND DISCUSSION

Figure 2 shows a 5 mm length of the embedded E-glass fiber in the epoxy dogbone. The OM image is shown in Figure 2A and the OCT image is shown in Figure 2B. The fiber in each image is indicated by the arrows. There is very good agreement between the measured fiber diameters when comparing the OM and OCT images. The measured fiber diameter of the OM image is 15 μm and 16 μm for the OCT image. The standard error for the OM image is 1 μm, and it is 2 μm for the OCT image.

Figure 3 compares the OM (A) and OCT (B) images for the fragmented E-glass fiber in epoxy dogbone. The location of the breaks in each image is indicated by the dotted arrow. There is very good agreement between the breaks in the OM and OCT images, indicating that OCT is capable of accurately capturing these events. However, the width of the fiber is much wider than in the unstrained case, about 30 μm. At this point in time, the origin of the abnormally large fiber diameter of the strained sample is not clear. It could possibly be degradation of resolution from the fiber being too far out of the focal plane of the sample. This issue will need to be resolved since it could potentially interfere with visualization of adjacent fibers.

CONCLUSIONS

This work has demonstrated that OCT is capable of detecting single 15 μm, E-glass fibers in a micro-mechanical test specimen. In a sample under strain, the fiber breaks detected by OCT agree well with OM images. Fiber diameter issues will be resolved by carefully controlling the optical configuration.

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6. Identification of a commercial product is made only to facilitate experimental reproducibility and to adequately describe experimental procedure. In no case does it imply endorsement by NIST or imply that it is necessarily the best product for the experimental procedure.

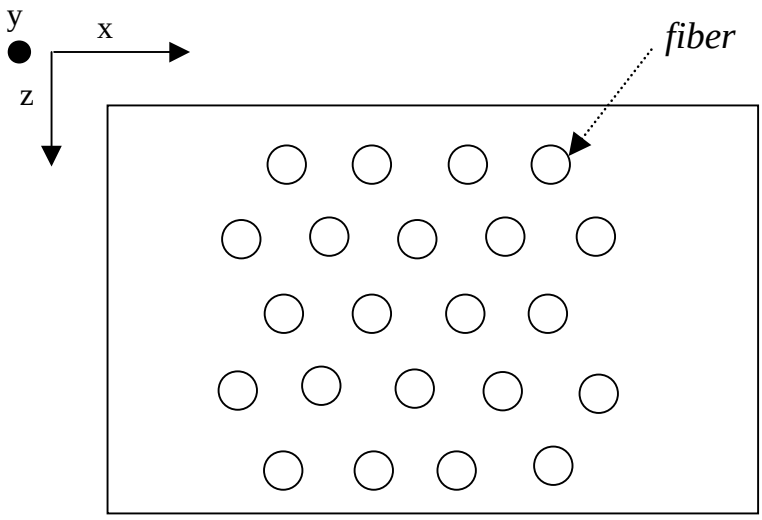

Figure 1: Cross-sectional schematic of multi-planar, multi-fiber micro-mechanical specimen.

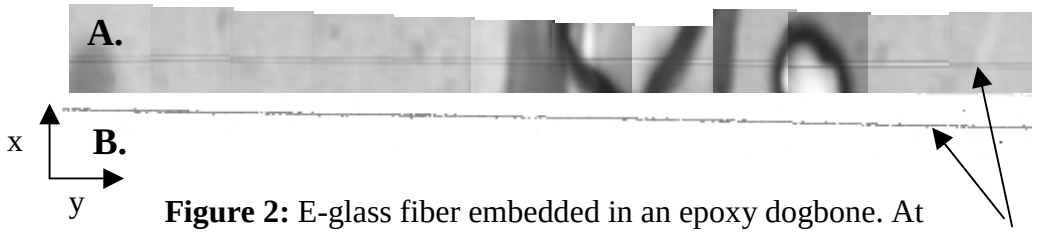

Figure 2: E-glass fiber embedded in an epoxy dogbone. At 0% strain. OM image (A.), OCT image (B.).

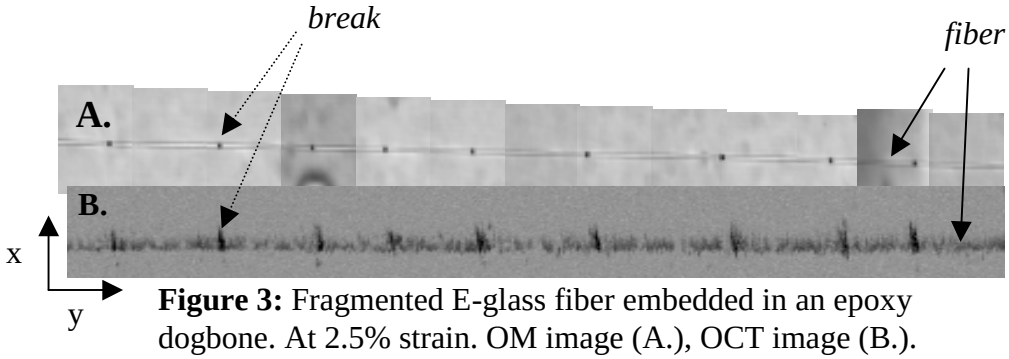

Figure 3: Fragmented E-glass fiber embedded in an epoxy dogbone. At 2.5% strain. OM image (A.), OCT image (B.).