Test Protocol for
Single-Fiber Fragmentation Test

International Round Robin

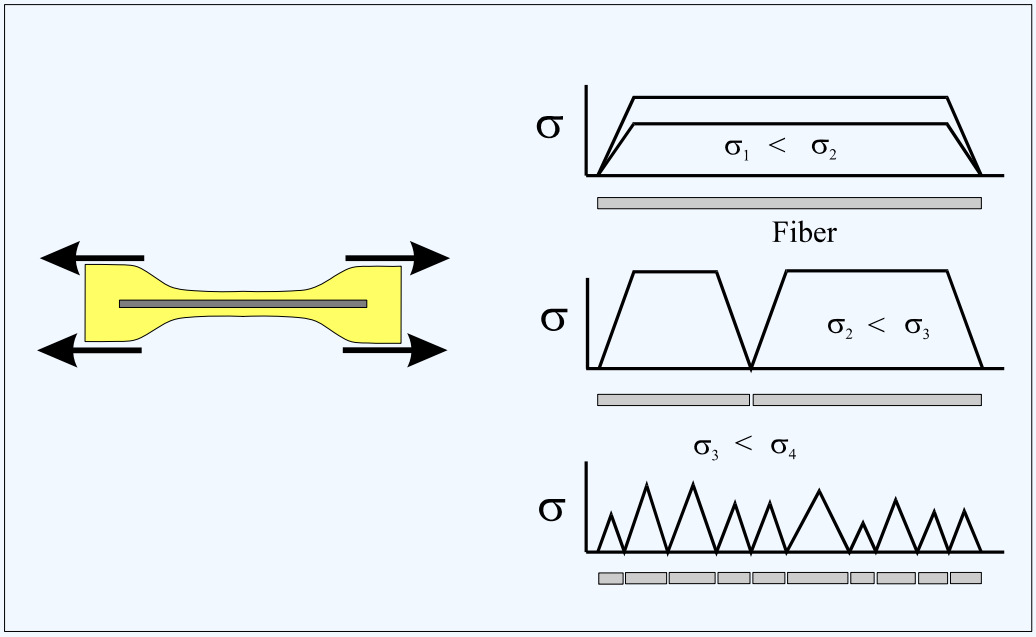

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Table of Contents

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General Comments Regarding Round-Robin Tests

Based or our experience in testing the specimens, we would offer the following advice in addition to the test procedure outlined below. First, we found more specimen to specimen variation than we would like, and as a result, it is important to average multiple results. As a result, please test all 10 specimens. We found it necessary to run several experiments before we could get the step size close to that specified in the protocol. In addition, some specimens may break before you reach saturation. Consequently, please mark as "questionable or bad" those experiments that you feel should not be included in the analysis and indicate why (for example, the loading deviated significantly for the protocol or saturation was not reached). Another important point is the measurement of the fiber diameters. This is a big potential source of error in the experiment so it is important for you to calibrate the procedure before doing the measurements.

The samples will be shipped in individual metalized bags sealed in a dry inert atmosphere. Please keep the samples in the sealed bag until you are ready to test them. Inside the metalized bag, the samples are contained in a plastic bag. This second bag is not sealed initially, but can be sealed to hold the samples when they are returned after testing. Both the sample itself and a card inside the bag are marked with the sample designation. Record this designation on the Sample line on the Data Sheet.

Basic Test

The test involves a resin dogbone with a single fiber extending the length of the sample along the dogbone's axis. There are a number of ways in which the test can be conducted; the protocol outlined here is the method selected for this round robin. To be successful with this protocol, the resin must be transparent and have a higher extension to failure than the fiber. The test involves loading the sample in tension. As the load increases, the fiber breaks repeatedly until the remaining fiber fragments are shorter than a critical length ℓ_c. Fragments of length ℓ_c or less are so short that it is not possible to transfer sufficient stress into the fiber through the fiber-matrix interface to cause the tensile stress within the fragment to reach the failure strength. As a result, the fragments no longer break with increasing strain of the dogbone. This is called saturation. Since any fragment with a length exceeding ℓ_c will be broken before the conclusion of the experiment, a distribution of fragment lengths between $\ell_c/2$ and ℓ_c will be obtained. In actual tests, the distribution of fragments is larger. One reason for this is that most high-performance engineering fibers have strengths that vary considerably along their lengths because of flaws introduced through handling or manufacturing or because of intrinsic anomalies of the material. These flaws or point defects introduce variations in strength, and this also contributes to the distribution of fragment lengths at saturation. By analyzing the fragment distribution with an appropriate model, a measure of the interface performance is obtained.

Samples

The sample dimensions are shown in Figure 1. Since the samples are prepared in an open mold, they initially have one flat side and one side with a meniscus. After fabrication, the samples have been polished to remove the meniscus, but it should still be possible to tell which side was initially flat by examining the edges. If this side can be determined, place the sample into the grips with this side down. Since the samples have been polished, there should no problem in seeing the fiber. Nevertheless, it may be useful to put some silicone oil on a 25 mm x 25 mm microscope cover slide (or at least having dimensions that cover the gage length used in the test) which is then placed on top of the sample in the gage area to aid in seeing the fiber

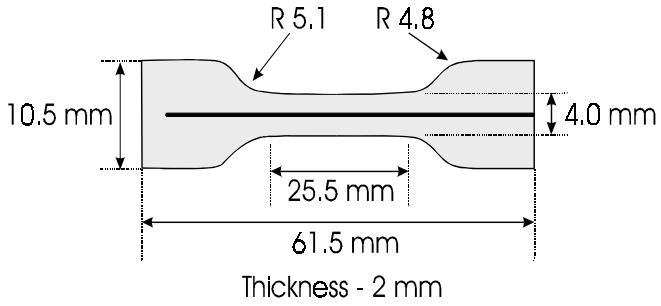

Figure 1: Typical dimensions for dogbone specimen. Dimensions for any given sample may vary from these values.

clearly. If the operator is still having trouble seeing the fiber, a second glass cover slide with silicone oil can be placed on the bottom (flat) side of the sample.

The test specimens are AS-4 carbon fibers encapsulated in a diglycidyl ether of bisphenol-A epoxy (Shell Epon 828) cured with a stoichiometric amount of meta-phenylene diamine. Before testing a sample, run your finger along the edge to see if any nicks or defects are present. If anything is found, it should be eliminated by a light sanding with fine grain paper (800 or 1000 grit).

Apparatus

The sample shall be tested in a micro-straining machine having the ability to load the sample in tension to a load that generates saturation using the strain history outlined below. It should be able to measure the fragment lengths over the complete gage length (about 1.5 cm) with an accuracy of ±5 μm or better. There must also be a procedure for measuring the fiber diameter throughout the gage length with an accuracy of ±1 μm. Both measurements are usually done with a microscope, and polarizers may be needed to assist in locating the fiber break points.

A recommended apparatus consists of a small loading frame mounted on a X-Y micrometer stage attached to a polarizing microscope (see Figure 2). The load frame should be capable of straining the specimen to at least 7 % (15 % for glass fibers). The micrometer stage

is used to manipulate the load frame under the microscope so the entire gage length can be scanned. An LVDT attached to the stage monitors the exact position of the load frame. When a fiber break or other feature is aligned with a cross-hair in the microscope view field, the LVDT reading quantifies the locations of that feature. The microscope should be configured such that there is one polarizer below and one above the test coupon. Since the embedded fiber is located in the center of the polymeric sample and therefore is difficult to observe at high magnification with standard objectives, the microscope should be equipped with a long working distance 20x or greater objective lens. Since the break is sometimes difficult to see in graphite fibers, magnification of at least 250x is recommended and 500x is preferable. The light source should provide enough illumination to see clearly events such as fiber breaks and birefringence. Some samples do not exhibit much birefringence. In this case, one approach is to insert the polarizer lens only part of the way into the scope. This can sometimes produce a condition where there are bright lines along the surface of the carbon fiber. These lines are disrupted when there is a break (see Figure 3). The load frame should be equipped with a displacement gage or other device to monitor the approximate strain applied to the sample. A load cell for monitoring stress on the sample is also helpful but not essential.

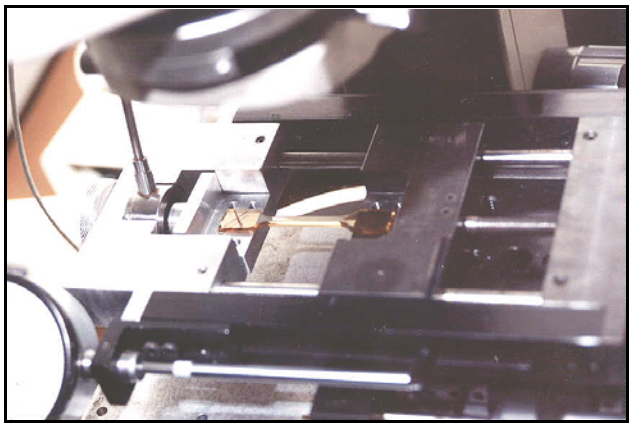

Figure 2: Loading jig with sample mounted in microscope.

A number of grip designs are possible. The key is to minimize slipping while avoiding stress concentrations that cause premature failure in the grips. Since the grip may have an effect on the experiment, please provide a brief description of the grips that you used. A diagram of the grips used at NIST is available for anyone interested.

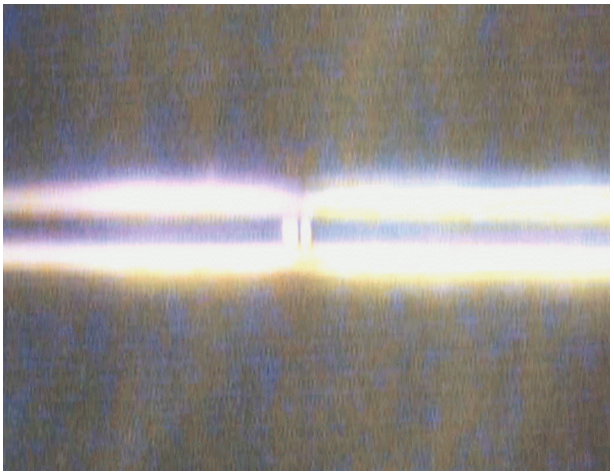

Figure 3: Microscope view with polarizer inserted part way in.

Measurements before Testing

A number of measurements need to be made before the test is started.

Fiber Diameter: The fiber diameter needs to be measured for each dogbone sample with a calibrated filar eyepiece or video calipers. Video calipers, although more expensive, tend to place less wear and tear on the operator's eyes, and because several researchers can see the fiber on a monitor, tend

to result in more uniform measurements from person to person. The diameter should be measured at least five times along this gage length region. If measuring the fiber diameter on the test stage, be aware that sometimes it is difficult to find the exact edges of the fiber, and this difficulty can lead to uncertainty in calculated values of the interfacial shear strength parameter. Careful calibration of the procedure against a dimensional standard just prior to the measurements is critical since determination of fiber diameter is a potential source of significant error in the experiment. Appendix I illustrates the determination of fiber diameter. Since this measurement is so important, we would request that you repeat the calibration process a number of times to determine an experimental uncertainty for the diameter measurement. A place for this uncertainty is included on the data sheet.

Shifting of Gage-Length Region: Depending on how the apparatus is designed, the gage-length region may shift as well as stretch during the experiment. For example, in many designs, one end of the sample is fixed while the other end is pulled to introduce the load. As a result, when numerous fiber breaks are present, it may become difficult to determine which are within the original gage length and which are not. One way to address this problem is to place marks on the specimen before testing to indicate the ends of the gage length. You can focus on the mark to determine its location and then focus on the fiber break and determine its location. We use a magic marker to draw two parallel lines on the specimen (about 12 to 14 mm apart in our case). These same marks can used to measure strain as outlined below. Another way is to measure the absolute location of each break after every strain step. The gage length can then be mapped onto these data to determine which breaks to monitor. In any case, some procedure is needed to address this problem.

Strain Measurement Marks: It is also necessary to measure the strain at each loading step in the experiment. In most cases, measurements based on grip displacements do not adequately characterize the actual strain in the sample. This is especially true if the sample should slip in the grips at any time during the test. Therefore, direct strain measurements are mandatory. The easiest way is to place marks on the sample before testing and then the positions of the marks can be monitored by the same technique used to determine the fiber fracture points. If these marks are placed at the end of the gage length region as outlined above, they can be use to indicate the gage length as well as to determine the strain.

Testing

The testing is carried out at room temperature and humidity; both of which should be recorded.

Zero Load: The experiment is conducted by first taking the slack out of the load frame but applying no load. If the device is equipped with a load cell, this is straight-forward. If the investigator does not have a load cell on their apparatus, another approach must be used. One approach is the following. After the grips have been tightened, manipulate the strain until you feel a slack in the sample that indicated that there was no strain present. Then add strain in the

sample up to the point where you just observed a change in the color of the matrix (under polarized light) and back off slightly. This is usually a good estimate of the point to call zero stress. Another approach can be used if the grips do not hold the specimen completely fixed. The sample is wiggled along the loading axis while grip separation is increased until it is no longer possible to wiggle the sample. The gage length marks on the sample are measured both before and after the procedure. If the readings are similar, it is safe to assume that no strain has been introduced into the sample by the zeroing procedure.

Zero Strain Measurement: Once the load is zeroed, the next step is to measure the location of the strain markers at the zero load. The separation distance of the strain markers serves as the basis for calculating strains when similar measurements are made for the sample under load.

Loading History and Measurements: The sample is now loaded with a series of cycles involving a step strain followed by a holding period during which the stain is held fixed. We recognize that most people in this field have manual instruments with very simple controls. Nevertheless, aiming for a standard strain history should reduce the variations from one laboratory to another even though actual loading will vary. Each strain step should be about 0.2 % strain. Since few instruments will be able to directly control strain, the usual procedure will be to control the cross-head displacement in a way to give approximately 0.2 % strain. For our instrument this means that each step is about 0.09 mm or 35/10000 of an inch. Each cycle should be 10 minutes long. The strain step is applied in the first couple of seconds. During the hold time, the strain marks are measured and the actual size of the strain step is calculated and recorded. This knowledge is used to adjust the next strain step so that it is as close as possible to the goal of 0.2 %. If the calculations shows that the last strain step was too small or too large, do not try to make up for this difference. Just aim to achieve the goal of 0.2 % in the next step. At the 8 minute point in the cycle, the gage length is scanned and the number of breaks in the gage length are counted and recorded. The first breaks will usually be observed when the total strain reaches a level of between 1.2 % and 1.8 %. At the 10 minute point, the next cycle is started by applying the strain step. The cycles continue until mechanical saturation is reached. We shall define mechanical saturation as the point at which, during three or more successive cycles that total 0.6 % strain, no additional breaks are recorded. The strain at this point should be between 3 % and 5 %. Once saturation is reached, the fragment lengths must be measured before the load is removed (Once the load is removed the breaks are difficult to see). Depending on your particular equipment, either the fragment lengths or the locations of each break within the gage length need to be measured and recorded. The approximate time of the experiment can be estimated by noting that loading to 3 % will involve 15 steps (150 min.) plus the time required to characterize the fragments. Loading to 5 % will require 25 steps (250 min.) plus characterization time.

Determining Location of Fiber Breaks in Specimen: As this test will involve using carbon fibers, special testing issues should be mentioned. With carbon samples, it is sometimes difficult to tell where the breaks occur. This is especially true near mechanical saturation (at the

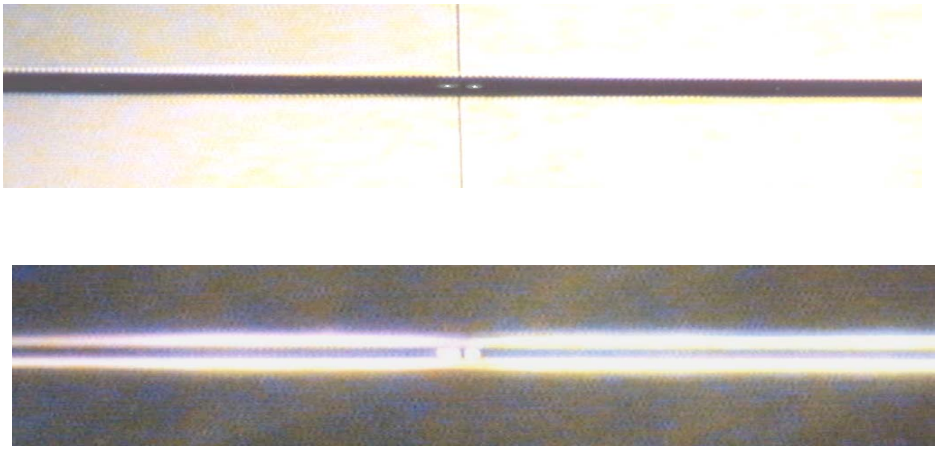

Figure 4: Microscope view of fiber break in carbon fiber. Part A shows fiber without polarizers, while part B has the polarizer partially inserted in the optical path.

end of the test) where the breaks are fairly close together. A magnification of 250x or more is very helpful. If the operator is looking for fractures under polarized light, then what appears to be a crack may just be two birefringent regions coming together. When looking for breaks without using the polarizers, there are also difficulties in determining where the fracture occurs. With glass fibers, the break regions are usually clearly defined, but this is not the case with carbon fibers. When examining the breaks in carbon fiber samples without the aid of cross-polarizers, the operator needs to look for, what can best be described as, white spots that indicate where the break location (Figure 4). Sometimes these spots are easy to find, sometimes they are not. It is helpful to switch between using and not using the polarizers to find the exact location of the break. Moreover, as mentioned earlier, it is sometimes helpful to insert the polarizer into the scope only part way. Under these conditions, bright lines along the surface of the carbon fiber may be observed. If these lines are present, they are disrupted by a break in the fiber. This can be used to help determine the position of each break (Figure 3).

Measuring Fragment Lengths: At mechanical saturation, the fragment lengths or the position of the fiber breaks need to be determined. Traditionally, this is done optically using a microscope equipped with a calibrated filar eyepiece, but an alternative is to use video calipers. As mentioned earlier, this is easier and more reliable. A third method is to record the location of each break using a procedure such as that described above for determining the strain.

Data Reporting

For the sake of generic testing and due to the variety of analysis methods that have been proposed in the literature to determine interfacial shear strength, this round robin will focus only on the experimentally measured quantities such as the distribution of fragment lengths at

saturation. Other characterization experiments such as fiber surface analysis, Raman data on the fiber stresses, etc. will be encouraged, and the information obtained will be added to the data base. All of this information will be collected and made available to participants in the program who would like to analyze the results with their own approach. In the final report, the source of each data set will be anonymous; labeled only with a test laboratory number.

There is a minimum set of data that should be reported for each fragmentation test. A general reporting form for this data is provided in Appendix II. You will need to make a copy of this form for each specimen. An electronic version of this form (Microsoft Excel 97) is included on a disk for those who would find this useful. The description of how the experiment was performed need only be entered on one data sheet unless there is something special about a particular test. Things to include are grip design, strain assessment method, procedure to determine which breaks are within the gage length, fragment length or location measurement technique, etc. If possible, two photographs should be provided. The photographs should show a typical fiber break under polarized light and under load at both low (~100x) and high (~400x) magnifications. Moreover, if you take additional information, we would be more than happy to include these results in the data base as well. One example would be the measurement of load during the experiment. We hope to obtain as much data as possible on this model system so researchers can use the data in the future. Consequently, anyone who would like to characterize the materials by other methods, such as fiber surface analysis, should let us know and we will try to provide materials.

What needs to be returned when the experiments are completed: When you have finished the testing, please place the tested sample back the resealable plastic bag and return the following items: (1) tested samples, (2) completed data sheets, (3) photographs of typical fiber breaks, (4) description of any differences in how the experiments were performed relative to the procedure described here (including the calibration procedure), and (5) any additional characterization information on the samples or constituents. If you have any questions or suggestions, please send them at once since other participants may have the same concerns. The address is

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Appendix I

The pictures below illustrate the determination of fiber diameter using a video caliper. Part A in the Figure shows the fiber and cursors. To measure the diameter, the inside edges of the cursors are aligned with the outer edges of the fiber as shown in Part B. To assure accuracy, the caliper should be calibrated prior to the measurements, and the calibration should involve alignment of the cursors edges in the same way as is done with the fiber.

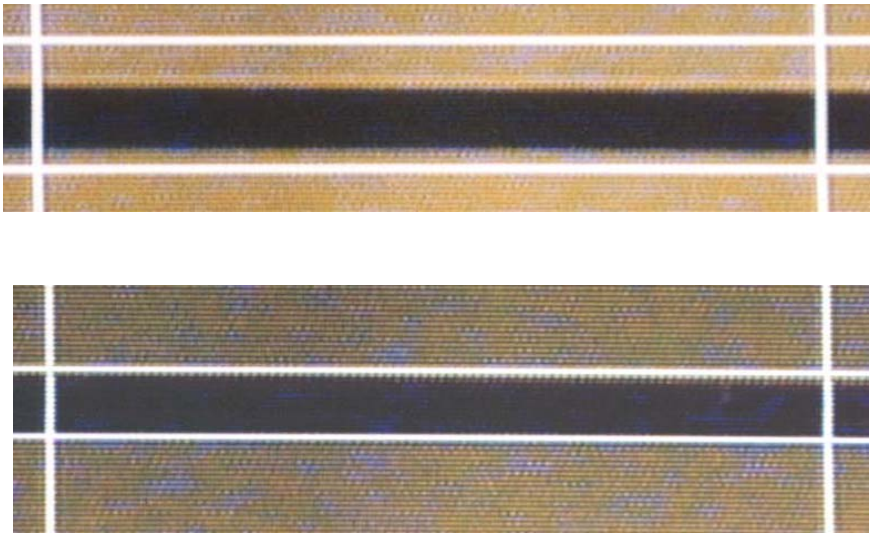

Figure 5: Two microscope pictures illustrate the use of a video caliper to measure the fiber diameter. The cursors are shown in part A at some distance from the fiber surface. In part B they are aligned to measure the diameter.

Appendix II
Data Sheet for Fragmentation Test Results

Sample _____

Laboratory_____

Date _____

Temperature (C)_____

Relative Humidity (%)_____

Fiber Diameter (μm) _____
_____
_____
_____
_____
_____

Average Diameter (μm)_____

Uncertainty in Diameter (μm)_____

UP TO SATURATION

Time (min)	Strain (%)	Number of Breaks	Time (min)	Strain (%)	Number of Breaks
0	0	0	168		
8			178		
18			188		
28			198		
38			208		
48			218		
58			228		
68			238		
78			248		
88			258		
98			268		
108			278		
118			288		
128			298		
138			308		
148			318		
158			328		

AT SATURATION

□[1] Location of fiber Breaks in gage section at saturation or

□[1] Fragment lengths at saturation

(mm)	(mm)

[1]Indicate with a check which type of data is provided by your equipment and reported here.

Page 2 of Results

Sample _____ Laboratory _____

Brief description of how experiments were performed (main thing of interest here is deviations from recommended procedure and equipment)

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Other comments: