HIGH STRENGTH FIBER TESTING: THE SINGLE FOLD TEST

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ABSTRACT

Questions about the field performance of a first responder's body armor prompted research to assess the long-term durability and effectiveness of current and future soft body armor. Prior work in this laboratory described a 10 % drop in tensile strength in poly(*p*-phenylene benzobisoxazole) (PBO) fibers after they were subjected to a single fold for 24 h. Regions of internal damage were also seen in the folded fibers. Here, that work is continued by investigating the response of poly(p-phenylene terephthalamide) (PPT) fibers to the single fold. The results presented in this paper show no change in tensile strength, modulus, or failure strain in the PPT fibers and a different surface morphology in the folded region.

1. INTRODUCTION

The National Institute of Standards and Technology's Office of Law Enforcement and Standards (NIST-OLES), under the auspices of the National Institute of Justice (NIJ), conducts and sponsors research to assess the long-term durability and effectiveness of current and future soft body armor. This program was established in response to the field perforation of armor made with poly(*p*-phenylene benzobisoxazole) (PBO) fibers at a threat level it had been designed to defeat.

A review of potential degradation mechanisms of PBO fibers pointed to ultraviolet (UV) exposure, exposure to moisture (associated with perspiration and humidity), elevated temperature exposure (resulting from storage in the trunk of an automobile), and folding (associated with normal wear) as possible factors that may compromise the structural integrity of the ballistic fiber during use.[1]

Prior work has focused on the role that folding may play in the degradation observed in soft body armor.[2] Using the modified single fiber test (m-SFT), the measured strain-to-failure and ultimate tensile strength of the single-folded virgin PBO fibers were found to

be reduced by about 10 % relative to the virgin fibers that had not been exposed to the fold. These results indicate that the property changes induced by folding should be quantifiable and that the m-SFT is sensitive enough to observe these changes. One of the questions remaining after examining these results was if other high strength fibers, such as PPT, show a similar reduction in strength after folding. The first objective of the work presented herein is to examine results from PPT fibers tested following the single fold procedure previously implemented for PBO.

Another aspect of this work is to examine the robustness of the single fiber test technique by using a second similar method. One of the main differences from the first method is that the fibers are clamped directly, so tabs are not needed. Instead of optically measuring the fiber diameters directly, they are calculated from linear density measurements determined by a vibroscopic method.[3] The vibroscopic method consists of a system for applying an oscillatory force of known frequency to a filament under tension and a means for detecting the mechanical resonance.[3] One of the potential drawbacks with vibroscopes is that changes in the cross-sectional area along the length of a fiber resulted in changes in the frequency measurement and may induce an error in the calculation of the average cross-sectional area and hence the average fiber diameter.[4]

Finally, it is acknowledged that the testing speed used in the single fiber testing is substantially slower than that seen in a real ballistic event. The purpose for using quasi-static single fiber measurement methods is to develop a fundamental understanding of possible damage accumulation in fibers used for ballistic applications after exposure to folding.

2. EXPERIMENTATION[i]

Two techniques were employed to test the single fibers. The first is the modified single fiber test (m-SFT), a method that has been used extensively in our lab to measure the tensile properties of single fibers[5] and is designated Method 1. The second involves a machine that is used extensively in industry to measure properties of ballistic fibers and is designated Method 2.

2.1 The Single Fold Exposure

The single fold test has been described previously.[2] Briefly, 50 fibers were removed from a single yarn of virgin PPT fibers and then were individually placed across two pieces of poster board that had been taped together. The poster boards were then folded together around a sheet of paper, and bricks (11.8 kg) were placed on the fiber fold region

[i] "Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose."

and left overnight. Subsequently, the 50 folded and 50 non-folded virgin PPT fibers were then tested with each method. This is an admittedly severe test but is one in which potential susceptibility to folding may be exposed.

2.1.1 Preparation of Single Fiber Test Specimens (Method 1)

The procedure for preparing single fiber tensile test specimens using the modified single fiber test has been described in detail elsewhere.[5] The salient points are as follows: individual fibers were temporarily attached to paper templates with double-sided tape. Subsequently, small strips of silver reflective tape were applied to the template at the top and bottom of the gauge area. The reflective tape allows a laser extensometer to measure elongation while the fiber is undergoing tensile testing. Using the extensometer obviates the need to measure the compliance of the testing system as the actual strain in the fiber is measured. The fibers were then permanently bonded to the template by epoxy glue. For the fibers exposed to the fold, the damaged region was positioned to be in the center of the gauge section. A gauge length of 6.0 cm was chosen for comparability to the length of fiber that may be deformed during ballistic impact and because the minimum gauge length needed with the laser extensometer is 5.1 cm.

2.1.2 Fiber Diameter Measurements

Fiber diameter measurements have been described previously.[5] Briefly, fiber diameters at five equally spaced locations along a 6 cm gauge length specimen were measured. A distinct advantage of making the five measurements along the gauge length is that variations can be detected. In previous research we found that if one of the five measurements was less than 11 μm, then there was a drastic reduction in the measured strain-to-failure.[5] However, for the purposes of statistical analysis, the average value of the individual fiber was used, not all of the measurements. It is noted here and in the similar section for Method 2 that there are several sources of uncertainty in diameter measurements. For the optical measurements, uncertainties can come from operator to operator variances, non-circularity of the fiber, problems with edge detection, and limitations of the video camera system. As will be seen in the results section, there were differences between the diameter measurements using the two methods. These differences will be addressed in another paper.

2.1.3 The Single Fiber Test for Method 1

The *m*-SFT samples were tested at a displacement rate of 2 mm/min. Again, we note the difference in the displacement rate for these tests with the strain rate the fibers experience in their ballistic applications.

2.2 Method 2

2.2.1 Preparation of Test Specimens

The fibers tested in the first machine had a 6 cm gauge length, so for comparison purposes, the same gauge length for the fibers tested was used for Method 2 (Textechno Herbert Stein GmbH & Co. KG). Yarns were cut to approximately 10 cm in length and

placed on a mat. Fibers were separated individually, a small weight was attached to one end of the fiber and, using tweezers, placed into grips. The grips were then closed. Figure 1 shows the gripping fixture for Method 2. Although the machine is capable of automatically testing the fibers, we chose to test manually so that we could retrieve the tested fiber for failure analysis. A major difference between the two methods is that in the first method, fibers were bonded to paper tabs and were attached to the grips through the tab ends. With the second method, the fibers were clamped by the grips directly.

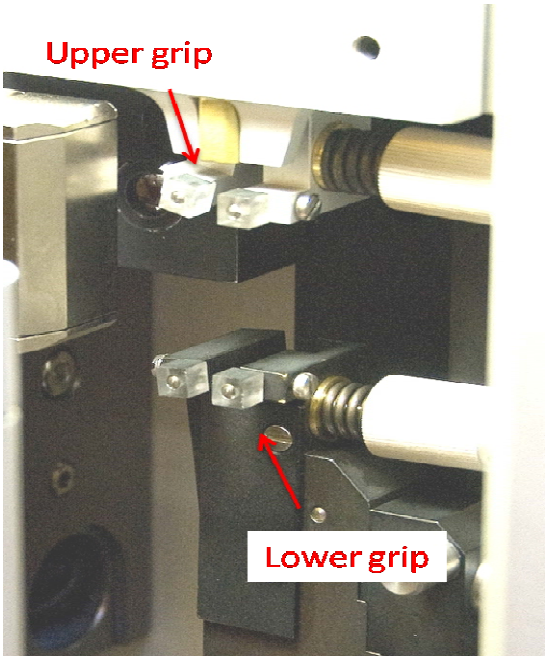

Figure 1. Close-up of the grips used in Method 2.

2.2.2 Fiber Diameter Measurements

Once the fiber is loaded, the linear density measurement is made through a vibroscope. The machine has a built-in measuring head that determines the fineness of single fibers according to ASTM D 1577.[6] The equation that is used assumes a uniform mass distribution and a circular cross section[7]:

$$T_t = F_v * 10^{11}/(4 * f^2 * L^2) \quad [1]$$

where:

T_t = fineness in dtex
F_v = pretensioning force in cN
f = resonance frequency in Hz
L = testing length in mm.

From the linear density, one calculates the average diameter of the specimen[8]:

$$d_f = SQRT[(4*N)/(9*10^5*\pi*\rho_f)] \quad [2]$$

where:

d_f = average diameter in cm
N = fiber denier (g/9 km)
ρ_f = fiber density (g/cm^3)

One difference between the first method and the second method is that Method 1 can detect possible diameter variations along the length of the fiber through multiple measurements along the gauge length, whereas the second method reports only a single diameter value averaged over the entire gauge length. One source of uncertainty in this method comes from the averaging of the diameter along the gauge length. As noted earlier, a uniform cross-sectional area is assumed, so if there are diameter changes along the length of a fiber, then this can cause changes in the frequency measurement. This may induce an error in the calculation of the average cross-sectional area and hence the average fiber diameter.[4]

2.2.3 The Single Fiber Test for Method 2

Once the linear density measurement has been made, the tension is released by the bottom clamp moving back to the start position. The fiber is then pulled in tension at a rate of 2 mm/min until fiber failure. At that point, the fiber sections are recovered and held in place on a mat using double stick tape for further analysis.

2.3 Scanning Electron Microscopy

The SEM images have been obtained on the Cold Field-Emission Scanning Electron Microscope (FE-SEM) Hitachi S4700. To emphasize the topographical features and surface morphology, the images were acquired at 1 KeV with the lower Everhart-Thornley detector.

2.4 Statistical Analysis[9,10]

The t test, and Mann-Whitney and Kolmogorov-Smirnov nonparametric tests were used to check for significant statistical differences among the four responses measured between Method 1 and Method 2 and between virgin and single fold test fibers. The t test assumes underlying Gaussian distributions for the data sets, whereas the non-parametric tests do not.

3. RESULTS

The mean tensile strength and modulus values for the two methods and associated standard deviations of the mean ($s/\sqrt{N}$), which are taken as the estimates of the standard uncertainties, are displayed in Table 1a, the strain and diameter measurements are shown in Table 1b and the force-at-failure is shown in Table 1c. N is equal to the number of fibers tested (47, 48, 42, and 44 for virgin-Method 1, single fold-Method 1, virgin-

Method 2, and single fold-Method 2, respectively). Modulus values were calculated from the linear portions (<1 % strain) of the tensile stress vs. strain curves.

Table 1a: Tensile Strength and Modulus values (PPT Fibers)

	Virgin	Single Fold	Virgin	Single Fold
	Tensile Strength (GPa)	Tensile Strength (GPa)	Modulus (GPa)	Modulus (GPa)
Method 1	2.77 ± 0.04	2.73 ± 0.04	69.60 ± 0.65	68.95 ± 0.61
Method 2	2.91 ± 0.04	2.95 ± 0.02	77.14 ± 0.56	77.69 ± 0.48

Table 1b: Strain and Diameter values (PPT Fibers)

	Virgin	Single Fold	Virgin	Single Fold
	strain %	strain %	Diameter (μm)	Diameter (μm)
Method 1	3.69 ± 0.04	3.66 ± 0.05	12.99 ± 0.07	13.09 ± 0.05
Method 2	3.56 ± 0.05	3.58 ± 0.03	12.76 ± 0.06	12.76 ± 0.05

Table 1c: Force Values (PPT fibers)

	Virgin	Single Fold
	Force (N)	Force (N)
Method 1	0.37 ± 0.01	0.37 ± 0.01
Method 2	0.37 ± 0.01	0.38 ± 0.01

Within each test method, there was no significant statistical difference between the PPT virgin and single fold samples. As was reported earlier, PBO fibers had shown a 10 % drop in tensile strength. There were significant statistical differences when comparing the results between the two methods. The main source of variation appears to be due to the difference in the values of the diameters. Since the tensile strength and modulus values are derived from the diameter measurements, we should expect these values to be statistically different, and they are. As can be seen in Table 1c, however, the force-at-failure values are not statistically different, thus clearly pointing to the difference in diameter measurements as something to examine further.

The result that the strain-to-failure values were different was surprising. Initial statistical analysis resulted in a disagreement between the t-test and the non-parametric tests. For the results from the t-test to be strictly valid, the populations underlying the two data sets must be normal (Gaussian). Figures 2 and 3 show plots wherein the Y axis represents the sorted experimental strains, the X axis the normal predictions for strain. If the data are

normal, the lines will be straight and both the t-test and non-parametric tests would be valid. When a graphical analysis of the virgin fiber strain data was performed, the strain data using Method 2 was not straight (Figure 2), thus making the nonparametric analyses (and statistical graphics) the more appropriate as these methods make no assumption of normality. In this case the results were not significantly statistically different. When a graphical analysis of the single fold strain data was performed, the strain data using Method 1 was not straight (Figure 3), again making the nonparametric analysis the more appropriate approach. In this case the results were significantly statistically different. These differences may be real or may indicate a need to test more samples.

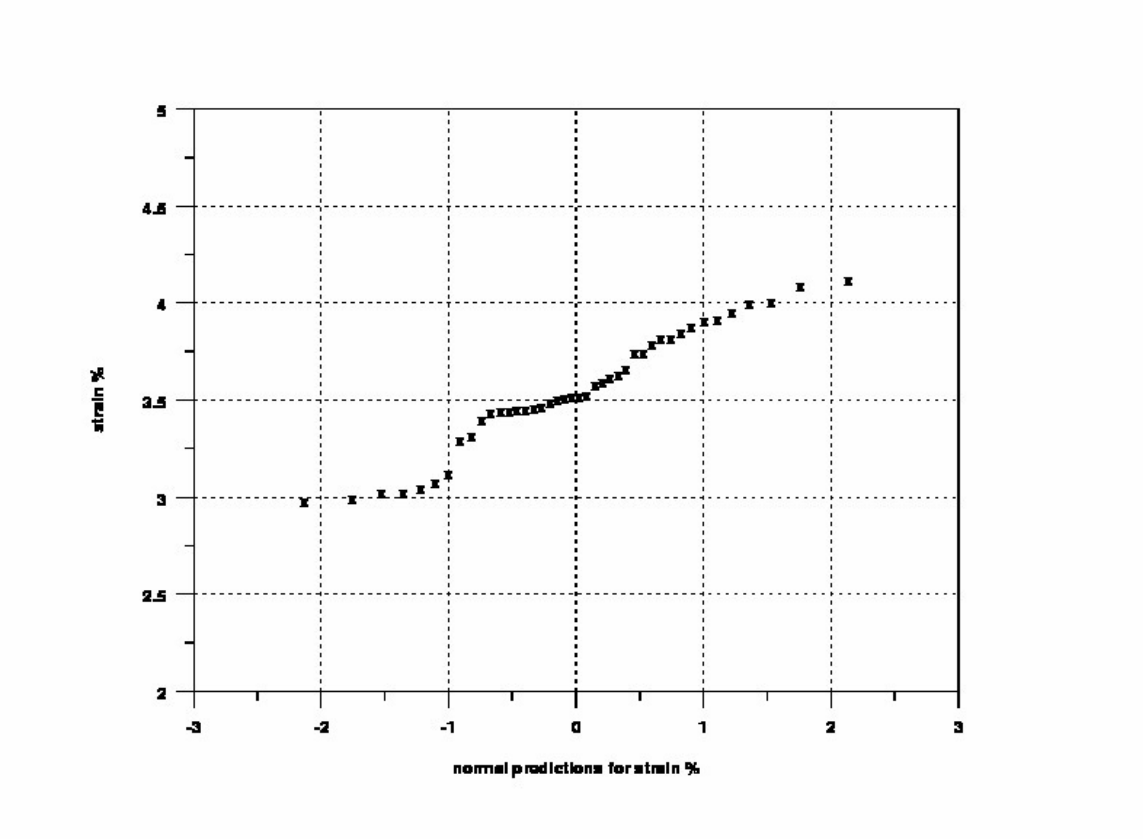

Figure 2: Graphical representation of the non-Gaussian distribution for strain values using Method 2 for the Virgin samples. The Y axis represents the sorted experimental strains, the X axis the normal predictions for strain.

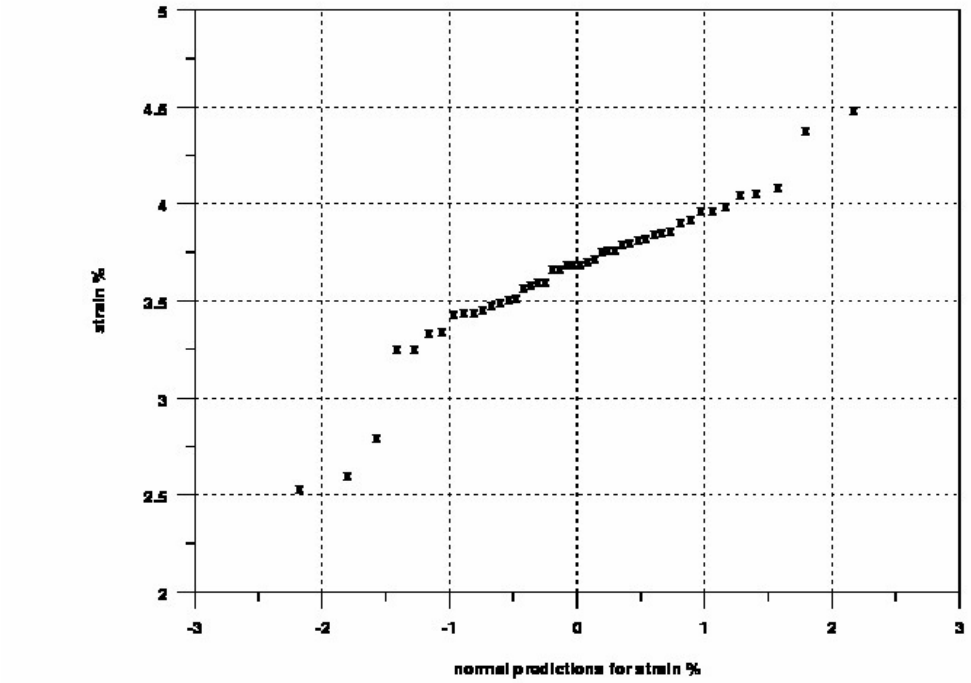

Figure 3: Graphical representation of the non-Gaussian distribution for strain values using Method 1 for the single fold samples. The Y axis represents the sorted experimental strains, the X axis the normal predictions for strain.

Interestingly, when measuring the fiber diameters, a different morphology was noted in the folded region for PPT fibers (Figure 4) than what had been observed previously with PBO fibers (Figure 5). The morphology of the PPT fibers exhibits significant surface damage similar to that observed by Iyer and Viljayan after they ultrasonically agitated PPT fibers.[11] They termed this type of damage kink bands. With the PBO fibers, we observed what has been termed macrobuckling by Iyer and Viljayan. SEM images of the PPT fibers before and after folding (Figure 6) and the before and after fold SEM images of the PBO fibers (Figure 7) give further indication of the damage being localized on the surface of the PPT fibers versus internal in the PBO fibers.

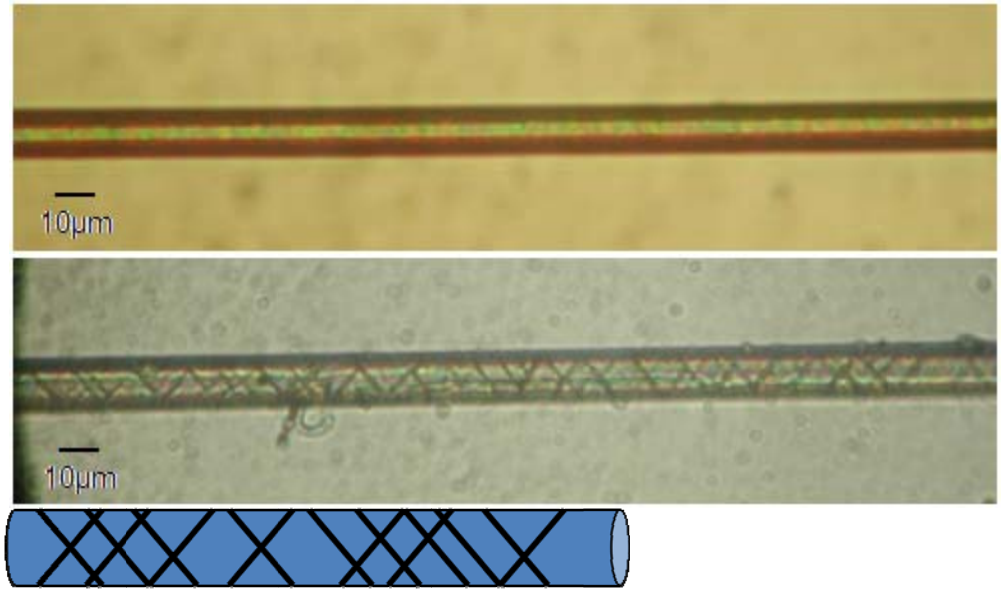

Figure 4: Top fiber: Non-folded virgin PPT fiber. Bottom fiber: Damage induced by single fold of PPT fiber.

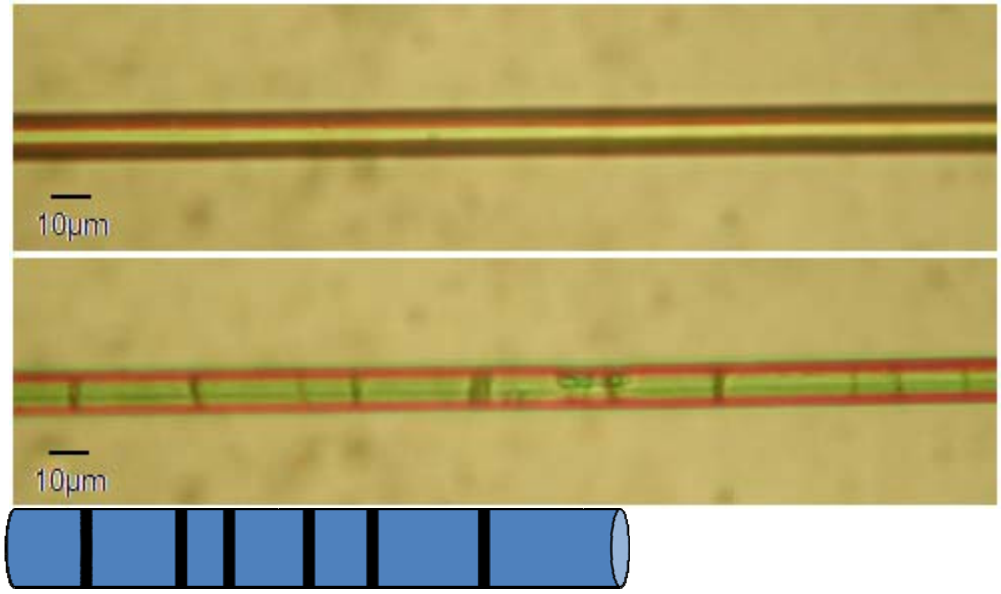

Figure 5: Top fiber: Non-folded virgin PBO fiber. Bottom fiber: Damage induced by single fold of PBO fiber.

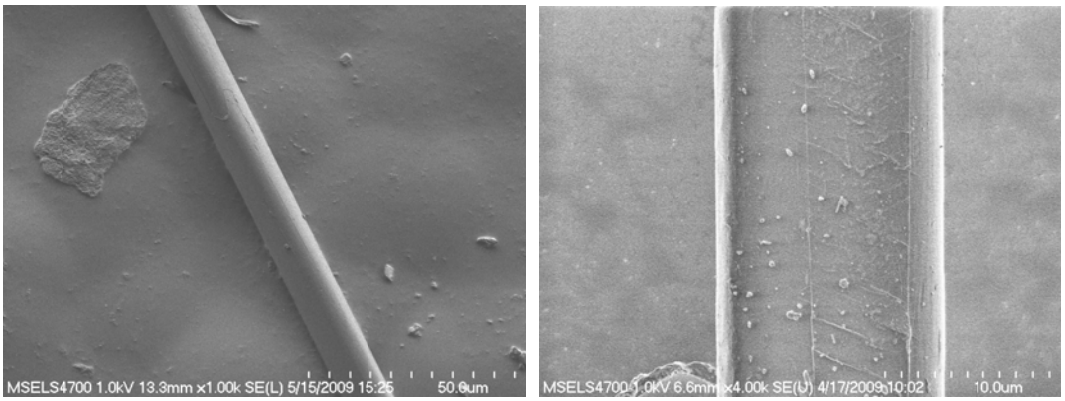

Figure 6: Left: Non-folded virgin PPT fiber. Right: Damage induced by single fold of PPT fiber.

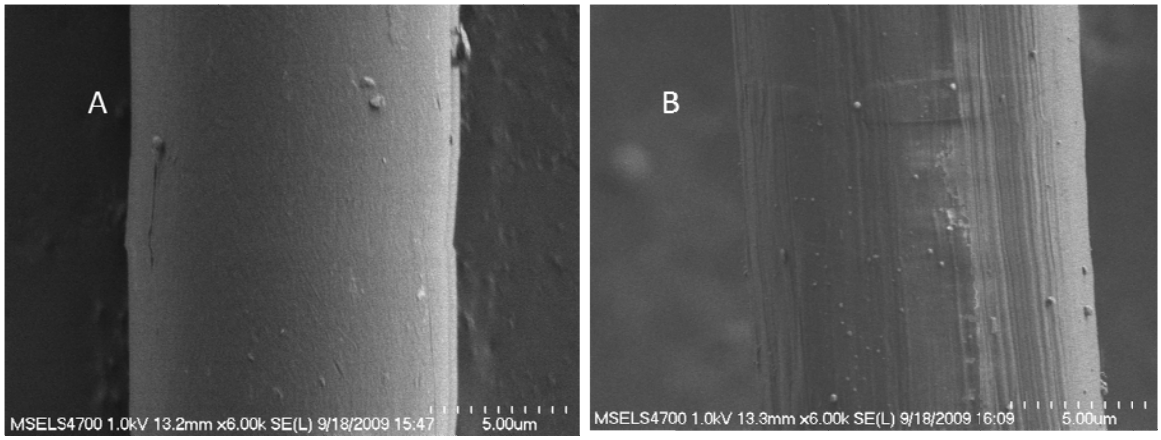

Figure 7: (A) Non-folded virgin PBO fiber. (B) Damage induced by single fold of PBO fiber.

Another point of interest is that, for the most part, failure occurs randomly along the gauge length rather than occurring primarily in the folded region. When the testing of the PBO fibers was repeated, a similar randomness was observed. One could speculate that, although a severe bend is placed on the fiber by the single fold technique, the mass of the brick may be damaging (crushing) the fibers, perhaps more so with the PBO fibers than with the PPT fibers, thus influencing the overall strength of the fibers. Two possible ways to examine the cause of the randomness of the failure in the folded fibers would be 1) localize the location of the brick to just the folded region, and 2) place the brick on unfolded fibers for 24 h and measure the strength.

Finally, as a check on this work, PBO fibers were tested again. These fibers were stored in the laboratory under yellow light and exposed to regular temperature and humidity conditions for around two years after the initial single fold tests were conducted. Originally, the strain-to-failure was measured as 3.5 % ± 0.5 %.[2] When retested, the

strain-to-failure was measured as 2.6 % ± 0.5 %. This reduction could be indicative of hydrolytic degradation of the fibers over time. The acid source for this degradation has recently been identified as polyphosphoric acid chemically bound to the PBO polymer chain.[12]

4. CONCLUSIONS

In previous work in this laboratory, a drop in tensile strength properties in PBO fibers after a single fold on the order of 10 % was found. In the current work, no loss was observed in strength, modulus, or strain within the identical test methods for PPT fibers. Whereas significant internal damage was observed in the folded region of the single fold PBO specimens, only surface wrinkling was observed in the folded region on the single fold PPT specimens. Although a single fold induced what appeared to be a specific damage zone, the observed point of failure was essentially a random process independent of damage zone. Follow-up analysis of the PBO fibers showed the same phenomena. Statistically different responses in strain values were observed when comparing results of the single fold fibers from the two methods. Finally, indications of hydrolytic degradation were observed in the PBO fibers.

5. ACKNOWLEDGEMENT

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