A detailed investigation of the mechanical properties of polybenzoxazole fibers within soft body armor

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Abstract Using the *modified*-single fiber test developed by Holmes and colleagues (J Appl Polym 2008), a detailed analysis of fibers extracted from soft body armor comprised of polybenzoxazole (PBO) fibers was performed. The data indicate that hydrolytic degradation of these ballistic fibers is accompanied by degradation associated with folding (or fatigue-induced degradation) and an undefined degradation mechanism associated with vest use that appears to target the horizontal yarns of the alternating $0^{\circ}/90^{\circ}$ woven layers. These additional failure mechanisms have the potential to create localized regions in the PBO soft body armor which are significantly lower than the homogeneous degradation expected from uniform hydrolysis. Results also indicate that the absence of ballistic penetrations in the initial study conducted at the National Institute of Standards and Technology may be associated with using the properties of the fibers from the back panel of a compromised vest as representative of the properties in the front panel that was penetrated. Analysis of a field return vest showed the front panel to be significantly more degraded than the back panel.

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Introduction

A soft body armor vest that had been in service was penetrated by a matched ballistic threat (bullet) within 6 months of the manufacture date. This led the National Institute of Justice (NIJ) to initiate a research program under the direction of the Office of Law Enforcement Standards at the National Institute of Standards and Technology (NIST) to determine the causes of the failure and to implement standards and testing protocols that would provide greater confidence that performance of body armor would be maintained throughout the projected lifespan. Since it is speculated that residual acid in the polybenzoxazole (PBO) fibers that comprised the vest coupled with moisture results in the rapid decline in the integrity of the vest, a controlled hydrolytic study was conducted in the NIST laboratories to simulate this exposure and quantify the impact of hydrolytic degradation on ballistic performance [1]. The targeted level of degradation was determined by the twisted yarn test and referenced to the fiber properties measured from the back panel of the compromised vest. Although spectroscopic changes were observed in the hydrolytically conditioned (HC) PBO fibers, and the V_{50}^{-1} (the velocity at which 50% of bullets will penetrate the vest) tests indicated a change in ballistic performance of the HC vests, these observed differences did not result in ballistic penetrations at the V_{ref} velocity² (the velocity at which no perforations are allowed to occur during new armor performance testing).

² The specified test velocity for performance testing of new armor. During compliance testing, no perforations are allowed to occur at or below this velocity.



¹ The velocity at which an armor panel of a given design has a 50% probability of stopping the projectile.

Since both empirical [2] and theoretical [3, 4] research have linked the ballistic performance of soft body armor to the mechanical properties of the fibers used in its construction (see Eq. 1), this result was unexpected, because literature results [5] indicate that the benzoxazole ring structure in PBO fibers is susceptible to hydrolytic action under acidic conditions and ultraviolet (UV) radiation. In order to better understand the cause of these results, a follow-up investigation was initiated utilizing the modified-single fiber test (*m*-SFT) developed by Holmes and colleagues [6] to accurately measure the material properties of ballistic fibers. [U*]^{1/3} is the product of the elastic storage capability of the fiber per unit mass and strain wave velocity and is expressed in terms of m/s.

$$\left[\mathbf{U}^*\right]^{1/3} = \left[\frac{\sigma_{\mathbf{f}}^{\mathbf{u}} \varepsilon_{\mathbf{f}}^{\mathbf{u}}}{2\rho} \sqrt{\frac{E_{1\mathbf{f}}}{\rho}}\right]^{1/3},\tag{1}$$

where $\sigma_{\rm f}^{\rm u}$ is the fiber ultimate axial tensile strength, $\varepsilon_{\rm f}^{\rm u}$ is the fiber ultimate tensile strain, ρ is the fiber density, and $E_{\rm 1f}$ is the longitudinal linear elastic fiber modulus.

Using Eq. 1, a test methodology that precisely measures the failure strain, failure stress, and modulus can provide an indication of the expected reduction in ballistic resistance with exposure or wear. The results presented here involve analyses of fibers extracted from a new vest (denoted by N), a HC new vest (denoted by A), the back panel of the compromised (evidence) vest (denoted by E), and a field return vest (denoted by F and B for front and back, respectively).

Important observations from the initial study

Initial inquiries of ballistic fiber manufacturers revealed that the twisted fiber yarn test, utilizing a gauge length of 25 cm, is the preferred method for measuring yarn properties. Due to the fiber grip requirements of 7.6 cm for the universal testing machine, this test requires specimens longer than 33 cm for testing. Soft body armor is generally constructed of multiple layers of textiles. The specific combination of ballistic materials, textile types, layer orientations, and stitch patterns varies between different models of body armor. The body armor model focused on in this study is constructed from multiple layers of woven PBO fabric with the fiber orientation in each layer alternating between $0^{\circ}/90^{\circ}$ and $\pm 45^{\circ}$, with the typical dimensions of the fibers in each layer of the vest as shown in Fig. 1. The yarn length requirements for the twisted fiber yarn test limited testing to the horizontal fiber yarns in the alternating 0°/90° woven layer, where a value of 2.5% strain was obtained by the yarn vest. Implicit in the testing of only these fibers is the assumption that the properties of the horizontal fibers reflect the properties of the vertical

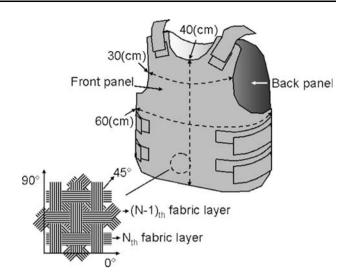


Fig. 1 Typical dimensions of the fibers in a vest

fibers in the same layer and the properties of the fibers in the $\pm 45^{\circ}$ layers after the vest have been worn or artificially conditioned.

Secondly, by referencing the targeted level of degradation to the properties of the evidence vest back panel, the initial study assumed that the degradation behavior of the back and front panels was the same. Although these assumptions are reasonable if hydrolytic action is the only degradation mechanism, they may not be appropriate if other degradation mechanisms, such as folding (fatigue-induced degradation), are also operative. Noting from a previous study [6] that the m-SFT is sensitive enough to detect the impact of a single fold on the mechanical property changes of PBO fibers, care was taken to ensure that the folded region that is often observed near the bottom of a worn vest in the vertical fibers of the $0^{\circ}/90^{\circ}$ woven layer was tested.

For this study we used four types of vests. The New vest has no known history. The HC vest is a new vest that has been conditioned; otherwise its history is as unknown as the New vest. The Evidence vest and the Field Return vest have no known histories, but as they have been used, there is the potential for UV, hydrolytic, and fatigue-induced degradation mechanisms. In all the cases, the PBO fabrics for the vests were enclosed in UV-protected armor panel covers, hence, UV degradation was not considered in this study.

Experimental

Fiber extraction procedure

A PBO fabric stack was removed from an armor panel inside of the body armor. Although the PBO fabric stack is interlocked together by stitching through the unit ply, the stitching thread was removed around the region targeted



for varn extraction in the PBO fabric stack. After removing the stitched thread from the vest, the layer of interest was collected from the PBO fabric stack. Horizontal yarns (denoted by H) were then removed, beginning at the hem of the fabric and continuing toward the target region. Vertical yarns (denoted by V) and ±45° yarns (denoted by 45L) were removed in a similar manner. Each collected yarn was placed on a piece of aluminum foil with the ends held in place by removable tape. The foil was then folded lengthwise over the yarn to keep out light. The wrapped fibers were then placed in the drawer of a darkroom until harvesting. During the time the fibers were being harvested, mounted onto the template, undergoing diameter measurements, or tensile testing, they were exposed to only yellow laboratory lighting that was free of UV frequencies. For fabricating a single fiber tensile test specimen, a single fiber from a yarn was manually separated and mounted onto a paper template having a 6 cm × 1 cm rectangular window.

Preparation of *modified*-SFT specimens

Fifty individual fibers, each approximately 30–40 cm long, were obtained from a harvested yarn and mounted onto a paper tensile testing template. The template, printed on a typical 21.6 cm (8.5 in.) by 27.9 cm (11 in.) printer paper that contained 1 cm major graduations and 1 mm minor graduations, held two rows of five fibers. Therefore, one fiber strand generated two test samples (A and B), each with a 6-cm gauge length. Individual fibers were initially attached temporarily to the paper template, outside the region of the fiber that would undergo diameter measurement and tensile testing, with double-sided tape (3M Stationary Products Division, St. Paul, MN 55119). Care was taken to mount all fibers from each yarn with the same orientation. For the vertical fibers, where folding was observed in the bottom region of the worn vests, care was taken to ensure that the folded region was in the gauge section of each specimen.

Prior to epoxy gluing, small strips (approximately $1.2~\rm cm \times 0.2~\rm cm$) of silver reflective tape (supplied by United Calibration Corp.) were applied to the template at the top and bottom of the gauge sections of each fiber sample. The reflective tape allows elongation measurements to be made by a United Calibration Corp., laser extensometer (Model EXT 62 LOE) while the sample is undergoing tensile testing. The fibers were then permanently bonded to the template by epoxy glue (Hardman

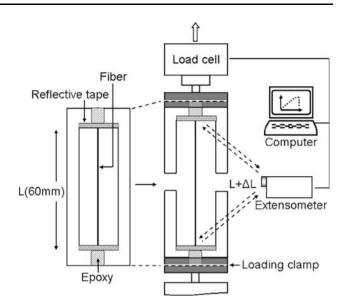


Fig. 2 Schematic of m-SFT specimen

Water-Clear Epoxy, Double/Bubble Green Package #04004). The epoxy glue was allowed to cover up to 0.1 cm thickness of the reflective tape in order to avoid slip between the fiber, paper template, and reflective tape. Extensive testing using E-glass fibers showed that this configuration minimized perturbations, with the failure strain and stress data from the E-glass fiber specimens conforming to the expected normal distributions. A schematic of the specimen used in the *m*-SFT is shown in Fig. 2.

Fiber diameter measurements

Since the post-mortem on the tested fiber recommended in ASTM C1557-03 is not practical for ballistic fibers that undergo fibrillation during tensile failure, the fiber diameter at five equally spaced locations along a 6-cm gauge length specimen was measured. This was accomplished by using an optical micrometer (Excel Technologies Inc., Model VIA-100) attached to a Nikon Optiphot-POL microscope equipped with a video camera (Optronix LX-450 RGB Remote Head microscope camera). The fiber image was viewed on a Sony PVM-1344Q color video monitor. The standard uncertainty in the diameter measurement of fiber diameters is 0.4 μm .

Previous research [6] has shown that minimum fiber diameter measurements less than $10.65 \, \mu m$ along the length of PBO fibers result in premature failure that causes the failure strain and stress distributions to be non-normal. However, initial results indicate that this effect may involve less than 10% of the tested fibers. Therefore, the published results in this report use averages of five measurements to determine the stress–strain response of the



³ Certain commercial materials and equipments are identified in this article to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the NIST, nor does it imply necessarily that the product is the best available for the purpose.

fiber. In between the mounting procedure, the measuring of the fiber diameter, and tensile testing, the fiber samples were stored in the dark, in wooden map cabinets.

Optical strain measurements

Although the compliance method in ASTM C1557-03 has been found to be satisfactory for quantifying the properties of new fibers, the use of non-contact extensometers to detect gauge section elongation directly is often suggested [7, 8] if a more accurate measure of strain is required, since specimen fragility prevents the use of normal strain-sensing devices such as strain gauges or mechanical extensometers. Consistent with this recommendation, a United Calibration Corporation Model EXT-62-LOE laser extensometer was used.

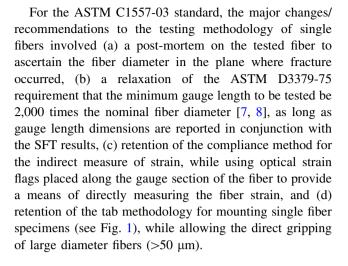
An initial gauge length of 5.1 cm or greater is required for the optimum performance of the laser extensometer. Furthermore, because fiber strength is typically gauge length dependent, a specimen length reflective of the amount of material that may be deformed during ballistic action is probably necessary. Therefore a gauge length of 6.0 cm was chosen. The laser extensometer was calibrated using an Epsilon extensometer calibrator Model 3590C that has 10 cm of length [6]. The standard uncertainty in the strain at 6.1 cm associated with this measurement is 0.0001 cm. The standard uncertainty in the load cell at 100 g is 0.001 g.

The *m*-SFT

The *m*-SFT used in this article is based on the ASTM C1557-03 SFT standard. As stated previously, since the post-mortem on the tested fiber recommended in ASTM C1557-03 is not practical for ballistic fibers that undergo fibrillation during tensile failure, the fiber diameter at five equally spaced locations along a 6-cm gauge length specimen was measured. The ASTM C1557-03 standard supersedes the original and widely used ASTM D3379-75 standard for testing single fibers that was discontinued in 1998 in part because of technical inaccuracies associated with the use of the average of the cross-sectional area of several fibers for the calculation of individual fiber strengths [9].

Table 1 Results for the new yest

Sample name	Tensile strength (GPa)		Strain-to-failure (%)		Modulus (GPa)	
	A	В	A	В	A	В
N2_45L	3.9 ± 0.5	3.8 ± 0.3	3.4 ± 0.6	3.4 ± 0.5	138 ± 21	136 ± 15
N3_VR	3.8 ± 0.6	3.9 ± 0.2	3.3 ± 0.6	3.4 ± 0.3	125 ± 29	129 ± 24
N3_HB	3.6 ± 0.3	3.8 ± 0.4	3.1 ± 0.3	3.4 ± 0.5	134 ± 22	126 ± 18



Results and discussions

The new vest

The m-SFT results from the new vest are shown in Table 1 for a $\pm 45^{\circ}$ yarn taken from the second layer (N2_45L), and vertical (N3_VR) and horizontal (N3_HB) yarns taken from the third layer of the vest. The L, R, and B labels at the end of each yarn designation denotes left (L), right (R), and bottom (B), respectively, to identify the general location on the vest where the yarns were extracted. All results are reported with values for one standard deviation. Since the strain-to-failure measures involve only differences in length, this parameter will be used in the discussions that follow as the primary reference.

In Table 1, the strain-to-failures of the A and B samples for the $\pm 45^{\circ}$ yarn and the vertical yarn from the third layer are statistically indistinguishable at the 95% confidence level with p-values of 0.62 and 0.58, respectively. However, the strain-to-failures of the A and B samples from the N3_HB yarn are statistically distinguishable with a p-value of 0.02. As expected the tensile strength data in Table 1 parallel the strain-to-failure data, whereas the modulus data are shown to be statistically indistinguishable among yarns with a p-value of 0.19.

Although no immediate explanation for the approximate 9% decrease in the strain-to-failure data of the N3_HB A specimens relative to the B specimens can be advanced at



this time, localized damage occurring in the fibers during the extraction process, the presence of a vertical fold in the new vest, and localized damage occurring during the manufacturing process are factors that must be considered as viable agents for the observed damage. Interestingly, five of the six sets of fibers from the new vest give strain-to-failure results consistent with the strain-to-failure results obtained from the yarn test on virgin fibers, where the strain-to-failure has a value of 3.5%. The small difference between the numbers suggests that minimal damage occurred to the fiber of the new vest during the manufacturing process. It is also worth noting that previous research [6] has shown that these fibers are sensitive to damage induced by folding, with a single sharp fold potentially resulting in a 15% decrease in failure strain.

Back panel of evidence vest

In contrast to the 3–12% reduction in strain-to-failure observed in the new vest (Table 1) relative to virgin PBO fibers (strain-to-failure of 3.5%), data from the second and third layers of the back panel of the evidence vest indicate reductions in the strain-to-failure of the fibers of 17-29% (see Table 2) relative to virgin PBO fibers. As generally observed in the new vest, the tensile strength, strain-to-failure, and modulus values from the A and B samples were statistically indistinguishable. The strain-to-failure of the horizontal fibers (E3 HB), by the m-SFT, was found to be 2.5%. This value is consistent with the value obtained on these fibers in the original study obtained by the Yarn test (see section "Important observations from the initial study"). An aggregate analysis of all the strain-to-failure values indicates that the horizontal fibers (E3 HB), which were used in the initial study to target the desired degradation level of 2.5%, are statistically different from the E3_VR and E2 45L fibers, with a p-value of 2.4×10^{-4} . Therefore, the m-SFT results for the fibers extracted from the vertical (E3_VR) and ±45° (E2_45L) yarns, which could not be

tested in the original study by the yarn test, indicate that they are 8–16% stronger than the horizontal fibers.

These results, therefore, suggest a more complex degradation process than the hydrolysis only mechanism assumed in the original investigation. Although differences arising from the manufacturing process cannot be discounted, further support for the more complex degradation process is provided by an aggregate analysis of the E2_45 L and E3_VR fibers. Even though these fibers are in different layers and have different orientations, their strain-to-failure values are statistically indistinguishable with a *p*-value of 0.21, suggesting a different stress history for the E3_HB fibers.

An aggregate analysis of the tensile strength, which usually tracks the strain-to-failure results, indicates that the three fiber types are statistically different with a p-value of 2×10^{-8} . Subset analyses of only two fiber types support this result. Although an aggregate analysis of the modulus values indicate that they are indistinguishable with a p-value of 0.19, the lower modulus value of the E3_VR fibers may be contributing to the results observed in the tensile strength analysis.

HC vest

The *m*-SFT data for a HC new vest prepared in the initial investigation are given in Table 3. Relative to virgin PBO fibers, these results indicate a strain-to-failure reduction of 23–46%, with the A and B samples from the A3_HB yarn yielding strain values of 2.7 ± 0.3 and $2.4 \pm 0.4\%$, respectively. The difference in the A and B fiber samples from the A3_HB yarn was found to be statistically significant (*p*-value = 4×10^{-3}). Recalling a similar result from the analyses of the new vest (see data for N3_HB in Table 1), the observed difference may be similarly attributed to localized damage occurring in the fibers during the extraction process, the presence of a vertical fold in the new vest, and/or localized damage occurring during the

Table 2 Results for the back panel of the evidence vest

Sample name	Tensile strength (GPa)		Strain-to-failure (%)		Modulus (GPa)	
	A	В	A	В	A	В
E2_45L	3.5 ± 0.4	3.2 ± 0.4	2.9 ± 0.5	2.7 ± 0.3	131 ± 27	131 ± 22
E3_VR	3.1 ± 0.4	3.2 ± 0.4	2.7 ± 0.3	2.9 ± 0.4	122 ± 20	129 ± 22
E3_HB	2.9 ± 0.4	2.9 ± 0.3	2.5 ± 0.4	2.5 ± 0.3	136 ± 19	135 ± 21

Table 3 Results for the HC vest

Sample name	Tensile strength (GPa)		Strain-to-failure (%)		Modulus (GPa)	
	A	В	A	В	A	В
A2_45L	2.5 ± 0.2	2.4 ± 0.2	2.0 ± 0.3	1.9 ± 0.2	138 ± 23	138 ± 26
A3_VR	3.3 ± 0.3	3.4 ± 0.3	2.6 ± 0.2	2.7 ± 0.3	133 ± 34	143 ± 29
A3_HB	2.9 ± 0.3	2.7 ± 0.4	2.7 ± 0.3	2.4 ± 0.4	117 ± 23	119 ± 26



manufacturing process. In addition to these factors, the much lower strain-to-failure of the A and B samples from the A2_45L fibers may be caused by non-uniform conditioning between the initial layers of the ballistic pack. In spite of these differences, these results indicate that the targeted degradation level in the vest was almost reached by the hydrolytic conditioning, with all fibers being generally weaker than those observed from the back panel of the evidence vest.

Field return vest

Since no ballistic penetrations were observed at the $V_{\rm ref}$ velocity for the HC vests, the results of the initial study suggest that Eq. 1 may be invalid for the hydrolytic degradation mechanism, or that Eq. 1 is valid, but the safety margin of the body armor is decreasing, but still sufficient to stop the projectile. In order to answer this question, a comparative analysis was performed on the vertical and horizontal fibers extracted from the front and back panels of a field return vest. The results given in Table 4, in addition to showing the fiber strain-to-failure properties to be extensively degraded, 25–54% relative to virgin PBO fibers, indicate that the fiber properties in the front panel are generally lower than those of the comparable fiber in the back panel.

If one assumes uniform hydrolytic degradation in each panel and takes the largest strain-to-failure value from each panel as quantifying this degradation mechanism, then hydrolytic action accounts for 25-43% of the observed reduction relative to virgin PBO fibers, with the most extensive degradation occurring in the front panel. The statistically significant 6% reduction in the strain-to-failure between the A and B samples from the FVR yarn (p-value = 1.7×10^{-3}) appears to be due to folding, since care was taken to ensure that the folding regions observed in the vest span the gauge section of the single fiber B samples. A similar interpretation can be invoked for the 9% reduction observed between the A and B samples for the BVR yarn $(p\text{-value} = 7.5 \times 10^{-3})$ results if one imagines that the lower part of the back panel (the B-sample) remains essentially straight when pressed against the car seat and flexing of the fibers in the A sample occur as the vest rises up the wearer's back during use. This latter statement refers to the observation that when an officer gets in and out of a car, the bottom portion of the back panel may get pinned to the seat cushion causing the back panel to rise up on the officer's back, resulting in the fibers at the top portion of the panel to be flexed or bent. Although the difference was not statistically significant (*p*-value = 0.16), it should be noted that a similar trend was observed in the E3_VR fibers from the back panel of the evidence vest.

Surprisingly, the horizontal fiber properties are generally lower than those of the vertical fibers, with additional reductions of 11 and 22% being observed relative to the maximum fiber strain observed in the front and back panels, respectively. Since this result was also observed for the back panel of the evidence vest and not in the new or HC new vests, this additional decrease in properties appears to be associated with use of the vest.

Conclusions

The strain-to-failure of the PBO fibers extracted from the second and third layers of the ballistic pack of the new vest showed a 3–12% reduction in strain-to-failure when referenced to the 3.5% failure of virgin PBO. This reduction may be associated with the manufacturing process. Analysis of the back panel of the evidence vest exhibited a 17–29% reduction in failure strain from the same layers. Although it was implicitly assumed in the initial study that the horizontal fibers tested from the evidence vest reflected the properties of the remaining fibers, a detailed analysis of the vertical fibers from the third layer and the $\pm 45^{\circ}$ fibers from the second layer showed that they were 8–16% stronger than the horizontal fibers, thereby suggesting a more complicated degradation mechanism than associated with just hydrolytic action.

Although differences in the strain-to-failure of fibers taken from the second and third layers of a HC new vest indicate some non-uniform hydrolysis on the topmost layers, the level of degradation in these vests was found to be close to or exceeding the targeted failure strain. These results suggest that Eq. 1 may be invalid for degradation caused by hydrolytic action, or Eq. 1 is valid but the safety margin of the body armor is decreasing, but still sufficient to stop the projectile, or that the assumption that the back

Table 4 Results for the field return vest

Sample name	Tensile strength (GPa)		Strain-to-failure (%)		Modulus (GPa)	
	A	В	A	В	A	В
FVR	2.5 ± 0.4	2.2 ± 0.2	2.0 ± 0.3	1.8 ± 0.3	134 ± 25	130 ± 24
FHB	2.3 ± 0.3	2.1 ± 0.3	1.6 ± 0.2	1.7 ± 0.4	145 ± 18	142 ± 18
BVR	3.0 ± 0.3	3.4 ± 0.4	2.3 ± 0.3	2.6 ± 0.4	149 ± 18	155 ± 19
ВНВ	2.5 ± 0.4	2.6 ± 0.4	1.8 ± 0.3	1.9 ± 0.3	142 ± 28	144 ± 28



panel of the evidence vest reflects the properties of the front panel of the vest is incorrect.

Additional analyses on a field returned vest showed that the properties of the front panel are significantly lower than the back panel, thereby indicating that material properties of the fibers in the front panel of the compromised vest are much lower than those observed in the back panel of the evidence vest. Additional analyses suggest that the hydrolytic degradation mechanism is accompanied by a folding-induced degradation mechanism and an undefined degradation mechanism associated with vest wear in the horizontal fibers of the alternating 0°/90° woven layers. These degradation mechanisms combine to significantly reduce the mechanical properties. These additional degradation mechanisms have the potential of producing localized regions of weakness in the ballistic pack that are weaker than the homogeneous degradation expected to be caused by uniform hydrolysis.

Finally, it should be noted that fiber strength variability with coefficients of variation of 15–20% have been observed by others [2], with the fiber strength showing a strong gauge length dependence. Noting these facts, the gauge length of the fibers tested in this report was kept constant at 6 cm and at least 40 specimens were tested for each fiber set to establish the variance in the measured values. It is also encouraging that test results from the *m*-SFT and yarn test on comparable samples, where localized folding is not a factor, yield comparable results. These consistent results further indicate the applicability of Eq. 1 for quantifying changes in ballistic performance that occur in degraded fibers.

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