

FATIGUE PERFORMANCE OF THERMOPLASTIC TUFF COMPOSITES

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ABSTRACT

Tailorable universal Feedstock for Forming (TuFF) is a composite material consisting of highly aligned short fibers able to achieve high fiber volume fraction (up to 60%). The high degree of alignment, high fiber volume fraction (FVF) and high level of fiber-matrix adhesion provides quasi-static mechanical properties comparable to continuous fiber composites using a fiber aspect ratio of 600. However, in structural applications, it is important to consider the fatigue behavior of TuFF, particularly because the complex load transfer paths among the discontinuous aligned fibers may affect the properties. In this work fatigue performance of unidirectional carbon fiber/polyetherimide TuFF composites (3mm IM7, 57% FVF) are tested in tension ($R = 0.1$) to generate the S-N curve. Digital Image Correlation (DIC) is used to measure effects of cyclic loading on modulus. Failure surfaces are examined using microscopy to determine failure modes. It is demonstrated that TuFF material has comparable fatigue performance to other continuous carbon fiber structural composite materials reported in literature.

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1. INTRODUCTION

The TuFF process produces highly aligned short fiber preforms and when consolidated with a matrix material leads to properties comparable to continuous fiber counterparts at high fiber volume fraction (57%) [1,2]. Using the IM7/8552 properties found in the National Institute for Aviation Research (NIAR) database as a reference material, 3mm IM7 carbon fiber/polyetherimide TuFF composite was shown to have comparable quasi-static mechanical properties (i.e., tension, compression, bearing, OHC and short bean shear) as shown in Figure 1. The high level of mechanical properties arises from the high degree of fiber alignment in the TuFF process where 95% of the fiber orientation distribution fall within $\pm 5^\circ$ of the loading direction [2]. The TuFF orientation distribution and idealized microstructure (fiber packing, aspect ratio, and volume fraction) are shown in Figure 1. In comparison, the fiber misalignment seen typically in a continuous fiber unidirectional material system, where prepreg tapes (both thermoset and thermoplastic) have 95% of the fibers within $\pm 3^\circ$ [3,4]. A key attribute of the aligned TuFF material/microstructure is the high degree of in-plane extension achievable during processing that enables single step stretch-forming of complex geometries at higher throughput of parts.

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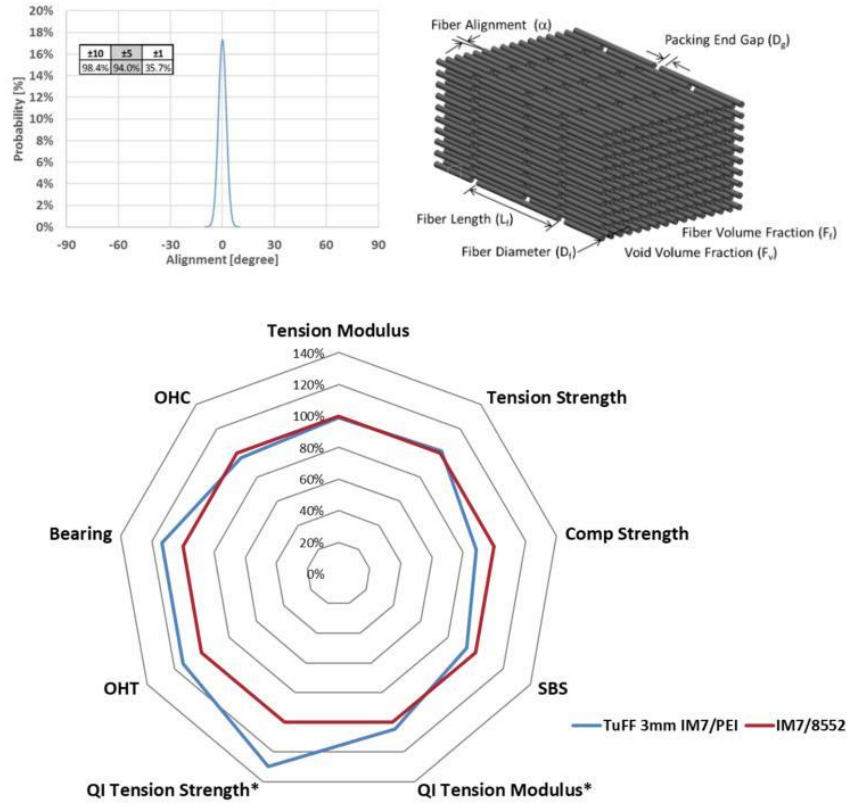


Figure 1. TuFF fiber alignment probability, idealized microstructure, and TuFF (IM7/PEI) comparison with IM7/8552 (NIAR database) [2].

Composite structural components will see repeated load-unload cycles throughout its lifecycle. Therefore, it is critical to assess the fatigue performance of TuFF materials for the first time to identify material design limits and estimate service lifetimes. For metal fatigue failure mechanisms, cyclic crack growth occurs in stages leading to final failure [5]. In composites there are multiple interacting failure mechanisms present, often initiated at the fiber length-scale. Damage mechanisms within a layer may include fiber debonding initiating at fiber ends, fiber breakage, matrix/resin yielding/plasticity and cracking [6,7]. This damage can lead to microcracking and delamination in multi-layer laminates that lead to stiffness loss and strength reduction during cyclic loading.

Fatigue data from the literature is shown in Figure 2 for different continuous carbon fiber thermoplastic and thermoset composites subjected to cyclic loading [8–10]. These studies highlight a reduction in tensile strength with increasing fatigue cycles. To assess the performance of TuFF, fatigue performance of unidirectional composites is used as the baseline comparison. Tensile loading of off-axis layers has been shown to reduce both the static and fatigue strength due to the multi-axial stress state (i.e., in-plane shear, transverse tension and tensile stress in the fiber direction). A study conducted by Jen and Lee tested a range of different fiber orientations in AS4/PEEK and showed a 15° fiber orientation resulted in lower fatigue performance than the 0° [8] as shown in Figure 2. Fiber breaks and fiber debonding have been shown to reduce overall stiffness in unidirectional composites subjected to fatigue loading[7]. However, it is difficult to detect the onset and growth of cracks at the filament level within the composite before

catastrophic failure occurs. Furthermore, with a short fiber thermoplastic material system such as TuFF, it is important to understand the critical micro-scale mechanisms that dictate macroscopic fatigue performance.

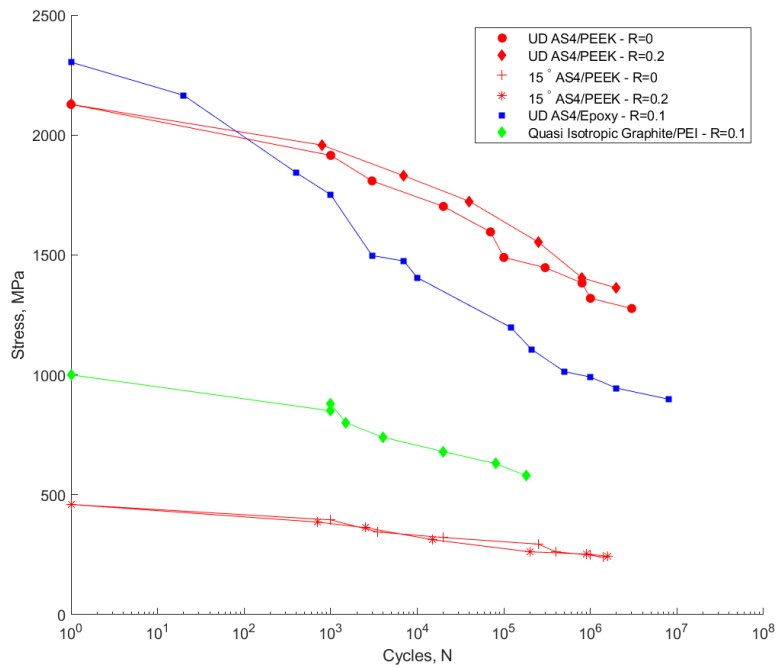


Figure 2. Comparison of thermoplastic and thermoset continuous fiber composites tested in fatigue [8–10].

In this work, the effects of tensile fatigue on strength and modulus of 3mm IM7/polyetherimide TuFF composites in the fiber direction are investigated.

2. EXPERIMENTATION

2.1 Fatigue Testing

Fatigue testing was conducted in a Instron 1331 hydraulic test frame equipped with a 10kN load cell and digital hydraulic controls. A tension-tension cyclic frequency of 3 Hz was used with an R=0.1. The test frame was used in load-control mode. Specimens were tested at 0.75, 0.5, 0.25 of the quasi-static ultimate tensile strength (UTS) defined as the average minus three standard deviations (quasi-static properties are shown in Table 1). VIC-3D digital image correlation (DIC) software was used for accurate strain measurement during the periodic modulus checks. Modulus checks were taken periodically to detect fatigue damage that would degrade specimen stiffness through cyclic loading. Strain-gauges were used to validate the DIC method and is described in detail below.

2.2 Preparing Test Specimens

Specimens were composed of Hexcel 3mm IM7/polyetherimide (PEI - Ultem1000) and were prepared in an autoclave to achieve low void content and uniform panel materials. Panels were C-scanned to ensure there were no material defects present in the region specimens would

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be harvested, and an example is shown in Figure 3a. Panels were tabbed using 3mm thick G-10 composite and attached using LOCTITE EA 9309NA AERO adhesive. A diamond wet saw was used to cut samples from the center portion of the panel. 26mm (1 inch) was trimmed off the edges to avoid possible material variations. A schematic cut diagram is shown in Figure 3b. Test coupons had a nominal geometry of 170 x 13 x 0.2 mm, and a nominal fiber volume fraction of 57%. One side of the specimen was speckle coated for DIC and the other was fixed with a strain gage (Micro-Measurement). Specimens are shown in Figure 4.

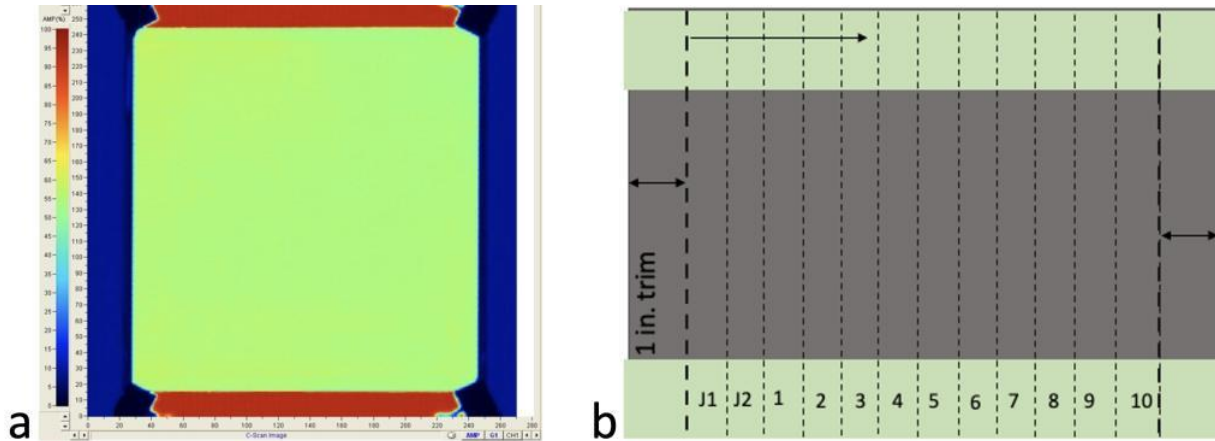


Figure 3. (a) C-Scan of TuFF panel. (b) Cutting layout of panel, after tabbing material is affixed.



Figure 4. TuFF test specimens, DIC speckle coated (top), back side (middle), strain gauged back (bottom).

2.3 Fatigue Test Validation

A series of validation experiments were conducted to confirm proper specimen loading conditions. Back-to-back strain gauges were used to identify possible asymmetry and if bending was present during the testing and modulus measurements. Modulus variation between the back-to-back strain gauges was on average ~0.7%, indicating symmetric loading conditions and specimen bending was negligible. Next, DIC modulus and strain gauge moduli were compared and shown to be within 1% error between 0 and 1000 cycles. A trial with several different DIC sampling areas in comparison to the size of the strain gauge measurement is shown in Figure 5.

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There was the concern of strain gauges debonding with cyclic loading, as evidenced by a significant decrease in strain gauge modulus in comparison to the DIC calculated value at and above 10k cycles. This present work utilized the DIC technique to monitor specimen strain because it offered a larger sampling area compared to strain-gauges and is able to capture possible strain localization due to fatigue damage. A handheld FLIR IR camera was used to confirm uniform specimen temperature and no significant temperatures above ambient conditions. The temperature change was minimal, $\sim -0.1^{\circ}\text{C}$ (-0.2°F) between 100 and 100,000 cycles, indicating a 3Hz frequency did not impart specimen heating, as shown in Figure 6.

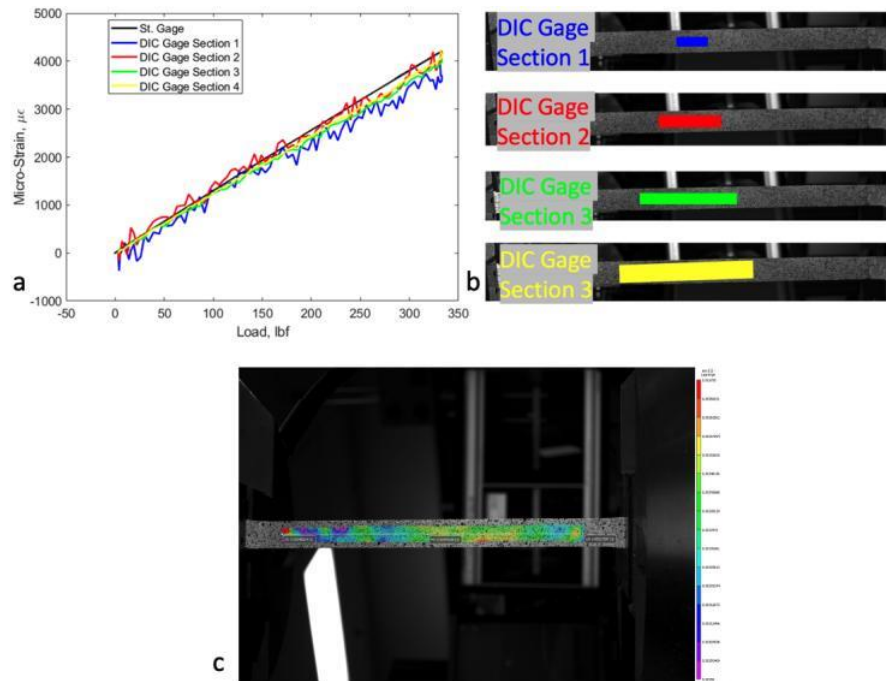


Figure 5. (a) Comparison of DIC strains to a strain gauge mounted on the back of the test specimen. (b) DIC sampling areas. (c) Total DIC field of view on the TuFF specimen.

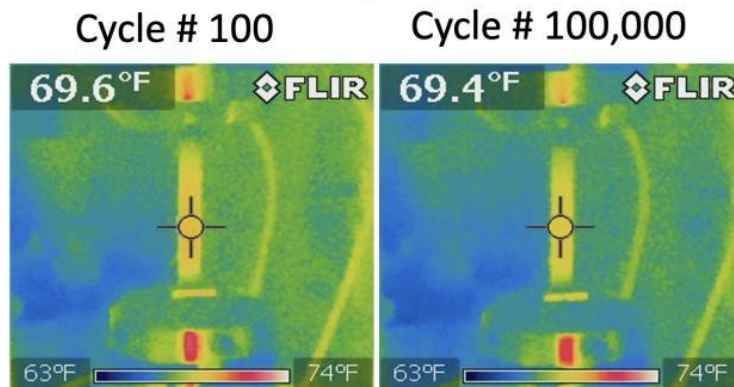


Figure 6. Thermal image of test specimen between 100 and 100,000 cycles (3Hz). No signs of sample heating.

3. RESULTS AND DISCUSSION

3.1 Quasi-Static Testing

High quality TuFF panels were tested in tension following the ASTM standard D3039 procedure. These panels had similar strength and modulus values as previously reported[2], a summary of modulus and strength are shown in Table 1.

Table 1. Static properties of 3mm IM7/polyetherimide TuFF tested in tension (FVF = 57%)

E_{11}	Strength	Strength Std	CV	UTS _{Mean} -3SD
GPa	MPa	MPa	%	MPa
161	2578	39.3	1.5	2460

3.2 Fatigue Testing

Fatigue testing results for TuFF are shown in Figure 7. The S/N curve follows a typical linear response on a semi-log plot. To compare the reduction in strength with increasing cycle count, the TuFF fatigue properties are compared to other continuous carbon fiber composites in Figure 8 (left). The results, normalized by the static strength, are presented in Figure 8 (right). The normalized S/N curves indicates that short fiber 3mm IM7/PEI TuFF has comparable performance to the other carbon fiber thermoplastic and thermoset matrix composites.

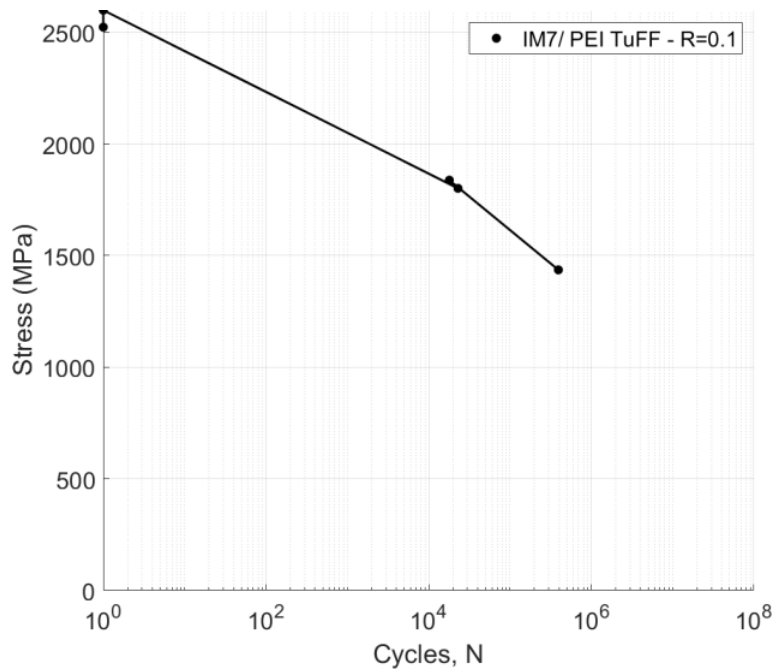


Figure 7. S/N curve for TuFF material (R = 0.1).

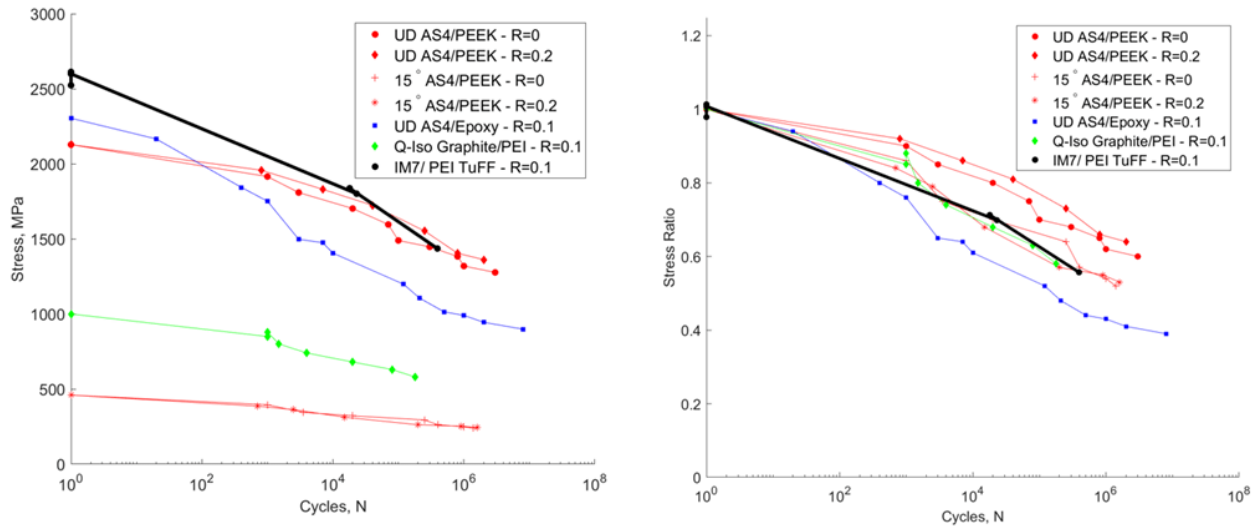


Figure 8. S/N curve (left) and normalized S/N curve (right) with TuFF (black) and comparison to literature (red[8], blue[9], green[10]).

Table 2. Slopes of strength reduction from semi-log plots.

	TuFF	AS4/PEEK-0°	AS4/PEEK-15°	AS4/Epoxy	Q-Iso/PEI
Slope Normalized Stress	-0.032	-0.024	-0.034	-0.043	-0.035

The normalized S/N curves can be compared quantitatively by comparing the slopes of the curves in Table 2. All of the carbon fiber composites have relatively low sensitivity to fatigue loading compared to other materials such as glass fiber composites and most metals. The short fiber TuFF material exhibits slightly more fatigue sensitivity than the continuous fiber AS4/PEEK but is superior in fatigue performance to the continuous fiber AS4/Epoxy. TuFF has comparable fatigue performance to the quasi-isotropic continuous carbon fiber PEI composite. One must also note there are resin differences, amorphous PEI versus semi-crystalline PEEK. Semi-crystalline polymers inherently have better mechanical properties due to the microstructure interlocking of spherulites. Interface strength is increased in a composite by the radial clamping on fibers arises from the shrinkage mismatch (CTE and crystallization) between the fiber and matrix[11][12]. These differences can contribute to differences in fatigue performance between TuFF 3mm IM7 amorphous matrix PEI composite and the continuous fiber AS4 semi-crystalline PEEK composite.

Table 2 also shows the sensitivity of fatigue S/N curves to off-axis fiber alignment in the case of the continuous fiber AS4/PEEK material where the degradation slope increases from -0.024 to -0.034 when nearly 100% of all fibers are off-axis by 15°. In-plane shear stress in off-axis loading is typically maximum in the 10-15° range and is known to significantly reduce quasi-static tensile strength by a change in failure mode (fiber dominated to matrix shear). At the fiber length-scale this can initiate matrix cracks and fiber matrix debonding in both static and fatigue loading. In contrast, TuFF has only a small percentage of the fibers within the orientation distribution (see Figure 1) that are off-axis and at a much lower angle (i.e., 95% of the fiber

distribution are within $\pm 5^\circ$ of the loading direction). Consequently, the reduction in static strength is negligible and the sensitivity to fatigue loading associated with the off-axis fibers is reduced.

However, TuFF as a short fiber composite has stress concentrations at the end of each fiber that can induce fatigue damage (matrix cracking or interface debonding), which can accumulate during cyclic loading, reducing the modulus and tensile strength. For the TuFF composite, modulus reduction with cycle count can be seen in Figure 9 for three stress levels ranging from 25% to 75% of ultimate strength ($R = 0.1$). Even at the highest fatigue stress level modulus reduction is less than 7% at 1 million cycles.

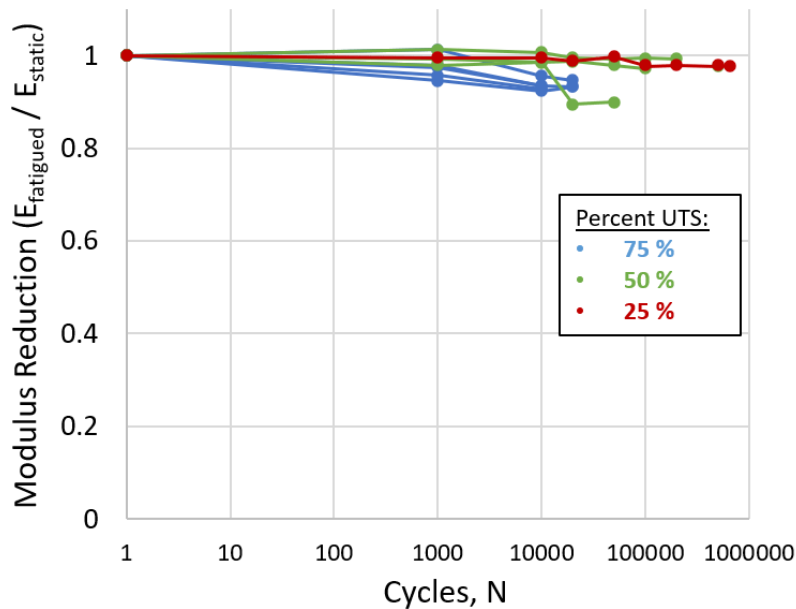


Figure 9. Modulus reduction versus cycle count at different applied peak loads.

Failure mode of TuFF coupons in quasi-static loading is an explosive failure very similar to the failure seen in continuous fiber coupons. An example of the failure surface can be seen in Figure 10. However, in the fatigue loading coupons there are large splits and cracks running along the fiber axial direction. This failure does not appear as explosive as in the quasi-static case as failure does occur at a lower stress level. From the remaining fragments, it is impossible to isolate the sequence of failure mechanisms at the microscale. From the microscopy, as shown in Figure 11, axial splintering and fiber pullout mechanisms can be identified, but it is hard to identify further details in a systematic manner. The large longitudinal splitting mechanism suggests that progressive debonding along the fiber/interface may occur prior to catastrophic failure. To isolate, understand, and validate these failure mechanisms and interactions, model micro-composites are being developed to visualize and confirm damage mechanisms under fatigue loading. This future work should provide better understanding on the role of interface properties, matrix properties, and fiber-fiber stress concentrations on fatigue performance of TuFF composites.

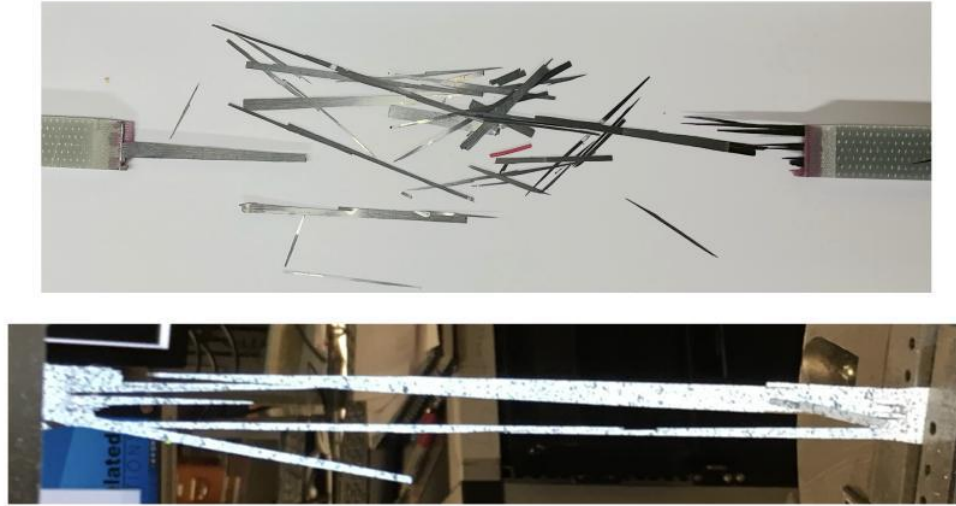


Figure 10. Examples of TuFF coupon failure, (top) quasi-static, and (bottom) fatigue loading.

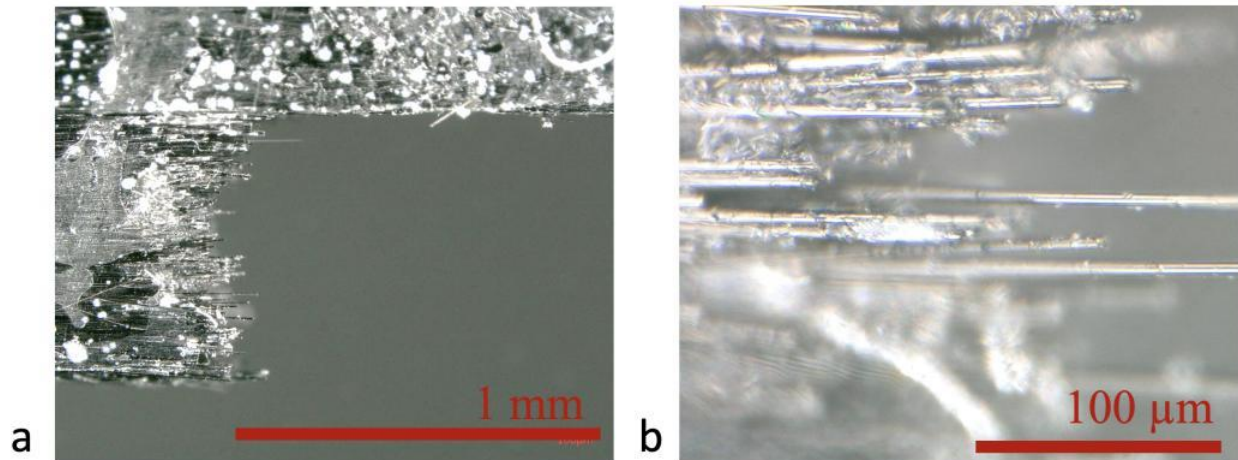


Figure 11. Failure surface of a TuFF coupon tested in fatigue.

4. CONCLUSIONS AND FUTURE WORK

Thermoplastic TuFF (IM7/PEI) comprised of highly aligned 3mm short fibers has comparable tensile fatigue performance (S/N curves) to other continuous carbon fiber reinforced thermoplastic composites and has superior fatigue performance compared to continuous carbon fiber/epoxy composites reported in the literature. Effects of fatigue at 25-75% of ultimate tensile strength on modulus reduction were measured to be in the 3-7% range out to 1 million cycles. Quasi-static failure modes observed consisted of explosive axial splitting similar to the failure mode observed for continuous carbon fiber. Fatigue loading initiated a more progressive axial splitting failure mode in the samples. Future work should focus on more in-depth failure analysis to determine lower length scale failure modes during fatigue loading. Micromechanical studies, using FE and experimental techniques, are underway to isolate and gain an understanding of these localized failure mechanisms. These mechanisms include interface characterization, fiber-debond growth with applied cyclic loading, and fiber-fiber interactions. Raman spectroscopy can

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be utilized to monitor in situ fiber strains and debond regions when compatible fiber/matrix constituents are used [11]. It is important to monitor the fiber-level loading that results from processing induced thermal stress, in addition to any applied mechanical loading. A FE model is being developed tying in all these factors to understand and ultimately control these mechanisms to improve the TuFF material performance.

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