MICROSTRUCTURAL EVALUATION OF ALIGNED, SHORT FIBER *TUFF* MATERIAL

Dirk Heider[1,2], John Tierney[1], Mohamed A. Henchir[1], Verena Gargitter[1], Shridhar Yarlagadda[1,2], John W. Gillespie, Jr.[1,3], Jessica Sun[1,4], Jennifer M. Sietins, [4] Dan Knorr[4]

1) Center for Composite Materials, Newark, DE 19716
2) Electrical &Computer Engineering Department, Newark, DE 19716
3) Department of Materials Science and Engineering, Department of Civil and Environmental Engineering, Department of Mechanical Engineering, Newark, DE 19716
4) Army Research Laboratory, Aberdeen Proving Ground, MD 21005

ABSTRACT

The Tailorable Universal Feedstock for Forming (*TuFF*) material is comprised of highly aligned discontinuous fibers that achieves a high level of mechanical properties with large in-plane extensibility for forming of complex geometries. The paper characterizes the *TuFF* microstructure in terms of areal weight and fiber volume fraction and statistical distributions of fiber length, fiber-fiber spacing and fiber alignment using microscopy and X-ray Computed-Tomography with custom developed algorithms for data reduction. The *TuFF* program has demonstrated full property translation of stiffness and strength compared to continuous prepreg (60% fiber volume fraction) when 95% of all fibers are aligned within 5° and have a minimum fiber aspect ratio (length over diameter) of 600.

1. INTRODUCTION

Carbon fiber reinforced composites are used extensively in a wide-range of primary structural aerospace applications. The material allows weight efficient designs and multi-functionally such as corrosion resistance and lightning strike protection. Microstructure based modeling and analysis have been used to study the mechanical behavior and other functional properties of continuous fiber reinforced composites. The ideal microstructure assumes that the fibers are straight parallel cylinders with uniform spacing at high fiber volume fraction (nominally 60%). The fiber spacing and fiber volume fraction information can be obtained from the cross section of a composite specimen and represents the bulk microstructure. A detailed study of the continuous fiber microstructure was performed by Gutowski et al. [1], which relaxed the assumption of straight fibers, and introduced a parameter that quantified multiple contact points along its length with the neighboring fibers due to the fiber waviness that follows a sinusoidal path (typical waviness angles were reported to be in the range of 0.5°). In contrast, discontinuous aligned fibers will have sinusoidal fiber waviness similar to continuous fiber based on the fiber aspect ratio and fiber packing [2-6] but can also have significant variations in fiber alignment distribution ranging from 2-D and 3-D random to highly aligned in a single direction. To achieve properties equivalent to a high fiber volume fraction continuous fiber composite, the discontinuous fiber composite must achieve a microstructure similar to Figure 1 that is comprised of a highly aligned fiber orientation distribution that allows for efficient packing of fibers during consolidation and an optimum fiber aspect ratio and excellent fiber-matrix adhesion for effective load transfer between fibers. Unidirectional composites fabricated from short fiber *TuFF* feedstock have achieved microstructure similar to Figure 1 achieving fiber volume fractions of 60% and full property translation [7] compared to continuous fibers. The short fiber material allows in-plane stretch approaching 50% enabling metal-like forming capabilities.

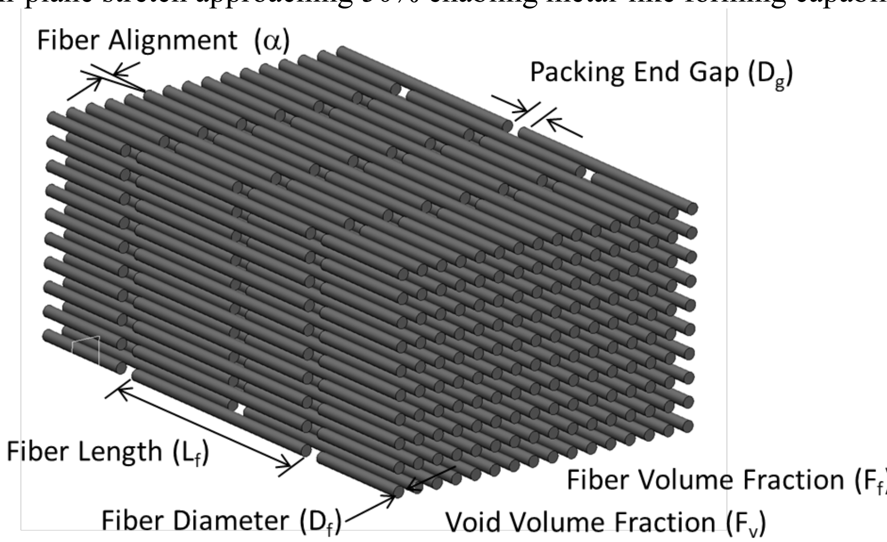


Figure 1: Idealized Microstructure of the *TuFF* Material

This paper evaluates the microstructure of thin (prepreg) and thick (coupon-level) impregnated *TuFF* material. The *TuFF* method aligns fibers creating a thin dry sheet, which can be assembled into a multi-layer stack preform and impregnated using various composite processes. In this paper, PEI resin is used as the matrix and panels are processed in an autoclave. The key microstructural properties are shown in Table 1. The fiber length and diameter correlate directly to the aspect ratio, which affects properties such as stiffness and strength but also the formability [8]. Discontinuous fibers can be obtained from various sources including waste and recycling stream, reducing fiber cost but may result in larger variation of the fiber aspect ratio. In this paper, short fibers were cut from continuous IM7 tows allowing good control of the incoming fiber length and direct correlation with the continuous fiber composite properties. Fiber aspect ratio is measured before and after alignment to quantify any length degradation effects. The packing efficiency is governed by the fiber alignment quality and fiber spacing. The *TuFF* alignment process is quantified by directly measuring fiber orientation distribution and indirectly by measuring the achievable fiber volume fraction in the composite (i.e. excellent alignment is required to achieve 60% fiber volume fraction composites).

Most microstructural properties can be obtained using microscopy of a cross-section of the composite sample including fiber diameter, fiber-to-fiber distances, fiber alignment (with an off-axis cut) and void/fiber volume fraction. Similar results can be obtained using computed tomography (CT) [9] but the limitation on voxel resolution may affect accuracy. Fiber alignment measurements from CT have been successfully demonstrated [10] and is used to evaluate the *TuFF* sample alignment quality in this paper. 3D volume information is needed to evaluate fiber end gaps and fiber length distribution within a composite sample. Table 1 shows the tests conducted in this paper (green) while other measurements (orange) will be discussed in future work.

Table 1: Microstructure properties and measurement method

	Microscopy	Computed-Tomography	Visual
Fiber Length (L_f)			
Fiber Diameter (D_f)			
Packing - End Gap (D_g)			
Packing - Fiber Distance (D_D)			
Fiber Alignment (α)			
Void Volume Fraction (F_V)			
Fiber Volume Fraction (F_F)			

2. MATERIAL PROPERTY EVALUATION

The paper evaluates the fiber length of the short fibers as received and after alignment, and all other *TuFF* microstructure properties at the prepreg and coupon level (Figure 2). Mechanical properties of the *TuFF* coupon are presented in [7] to demonstrate full property translation with the measured microstructure.

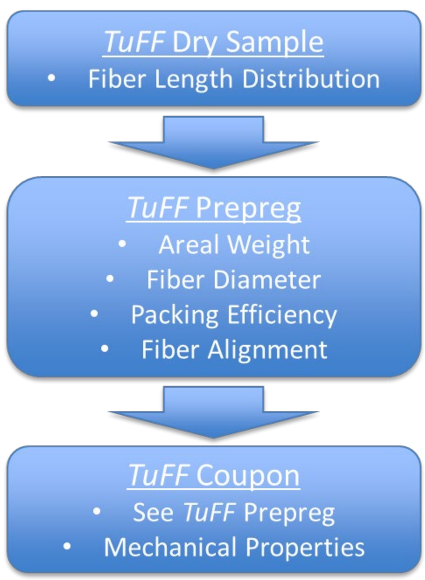


Figure 2: Microstructure Evaluation at the Different Material Form Scale

2.1 Fiber Length Distribution of Dry Samples

IM7 fibers were cut between 1-7mm and processed using the alignment system. A small amount of the fibers were separated, dispersed in a fluid, sonicated and evaluated under a microscope to measure the fiber length distribution (Figure 3, Baseline plots). Very good control of the incoming material is achieved with a cutting accuracy of approximately 0.25mm (68% of population) for all fiber lengths. To assess whether the *TuFF* process can cause fiber breakage, a wide range of fiber lengths were aligned, measured and compared to the incoming length distributions as shown in Figure 3 (*TuFF* sheet plots). For fiber lengths 5 mm and below, fiber breakage is negligible (less than 10% of fibers were broken). Some evidence of fiber breakage is seen for the 6 and 7mm long fibers (80% of all fiber length were maintained after alignment was completed; 95% of the fibers remain longer than 3mm). For the 3mm fibers used in this paper the coefficient of variation of the fiber length after alignment is approximately 10%. Full translation of properties has been achieved for the 3 and 5mm materials (testing of composites using the higher fiber lengths has not yet started).

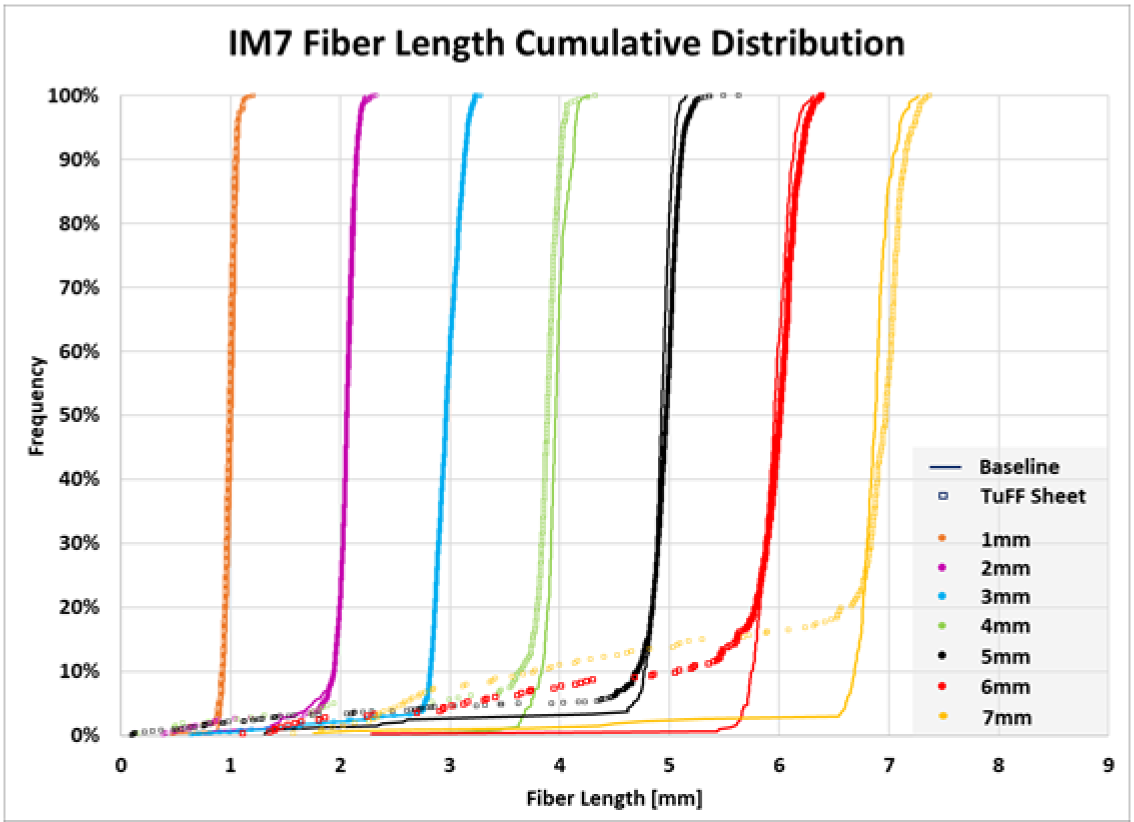


Figure 3: Fiber length distribution of as-received (Baseline) and aligned (*TuFF* sheet) material

2.2 Microscopy Evaluation

Optical microscopy images of the *TuFF* prepreg and coupon were taken with a 50x lens, VK-X200 3D Color Laser Scanning Microscope, at a resolution of 0.2μm per pixel, and are shown in Figure 4. Figure 4a and Figure 4c show the cross-section (90° cut from the fiber direction) of the prepreg and coupon, respectively. Figure 4b and 4d show the microscopic image of the 75° cut, which is used to measure fiber alignment. The cross-section images show the location and diameter of the fibers, which can be used to measure areal weight and fiber spacing. Void and fiber volume fraction can also be obtained from the cross-section. The images are processed with custom developed software to locate the fiber center, diameter and aspect ratio in case of the fiber ovals in the 75° cut. Figure 5 shows the detected fiber perimeters of subsections of the prepreg microscopic images.

Figure 4: Microscopic Image of a) cross-section and b) 75° cut of prepreg, c) cross-section and d) 75° cut of coupon

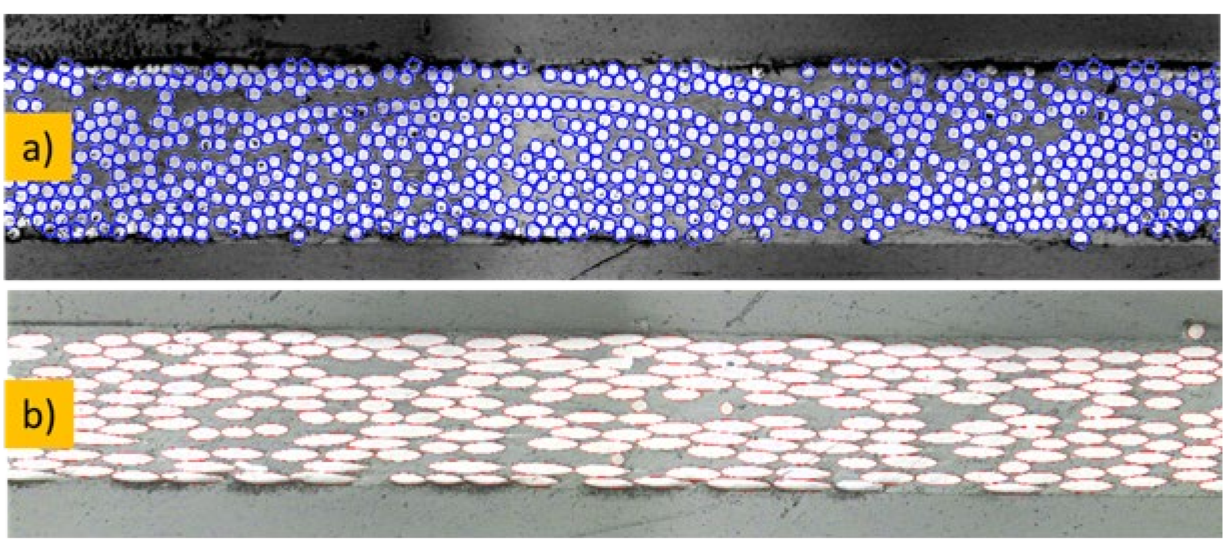
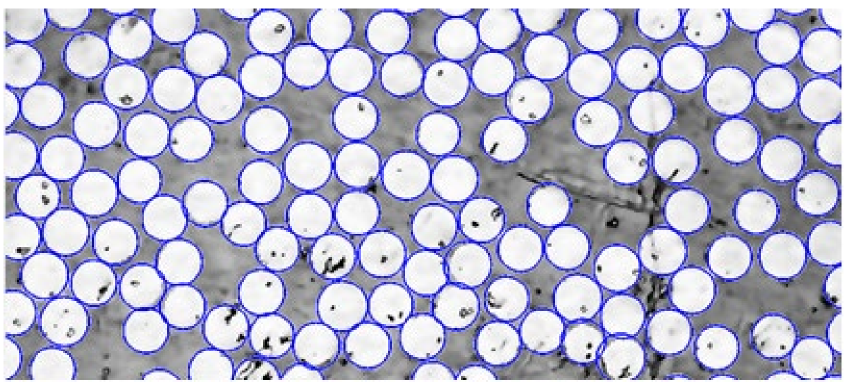


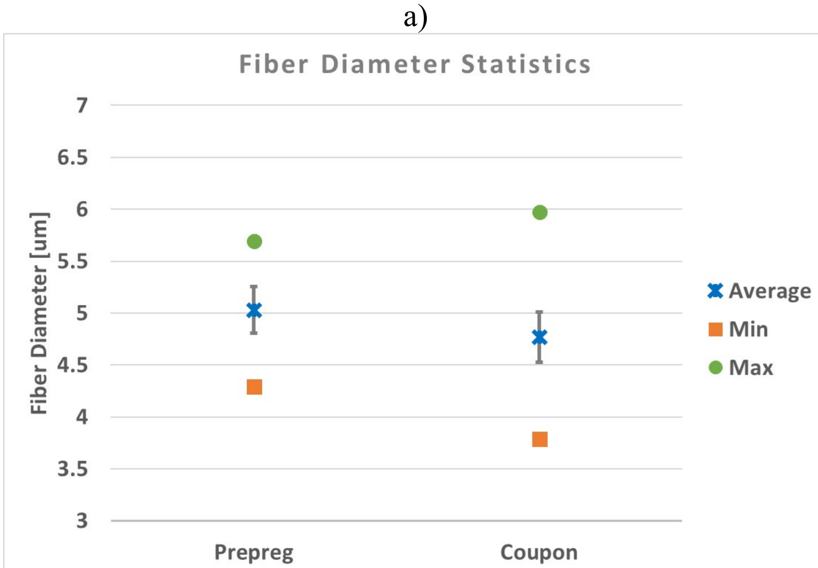
Figure 5: Fiber detection algorithm identifying fiber perimeters of sub-sections from Figure 4a and 4b.

2.2.1 Fiber Diameter

The fiber diameter algorithm automatically determines the fiber center and diameter of each fiber using an edge detection followed by a Hough transformation algorithm. Figure 6a shows a close-up of the detected fiber perimeters and Figure 6b shows the statistics of the prepreg and coupon samples. The average fiber diameter is measured to be 5.03µm (prepreg. ~2000 fibers measured) and 4.77µm (coupon, ~4000 fibers measured) with a COV between ~5%. The IM7 data sheet specifies a diameter of 5.2µm [11], which is within one standard deviation of the measured coupon data and two standard deviations at the prepreg level. As the sample size increases from prepreg to coupon, min-max fiber diameter range is spreading from 4.29µm-5.69 µm as population size increases showing the inherent variability of the fiber. The fiber diameter variation affects the aspect ratio, which is important to measure accurately for full property translation. Currently, fiber diameter and fiber length cannot be measured on each individual fiber and thus aspect ratio information can only be constructed from the distributions of the fiber length and diameter information. Additional measurements are needed and is ongoing.



a)



b)

Figure 6: a) Close-up of the micrograph showing fiber perimeters and b) diameter statistics

2.2.2 Fiber Count/Areal Weight

The fiber density per unit width can be used to calculate the areal weight of the specimens at the mm-scale. First the fiber location is determined by the center point and the number of fibers located within a cross-sectional areas (full sample height and partial section width in the in-plane direction) is calculated. The data can be converted into areal weight for a known fiber density and fiber diameter. Figure 7 plots the areal weight of the prepreg and coupon as a function of in-plane center location and section width over the complete sample width (~0.8mm wide). The average areal weight is ~70gsm for the prepreg and ~194gsm for the coupon in this example (the areal weight of the *TuFF* dry sheets can be tailored based on the application requirements). As the section size increases (height of the sample as well as width), the variation and thus coefficient of variation reduces (17% for the 5µm section width prepreg going to 4% for 500µm section width, 9% for the 5µm coupon going to 0.3% for 500m section width). The coefficient of variation is well within the typical acceptable level of commercial available continuous prepreg and shows that at the mm-scale, variability of the *TuFF* material is acceptable.

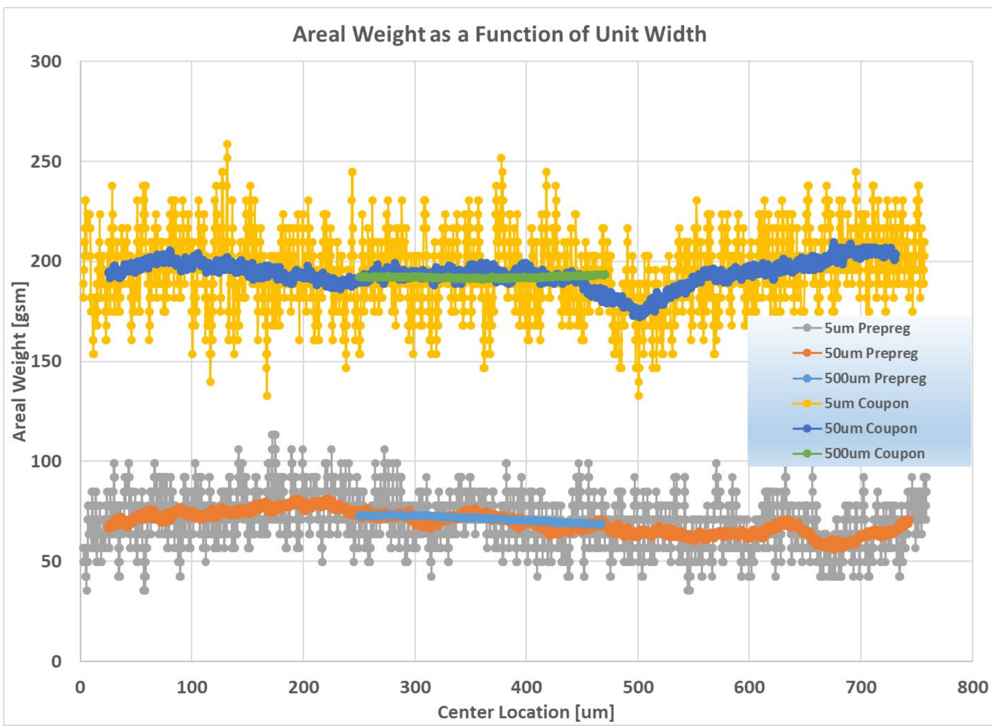
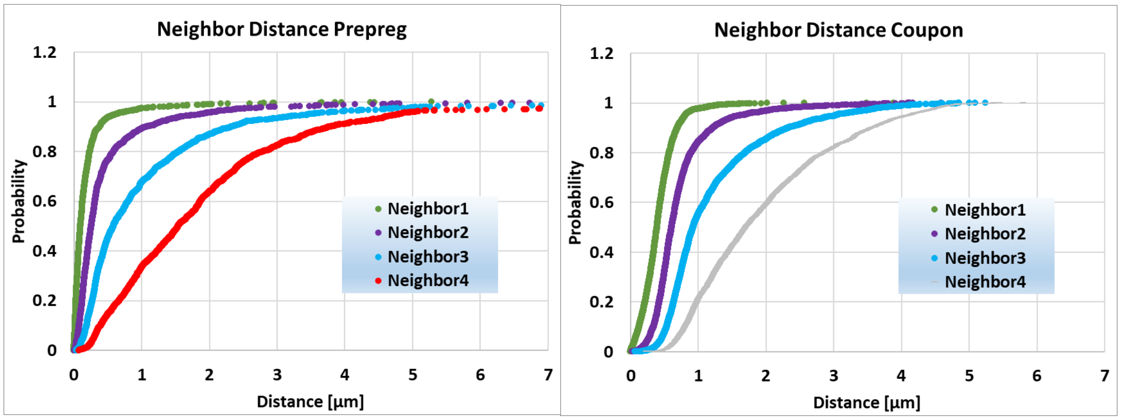


Figure 7: Areal Weight versus sample location for 5, 50 and 500µm wide sections in the a) prepreg and b) coupon sample

2.2.3 Fiber-to-Fiber Spacing

Fiber spacing is an important microstructural property as it determines the fiber packing, interactions between fibers and bulk fiber volume fraction. Fiber spacing for each fiber is calculated by finding the nearest neighbors and plotting the fiber spacing statistics. The probability density function for the first four neighbors is plotted in Figure 8. Most neighboring fibers are located within 0.3, 0.5, 1.5, 3µm and 0.6, 0.9, 1.5, 3 µm (80% of population of 1st, 2nd, 3rd, and 4th neighbor) with highest probability of the nearest neighbor being 0.2 µm and 0.5 µm for the prepreg and coupon, respectively. The packing is not ideal (hexagonal or square packing have a single distance for all fibers) and would have to be considered to model the mechanical behavior in detail.

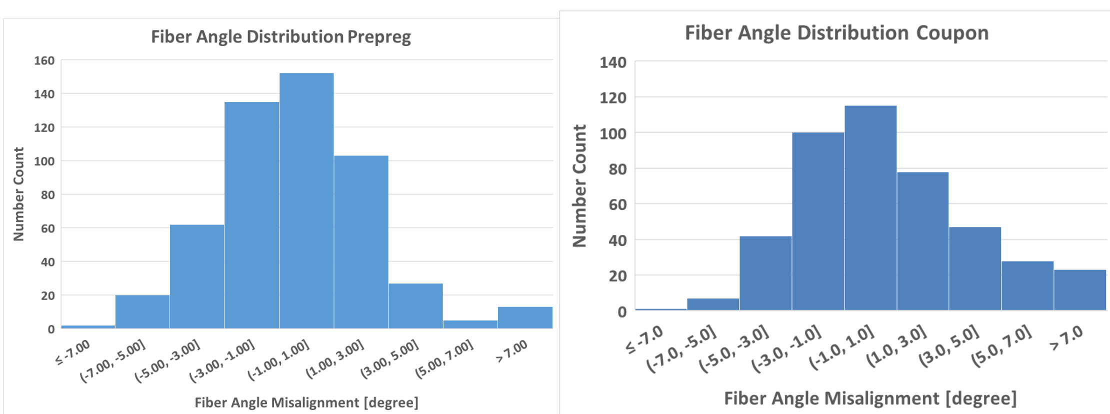


a) b)

Figure 8: Probability Density Function of Neighbor Spacing a) Prepreg and b) Coupon

2.2.4 Fiber Orientation

Fiber orientation is the key parameter to evaluate alignment quality. Two approaches are evaluated: 1) a microscopic image of the sample cut 75° from the fiber direction allows calculation of each individual fiber orientation through the resulting aspect ratio of the minor and major axes and 2) 3D volume information using CT is used to measure fiber orientation directly (the measurement method has been discussed in detail in [10]). Data for both coupons has been generated using both techniques while the CT-approach has also been applied for other coupons made using the *TuFF* process. Figure 9 shows the fiber orientation measured by the microscopy approach for the prepreg (a) and coupon (b) sample. Most fibers are aligned within ±5° (~90%-95% of fibers within that range).



a) b)

Figure 9: Fiber Angle Distribution of 1) Prepreg and b) Coupon measured with Microscopy

The microscopic approach is evaluating a small number of fibers (around 400 for both the prepreg and coupon) depending on the microscopic area tested. CT can be used to evaluate a 3D-volume with more fibers and considers any fiber undulation as well. Data was generated for multiple samples including the 3mm coupon from different locations (see Figure 10) based on the algorithm developed in [11]. In general, the alignment probability is very repeatable as sample-to-sample variation is low. Smaller fiber lengths tend to increase alignment quality. The standard deviation is well below 5° without a significant amount of outliers.

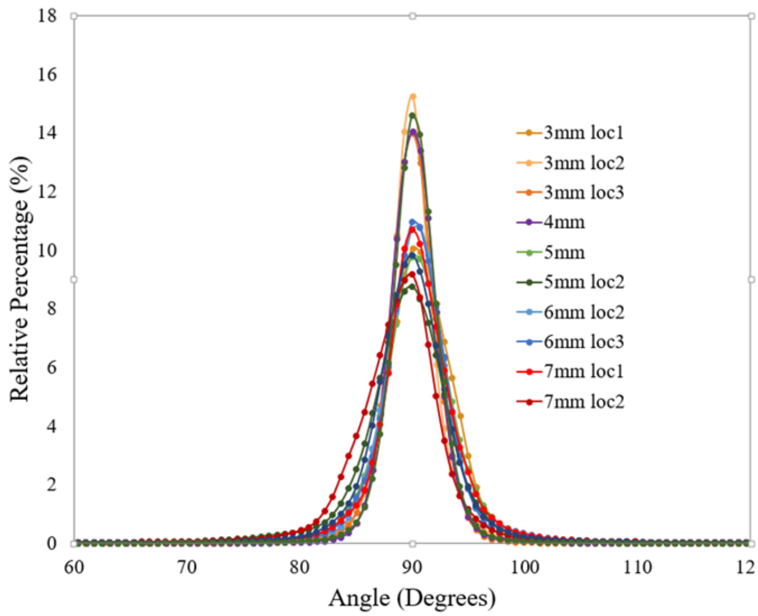


Figure 10: Alignment Quality of *TuFF* samples measured via CT

The CT approach also allows a total volume view of any potential gross misaligned fibers. Figure 11 shows a 3D view of a *TuFF* sample showing a small amount of misaligned fibers being highlighted. Misaligned fibers seem to be well separated and dispersed throughout the microstructure reducing local stress concentration typically seen with other defects such as voids.

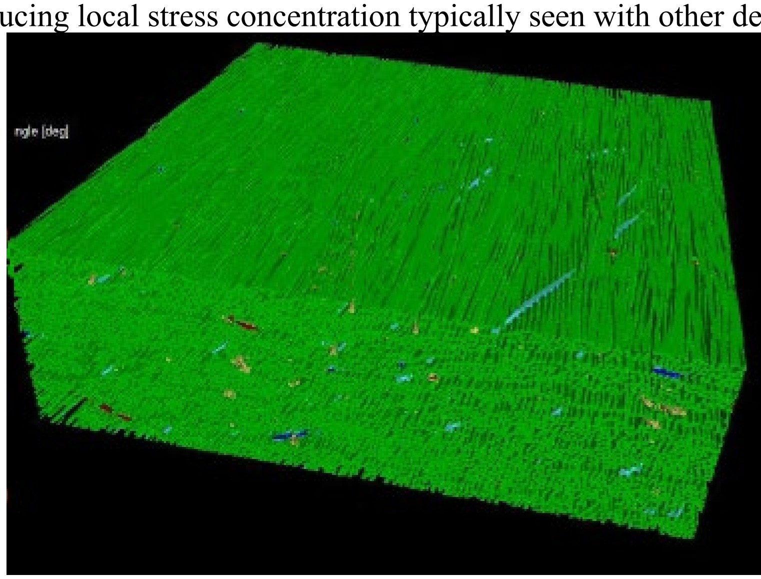


Figure 11: 3D view of alignment quality

The two-orientation measurement techniques provide very similar alignment statistics. The CT evaluates more fibers and could be more representative of the overall microstructure.

Nevertheless, the data suggest a high degree of alignment. Future work will compare the data with continuous prepreg, which has shown to have significant undulation and misalignment as well [12].

Table 2: Percentage of Fibers within an alignment range

	Alignment Range		
	±1	**±5**	**±10**
CT	**36%**	**94%**	**>99%**
Microscopy	**26%**	**87%**	**>99%**

2.2.5 Fiber Volume Fraction, Void Content and Mechanical Properties

Fiber volume and void fraction can be estimated from the microscopic images and confirm 57% fiber volume fraction and low void content leading to mechanical properties reported in [7]. This microstructure is key to 100% property translation resulting in mechanical tensile stiffness and strength comparable to continuous fiber composites.

3. CONCLUSIONS

The paper quantifies the microstructure of *TuFF* composites that has achieved full property translation of continuous fiber composites. Features include both averaged quantities such as areal weight and fiber volume fraction as well as statistical distributions in fiber length and diameter, fiber-fiber spacing and angle variation using optical microscopy and CT with custom developed algorithms for data reduction. Fiber length and aspect ratio are important short fiber properties to ensure full strength translation and are measured for the *TuFF* material. Fiber length can be controlled via the cutting process and showed very low variability (less than 10% coefficient of variation for the 3mm fiber). Fiber diameter was measured with coefficient of variation around 5%. Areal weight consistency is important and was very high for coupons larger than 100 fiber diameters (less than 1% coefficient of variation at the coupon level). The *TuFF* program has demonstrated full property translation compared to continuous prepreg when 95% of all fibers are aligned within 5° at a fiber aspect ratio (length over diameter) of 600. Future work will compare the microstructure with continuous fiber composites and develop a technique to measure fiber location and diameter throughout a 3D volume using CT. This will allow to direct measurement of aspect ratio and fiber end gaps.

4. REFERENCES

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5. ACKNOWLEDGEMENTS

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