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Characterization of Continuous Fiber-Reinforced Composite Materials Manufactured Via Fused Filament Fabrication

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14. ABSTRACT

The current work has focused on characterizing the tensile performance of continuous fiber reinforced specimens manufactured via Continuous Filament Fabrication (CFF). The specimens were tested in multiple orientations with and without continuous carbon fiber reinforcement. When comparing 0° carbon fiber reinforced specimens to specimens without continuous reinforcement, the average yield strength, tensile strength, and elastic modulus increased by factors of 20X, 15X, and 240X, respectively. When comparing the results for specimens with 90° oriented continuous reinforcement to the 0° specimens, there was a 60% drop in yield strength, 62% drop in tensile strength, and 52% drop in elastic modulus. These results indicated that mechanical performance is reduced significantly when load is applied perpendicular to the fiber orientations. The adhesion between adjacent layers was tested by printed specimens standing vertically on the print bed. These specimens had the lowest strength of all specimens. The authors recommend follow on testing using rectangular specimens with bonded tabs per ASTM D3039-17 to reduced issues with fiber alignment that were encountered with the dog bone specimens.

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Introduction

While additive manufacturing (AM) technology has evolved rapidly over the past several years, there is still limited understanding into the fundamental behaviors of as-manufactured AM materials. One fact, however, is well known: AM materials do not behave the same as traditionally manufactured parts. For instance, the most common AM processes, commonly referred to as Fused Filament Fabrication (FFF) or Fused Deposition Modeling (FDM), can produce parts with the same geometry and materials as injection molding, however the material properties of the as-manufactured parts can be significantly different. Since most AM parts are built from the bottom up in a layer-by-layer process, it is very common for the out-of-plane material properties to be weaker than the in-plane material properties. A previous study reported that the mechanical properties of the bond between layers (out-of-plane) can be 10-25% weaker than in-plane properties (Duty, et al., 2017). Very recently, new commercial technology has been introduced that enables printing of continuous fiber-reinforced thermoplastic composites. This technology is referred to as Continuous Filament Fabrication (CFF) (Markforged, 2018). When continuous (carbon, glass, and/or aramid) fibers are printed in-plane, they have the potential to significantly increase in-plane strength and stiffness of the AM part compared to traditionally printed parts. However, it is not well understood how these continuous fiber reinforcements would affect the mechanical anisotropy of the as-manufactured part. In order for design engineers to utilize continuous fiber-reinforced AM parts in structural applications, they will require the mechanical properties of these materials in three dimensions. This study aims to characterize continuous carbon fiber-reinforced thermoplastic composite parts produced using a Markforged Mark Two desktop printer. Methods for characterizing continuous fiber-reinforced AM parts are not well established, so methods have been adapted from AM, plastics, and composites communities.

Description of Test Specimens

Test items were manufactured using a Markforged Mark Two desktop printer, which is capable of printing thermoplastic (nylon) parts with continuous fiber reinforcements using fibers such as carbon, glass, and aramid fibers. In the current study, only carbon fibers were utilized in the printed parts. In order to print a continuous carbon fiber-reinforced part on the Mark Two, two spools of filament are required. The first filament is a material that Markforged sells under the name of Onyx, which is a nylon-based thermoplastic that is said to have chopped carbon-fiber reinforcement already mixed into the filament. The second filament is a roll of continuous carbon fiber tow coated with a binder material.

The Mark Two printer utilizes Eiger, a cloud-based software package, to slice the cad geometry and specify the material parameters. The Eiger software allows the user to specify many parameters at the single layer level. Parameters relevant to this study included: layer height, % infill, type of reinforcement (fiber), and fiber orientation. For traditional laminated composite materials the percentage of air pockets, or voids, can have non-linear impact on mechanical performance. These voids can complicate interpretation of test data, therefore in this study, all specimens were printed with 100% fill. It is important to note that 100% infill for FDM and CFF processes does not mean that the as-manufactured part is void free. FDM and CFF processes lack the consolidation pressure required to eliminate all voids, therefore voids would still be expected in 100% infill parts. The specimens were printed to net shape as shown in Figure 1, fiber orientations shown in Figure 2, and with dimensions in Table A.1. In order to best understand the influence of the continuous carbon fiber-reinforcement, the following test specimens were printed:

- Group 1: Onyx (in plane, Nylon/Carbon plastic): ID# 1-1, 1-2, 1-3
- Group 2: 0^{o} fibers (in-plane, aligned carbon fibers):: ID# 2-1, 2-2, 2-3
- Group 3: 90^{o} fibers (in-plane, perpendicular to carbon fibers): ID# 3-1, 3-2, 3-3
- Group 4: z direction (out-of plane, perpendicular to carbon fibers): ID# 4-1, 4-2, 4-3

The decision to test only specimens with unidirectional fiber orientations was to simplify analysis of the results. The specimens 1-1, 1-2, and 1-3 were approximately 1.8 mm thick and printed out of pure Onyx material with 100% infill. These specimens were used as a baseline to understand the performance of the Onyx material, which is used as the skin material for the outer shell of the fiber-reinforced specimens. The 0 degree fiber-reinforced specimens 2-1, 2-2, and 2-3 were printed with two 0.125mm layers of Onyx on the roof and floor and two layers of Onyx on the side walls. The remaining interior core of the material was filled with carbon fiber that was oriented longitudinally in the direction of pull for a tensile test (see Figure 2).

It is noteworthy, that the "unidirectional" fiber-reinforced specimens in this report are not true unidirectional specimens due to the printing process used to lay down the carbon fiber filament. For example, when the print head changes direction from one pass to the next, the carbon fiber filament must turn the corner to prepare for the next pass. In addition, the radii from the grip section to the gauge section force additional non-linear paths for laying down the fiber-reinforcement. However, within the gauge length itself, the fiber-reinforcement maintained uniform orientation.

Additional specimens 3-1, 3-2, and 3-3 were printed with fibers oriented perpendicular to the tensile pull direction. These specimens had the same thickness of Onyx on the roof, floor, and walls as the previous set of specimens (2-1, 2-2, and 2-2). It is noteworthy that for these specimens, since fibers were oriented perpendicular to the direction of tensile pull, the print head must turn corners within the gauge section, and therefore, the fiber orientation within the gauge section was not perfectly unidirectional.

The final set of specimens 4-1, 4-2, and 4-3 were printed standing vertically on the print bed. This specimen orientation presented challenges since the height of the build was much larger than the base of the tensile bar connected to the print bed. First, the standard length of tensile bar per ASTM D638-14 was greater than the maximum print build of the specimen. In order to fit within the printer capabilities, the tensile bar was scaled down to a height of 150mm, instead of the standard 170mm length of the other specimens. Moreover, the tensile bar thickness was increased to 6mm thick. These specimens were printed with 2 x 0.125mm layers of Onyx on the roof, floor, and side walls. This set of specimens was tested to evaluate the adhesion between layers of fiber-reinforcement, which is typically referred to as z-direction strength.

Upon receipt and inspection, 11 of the 12 test items (samples) were acceptable for testing. The 12th test item (ID#004-03) experienced a malfunction during the print, and therefore was excluded from tensile testing.

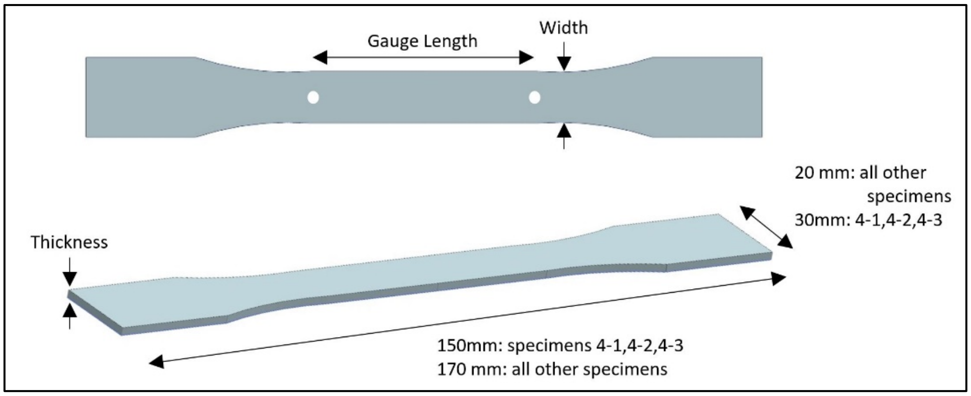

Figure 1: Geometry of Tensile Specimens, referenced from ASTM D638-14. Measurements for each specimen are given in the Appendix.

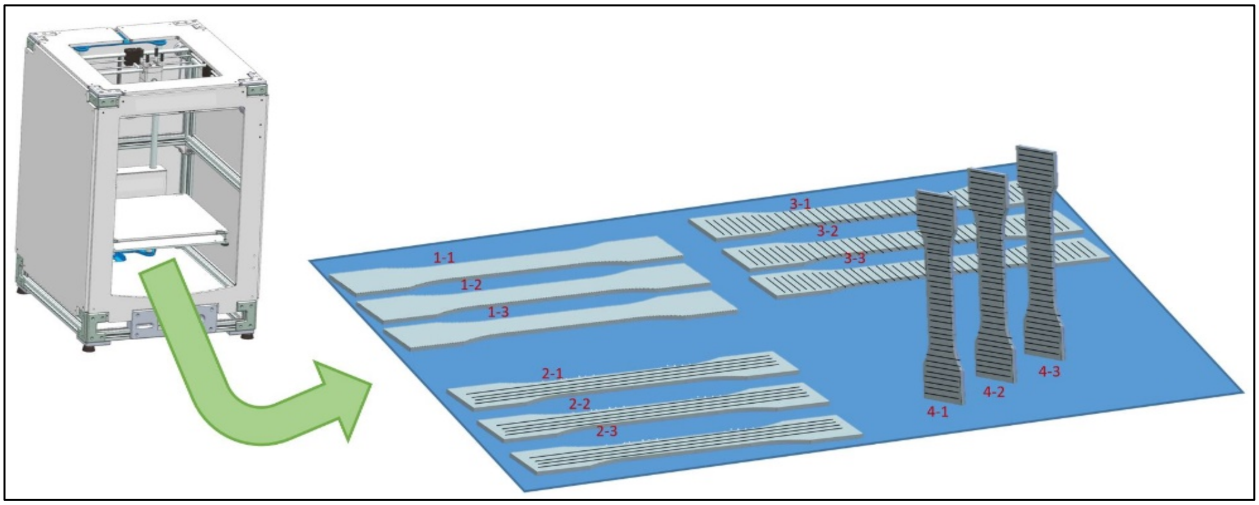

Figure 2: Schematic of specimens on print bed to show specimen placement and fiber orientation (where relevant).

Methods

Tensile testing conducted was in accordance with the following test methods: ASTM D638-14 *Standard Test Methods for Tension Testing of Plastics* and ASTM F2971-13 *Standard Practice for Reporting Data for Test Specimens Prepared by Additive Manufacturing*. In total, 11 tensile specimens were tested and results were averaged to produce the desired mechanical properties. Tensile tests were conducted using an Instron 5984 Tensile Tester with an integrated video extensometer. The tensile testing was conducted at extension rate of 5mm/min. The video extensometer was used to capture axial and transverse strain during the tensile test. Bluehill 3 software used data such as load and axial displacement to calculate the material properties such as: elastic modulus, yield strength, and tensile strength.

In addition to tensile testing, Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) Analysis were conducted on the Onyx material to better understand the thermal characteristics of this thermoplastic filament. The TGA tests were performed on a 12.136 mg sample of Onyx filament as supplied by the manufacturer (i.e. virgin filament that had not been printed). The TGA test was conducted on a TA Instruments Q5000 from room temperature to 800 ^{o}C at a ramp rate of 30 ^{o}C per minute. The FTIR Analysis was performed on a TA Instruments FTIR Spectrometer.

Results

Tensile Test Results

All tensile tests were performed until complete specimen failure, and the average strength results are shown below in Table I. Individual stress versus strain plots are displayed for select specimens in Figure 3. Based on the test data, the ultimate tensile strength was highest for the group 2 test specimens with 0 degree fibers (ID# 2-1, 2-2, and 2-3). The ultimate tensile strength was reduced when rotating the fibers 90 degrees for the group 3 specimens (ID# 3-1, 3-2, 3-3) compared to the 0 degree test specimens (ID# 2-1, 2-2, and 2-3). The z-direction ultimate tensile strength for group 4 specimens (ID# 4-1, 4-2) was significantly lower than all other test specimens included in this study. Based on these results, the carbon fiber reinforcement had a significant impact on both the stiffness and strength of the as-manufactured materials.

When comparing 0^{o} carbon fiber reinforced specimens (group 2 in Table I) to pure onyx specimens (group 1 in Table I), the mechanical properties increased by orders of magnitude. For example, the average yield strength, tensile strength, and elastic modulus increased by factors of 20X, 15X, and 240X, respectively. When comparing mechanical performance of the fiber-reinforced specimens to the Onyx material, the significant improvement in mechanical performance is consistent with traditional laminated composites, where unidirectional specimens have strength and stiffness orders of magnitude higher than a homogenous epoxy matrix material. When comparing the results for the 90^{o} specimens (group 3 in Table I) to the 0^{o} specimens, there was a 60% drop in yield strength, 62% drop in tensile strength, and 52% drop in elastic modulus. These results indicated that mechanical performance is reduced significantly when load is applied perpendicular to the fiber orientations. However, the relative drop in mechanical performance was not as significant as what is observed for many traditional unidirectional composites tested at 90^{o} orientation. For example, the 90^{o} strength of an aerospace grade unidirectional laminate may be 96% lower than the 0^{o} strength of that same laminate (Hexcel Corporation, 2016). While the materials tested in this study are nowhere near aerospace grade performance, it is important to note that these materials may not experience as severe mechanical anisotropy compared to typical carbon fiber laminates. The exact reason is not known at this time, but the lower directional anisotropy may be due to the fact that in the CFF process, the continuous fibers must be steered, both to conform to the part geometry and to proceed from one pass to the next. The steered fiber reinforcement is therefore not perfectly unidirectional. While the decrease in mechanical performance is significant compare to 0^o specimens, these 90^o specimens still had strength and stiffness 1 order of magnitude greater than the Onyx material. This was not the case when testing the adhesion between successive layers in the z-direction specimens (group 4 in Table I). When testing the adhesion between layers, the group 4 specimens broke very quickly, well below the failure stress and strain of the pure Onyx material. It is noteworthy that in these tensile tests, the strength values reported are based on the cross-sectional area of the gauge length. However for some specimens failure occurred outside of the gauge length, which is not desirable for a tensile test. The strength values reported represent the equivalent strength of a homogenized orthotropic material within the gauge length. However, it is understood that these continuous fiber reinforced specimens are neither homogenous, and failure occurs due to local stresses within the fibers and/or matrix materials. Further insight is gained when studying the failure mechanisms of the individual specimens.

Table I: Average Tensile Strength Results for Each Type of Specimen.

Test Item Group	**Fiber Description**	**Avg. Yield Strength (0.2% Offset) (KSI)**	**Std. Dev. (KSI)**	**Avg. Tensile Strength (KSI)**	**Std. Dev. (KSI)**	**Avg. Elastic Modulus (KSI)**	**Std. Dev. (KSI)**
1	Pure Onyx	1.85E+00	3.00E-02	3.33E+00	3.70E-01	1.94E+01	4.41E+00
2	0^o Carbon	3.78E+01	9.26E-01	4.94E+01	1.62E+00	4.68E+03	2.18E+01
3	90^o Carbon	1.49E+01	2.04E+00	1.90E+01	2.13E+00	2.14E+03	2.21E+02
4	Z Carbon	1.08E+00	2.12E-02	1.39E+00	1.40E-01	2.00E+02	2.19E+01

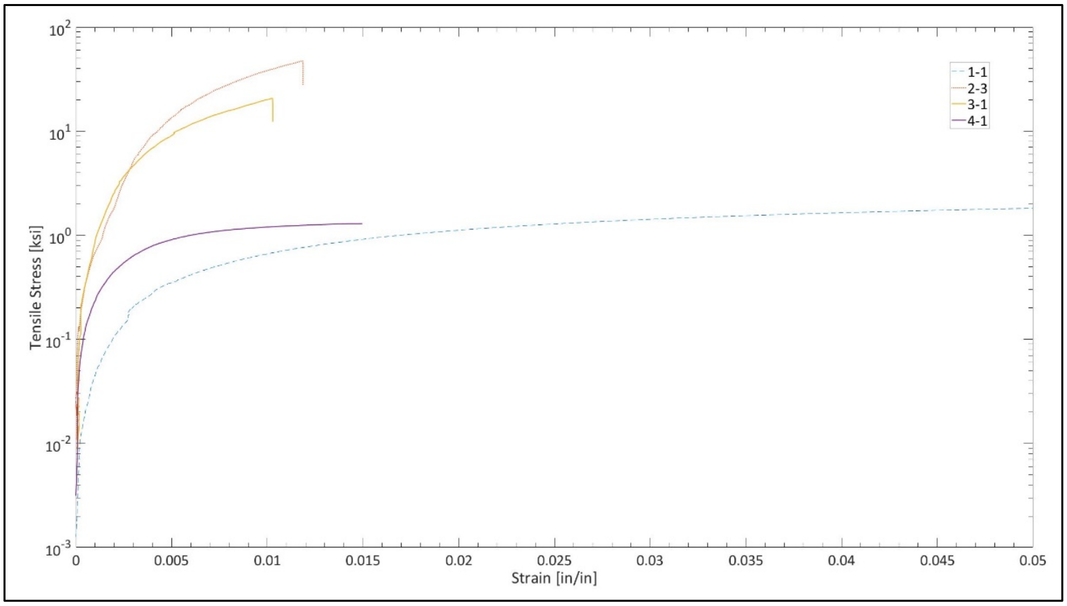

Figure 3: Tensile stress versus strain for individual test specimens made from pure Onyx material (1-1), 0^o carbon fiber reinforcement, and 90^o carbon fiber reinforcement. Note: Specimen 1-1 failed at strain of 0.25 in/in, though this is not shown in the plot.

Failure Analysis

When observing the failure of the group 1 Onyx specimens in Figure 4, all three specimens failed within the gauge length with a fracture surface oriented approximately 45° from the direction of tensile pull. This failure pattern may be a result of the splicing/manufacturing of the specimens. By default, the Eiger software orients layers at ± 45°, as visible by the witness lines prominently shown in Figure 4 (a) – (b). In Figure 4 (c), Onyx filament can be seen sticking out perpendicular to the fracture surface. This type of failure is reminiscent of fiber pullout in traditional composite materials and indicates that the extruded filament remains distinct within the printed part and the primary filament-to-filament adhesion mechanism is only partial sintering of the exterior skin of the Onyx filament. This is typical of plastic materials manufactured via FDM. The failure of the fiber-reinforced specimens was quite different and highly dependent on the orientation of the carbon fiber.

In contrast to the Onyx specimens, the 0° group 2 specimens all failed near the grip section at the gauge length radius, as shown in Figure 5. The failure surface of these specimens was very jagged and included unbroken fibers that bridged the fractured zone. Figure 6 shows detailed views of the failure surface with visible matrix cracking, fiber breakage, and fiber pullout. The 90° group 3 specimens also failed near the grip section at the radius, however, the failure was a much cleaner break, as shown in Figure 7. For these specimens with the carbon fibers oriented perpendicular to the direction of tensile load, the failure occurred due to matrix cracking that occurred between the fiber reinforcements. This failure is typically of traditional fiber-reinforced composite laminates. For the z-direction group 3 specimens (see Figure 8), both specimens failed within the gauge length via de-bonding of adjacent layers.

For both the 0° and 90°, the failure occurred outside of the gauge length, which is not desirable for a tensile test. In order to most accurately determine failure strength, the failure should occur within the gauge length. Therefore, the strength results reported in Table I and Table A.1 should only be analyzed within the context of the current study. The authors surmise that the failure near the radius was caused by the non-uniform alignment of carbon fibers near the radius between the grip section and gauge section. Using the CFF manufacturing process, it is not possible to lay down linearly oriented fiber along a curved feature. The fiber placement method requires some minimum space to turn corners resulting in a change in local fiber orientation. For this reason, the authors recommend follow on testing using rectangular specimens with bonded tabs per ASTM D3039-17 *Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials* rather than the dog bone style specimens per ASTM D638-14 S*tandard Test Method for Tensile Properties of Plastics* and ASTM F2971-13 *Reporting Data for Test Specimens Prepared by Additive Manufacturing*. It is noteworthy that ASTM D638-14 and ASTM F2971-13 were not developed with the CFF process in mind, and therefore may be the best method for testing additively manufactured materials with continuous fiber reinforcement. Further investigation is required.

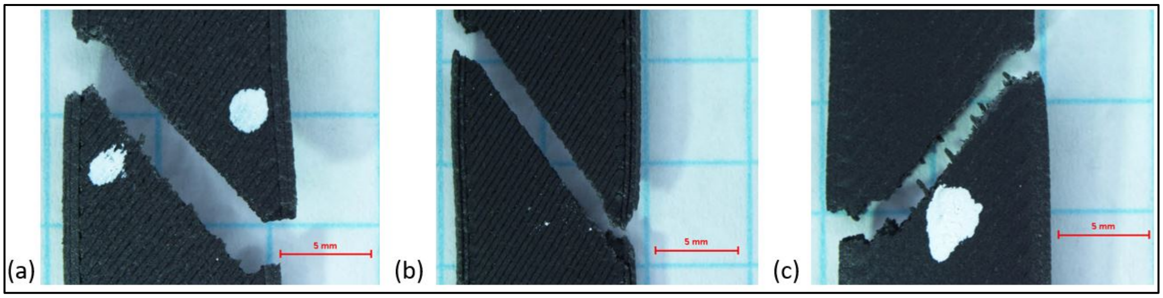

Figure 4: Fractured tensile specimens (a) 1-1, (b) 1-2, and (c) 1-3 printed flat on print bed with 100% infill of Onyx® material.

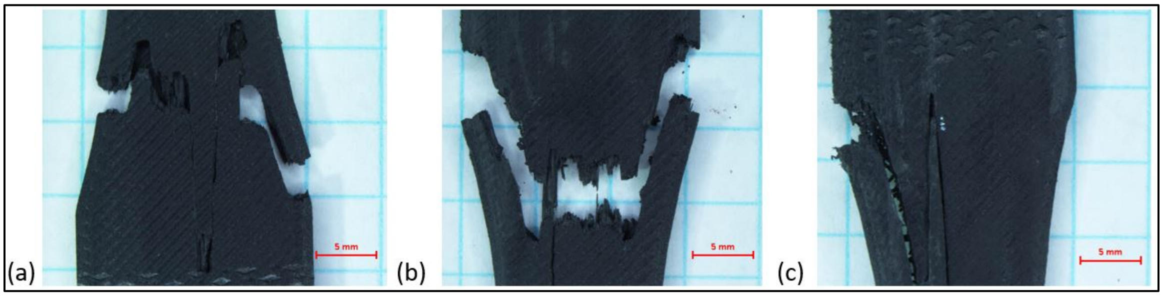

Figure 5: Fractured tensile specimens (a) 2-1, (b) 2-2, and (c) 2-3 printed flat on print bed with Onyx® skin and 100% infill of unidirectional carbon filament oriented in direction of tensile load.

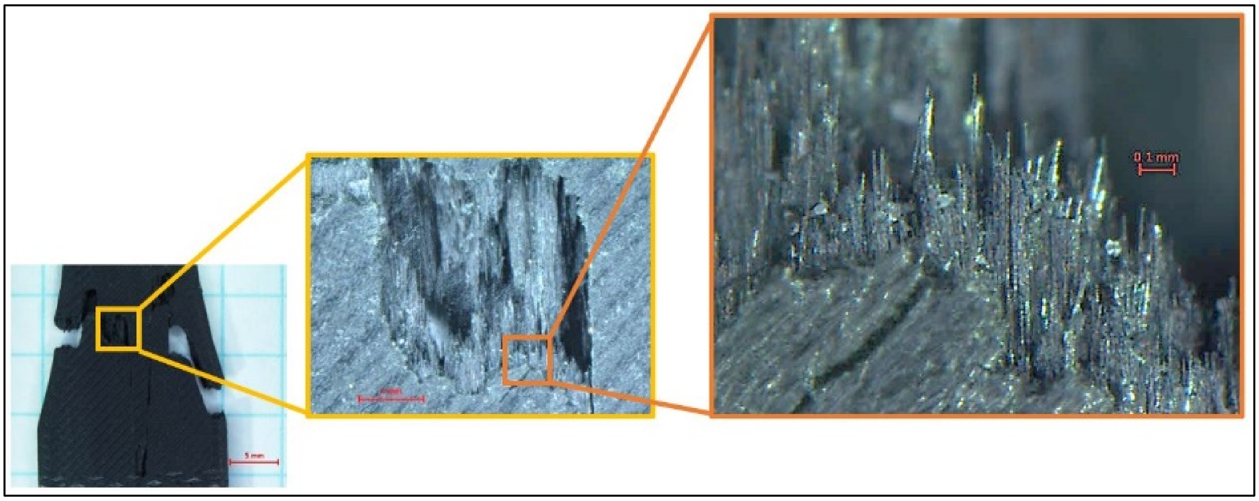

Figure 6: Detailed views of fracture surface of specimen 1-1, showing fiber breakage, fiber pullout, and matrix cracking.

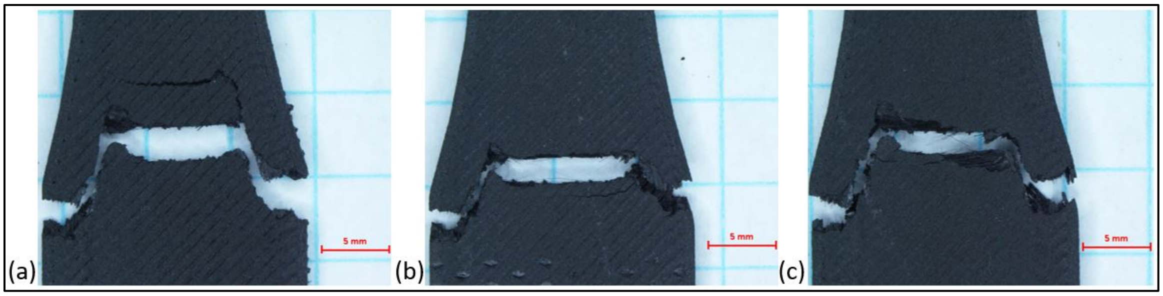

Figure 7: Fractured tensile specimens (a) 3-1, (b) 3-2, and (c) 3-3 printed flat on print bed with Onyx® skin and 100% infill of unidirectional carbon filament oriented perpendicular to direction of tensile load.

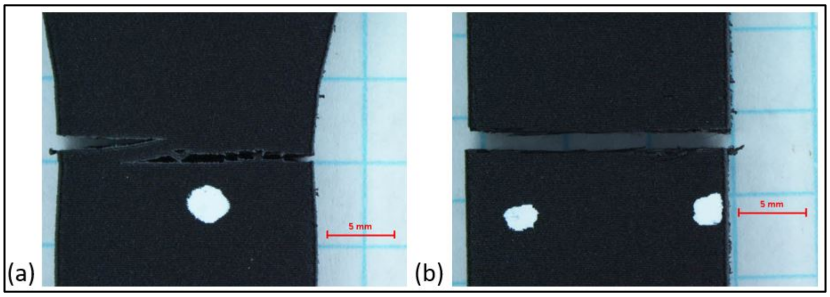

Figure 8: Fractured tensile specimens (a) 4-1, and (b) 4-2 printed vertically on print bed with Onyx® skin and 100% infill of unidirectional carbon filament oriented perpendicular to direction of tensile load.

TGA and FTIR Analysis

Thermogravimetric Analysis (TGA) was performed on virgin Onyx filament to observe the change in mass versus temperature. TGA testing can be useful for understanding the thermal degradation of the material so that engineers can match the correct material with the expected operating environment. While the thermal degradation of nylon is well understood, there is limited data on these new nylon/carbon fiber filaments for 3D printing. For aerospace grade carbon fiber composites, the resin typically degrades up to approximately 500 ^{o}C. Beyond 500 ^{o}C, the composition is mainly carbon fiber (Quintiere, Walters, & Crowley, 2007). For the Onyx material, however, the exact resin system was not known, so Fourier Transform Infrared (FTIR) Analysis was conducted to characterize the material. The results of the FTIR analysis are shown in Figure 9. Results indicated that the thermoplastic resin used is a Nylon 6 or Nylon 6,6 resin or a combination of the two. The ignition temperature of nylon 6 is 329 ^{o}C, compared to 377 ^{o}C for nylon 6,6 (Taylor Edge, 2018). Considering that both grades of nylon ignite well below 500 ^{o}C, it is reasonable to assume that mass lost below 500 ^{o}C is attributed to the resin system, whereas mass remaining beyond 500 ^{o}C would be primarily carbon fiber. Based on the TGA results in Figure 10, 21% mass remained at 500 ^{o}C. Based on the temperature ramp rate of 30 ^{o}C per minute, there was a 10 minute period between 500 ^{o}C and the end of the test at 800 ^{o}C. Whether significant or not, it is noted that 3.2% mass remained at 700 ^{o}C, which is the ignition temperature of carbon.

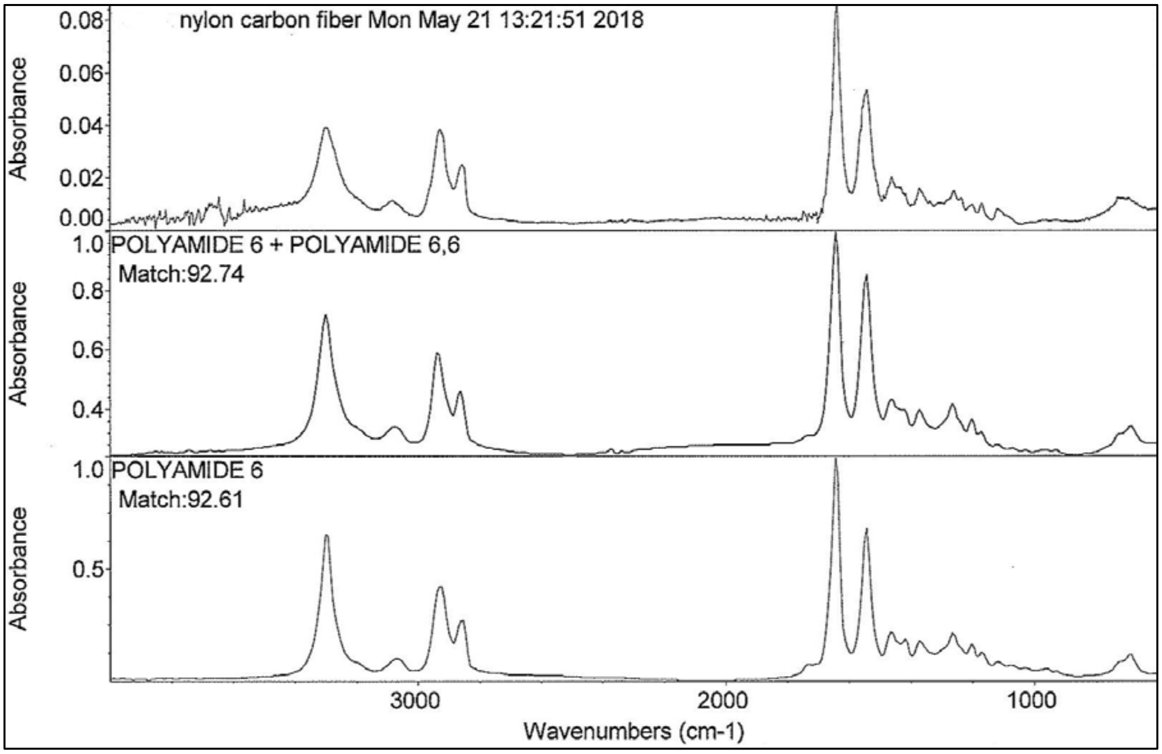

Figure 9: FTIR analysis of virgin filament.

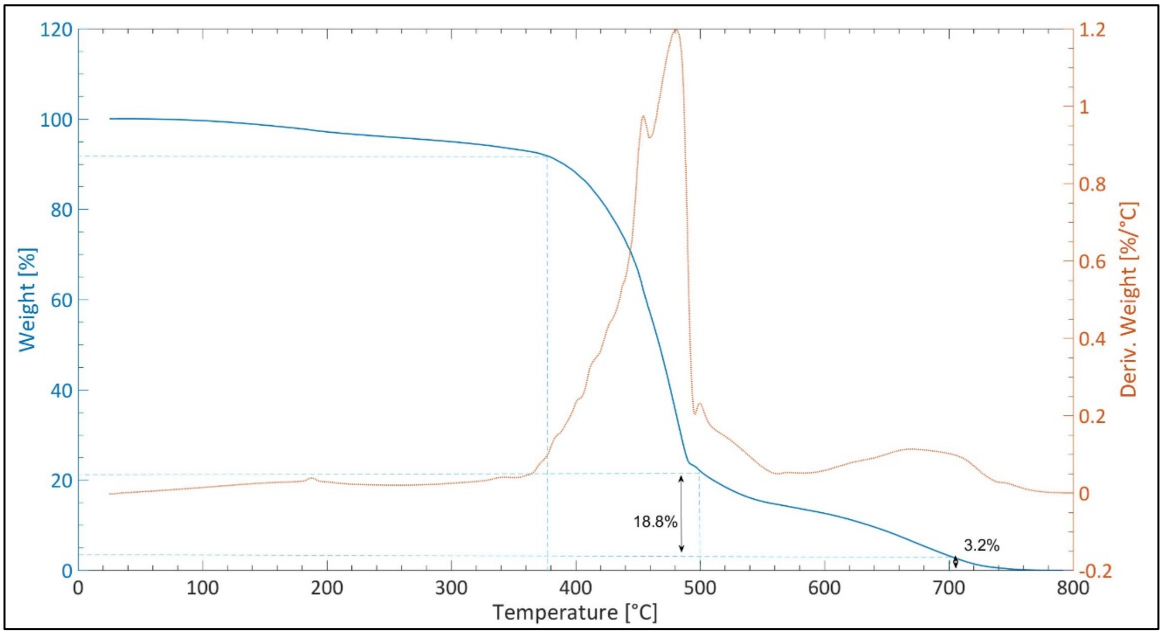

Figure 10: TGA analysis of filament. Vertical dashed line at temperature of 377 ^{o}C corresponds to ignition temperature of nylon 6,6. Vertical dashed line at temperature of 700 ^{o}C corresponds to ignition temperature of carbon.

Conclusions

The current work focused on characterizing the tensile performance of continuous fiber reinforced specimens manufactured via Continuous Filament Fabrication (CFF). The specimens were tested in multiple orientations with and without continuous carbon fiber reinforcement. When comparing 0^{o} carbon fiber reinforced specimens to pure onyx specimens, the mechanical properties increased by orders of magnitude. For example, the average yield strength, tensile strength, and elastic modulus increased by factors of 20X, 15X, and 240X, respectively. When comparing mechanical performance of the fiber-reinforced specimens to the Onyx material, the significant improvement in mechanical performance is consistent with traditional laminated composites, where unidirectional specimens have strength and stiffness orders of magnitude higher than a homogenous epoxy matrix material. When comparing the results for the 90^{o} specimens to the 0^{o} specimens, there was a 60% drop in yield strength, 62% drop in tensile strength, and 52% drop in elastic modulus. These results indicated that mechanical performance is reduced significantly when load is applied perpendicular to the fiber orientations. However, the relative drop in mechanical performance was not as significant as what is observed for many traditional unidirectional composites tested at 90^{o} orientation. The adhesion between adjacent layers was tested by printed specimens standing vertically on the print bed. These specimens had the lowest strength of all specimens. The results of this study indicate that there is a high degree of mechanical anisotropy in these materials, and that the 3D anisotropic mechanical properties must be considered when implementing these materials in structural applications.

This study found that use of traditional dog bone shaped tensile bars were not ideal for CFF specimens due to the unique fiber placement process and local variations in fiber angle around the curved radii. The authors recommend follow on testing using rectangular specimens with bonded tabs per ASTM D3039-17 *Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials* rather than the dog bone style specimens per ASTM D638-14 S*tandard Test Method for Tensile Properties of Plastics* and ASTM F2971-13 *Reporting Data for Test Specimens Prepared by Additive Manufacturing*. It is noteworthy that ASTM D638-14 and ASTM F2971-13 were not developed with the CFF process in mind, and therefore may be the best method for testing additively manufactured materials with continuous fiber reinforcement. Further investigation is required.

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Appendix A

Table A.1: Tensile Test Data ("o" indicates original dimension, "f" indicates final dimension)

Test Item ID	Thick$_o$ (in.)	Gauge Width$_o$ (in.)	Gauge Length$_o$ (in.)	Thick$_f$ (in.)	Gauge Width$_f$ (in.)	Gauge Length$_f$ (in.)	Yield Strength 0.2% offset (KSI)	Tensile Strength (KSI)	Tensile Modulus (KSI)
1-1	7.55E-02	5.17E-01	2.00E+00	7.00E-02	4.69E-01	2.43E+00	1.85E+00	3.22E+00	2.04E+01
1-2	7.30E-02	5.17E-01	2.00E+00	6.85E-02	4.44E-01	2.62E+00	1.88E+00	3.75E+00	2.33E+01
1-3	7.80E-02	5.17E-01	2.00E+00	7.55E-02	4.56E-01	2.38E+00	1.82E+00	3.03E+00	1.46E+01
Average	**7.55E-02**	**5.17E-01**	**2.00E+00**	**7.13E-02**	**4.56E-01**	**2.48E+00**	**1.85E+00**	**3.33E+00**	**1.94E+01**
Std. Dev.	**2.50E-03**	**0.00E+00**	**0.00E+00**	**3.70E-03**	**1.25E-02**	**1.24E-01**	**3.00E-02**	**3.70E-01**	**4.41E+00**
2-1	7.20E-02	5.17E-01	2.00E+00	7.15E-02	5.14E-01	2.10E+00	3.81E+01	5.06E+01	4.66E+03
2-2	7.65E-02	5.17E-01	2.00E+00	7.50E-02	5.16E-01	2.11E+00	3.84E+01	5.00E+01	4.67E+03
2-3	7.80E-02	5.18E-01	2.00E+00	7.90E-02	5.25E-01	2.11E+00	3.67E+01	4.75E+01	4.70E+03
Average	**7.55E-02**	**5.17E-01**	**2.00E+00**	**7.52E-02**	**5.18E-01**	**2.11E+00**	**3.78E+01**	**4.94E+01**	**4.68E+03**
Std. Dev.	**3.12E-03**	**5.00E-04**	**0.00E+00**	**3.80E-03**	**5.90E-03**	**7.20E-03**	**9.26E-01**	**1.62E+00**	**2.18E+01**
3-1	7.25E-02	5.15E-01	2.00E+00	7.15E-02	5.13E-01	2.08E+00	1.68E+01	2.08E+01	2.28E+03
3-2	7.55E-02	5.11E-01	2.00E+00	7.35E-02	5.09E-01	2.11E+00	1.53E+01	1.94E+01	2.25E+03
3-3	7.75E-02	5.13E-01	2.00E+00	7.70E-02	5.11E-01	2.13E+00	1.27E+01	1.66E+01	1.88E+03
Average	**7.52E-02**	**5.13E-01**	**2.00E+00**	**7.40E-02**	**5.11E-01**	**2.10E+00**	**1.49E+01**	**1.90E+01**	**2.14E+03**
Std. Dev.	**2.52E-03**	**2.30E-03**	**0.00E+00**	**2.80E-03**	**2.00E-03**	**2.54E-02**	**2.04E+00**	**2.13E+00**	**2.21E+02**
4-1	2.37E-01	7.75E-01	2.00E+00	2.37E-01	7.75E-01	2.00E+00	1.06E+00	1.29E+00	2.15E+02
4-2	2.40E-01	7.79E-01	2.00E+00	2.40E-01	7.79E-01	2.00E+00	1.09E+00	1.49E+00	1.84E+02
4-3	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Average	**2.38E-01**	**7.77E-01**	**2.00E+00**	**2.38E-01**	**7.77E-01**	**2.00E+00**	**1.08E+00**	**1.39E+00**	**2.00E+02**
Std. Dev.	**2.12E-03**	**2.50E-03**	**0.00E+00**	**2.10E-03**	**2.50E-03**	**0.00E+00**	**2.12E-02**	**1.40E-01**	**2.19E+01**