Ultimate Tensile Strengths of 3D Printed Carbon-fiber Reinforced Thermoplastics in Liquid Nitrogen

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Abstract. 3D printed composites have potential to satisfy needs for lighter and more sophisticated materials for aerospace, medical, and other sectors. However, few mechanical property measurements of 3D printed parts at cryogenic temperatures are available. To address this need, this work performs ultimate tensile strength testing of 3D printed thermoplastics immersed in liquid nitrogen at approximately 77 K. Materials tested include carbon-fiber reinforced PETG and carbon-fiber reinforced Amphora AM1800 filament. The carbon-fiber reinforced PETG increased in ultimate tensile strength and modulus of elasticity by 49% and 43.2% from room temperature tests, respectively. The carbon-fiber reinforced Amphora AM1800 filament decreased in ultimate tensile strength by 29.9% from room-temperature.

1. Introduction

The availability of thermoset and thermoplastic polymers embedded with carbon fibers, especially with fused deposition modeling (FDM) techniques, continues to increase. Polymers currently offer superior light-weighting design potential compared to conventional metal components due to lower thermal conductivity, density, and often reduced associated manufacturing costs. However, the ultimate strength of these components is often inferior to conventional materials. Embedding carbonous additives, including chipped strand and graphene sheets into 3D printable polymers, is one promising method for increasing strength of the bulk material. [1, 2] Thus, there is a persistent need for material property information of these 3D printed materials, including 3D printed carbon fiber samples at cryogenic temperatures. [3]

An additional consideration is the fact that 3D printing methods can considerably vary the anisotropy of an already complex material. [4] Prior work has shown that polymer blends printed perpendicular to the axis of loading fracture under the lowest load at cryogenic temperatures. [5] This is indicated as the XY orientation.

2. Materials and Methods

Test specimens are printed using a Felix Pro 1 3D printer on a bed made of Mylar, with coatings of hairspray between prints to improve adhesion between the polymer and the bed. Five specimens of carbon-fiber reinforced polyethylene terephthalate glycol (PETG) and carbon-fiber reinforced Amphora AM1800 filament (Amphora) were printed in an XY orientation. Specimen geometry is tested in accordance with the ASTM D638 Type 1 sample standard. [6] Figure 1 shows the specimen dimensions and the extensometer used. Material properties at cryogenic temperatures were compared to the manufacturer’s ambient temperature properties and are listed in the results section.
Figure 1. Testing sample dimensions and linear extensometer setup on sample. All dimensions are in millimeters.

A custom extensometer with knife-blade edges was used to measure the strain on the specimens. Manufacturer data for the error in the extensometer are not available. The extensometer uncertainty is measured through calibration between room-temperature and 77 K. Table 1 lists the associated uncertainties for the devices used.

Table 1. Instrumentation Bias Uncertainties and Manufacturer

| Device             | Manufacturer | Uncertainty   |
|--------------------|--------------|---------------|
| 600DX Load Frame   | Instron      | 0.1 N         |
| Extensometer       | Unknown      | 0.005 mm/mm   |

A zero-strain measurement is taken with the extensometer for each sample prior to cooling with zero load. The distance between the contact points of the extensometer on the sample is measured to provide a gauge length. This zero-strain reading provided an initial indication that the change in gauge length could then be determined during the experiment. Stress is computed from the measured load divided by the measured cross-sectional area of each specimen. The average measured cross-sectional areas of the specimens are provided in Table 2.

Table 2. Average cross-sectional area of virgin specimens

| Material                        | $A_0$, $10^5$ m$^2$ |
|---------------------------------|---------------------|
| Carbon Fiber Reinforced PETG    | 5.032               |
| Carbon Fiber Reinforced Amphora AM1800 | 4.995              |
The cryostat design allows for full submersion of the specimen and clamps in liquid nitrogen. An unspecific aluminum alloy is used as the main body of the cryostat due to its high thermal diffusivity and easy machinability. Furthermore, a passthrough hole drilled into the bottom of the cryostat allows the bottom extension rod to fit. To seal the passthrough, silicone putty is compressed between the cryostat and the extension rod. The silicone’s malleability at room temperature is useful for filling gaps, and the putty is not significantly brittle at 77 K. A layer of aerospace insulation is wrapped around the cryostat to decrease boil-off. Figure 2 shows the load frame and the experimental setup diagram.

![Figure 2. Experimental setup diagram for tensile testing in liquid nitrogen.](image)

3. Results and Discussion

The ultimate tensile strength, percent elongation at fracture, and modulus of elasticity for the PETG were calculated for the XY prints. PETG showed an increase in tensile strength of 49% at 77 K compared to room temperature. This is represented by an increase from 55.5 MPa to 82.8 MPa. The PETG showed a decrease of 0.659 percent elongation at fracture when cooled, representing a decrease from 2.5% to 1.84%. The modulus of elasticity of the PETG increased by 43.2% from 4928 MPa to 7057 MPa. [7]

Similar tests to the PETG specimens are performed for the Amphora, however only ultimate tensile strength data could be collected for the Amphora specimens. It should be noted that there are changes and limitations to the Amphora tests due to maintenance on the load frame used for the tests. The Amphora specimens are moved to testing on a separate load frame and extensometer measurements are not available for these tests. Therefore, strain values for the specimens are not accurately reported.

The tests performed with this material show an average maximum tensile strength of 53.3 MPa under the same testing conditions of the PETG filament. This is a decrease in tensile strength
of 29.9% from a room temperature ultimate strength of 76.0 MPa to 53.3 MPa. [8] Table 3 lists the available ultimate tensile strengths and elastic moduli of the tested materials at room-temperature and 77 K.

Table 3. 77 K ultimate tensile strength and modulus of elasticity. Manufacturer room temperature values given in parentheses for reference.

| Material                  | Temperature (K) | Ultimate Stress (MPa) | Elastic Modulus (GPa) |
|---------------------------|-----------------|-----------------------|-----------------------|
| Carbon Fiber Reinforced PETG | (295)           | (55.5)                | (4.93)                |
| Carbon Fiber Reinforced Amphora AM1800 | (295)           | (76)                  | -^                   |
| Carbon Fiber Reinforced Amphora AM1800 | 77              | 53.3 ± 1.85           | -^                   |

^ Modulus of elasticity not available from the manufacturer.
^ Modulus measurements not taken.

Figure 3 compares the mean ultimate stress of the tested materials, with associated manufacturer data. Figure 4 shows the mean elastic modulus of the PETG.

![Mean Ultimate Stress](image-url)

**Figure 3.** 77 K mean ultimate stress with standard error. The blue bars represent the manufacturer data.
Figure 4. 77 K mean modulus with standard error. The blue bar represents the manufacturer data.

4. Conclusions

This paper presents tensile strength tests for two carbon fiber reinforced polymers at 77 K. The PETG shows results that agree with the general understanding of increasing tensile strengths as a function of decreasing temperature. The ultimate strength of the PETG increased by 49%, and the elastic modulus increased by 15.8%. The ultimate strength of the Amphora decreased by 29.9%.

The manufacturer data for the extensometer is unknown, therefore its bias uncertainty is calculated through calibration. Due to the nature of the experimental set-up it is difficult to ensure thermal equilibrium of the specimens, in part due to a lack of temperature measurement. Furthermore, partway through testing the experiment was moved to a different load frame therefore the associated uncertainties were not consistent throughout.

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