Drivers of mechanical performance variance in 3D-printed fused filament fabrication parts: An Onyx FR case study

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Abstract
Effects on mechanical properties of user-controlled and user-uncontrolled factors in a 3D printing system were investigated using a flame retardant carbon fiber reinforced nylon filament. Moisture and print orientation, both user controllable elements, dominated mechanical property variation. Dry samples exhibited tensile (flexural) strength 2.4 (2.1) times greater than that of wet samples, while ZX samples were measured to have about half the tensile strength of XY and XZ samples. User-uncontrolled factors including material to material variation and printer to printer variation were found to have minor impact (<5%) on part tensile strength. Reinforcing the part with 21 vol% continuous fiber lowers the moisture induced variance from 13% to 2% in addition to increasing the measured strength by about 10 × (to 451 MPa).

KEYWORDS
3D printing, fiber reinforcement, fused filament fabrication, mechanical properties, moisture, print orientation

1 | INTRODUCTION

The 3D printing industry has grown over 25% per year for the past decade.[1] As the industry has grown, many different types of high-performance plastics as well as new printers have come to market to meet functional needs. Today, with fused filament fabrication machines alone, we have the ability to print with hobbyist filaments such as PLA and ABS, as well as engineering thermoplastics such as nylon (PA), polyether ether ketone (PEEK), and polyetherimide (PEI). In the first phase of additive manufacturing adoption, 3D printing was often used for rapid prototyping—to design and test a part before the final part is machined or injection molded. A second phase, 3D printing for end-use applications, has gained momentum with end-use parts found in nearly every commercial sector. Designing 3D-printed parts for end-use applications introduces new design difficulties inherent in the various print parameters, materials, and end-use environments the part may experience. Due to the layer-by-layer method to create 3D-printed parts, it is vitally important to understand the different variables and how they affect the final mechanical properties.

Researchers have investigated property–structure relationships in 3D printing to understand the effects of several parameters. Many studies investigate common print parameters such as print temperature, infill angle, infill density, bead width, and layer height, among others.[2–10] For example, variations of up to 50% in the mechanical strength of 3D-printed samples have been demonstrated when tuning the printing design process.[2] Additionally, Afoze et al. reported a 20% increase in strength for unreinforced PLA by printing the infill at 0° compared to at 90° with respect to the loading...
direction.\[^3\] Furthermore, Durgun et al. tested build orientation with respect to mechanical strength and found samples printed in the upright orientation exhibited \(~50\%\) reduced strength compared to beams printed flat.\[^4\] Nevertheless, there remain less discussed, but no less important, parameters that also affect the mechanical properties of the final part. For example, multiple parts are often printed on the same build plate to improve printing efficiency and reduce downtime; however, the effects of a multiple-part build on the mechanical strength of the final part are not well explored in the literature. As another example, many commonly used 3D printing filaments have hygroscopic properties and therefore understanding the moisture absorption behavior and predicting the part properties in the end-use environment is vital to the design process.\[^11\]-\[^13\]

We report here a case study of Onyx FR, a flame-resistant, nylon-based filament reinforced with discontinuous carbon fiber, and we examine the relative effects on mechanical properties of user controllable and user noncontrollable factors. The Onyx FR is a certified plastic with an Underwriters Laboratory (UL) Blue Card; therefore, the production is audited each year. External validation of the materials composition and properties (V-0 flammability rating) ensures relevance of this study throughout the product life cycle.\[^14\] Similar to other hygroscopic polymers, the strength of nylon-based filaments exhibits moisture sensitivity, which we explore in this study. In addition, we explore user-controlled effects such as build orientation, printing single or multiple parts on the same build plate, and the location of the print on the build plate, as well as effects ultimately out of the user’s control such as, filament spool-to-spool variations, filament lot-to-lot variations, and printer-to-printer variations. Furthermore, we investigate the effects on the final mechanical properties of replacing the nozzle and recalibrating a printer’s nozzle temperatures. In each test scenario, control samples following ASTM standards were included for comparison. The same control settings were used when possible such that an overarching control variance could be determined and the experimental parameters could be put in appropriate context. The results of this study identify factors that cause the highest variation in mechanical strength in the Markforged composite printing ecosystem, and inform readers how to better design their end-use parts to account for these variations. This study is not meant to represent a qualification suite, such as provided by the National Center for Advanced Materials Performance; however, it can serve as a guide on property variation in the Markforged system, and can be used as a foundation to see if similarities exist in other printing platforms.

2 | EXPERIMENTAL

Many factors affect a 3D-printed part’s material properties. To simplify the complexity and clearly exhibit cause–effect relationships, a baseline was established and specific characteristics were varied and tested. For comparison of results, mechanical properties were analyzed—primarily tensile strength as it is most commonly compared.\[^5\]

All of the printing material and printers used in this study were supplied from Markforged (Watertown, MA, USA). Materials tested were Onyx FR (a UL94 V-0 rated micro-carbon fiber reinforced nylon) and Continuous Carbon Fiber (CCF). V-0 is the most stringent UL94 classification (most flame resistant), for plastics that, among other characteristics, self-extinguishes a flame within 10 s. Test specimens were printed on Markforged’s Industrial X7 3D printer using the company’s Eiger Software. The printers were leveled using the laser automatic bed leveling feature. All samples were printed using 100% solid infill at 0\(^\circ\). Onyx FR tensile dogbone samples were printed in the ASTM D638 Type 1 configuration (50 mm gage length, 13 mm width, and 3 mm thickness) and tested following ASTM D638. Rectangular flexural beam samples (140 mm length, 10 mm width, 6 mm thickness) were printed in the XY orientation and tested via ASTM D790. Rectangular CCF tensile samples (i.e. Onyx FR reinforced with CCF, 175 mm length, 25 mm width, 3.6 mm thickness) were printed in the XY orientation with 8 vol% and 21 vol% CCF and tested following ASTM D638. Tensile testing was conducted on an Instron 3369 with a 50 kN load cell and serrated wedge grips. Strain data were collected using an Instron SVE 2 Non-Contacting Video Extensometer. After conditioning and testing the specimens, moisture of a cross section of the beam was analyzed with an AMETEK Brookfield Compturac® Vapor Pro® XL. Mass of beams as printed and after respective conditioning periods were also used to confirm moisture content.

Experiments done in this study can be categorized into three groups: parameter-to-parameter, material-to-material, and printer-to-printer. Our standard control test specimens were printed four at a time in the center of the print bed in XY orientation and conditioned at 52% ± 4% relative humidity (RH) for 44 ± 1 h. This conditioning is consistent with ASTM D638, but with tighter limits so that factors could be thoroughly probed.

2.1 | Parameter-to-parameter

The effects that moisture, part orientation, number of parts per build, and print bed location have on tensile properties of a 3D-printed part were examined. These are all parameters that a user can control when preparing or using samples.
Effect of moisture was tested by conditioning printed samples in three different humidity environments (dry, 52% RH, wet) for specific lengths of time. Dry conditions were created with clay desiccant packs (2 Unit Pak, Desiccare, Inc., Las Vegas, NV, USA) stored in a 9.5 L air-tight container. The 52% RH chamber was created with a saturated magnesium nitrate hexahydrate solution (ACS grade, HiMedia Laboratories, Kennett Square, PA, USA) and monitored with a wireless temperature and humidity sensor (EasyLog EL-WiFi-TH+, Lascar Electronics, Erie, PA, USA). A wet environment was simulated by a container filled with distilled water. Test samples were placed in these environments at ambient temperature (23°C) for set durations (41 h, 44 h, 47 h, 1 week, and 2 weeks, respectively), then removed for immediate mechanical testing.

Effect of part orientation was tested by printing dogbone specimens in XY, XZ, and ZX orientations (as illustrated in Figure 5B). These beams were printed four per build. The XZ orientation includes printed breakaway supports under the thinnest portion of the dog bone, which were manually removed before mechanical testing.

The effect of the number of parts per build was tested by printing one sample per build four times and four samples per build one time.

Effect of print bed location was tested by printing samples one at a time, in the center and in each of the four quadrants (see Figure 6B for schematic).

### 2.2 Material-to-material

Effect of spools within a lot was tested by printing samples from Onyx FR Spool IDs EX01_OFR_190823_0244 and EX01_OFR_190822_2040 referred to as Spool 0244 and Spool 2040, respectively. These two spools were made using the same extruder, but 4 h apart.

Effect of different lots was tested by printing samples from Lot 6879 and Lot 6920. In this context, a lot refers to a distinct production compounding run, which incorporates different batches of raw materials (e.g. nylon resin, micro carbon fiber).

### 2.3 Printer-to-printer

Effect of a specific printer was tested by printing samples with the same settings on three different X7 printers.

Printer components which were hypothesized to effect properties were the nozzles and heater elements. Effect of the nozzle was tested by replacing the nozzle and reprinting samples. Effect of heater elements was tested by completing a nozzle temperature calibration. Using a custom built temperature probe fixture, the error between the actual temperature and set point was determined, and the set point was reset such that the actual temperature hit the desired temperature. This resulted in ~5°C offset from the original value.

### 3 RESULTS AND DISCUSSION

In order to compare factors which affect mechanical properties of Onyx FR-printed samples, we selected a baseline sample and condition. This condition aligns with the Markforged Datasheet, and consists of an ASTM D638 Type 1 style dogbone, printed in solid infill, 4 beams at a time in an XY orientation, and conditioned for 44 ± 1 h, at 52% RH ± 4%. We refer to this as the control condition, and typical stress–strain curve results for tensile testing are shown in Figure 1.

First, we discuss factors that an end user can readily control. Second, we discuss material supply factors. Third, we discuss printer supply and maintenance factors. Factors which an end user can readily control include (1) the environmental condition of the part, as measured by retained moisture; (2) the print orientation of a part; (3) the location on the print bed that a part is printed on; and (4) whether a single part is printed at a time or a build of multiple parts is printed at once. Factors that cannot be controlled by the user include material supply such as variations in spools from the same lot and variations between lots, as well as printer to printer variations, effects of changing the print nozzle, and temperature calibration drift. Finally we report the results of loading continuous carbon fiber within the part to reduce the effects of the most prominent variation observed in the study, moisture.
3.1 Environmental conditions (moisture)

Moisture is well known to act as a reversible plasticizing agent in nylons.\textsuperscript{[12–13,15]} As can be seen in Figure 2, the humidity conditioning of a 3D-printed Onyx FR part can affect the tensile strength by a factor of 2.4, and represents one of the most dominant sources of variance that we see in this study. A dry sample exhibits an ultimate tensile strength of almost 60 MPa, while a sample soaked in water for 14 days has a strength of 25 MPa. Of note, the authors have found that many material suppliers and some 3D printer manufacturers will quote the properties of dry samples in their technical data sheets; thus, it is worth evaluating the properties in the expected use environmental conditions.

Properties change significantly over time, until equilibrium with the environment is met. For instance, in a humid environment ($\sim$52% RH, 23°C) a printed part can decrease in tensile strength by 20% in a week, before leveling off. The change is the most prominent in the first day, which is a reason that the ASTM Procedure A testing conditions used as the control in this study recommend at least 40 h of conditioning. As can be seen in Figure 3, testing at 41 h compared to 44 and 47 h has negligible impact on the results, and it is not a significant source of variance seen in this study. This is fortunate as it takes several minutes to test each sample, and so a large sample set could span several hours. Corresponding with the strength decrease is an increase in the elongation at break of the part. For instance the tensile strain of elongation increases from about 10% for a dry part to about 30% for a wet part (Figure 3B). Although a part continues to absorb water when submerged, after 41 h, the mechanical properties stabilize.

To better understand the effect of conditioning, we took moisture content measurements of the beams after each break. As seen in Figure 4, there is a clear relation between moisture content and flexural strength, similar to the trends observed in the tensile studies. The equilibrium moisture content is determined by the environmental humidity and will vary depending on the end-use application. In a desert environment, with RH of 0%–20%, the samples exhibit a more brittle but stronger behavior. In Massachusetts, USA, we routinely measure the equilibrium moisture content of a printed part to be $\sim$1.6%, although this varies with seasons and is dependent on temperature and humidity. In a submerged application,
such as some of those used in marine environments, one can expect a part to absorb up to 8% water. Although Kikuchi et al. showed a higher water absorption (9.7%) for the Markforged Tough Nylon material, Onyx FR has additives that do not uptake water.\cite{16} Furthermore, when reporting moisture, it is important to note that artifacts due to measurement techniques are possible. For instance, many nylons contain caprolactam at up to 10 wt% which vaporizes at elevated temperatures. Thus, a thermal moisture balance would provide erroneously high moisture results and is not recommended. Karl Fisher titration and moisture-specific sensor methods can provide reliable moisture measurements in this system.

3.2 | Print orientation

Interlayer adhesion in 3D-printed materials relies on local polymer melting of the previous layer during deposition of the active layer. The layer-by-layer deposition process has been shown to adversely affect interlayer strength, often represented by the Z-strength of the part.\cite{2,4} It is common for manufacturers to claim Z-strength anywhere from 16%–90% of the properties in the XY direction.\cite{17–18} As seen in Figure 5, we also see anisotropy in the tested parts, with ZX samples measuring at about half the strength of the XY and XZ beams.

3.3 | Single versus build

The mechanical strength, specifically in the Z-direction, can be sensitive to long delays between printed layers that cause the polymer to cool and reduce interlayer adhesion. As the number of parts per build increases, the delay between each deposited layer increases, which can lead to a decrease in the interlayer strength. For our standard XY beams printed with one beam per build versus four beams per build, the force applied during mechanical testing is perpendicular to the layers, so any impact on the interface is not expected to impact the measured properties, which is consistent with what we see in this study (Figure 5).

Although not shown, we have observed that multiple-part builds affect strength on thin features. The Z strength is particularly reduced for small features as each layer has time to cool before the next layer is deposited. In previous studies with Onyx filament material, we have seen that printing hollow towers and then machining out dogbones or printing ZX beams one at a time can increase the ZX strength by 25%–30%.

3.4 | Print quadrant

Temperature uniformity, bed leveling, and material deposition paths were hypothesized to be different in the four
quadrants which could affect the overall mechanical performance. We examined the tensile strength of parts printed in the center and each quadrant of the print bed, as shown in Figure 6. No significant difference was observed between quadrants in the X7 system, although there is a slight decrease of strength (~2 MPa) measured in the center of this sample set. As the default print location is the center, this effect, albeit small, could be due to wear on the print bed.

3.5 Material supply

Material supply factors include the variance expected between two spools of a single lot of material, and between spools from two lots of material. Both filament area and material composition could affect how a part prints and, in turn, properties of that part. As the filament is a purchased consumable, an end user would not be able to control for this variation. In this study, we observed strengths of parts from the two spools of a single lot (Spool IDs 0244 and 2040 respectively, produced 4 h apart) and of parts from two lots (Lot IDs 6879 and 6920 respectively, produced 2 months apart) to be about 45 MPa, in line with the control sample (Figure 7).

3.6 Printer and printer component variances

Printer supply and maintenance factors include printer-to-printer variation in general, the effect of switching nozzles, and the effect of temperature calibration drift. A printer is a complex machine with many factors that can affect print quality and part properties. In some printers, there are many manual steps which can add to the variability. The X7 used in this study has auto-bed level functionality, and other maintenance was followed as per the user guides. Three X7 printers manufactured over a year apart were tested and resulted in samples with a tensile strength between 41 and 46 MPa (Figure 8). To further test potential differences between units, we swapped out the primary consumable, the nozzle. No significant deviation was seen, implying that either the nozzles are very uniform or that they do not play a strong role in affecting mechanical properties of the printed part. Lastly, we retuned the temperature calibration of a print head to see how sensitive the part properties were to this machine state. About a 5°C adjustment was made, yet, no significant change in tensile properties was observed with this change.

FIGURE 6 (A) Tensile strength versus print location and comparison of single part printed to four parts printed in one build for Onyx FR dogbones. Orange represents one sample per print, while purple represents four samples per print. Quad is short for quadrant. The green shaded region indicates two standard deviations above and below the typical control sample. (B) Schematic depiction bed locations, as generated in Eiger software

FIGURE 7 Tensile strength versus material supply conditions. The green-shaded region indicates two standard deviations above and below the typical control sample
Through these studies, we can clearly state that two factors dominate the properties expected from Onyx FR printed parts—moisture and print orientation. In fact, deviations in moisture uptake—from changes in humidity, temperature, or soak time—quickly overwhelm all other factors measured except for ZX print orientation, and, if not carefully controlled for, moisture level can dominate tested values. While moisture acts as a plasticizer and increases elongation at break and part toughness, the ZX orientation has reduced elongation at break in addition to reduced strength. The stress–strain relationship based on the conditioning environment and print orientation studies is highlighted in the Ashby type diagram found in Figure 9. For pure Onyx FR parts, it is important for engineers to design for the environmental use condition they expect the part to be in. Furthermore, it is also important to align stresses in-plane with the print direction when possible to maximize part strength.

An effective way to overcome limitations of the pure plastic part, especially the variance due to moisture uptake, is to incorporate continuous fiber into a part. Tensile samples were prepared with 8 vol% and 21 vol% CCF reinforcement and conditioned at 52% ± 2% RH for 47 h. A part reinforced with 8 vol% CCF achieved an ultimate tensile strength of 240 MPa strength dry and 221 MPa strength conditioned. Increasing the CCF loading to 21 vol%, we see a strength of 460 MPa dry and 451 MPa conditioned. While the tensile strength drops by about 13% for Onyx FR-only parts, parts reinforced with continuous fiber experience only an 8% and 2% reduction in strength with 8 vol% and 21 vol% CCF, respectively. This result aligns with the work of Kikuchi et al. who saw no significant change in tensile or compressive strength between wet and dry samples.\textsuperscript{[16]} Their study did note a decrease in modulus for wet composite samples (a behavior we did not see in this study). This difference could be explained by the different plastic matrix materials used in the studies (Tough Nylon in their study versus Onyx FR in the present study). The relationship between moisture and dimensional stability was out of scope of this study; however, one can expect to see similar benefits with reinforcement in this area as well.

This study looked at 3D part printed performance; however, 3D filament properties are often reported from injection molded values. Our testing of injection molded parts, flow along the direction of the beam, shows ultimate tensile strength values about ~40% higher than 3D-printed parts. While Markforged optimizes for part quality, with additional strength needs able to be met with continuous fiber reinforcement, other manufacturers may optimize for part strength. Even so, Kim et al. report printed strength values are 26%–56% lower for printed parts than the injection molded reported value, which would match expectations from this study.\textsuperscript{[19]} Furthermore, if a dry value is reported and the material is water sensitive, like nylon, to be comparable to a conditioned part a further drop in strength by 20% may be expected.

Although 3D printing enables arbitrary parts to be built, to compare across a broad suite of conditions, we limited ourselves to testing solid infill tensile dogbones.
and flexural samples, as outlined in the Experimental section. These simplistic samples provide relative insights into the properties of printed parts; however, we acknowledge that specific parts will have much more complex interactions, which have been discussed more extensively in other studies. For instance, Terekhina et al. show that infill parameters can greatly affect part properties. Specifically, they tested five different inﬁlls in 3D-printed nylon with dogbones printed at 20% inﬁll having an average inﬁll tensile strength of 0.83 versus 32.7 MPa printed at 100% inﬁll. Another complex interaction is that the time between layers can affect the surface temperature of parts, as well as the cooling dynamics—a factor that has been shown to affect semi-crystalline and amorphous polymers alike. Delving into these factors has led to rich studies in themselves. Faes et al. quantify the effects that interlayer cooling time has on ABS part tensile properties. They accomplished this by varying the number of parts per build in a print and discovered that printing 1, 3, and 5 XY dogbones per build had little effect on tensile strength, similar to what we saw in our data. However, when dogbones are printed in ZX, strength for a one-part build is 20.5 ± 2.5 MPa compared to 13.3 ± 0.44 MPa for a 10-part build. Yang et al. discuss how crystallinity and temperature affect part properties—both during printing and in post-treatment conditions for PEEK. By printing at different ambient temperatures (20–200°C), they varied the crystallinity of the material (21.3%–32.5%). Their results showed that as crystallinity increases, tensile and ﬂexural strength increase and elongation at break decreases signiﬁcantly. For example at a crystallinity of 21.3%, PEEK parts had a strength of about 49 MPa, while at 35.2% crystalline, the strength was about 138 MPa. Controlled testing such as completed in this study continues to help users understand the expectations from 3D-printed parts, and is important to build trust within industry as adoption for end-use parts continues to rise.

4 | CONCLUSION

The interlayer adhesion, or Z-strength, is an often cited 3D printing design consideration, and our results conﬁrm that print orientation is a major source or mechanical property variation in printed parts. Speciﬁcally, a ZX part can have less than 50% the tensile strength of an XY or XZ sample. Even so, arguably, moisture has an even more profound effect on the tensile and flexural properties of a 3D-printed Onyx FR part. While a dry part exhibits an ultimate tensile strength of ~55 MPa, a similar part soaked in water becomes more elastic and tougher, with a strength of ~27 MPa. Because of the growing utilization of 3D printing for end-use parts, taking into consideration the type of environment the part will be in is important, especially for 100% Onyx FR parts. No other factor analyzed including material-to-material variation and printer-to-printer variation had a signiﬁcant impact in the part properties.

Although part properties were shown to be highly dependent on moisture content, this can be mitigated with the inclusion of continuous ﬁber reinforcement. In addition to signiﬁcantly increasing the tensile properties (to 451 MPa for 21 vol% loading), continuous carbon ﬁber reinforcement was shown to reduce the strength variation due to moisture conditioning to <2%. As 3D-printed parts continue to be incorporated to end-use applications, building an understanding of and trust in the as-printed properties will have increasing importance. Studies like this one, or undertaken through standard bodies or national testing facilities, will be essential to build this trust.

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