Chapter 8
Assessing the Effects of Strain Rate, Print Orientation and Post Print Annealing in FDM PLA Specimens in an Undergraduate Materials Science Course

Matt Schaefer

Abstract This paper describes the development of a lab experiment for an undergraduate Materials Science course which demonstrates the influence of print orientation, strain rate and post-print annealing in FDM specimens of PLA polymer. The objective of experimentation in a materials science course is for students to have firsthand experience with common material property tests and understand how design properties are determined from those tests. But materials selection is a first step, not the end of story. A stretch goal is to understand some subtle factors beyond just designing to tensile, yield, and % elongation from a standard test. This experiment utilized tensile test specimens that were created via FDM using three different print orientations. This provided a clear demonstration of the anisotropic nature tensile properties in FDM. As expected, cross-layer mechanical properties are significantly reduced in specimens tested across print layers. In a second part of the experiment, influence of strain rate (testing at 0.5, 15 and 500 mm/min) was successfully demonstrated, with 20% higher tensile strength and lower elongation for the highest strain rate. Additionally, some samples were annealed after printing to improve mechanical properties. Results for this were mixed and mostly demonstrated the practical difficulties with trying to improve properties of PLA printed parts via heat treatment. For students, this lab was effective for the target objective and the stretch goal: they correctly analyzed tensile results and started to appreciate some of the finer points of material selection within mechanical design.

Keywords Fused Deposition Modeling · Tensile Test · PLA · Strain Rate · Print Orientation

Introduction

The idea that students learn best in their preferred learning style is not backed up by the research. Far more effective is to present concepts using a combination of teaching modes. Even more important is to suitably match the mode of delivery to specific course topics [1] [2]. Materials Science is well suited to experimentation for course topics involving concepts we can see and feel and test. These would include: How are polymers different than metals? What are mechanical properties and how do we test them? These questions are the focal points for an experiment involving tensile testing of FDM printed SLA polymer specimens. The course in Materials Science 1 is required third year course in the mechanical engineering program at Milwaukee School of Engineering (MSOE). A follow-on course, Materials & Manufacturing Processes is also required.

A good basic experiment will support a lecture topic with a physical demonstration of some principle of materials science in line with stated course learning objectives. And in this case, the "ability to perform experiments" is also a stated course outcome. A great experiment will go beyond this basic approach. Curriculum is a continuum, and experiments provide a good opportunity to review material learned in prerequisite courses and to preview material that students may study in future courses. Planning a lab-lesson can follow three basic questions; 1) What do we know already? 2) What are we learning today? 3) How does this lead into things you will learn in the future? As an example, mechanics of materials is prerequisite course for materials science. Students already know the concept of Young's modulus and that it describes elastic behavior up the yield. Or does it? When watching a tensile test in progress, there is time to point out that the "linear" portion may not really be so close to linear. When is this important? If those students take a future course in Fatigue & Fracture in

Matt Schaefer
Department of Mechanical Engineering, Milwaukee School of Engineering, 1025 N. Broadway, Milwaukee WI 53202 USA
e-mail: schaefma@msoe.edu

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Mechanical design, they will learn that this “slight” deviation (which occurs below yielding) is the key factor in describing the accumulation of fatigue damage in metals. These points are part of the in-lab discussion.

The same past-present-future approach applies within the course. One course learning outcome for the materials science is: Identify typical properties… for broad categories of materials (metals, polymers, ceramics, and composites). A week-1 course assignment was to simply do MatWeb research and compare mechanicals for metals versus polymers versus ceramics [3]. In a week’s prior experiment, students did tensile testing on metals. Following up with tests run on polymer samples provides the physical demonstration of the differences: 1) polymers are much lower strength and lower modulus materials, 2) polymers can be strain-rate sensitive, 3) polymers are, in general, more non-linear from the start of the stress-strain response. These three points support the primary course objective, to answer what is different about polymers versus metals. The second half of the materials course sequence concentrates more on manufacturing processes. Consideration of fabrication method and post-processing (FDM printing, print orientation as an experiment variable, post-print annealing) are not really necessary for the objectives of the basic materials 1 course. But these concepts do support course objectives for the follow-on manufacturing course.

Background

The lab experiment, which is the subject of this paper, aims to demonstrate typical mechanical properties of polymers. This could be accomplished using injection molded or machined test specimens. However, basing the experiment on 3D printed specimens helps spur interest and curiosity in the students. Simple FDM printers using PLA polymer are familiar to all the students, and many have made parts via this method. Tensile testing is the most fundamental technique for obtaining important design properties of strength, ductility and elastic modulus. The method used here follows closely to the ASTM D638 Standard Test Method for Tensile Testing of Plastics [4]. To expand beyond the basics, three additional elements were added to the experiment, effect of print orientation, effect of strain rate, and possible improvement of properties by post-print annealing.

With FDM printing, it is a well understood phenomenon that the mechanical properties will depend on print direction. The nature of the print method will produce incomplete bonding between successive layers as well as residual flaws or voids in the layering. These factors result in lower strength and elongation in tensile properties. Much data is available in the literature. Some FDM filament suppliers will provide data regarding this influence. Overture technical data sheet for PLA [5] shows tensile strength as 47.1 MPa in X-Y directions (lateral) and 38.9 MPa in the Z direction (vertical). Modulus, however, is only reported in the lateral direction, as E = 2641 MPa. Another supplier for materials used in this study, Polymaker - PolyLite™ PLA [6], gives tensile strength at 52.3 MPa in X-Y directions versus 40.5 MPa in Z direction, and also E = 3426 MPa in X-Y direction versus E = 3064 MPa in Z direction. It is expected that the current experiment would show similar results with those two materials. However, the current experiment explores the concept of “directionality” in a slightly different manner. Printing a flat tensile specimen flat to the printing platen, but in a different orientation is what the suppliers would refer to as X or Y. In the current study, an option of printing the flat sample but with the edge towards the platen is considered. This variation could be important when applying test results to practical parts made via FDM.

Polymers are more sensitive to changes in strain rate compared to other classes of material. Generally, increasing strain rate will increase both tensile strength and elastic modulus and will result in decreased strain to failure. While many polymers exhibit these trends, there is evidence that PLA is even more sensitive to strain rate effects compared to other common FDM materials [7] and therefore PLA is a good choice for demonstrating the effect in the current experiment. In one study the authors used only modest variation in crosshead speeds, from 1 to 5 mm/min, and still observed the effects [8]. Other studies used much wider range of speeds, from 0.001 to 100 strain/minute, and demonstrated approximately double the tensile strength and modulus at the higher strain rates [9].

Annealing of printed parts is a possible remedy for some of the problems associated with inter-layer weakness in FDM. Effectiveness of such treatments requires careful selection of both the annealing temperature and the cooling rate after annealing. If done correctly, post-print annealing can change the degree of crystallinity of semi-crystalline polymers, which then can improve mechanical strength and modulus, but with some loss of toughness [10]. Annealing at sufficient temperature followed by rapid cooling may have two-fold effect of 1) reducing internal flaws in essence “healing” some of the inherent voids produced during printing, and 2) promoting more amorphous character in the polymer. If samples are cooled slowly post-annealing the results can be worse than as-printed samples. In the current study, this was a factor in the experiment but was not thoroughly researched to achieve a workable yet beneficial annealing and cooling protocol. The trick is to obtain the desired benefit but without causing undue warping or dimensional changes in the specimens. In Hart’s study, annealing was carried out at 160°C for 2 hours (relative to T_{MP} of 147°C for the polymer) with 10 different cooling rates tested in that study. Cooling rate was a significant factor related to properties of the final samples [10].

Analysis

Materials & Methods

Tensile specimens for this study were made of FDM printed PLA polymer, Overture PLA 3D printer filament on a Prusa™ model MK3™ printer. Printing process parameters were within standard recommended for the material:

Table 1 Material, printer and print parameters used in this study.

Material	Overture® PLA 3D printer filament
Printer	Prusa® MK3
Nozzle Temperature	210°C (215°C for the first layer)
Bed Temperature	60°C
Print Speed	80 mm/sec.
Fill	100%
Samples tested in as-printed condition	For orientation & strain rate
Annealed samples for comparison	Annealed 120°, 2 hours, water quench

Two different part files were used to produce the test specimens. The first part file contained 6 total specimens: two printed lengthwise to the bed, two printed edgewise to the bed, and two printed flat to the bed [Figure 1]. These were used in testing to assess the influence of print orientation on tensile properties. These print pattern (three orientations) was also used in the study of annealing response of 3D printed samples. The second part file contained 6 total specimens with all parts produced flat to the print bed. These flat-printed samples were used to assess the effect of strain rate.

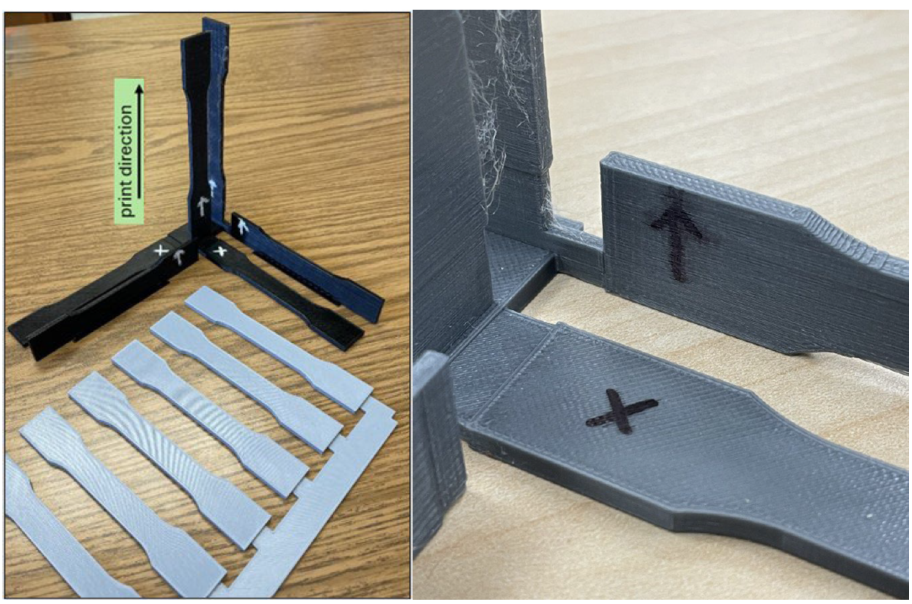

Fig. 1 Two print files for tensile specimens used in this study. Parts marked "x" were printed flat to the printing bed. Up arrow on the other specimens indicates the build direction.

For evaluating print orientation and strain rate, specimens were used in as-printed condition. For the third part of the experiment, samples were annealed prior to testing. The annealing process involved placing samples into an aluminum holding fixture, to prevent warping or dimensional changes of the samples [Figure 2]. The fixture and samples were placed in a lab furnace at 120°C for two hours. The entire fixture with samples was removed and quenched in room temperature water for at least 5 minutes, after which the specimens were removed from the holding fixture.

Tests were run at room temperature. Samples were printed about two weeks prior to testing and stored at room temperature in the lab, with no special pre-conditioning prior to testing.

Dimensions for the test specimens are following ASTM D638, Standard Test Method for Tensile Properties of Plastics for a Type I specimen. There was one minor difference. To accommodate the physical size limitations of the 3D printer

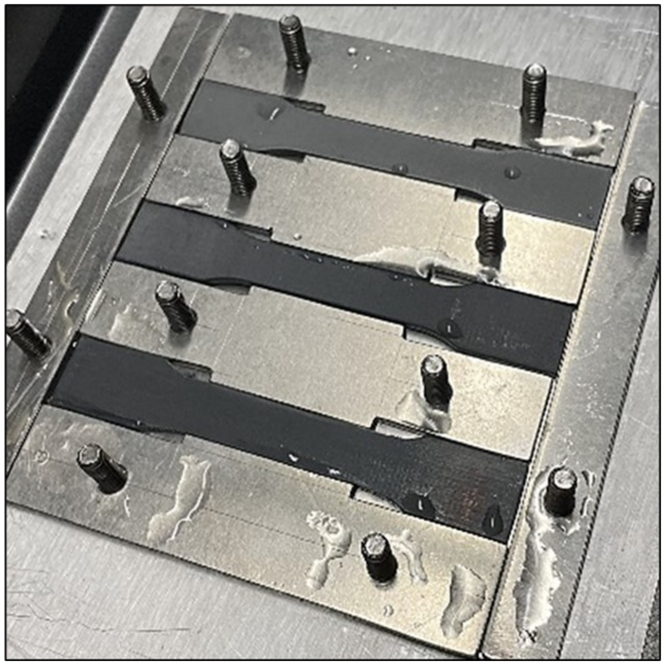

Fig. 2 Holding fixture used to restrain the specimens during annealing and subsequent quenching. There is an aluminum top cover (removed in the photo) to hold the specimens flat and reduce warping.

(specifically, for printing the lengthwise specimens) the overall length of parts was reduced and thus the length of the narrow section of the specimen was also slightly reduced. The narrow test section of the specimens was 50 mm long (versus 57 mm according to the standard), 13 mm wide, by 3.2 mm thick [Figure 3].

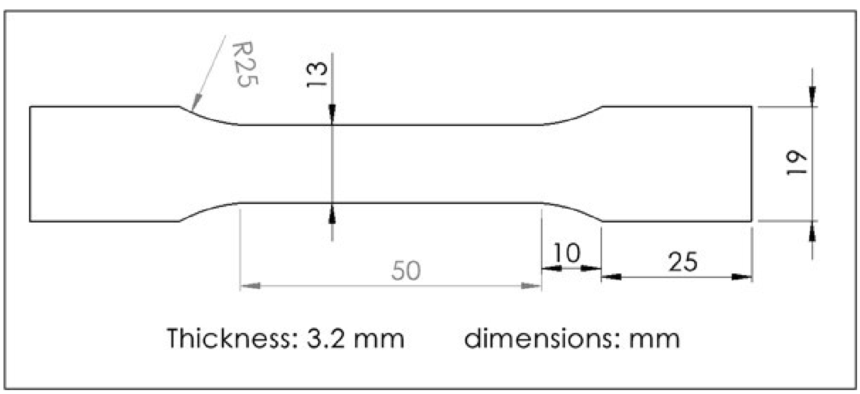

Fig. 3 Nominal dimensions of test specimens are slightly modified from ASTM standard, with reduced length in the middle section. The was done to accommodate printing in the lengthwise orientation in the Prusa printer used.

Method 1 & 3 – Print Orientation & Annealing Response

Tensile testing was done on an Instron 6800-series machine with a model 2580 (50 kN) load cell. Specimens for testing print orientation and annealing response were run at crosshead speed of 0.5 mm/min., for a nominal strain rate of 0.01 mm/mm per minute (1% per minute). This rate is in accordance with the standard, which states "use the lowest speed that produces rupture in 0.5 to 5 minutes." Strains in the experiment were not measured directly using an extensometer, rather they are derived (approximated) in the BlueHill software (Instron) based on crosshead displacement and specimen length in the reduced cross section area. This was done as a compromise which allowed quicker testing and more samples to be completed in limited lab time. All specimens were tested to rupture. Stress and derived strain were recorded as the representative results.

All specimens for annealing and print orientation tests were of parts printed in 3 different orientations in one file, the only difference was in the sample preparation. Testing during lab time was carried out by the instructor, with students observing.

Method 2 – Strain Rate

The only procedural change in the strain-rate method is that crosshead speed was set at three different rates: 0.5 mm/min, 15 mm/min and 500 mm/min (the maximum for the machine). This produced nominal strain rates of 1% per min., 30% per min., and 1000% per minute for the specimens. All specimens for strain-rate testing were from the flat printed parts.

Results

Experiment 1 – Print Orientation

Lengthwise printing clearly underperformed in terms of elongation and strength. This was the expected result. Print layers across the tensile stress area would lead to lower strength and ductility. Test results are shown in Figure 4 and summarized in Table 2. There was only a slight decrease in tensile modulus with print orientation in the lengthwise direction. Between the edgewise and flat printed specimens, edgewise produced the highest strength, while flat-printed produced the highest elongation to failure. In some preliminary testing, the elongation measurement seems to be quite variable, therefore the limited data here does not support a definitive conclusion. One possible explanation would faster cooling of each layer for the edgewise samples. That could potentially change the crystalline/amorphous balance and thus the mechanical properties. This was a repeatable and likely real effect and not just a test anomaly. But more data are needed to make a conclusion.

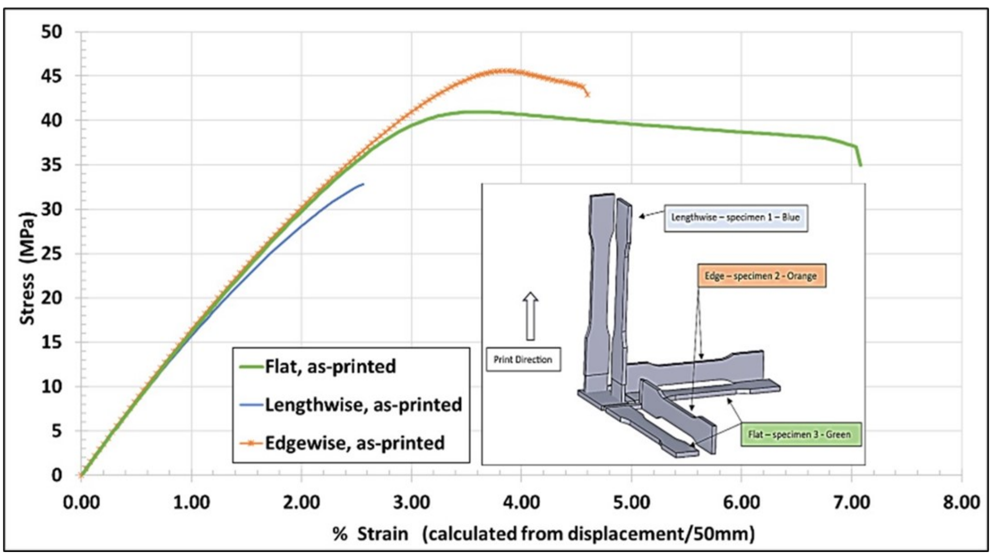

Fig. 4 Stress-strain results comparing the 3 different print orientations.

Table 2 Summary of mechanical properties comparing the 3 different print orientations. Above samples as-printed and tested at 0.5 mm/min (nominal strain rate of 1% per minute).

	Modulus, E [MPa]	Tensile Strength [MPa]	Elongation at Break [%]
Lengthwise	1,570	32.8	2.6
Edgewise	1,640	45.6	4.6
Flat	1,610	41.0	7.1

The test results of this study agree reasonably well but slightly under what the Overture® technical data sheet gives (for comparison, the supplier's values: tensile strength, 47.1 MPa in x-y directions, 38.9.3 MPa in z direction). The ratio of strength in z-direction of 76 to 80% of maximum for the current study, is comparable to the same ratio based on supplier information (83%). Modulus values in the current study are lower compared to published values (Young's modulus, 2641

MPa). Elongation at break was not as high as reported in manufacturer's data (8.2 % in x-y direction, not reported for z-direction). As expected, lengthwise printing gave lower strength and ductility. Mixed results make is difficult to say which is "better" between flat-printed and edgewise. Flat printed had higher elongation but edgewise had higher tensile strength.

Experiment 2 – Strain Rate

Trends shown in the tests on strain rate demonstrate all three expected behaviors. As strain rate increases the ultimate tensile strength increases. As strain rate increases, the elongation to rupture decreases. As strain rate increases, there is a slight increase in the elastic tensile modulus. Test results are shown in Figure 5 and summarized in Table 3.

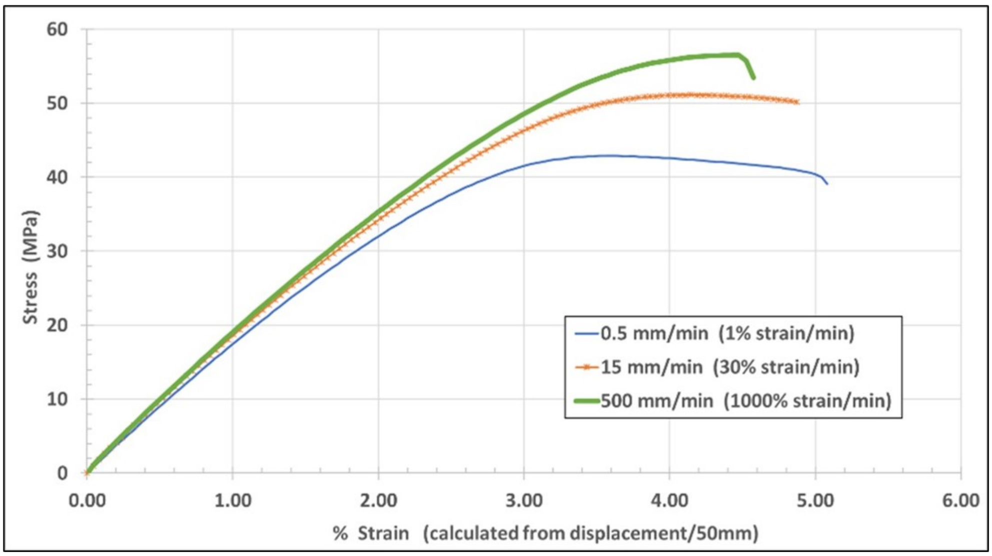

Fig. 5 Stress-strain results comparing strain rates. Flat-printed samples, tested in as-printed condition.

Table 3 Summary of mechanical properties, comparing 3 different strain rates tested.

Crosshead (strain rate)	Modulus, E [MPa]	Tensile Strength [MPa]	Elongation at Break [%]
0.5 mm (1%) /min.	1,750	42.9	5.1
15 mm (30%) /min.	1,870	51.1	4.9
500 mm (1000%) /min.	1,990	56.5	4.8

Experiment 3 – Annealing of FDM Samples, post-printing

Test results for this part of the experiment were counter to what was expected. Most significant was an approximately 30% reduction in elongation at break. It was expected that annealing after printing might improve elongation. Both strength and elastic modulus were improved, but only slightly (2 to 4% increase in strength). Figure 6 shows comparison for two of the annealed samples versus earlier data from the print orientation study.

This part of the experiment was most problematic, both in terms of devising a method for annealing and with generating clear trend in the results. A first attempt at annealing was done without the holding fixture and resulted in severe dimensional changes in the specimens. Using a holding fixture to restrain samples during annealing and cooling helped solve that issue. However, the entire process was quite cumbersome and time consuming.

Annealing response study needs further development to: 1) improve the ease-of-use of the holding fixture and 2) revise the annealing steps to come up with a more favorable response. Ideally, there would be increase in both strength and elongation. Variations in annealing temperature and cooling rate are the key parameters. Both these factors require further study and experimentation.

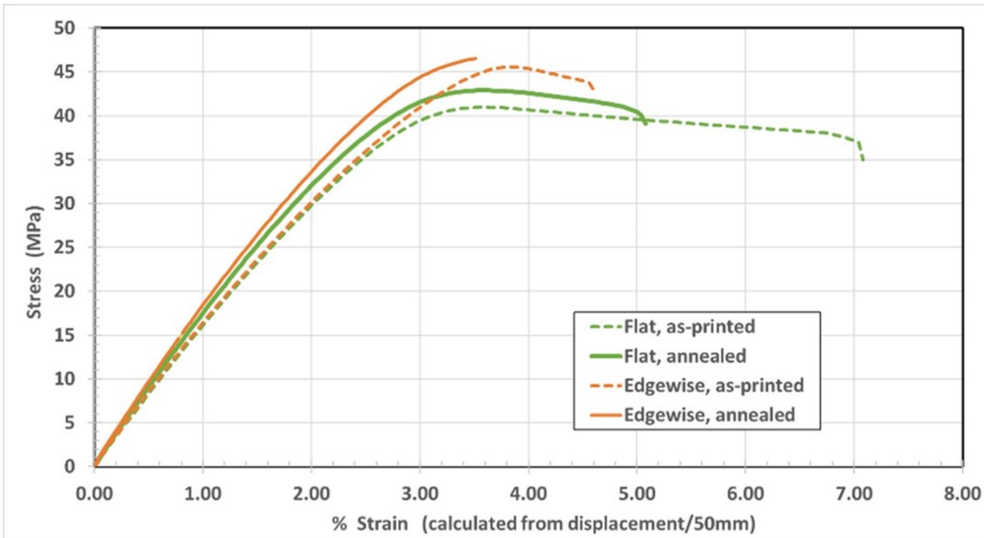

Fig. 6 Comparison of annealed specimens (solid lines) to as-printed specimens (dashed lines).

Table 4 Summary mechanical property comparison for annealed versus as-printed specimens.

	Modulus, E [MPa]	Tensile Strength [MPa]	Elongation at Break [%]
As-printed, Flat	1,610	41.0	7.1
Annealed, Flat	**1,750**	**42.9**	**5.1**
As-printed, Edgewise	1,640	45.6	4.6
Annealed, Edgewise	**1,890**	**46.6**	**3.5**

Conclusions

– Test results for print orientation demonstrated the expected results, were elastic modulus and tensile strength and elongation at failure were measurably lower for lengthwise printed samples.
– Test results for strain-rate also demonstrated that for higher strain rate, 1000% per minute versus 1% per minute, modulus increased by 13% and tensile strength increased by 30%.
– Annealing specimens post-printing proved procedurally difficult to accomplish in a way that both avoided warping and allowed rapid cooling after annealing. Strength was 9-15% higher with annealing, but elongation was about half.
– From the viewpoint of using this as an educational experience, the lab is a successful one. It clearly demonstrates differences in mechanical behavior of polymers versus metals. Additional concepts relating to prior courses can be effectively reviewed, and some new concepts relating to manufacturing processes are introduced.

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