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TENSILE MECHANICAL PROPERTIES OF POLYPROPYLENE COMPOSITES FABRICATED BY MATERIAL EXTRUSION

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ABSTRACT

In the material extrusion additive manufacturing process, a thin filament of material is deposited in a layer-by-layer manner to fabricate a three dimensional part. The filament deposition pattern can result in voids and incomplete bonding between adjacent filaments in a part, which leads to reduced mechanical properties. Further, the layer-by-layer deposition procedure typically results in mechanical property anisotropy, with higher properties in the layer compared to those across layers. The study reported in this paper explored various polypropylene composite formulations to address these issues: low residual stress and warpage, good mechanical properties, and reduced anisotropy. The reduction in anisotropy will be the focus of this paper as a function of thermal properties and process variable settings. A series of process simulation models was developed to explore ranges of thermal properties and process settings, which provided insights into tensile specimen behaviors. Results demonstrate that anisotropy can be reduced almost completely if the material can be formulated to have low crystallinity, low coefficient of thermal expansion, and moderate to high thermal conductivity (for a polymer).

1 INTRODUCTION

During the material extrusion (MEX) process, the part goes through a repetition of heating and cooling as the filament is liquefied in the liquefier chamber and is deposited onto a build platform to fabricate a three-dimensional part [1]. This filament deposition procedure causes voids to form in each layer, which reduces mechanical properties in the part. Furthermore, layer-to-layer bonding tends to be weaker than the filament strength, causing significant variations in mechanical properties in the layer vs. out-of-plane. In this paper, mechanical properties in tension of material extrusion fabricated parts are investigated as a function of process settings and material composition.

One of the challenges in material extrusion is the limited availability of materials. With additive manufacturing (AM) processes, many of the part geometries that are unachievable using conventional manufacturing processes can be realized. As different material compositions are investigated, AM technology will be improved further by expanding the portfolio of available materials. Polypropylene, a widely used thermoplastic that is inexpensive and flexible compared to acrylonitrile-*co*-butadiene-*co*-styrene (ABS), is the material of interest of this study. However, polypropylene is a semicrystalline thermoplastic unlike ABS, which is an amorphous thermoplastic, and there are processing issues associated with material extrusion of polypropylene. The molecules in semi-crystalline thermoplastics are drawn together and ordered during the crystallization process, so they shrink more than amorphous thermoplastics upon solidification [2]. This increased shrinkage causes parts that are fabricated with polypropylene to warp more and detach from the build platform, compared to those with ABS.

Alternatives to reduce warpage are to create polypropylene-based composite materials by combining polypropylene with additives and/or investigate polypropylene copolymers with reduced crystallinity. Several types of additives exist, such as particles, fibers, and agents that affect viscosity and thermal conductivity. In this study a total of 10 polypropylene formulations were investigated, of which 5 showed promise as a MEX material and were processable in our Hyrel System 30M machine [3]. Several composite formulations for some of these five polypropylenes were tested. Two materials were investigated further since they exhibited similar lack of warpage for their 3D bonding and mechanical properties. Although the materials processed equally well, they exhibited substantially different surface finish and levels of anisotropy in tensile mechanical properties. In this study, layer thickness, deposition (extruder) temperature, and fill angle were varied for the tensile specimens, while yield and ultimate strength and elastic modulus were measured. Note that all specimens were fabricated flat and horizontally.

2 LITERATURE SURVEY

Mechanical properties of parts fabricated using material extrusion are of great interest, as is the reduction in anisotropy. As is well recognized, properties are typically higher for parts built in the XY plane, compared to properties in the Z direction, since Z direction properties depend entirely on filament bond strength. In this process, bonds are weaker than filaments. Many researchers have investigated mechanical properties of MEX parts. In an early study, Rodriguez et al. [4] quantified the effects of mesostructure (road deposition pattern and pore size) on tensile strength and compared with monofilament strength. They also related process variables to pore size and mesostructure in order to identify process settings that maximize part strength through an understanding of bonding potential [5]. Sun et al. [6] showed that a correlation exists among road-to-road neck radius and flexural strength of test specimens which helps to explain these results.

More recently, a group tested tensile properties of parts in a Dimension system (Stratasys) with the ABS-M30 material [7]. Specimens were built flat, on edge, and vertically at various angles and tests indicated that properties were anisotropic, particularly for tensile strength. Parts built vertically were the weakest, as expected, since their strength was primarily dependent on bond strength between layers. Additionally, for the perpendicular specimens, the high surface roughness caused by layer boundaries and internal pores may have acted as stress concentrations and fracture initiation sites, which caused lower strength. Elastic modulus was fairly uniform across all sets of specimens and all orientations; interestingly, the highest values were for parts built vertically. It is important to note that elongation at break was highly dependent on orientation, with results of 7% for XZ orientation, while ZX orientations exhibited elongation of only 2%. These results are consistent with the Stratasys ABS-M30 specification sheet [8]. This trend indicated that while vertically built specimens may be stiff, they failed much earlier (lower load, less strain) than parts in other orientations.

Another group investigated tensile and compressive properties, as well as failure mechanisms, of ABS specimens built on a Zortrax M200 machine [9]. Similar anisotropic properties were reported. Failure mechanisms included ductile failures for some specimen and fill orientations, while other orientations exhibited fracture along filament interfaces, particularly for vertically built specimens. Compression results also exhibited anisotropy.

Of interest in this paper is research on polypropylene (PP) materials. One group compared tensile properties of two commercially available PP homopolymer extrusion grade materials, one a neat PP formulation and the other a glass fiber reinforced PP [10]. Specimens were fabricated on a Prusa i3, available from the RepRap platform. The authors reported significant part shrinkage and warpage. They investigated various fill orientations, infill percentages, and layer thicknesses; all specimens were built flat and horizontally. Tensile properties for the neat PP exhibited anisotropy for different fill orientations, which is consistent with ABS results referenced above. In contrast to [9], a larger layer thickness resulted in high ultimate strength. Little was reported about the glass fiber PP, except to compare strength and Young's modulus to the neat PP; properties for the glass fiber PP were significantly better. Another recent study [11] investigated impact strength of a different PP homopolymer composite material, where specimens were fabricated on a Makerbot Replicator 2X.

Two different extrusion temperatures (200, 250 °C) and layer thicknesses (0.1, 0.3 mm) were investigated. Results showed that specimens fabricated at the lower extruder temperature had significantly higher impact strength, which was comparable with injection molded specimens of the same material. X-ray diffraction experiments showed that the specimens extruded at 200 °C has high β-crystal content (75%), compared to 5.6% (0.3 mm layers), 11.4% (0.1 mm layers), and 4.6% (injection molding). Apparently, the higher crystallinity of the specimens extruded at the lower temperature compensated for the higher density of the injection molded specimens.

3 MATERIAL FORMULATIONS

During this research, ten different neat polypropylene-based polymers were investigated. Out of those, test specimens were successfully fabricated on our HYREL System 30M (HYREL 3D) with five of the polypropylenes (Polypropylenes A through E). The top and side views of the test specimens as well as percent crystallinity are presented in Table 1. Process variables under investigation included layer height, deposition temperature, and filament deposition angle. These five polypropylenes (two homopolymers and three copolymers) were studied and various composite materials were formulated with them using common filler materials. Note that sponsor restrictions prohibit publishing the specific material formulations. After screening based on printed part quality, two copolymer polypropylenes (C and D) were identified as favorable for further study. Although PP E had the smallest crystallinity and little warpage, printed parts did not exhibit good dimensional integrity; hence PP E was not selected for further study.

Table 1. Test specimens and percent crystallinities of candidate neat polypropylenes

Polypropylene	**Test Specimen**		**% Crystallinity**
Polypropylene A			52
Polypropylene B			39
Polypropylene C			34
Polypropylene D			13
Polypropylene E			10

Since polypropylene is a semi-crystalline thermoplastic polymer, it experiences a higher degree of shrinkage upon cooling than ABS, which is amorphous. This increased shrinkage led to increased part warpage, so polypropylene polymers with different levels of crystallinity were explored. Table 1 shows that warpage was indeed related to the percent crystallinity of the material. Polypropylene A had the highest percent crystallinity, and its test specimen showed the most warpage. In fact, the part fabrication with Polypropylene A could not be completed since it detached from the build platform completely during the fabrication process. In contrast, Polypropylene D and E had the lowest percent crystallinity and their test specimens showed the least warpage.

The addition of additives, such as particles and fibers, can reduce shrinkage, warpage, and residual stress. Two mechanisms are typically credited with these reductions: mechanical interference with shrinkage and prevention of crystal formation. Several types of additives were explored in Polypropylenes C and D, such as particles, fibers and agents that affect viscosity and thermal conductivity. One composite material (Polypropylene C1) was created with Polypropylene C as the base material, and three composite materials (Polypropylenes D1 through D3) were created with Polypropylene D as the base material. Test specimens were fabricated with these composite materials as well, but no significant differences in warpage were observed with respect to each other. Polypropylenes D1 and D2 will be explored further in this paper.

4 MATERIAL EXTRUSION SIMULATION

A series of simulation models has been developed and validated in our lab recently [12], which built on the work of others, e.g., [13, 14]. The objective of these models is to predict temperature distributions, deposited filament shapes, residual stresses, and warpages/deformations of fabricated parts. Inputs include material properties, process variable settings, and process conditions. A commercially available polypropylene-based polymer was used here as a model system for study. The simulation model overview is presented in Figure 1.

The simulations were developed using ANSYS® Polyflow and Mechanical. To capture the thermal processes experienced during material deposition, several simulations were developed, and these sequential simulations were linked to one another through the temperature profiles developed in previous steps. A final simulation was developed using ANSYS Mechanical to predict residual stresses and warpage. Each simulation model will be summarized here; additional information is available in references [12, 15].

The first simulation model was the deposition and cooling of the first layer of filament. The first layer was deposited onto a build platform, which was assumed to be at a constant temperature of 80 °C. By applying the calculated volumetric flow rate at the nozzle entrance and gravitational force, and using the remeshing technique in ANSYS® Polyflow, the deposition of the first layer was performed. In this simulation model, the filament was extruded through the nozzle in the vertical direction, while the deposition velocity was applied in the horizontal direction. In order to simulate the relative motion between the nozzle and the build platform, the nozzle was maintained in a fixed position, while the build platform translated in the horizontal direction with a deposition velocity.

The second simulation model was the deposition of the second layer of filament on top of the first layer and the cooling of both layers. The temperature distribution after the first layer cooling was exported from the previous simulation. Conduction heat transfer between the two layers was accomplished using the fluid-to-fluid contact capability in ANSYS® Polyflow.

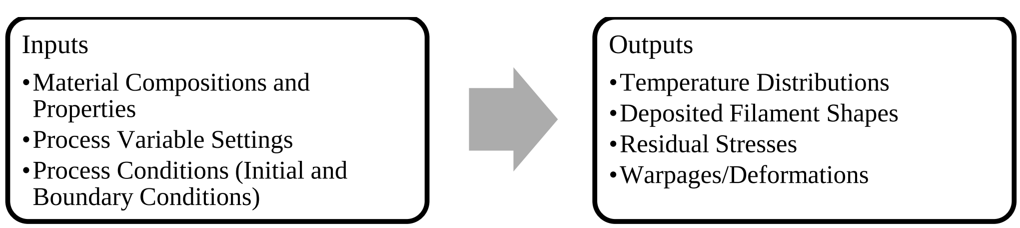
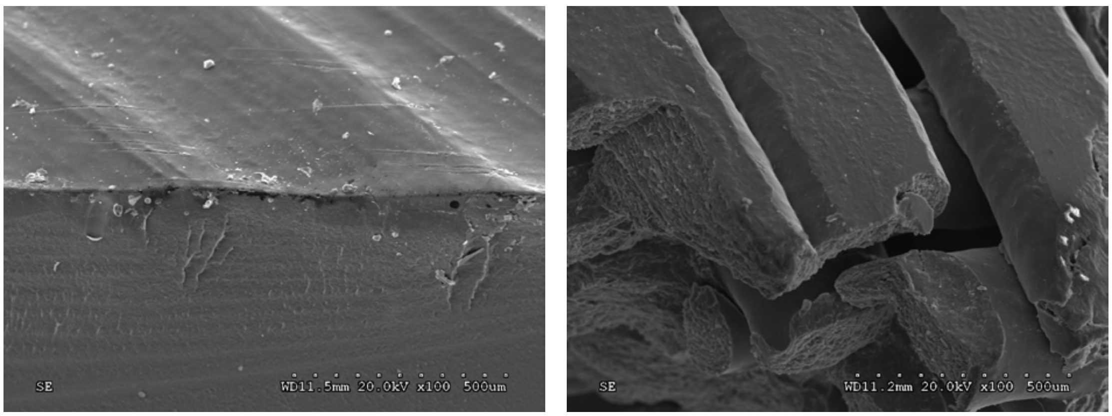


Figure 1. Overview of material extrusion process simulation models

5 MATERIAL PROPERTIES

Although test specimens with polypropylene-based composite materials showed no significant differences in warpage with respect to each other, differences in surface finish were observed, which was related to the bonding between the extruded filaments. The surface topography of test specimen was examined for each composite material using a SEM. The two extreme cases of surface finish are shown in Figure 2. The topographies are shown of the top surface and the cross section of the test specimens fabricated with Polypropylenes D2 and D1. The surface finish and bonding quality of Polypropylene D2 were remarkable as all of the extruded filaments seemed to have coalesced. The lumps on the top surface indicated each extruded filament, but no voids were visible from the SEM image. In contrast, the surface finish and bonding quality of Polypropylene D1 were poor as each extruded filament could be distinguished in the SEM image and broke during SEM specimen preparation.



(a) (b)

Figure 2. Images of (a) top surface of Polypropylene D2 and (b) cross section of Polypropylene D1

One of the disadvantages of MEX is known to be the pronounced anisotropy of mechanical properties of fabricated parts that is caused by incomplete bonding between the extruded filaments as well as preferred orientation of polymer chains and crystals due to the imposed flow [4, 16]. However, no voids were observed in the test specimen fabricated with Polypropylene D2, which meant that a complete bonding was accomplished between the extruded filaments and a solid part was created. This suggested that anisotropy was perhaps reduced with this composite material. In order to investigate this phenomenon further, tensile tests were conducted using Polypropylene D2.

For completeness, properties of Polypropylene D2 are presented in Table 2 [12].

Table 2. Material properties of polypropylene copolymer

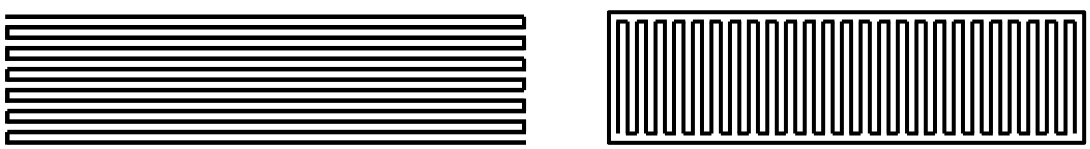
Viscosity Expression	$\eta = e^{\left[1318.9\left(\frac{1}{T}-\frac{1}{503.15}\right)\right]} 3346.4(\dot{\gamma})^{-0.54}$
Coefficient of Thermal Expression	1.50 x 10^{-4} m/(m-°C)
Thermal Conductivity	0.2 W/(m-°C)
Specific Heat	1920 J/(kg-°C)
Density	900 kg/m^3
Melting Temperature (T_m)	151.0 °C
Crystallization Temperature (T_c)	104.0 °C

6 MECHANICAL PROPERTY ANISOTROPY

Tensile experiments were performed to determine tensile strength at yield, tensile strength at failure, and elastic modulus for Polypropylene D2. Correlations between mechanical property anisotropy and the bonding quality of extruded filaments were examined experimentally by producing tensile property data of fabricated parts with different fill angles. The efficacy of the process simulation models was evaluated by comparing the experimental and simulation model results.

By producing tensile property data with different fill angles, the filament bonding performance can be tested and the degree of anisotropy can be assessed. Thin flat strips of material (12.5 mm x 87.5 mm) having a constant rectangular cross section were fabricated with two fill angles, 0° and 90° and were tested following a method similar to ASTM D3039/D3039M-14 [17]. Standard “dog bone” specimens were not used in order to avoid the stress concentrations that occur where along the curved regions between the gauge and grip regions of the specimen. The 0° fill angle specimens were fabricated without perimeters, but the 90° fill angle specimens required three perimeters since the fabrication process was unsuccessful without them. The schematics of fill angles are shown in Figure 3. Five specimens were tested using an Instron 5566 at a speed of 20 mm/min in order to produce failure within approximately 1 to 10 minutes.

Representative stress-strain curves with yield and filament failure points with 0° and 90° fill angles are shown in Figures 4 and 5, respectively. Yield point was defined according to the testing standard as the first point on the stress-strain curve at which an increase in strain occurs without an increase in stress. The filament failure point was estimated to be the point where filaments began to fail during the test. Since these test specimens deformed differently over the entire length of the sample between the grips, the nominal strain was calculated and was used on the stress-strain curves. The nominal strain was calculated by dividing the crosshead extension by the distance between grips, which was 62.5 mm. It should be noted that the test specimens with a 0° fill angle never failed during this test. Instead, the specimens continued to extend until they were too thin for the Instron machine to grip.



(a) (b)

Figure 3. Anisotropy test specimens: (a) 0° fill angle and (b) 90° fill angle

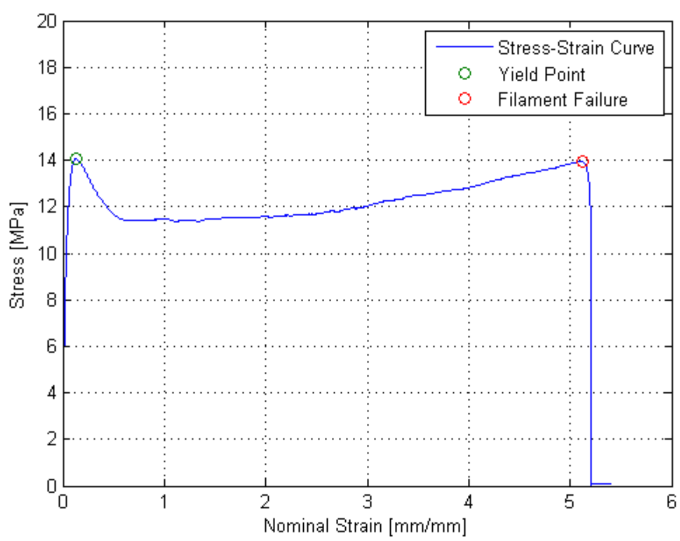


Figure 4. Stress-strain curve of Polypropylene D2 with a 0° fill angle

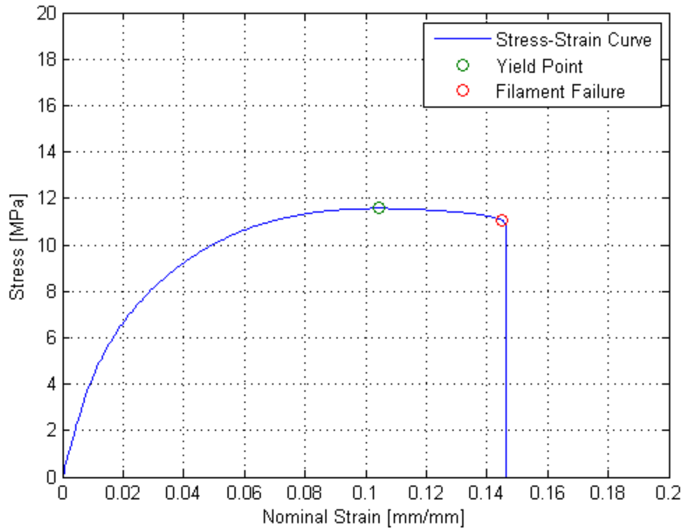


Figure 5. Stress-strain curve of Polypropylene D2 with a 90° fill angle

Various deposition temperatures and layer heights were also explored to see if these process variable settings affect mechanical property anisotropy and filament bonding performance. The settings are summarized in Table 2.

Table 2. Process variable settings for mechanical property anisotropy

Process Variable Settings	Values		
Deposition Temperature	240 °C	260 °C	280 °C
Layer Height	0.1 mm	0.2 mm	-

6.1 Deposition Temperature

From the stress-strain curves, tensile stress at yield point, tensile stress and nominal strain at filament failure point and modulus of elasticity were determined with various deposition temperatures, and are shown in Figures 6-9, respectively. In this case, the layer height was kept constant at 0.2 mm. Since there were overlaps of the error bars, statistical analyses were performed on these experimental results. Single factor analysis of variance (ANOVA) was run to test the null hypothesis that the means are all equal. For all four plots, the means were determined to be statistically equal for each fill angle. Tensile stress, nominal strain and modulus of elasticity with both 0° and 90° fill angles were not dependent on temperature.

At 240 °C, the tensile stress at yield point was higher with a 0° fill angle than with a 90° fill angle, which implied that anisotropy existed at this temperature. When the deposition temperature was increased to 260 °C and 280 °C, the tensile stresses at yield point were determined to be statistically equal. A similar trend was observed with the tensile stress at filament failure point in Figure 7. At 240 °C and 260 °C, the tensile stresses were higher with a 0° fill angle compared to a 90° fill angle. However, statistical analysis showed that they are equal at 280 °C. Therefore, a reduction in anisotropy was accomplished by increasing the deposition temperature. In addition, the typical value of tensile stress at yield point of Polypropylene D is 15.8 MPa, which is slightly lower than the base polypropylene.

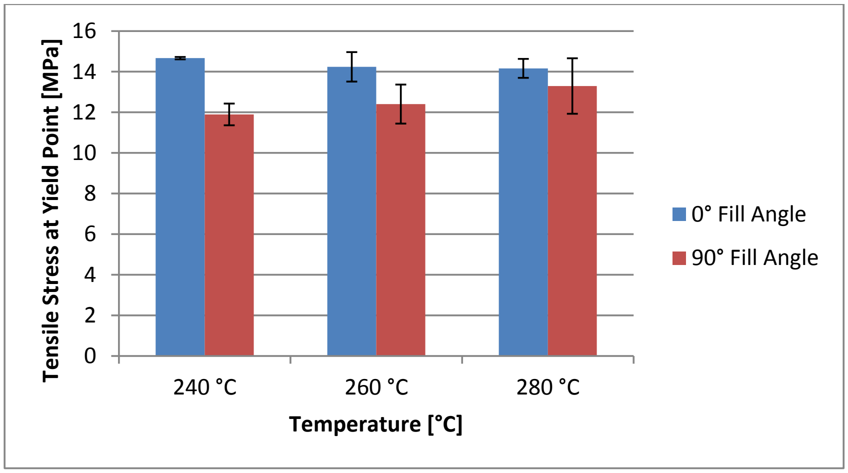


Figure 6. Tensile stress at yield point with various deposition temperatures

It can be observed from Figure 8 that the tensile nominal strain at filament failure point was highly dependent on fill angle. The nominal strain with 0° fill angle was approximately 5.1 mm/mm, and that with 90° fill angle was approximately 0.2 mm/mm. Although there were differences between the strain values, those with a 0° fill angle were significantly higher compared to those with a 90° fill angle. In addition, the typical value of elongation at break of Polypropylene D was reported to be 617%; the elongation at break for Polypropylene D2 with 0° fill angle was approximately 17% lower than the base polypropylene.

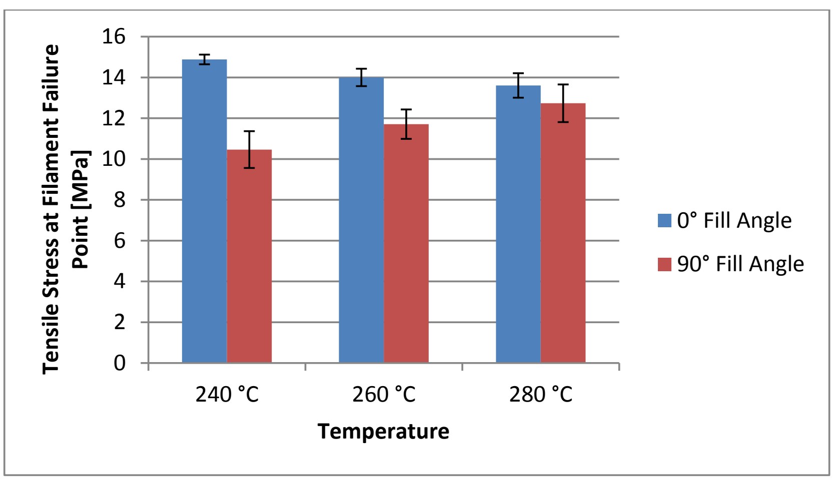


Figure 7. Tensile stress at filament failure point with various deposition temperatures

Figure 9 shows that the modulus of elasticity was fairly uniform and was not dependent on fill angle, although tensile nominal strain at filament failure point is highly dependent on fill angle as previously stated. It was determined that the moduli of elasticity with different fill angles were statistically equal at each temperature as well. This was due to consistent nominal strain values in the elastic region, and the range of nominal strain was approximately 0.005 and 0.015 mm/mm in all cases. However, the average value of modulus of elasticity with 0° fill angle was approximately 383 MPa, and that with 90° fill angle was approximately 357 MPa, which was a 7% decrease. The flexural modulus of Polypropylene D, the base material of this composite material, was slightly higher than the experimental data of the fabricated parts with a reported value of 393 MPa. In addition, Stratasys reported the tensile modulus of ABS-M30 was 2,230 MPA for 0° fill angle, while 90° fill angle exhibited tensile modulus of 2,180 MPa, which was a 2% difference [8].

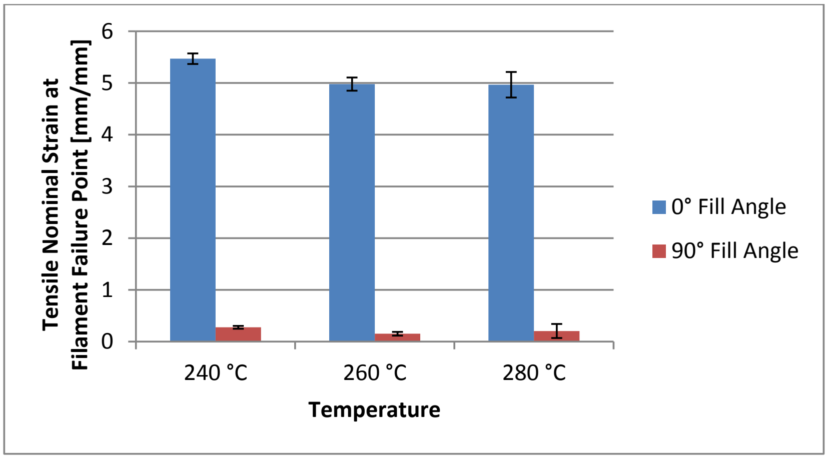


Figure 8. Tensile nominal strain at filament failure point with various deposition temperatures

Using the material extrusion process simulation models, the temperature distributions of two layers of filaments were determined and are shown in Figure 10. The difference in fill angles was simulated by changing the deposition length. In order for the deposition length to be directly proportional to the anisotropy test specimen dimensions shown in F, it was set to 5.0 mm for 0° fill angle and 0.7 mm for 90° fill angle. The temperature contour plots are shown in two colors only, in which green represents below melting temperature (108 °C) and red represents above melting temperature. In all cases, the temperature at the interface between the first and second layers was above melting temperature, which means that good bonding was achieved. In addition, no significant differences in the contour plots could be observed at different temperatures. This agreed with the experimental results that tensile stress, nominal strain and modulus of elasticity were not dependent on temperature.

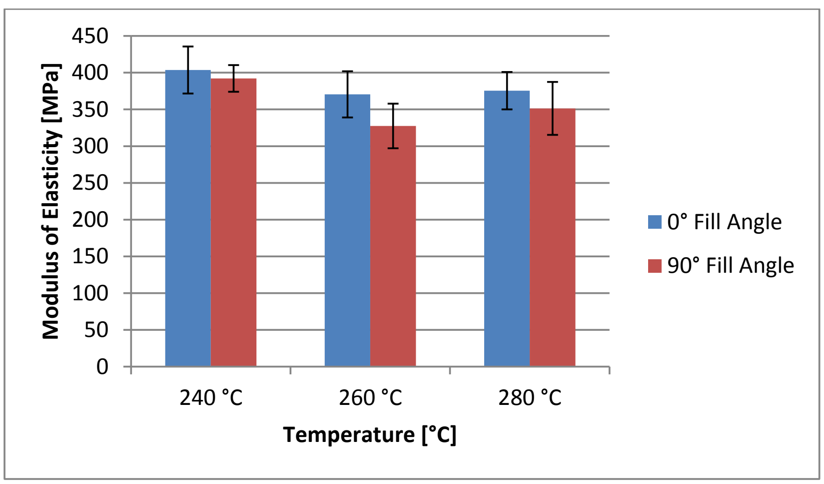


Figure 9. Modulus of elasticity with various deposition temperatures

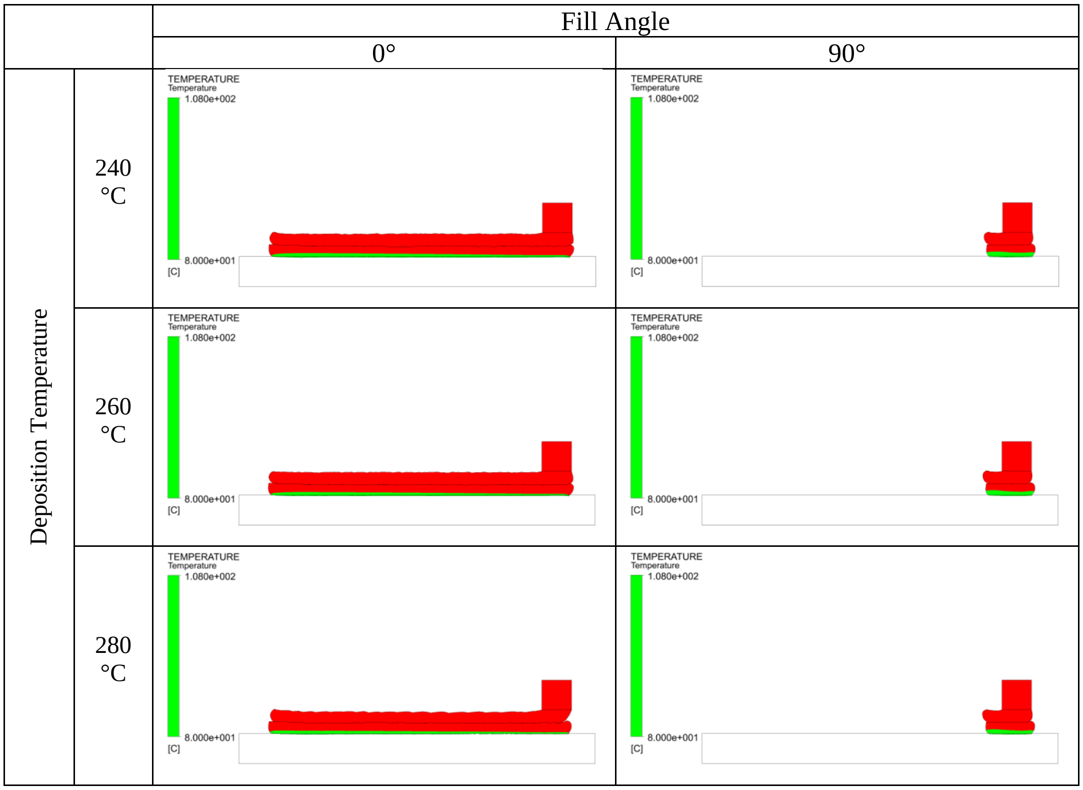


Figure 10. Temperature distributions from process simulation models with various fill angles and deposition temperatures

6.2 Layer Height

Tensile stress at yield point, tensile stress and nominal strain at filament failure point and modulus of elasticity were determined with various layer heights, and are shown in Figures 11-14, respectively. In this case, the deposition temperature was kept constant at 260 °C. Statistical analyses were performed on these experimental results as well due to the error bar overlaps. For all four plots, the means were determined to be statistically equal for the 0° fill angle, however, the means were determined to be statistically not equal for the 90° fill angle. In fact, the values with a layer height of 0.1 mm were determined to be higher than those with a layer height of 0.2 mm in all cases. Tensile stress, nominal strain and modulus of elasticity with a 0° fill angle were not dependent on layer height, but those with a 90° fill angle were dependent on layer height.

The tensile stresses with two different fill angles were also compared at each layer height. From the experimental results shown in Figure 12, the tensile stresses at yield point with a layer height of 0.1 mm were statistically equal, and those with a layer height of 0.2 mm were statistically equal. The same trend was observed with the tensile stresses at filament failure point, which implied that statistical anisotropy did not exist at each layer height. However, slightly larger differences in the average tensile stress values were observed with a 0.2 mm layer height from the two plots. Therefore, a reduction in anisotropy was perhaps accomplished by decreasing the layer height.

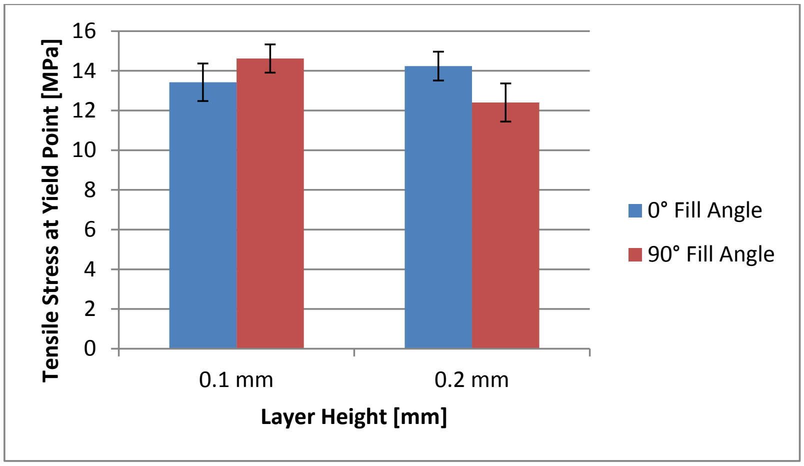


Figure 11. Tensile stress at yield point with various layer heights

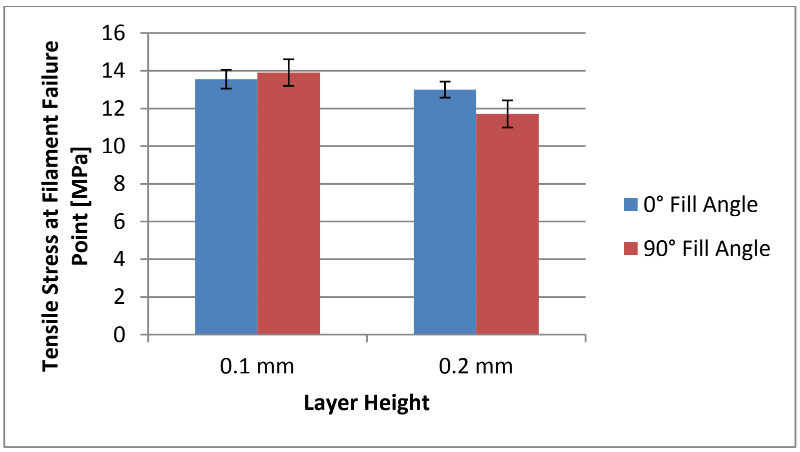


Figure 12. Tensile stress at filament failure point with various layer heights

It can be observed from Figure 13 that tensile nominal strain at filament failure point was highly dependent on fill angle. Although the nominal strains for the 0° fill angle specimens were higher than those for the 90° fill angle for both layer heights, the value with a 0.1 mm layer height was significantly higher than that with a 0.2 mm layer height for the 90° fill angle specimens. This indicated that a reduction in anisotropy in nominal strain was achieved by decreasing the layer height.

The moduli of elasticity with two different fill angles were compared at each layer height. From the experimental results shown in Figure 14, the moduli of elasticity with a layer height of 0.1 mm were statistically equal, and those with a layer height of 0.2 mm were statistically equal. This suggested that the modulus of elasticity was fairly uniform and was not dependent on fill angle. However, once again, the difference between the average modulus of elasticity with a 0.2 mm layer height was larger compared to that with a 0.1 mm layer height. This supported the hypothesis that anisotropy could be reduced with a lower layer height.

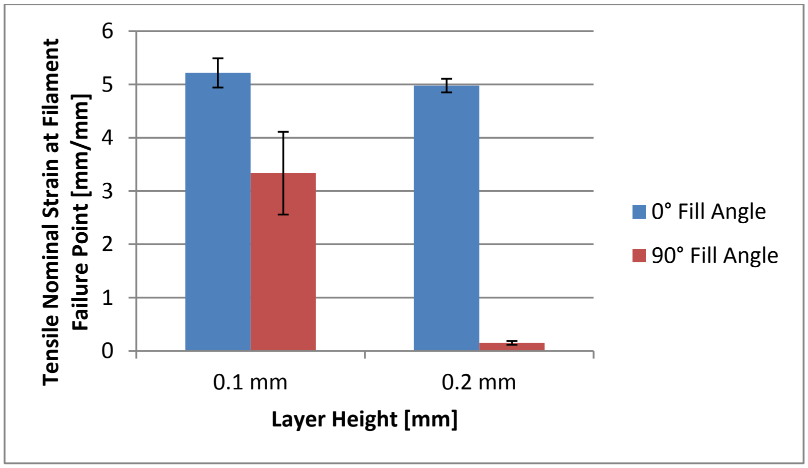


Figure 13. Tensile nominal strain at filament failure point with various layer heights

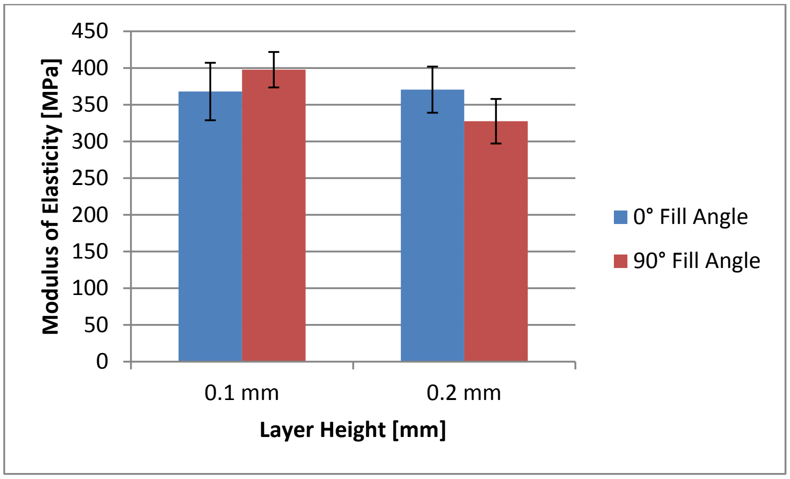


Figure 14. Modulus of elasticity with various layer heights

The temperature distributions of two layers of filaments with different layer height values were determined from the process simulation models, and the results are shown in Figure 15. Since the number of layers was kept constant, this led to differences in part thickness. Therefore, when comparing the red region in the vertical direction, the results needed to be normalized to the part thickness. It was determined that a larger percentage of the thickness was at a higher temperature with a lower layer height. This meant that a greater portion of the first layer with a 0.1 mm layer height was re-liquefied and a better diffusion across the interface was obtained. It can be concluded that a better bonding was achieved with filaments with a lower layer height. This agreed with the experimental results that tensile stress, nominal strain and modulus of elasticity with a layer height of 0.1 mm were higher than those with a layer height of 0.2 mm for the 90° fill angle specimens. It can also be observed that there was a large green region for the 0° fill angle specimen with a layer height of 0.1 mm. This was most likely due to this specimen being thinner than the specimen with a layer height of 0.2 mm. The simulation result indicated that the green region had cooled down at this instant but a good bonding between the layers had already been achieved as it can be observed from the red region in the current time step.

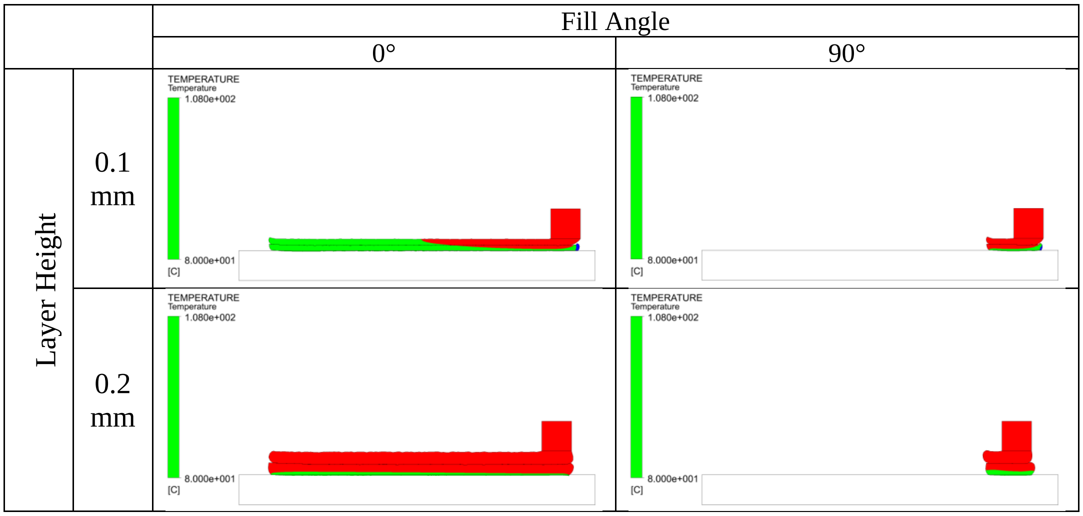


Figure 15. Temperature distributions from process simulation models with various fill angles and deposition temperatures

7 CONCLUSIONS

Tensile mechanical properties of polypropylene composite formulations were investigated as a function of some material extrusion process variables and the thermal properties of these formulations. Results demonstrated three main conclusions for tensile specimens fabricated horizontally and flat, with two different fill angles:

- Results demonstrate that anisotropy can be reduced almost completely if the material can be formulated to have low crystallinity, low coefficient of thermal expansion, and moderate to high thermal conductivity (for a polymer). Low crystallinity is critically important for good MEX processibility.
- Tensile stress, nominal strain and modulus of elasticity were not dependent on temperature with both the 0° and 90° fill angle specimens. However, a reduction in tensile stress anisotropy was achieved with an increase in deposition temperature. In addition, the tensile properties with a 0° fill angle test specimens were not dependent on layer height, but those with a 90° fill angle test specimens were dependent on layer height. The experimental results also showed that a reduction in tensile property anisotropy was accomplished with a decrease in layer height.
- Simulation model results exhibited good correlations with experimental results. Temperature contour plots at various deposition temperatures depicted no significant differences, which agreed with the experimental results that tensile properties were not dependent on temperature. The temperature contour plots with various layer heights showed that there is a greater region with higher temperature in the vertical direction with a lower layer height. This represented that a better bonding was achieved between the extruded filaments with a lower layer height, which leads to a reduction in mechanical property anisotropy. This agreed with the experimental results that tensile properties with a lower layer height were higher than those with a higher layer height for the 90° fill angle specimens.

ACKNOWLEDGEMENTS

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