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Reviewed Paper**

DEVELOPMENT OF PROCESSING PARAMETERS FOR THE SELECTIVE LASER SINTERING OF CARBON FIBER REINFORCED POLYPHENYLENE SULFIDE WITH A TOOLING APPLICATION

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Abstract

The ongoing development of processing parameters for advanced thermoplastic materials to be fabricated via selective laser sintering (SLS) is rapidly advancing the potential industrial applications of the manufacturing method. This research focuses on the development of SLS processing parameters and a tooling application for carbon fiber reinforced Polyphenylene Sulfide (CF-PPS), a composite material that is novel to SLS. A high temperature SLS research machine was used to identify suitable processing parameters for the material along with the tensile strength and geometrical accuracy associated with those parameters. Utilizing the previously identified parameters, a repair mold for an electronic cable assembly was fabricated. Mechanical tests were performed on fabricated CF-PPS parts to evaluate the performance of the material under the mold's normal operating conditions. The additively manufactured CF-PPS mold was deemed viable for production and was shipped to our sponsor for further evaluation.

Introduction

Selective Laser Sintering (SLS) is an additive manufacturing process capable of fabricating fully dense polymer parts with complex geometries that cannot be produced by other manufacturing methods [1] [2]. The process begins with a counter-rotating roller spreading a thin layer of powder over a piston. A laser then scans the cross-sectional area of the desired part(s). This raises the pre-heated polymer powder above its melting temperature, subsequently fusing the powder material together upon cooling. The piston then moves down a single layer and another layer of powder is spread on top of the previously sintered material. The process then repeats itself, layer-by-layer, to build up a three dimensional part(s) [3]. The SLS manufacturing method excels in quickly delivering high efficiency, low cost parts for low volume, high complexity component production. Seeking these benefits, the medical, aerospace, and tooling industries have been rapidly expanding their applications of this developing technology.

The ongoing development of processing parameters for advanced thermoplastic materials is also broadening the potential applications of the SLS process. This research focuses on the development of processing parameters and potential industry applications for carbon fiber reinforced polyphenylene sulfide (CF-PPS), a composite material that is novel to SLS. Polyphenylene sulfide (PPS) is a high performance thermoplastic that offers exceptional mechanical strength, chemical resistance, and high operating temperatures [4]. The addition of carbon fibers to PPS enhances both the stiffness and strength to weight ratio of the material while also elevating the material's heat distortion temperature. The combination of these qualities make it a desirable material to be used in tooling applications. An unnamed sponsor desired to develop processing parameters for CF-PPS in order to fabricate a mold for repairing an electronics cable assembly in adverse situations and conditions. The cable assembly has an exterior plastic casing that is in place to protect the electronics cable inside, but the casing frequently deteriorates causing the cable to be exposed. The fabricated mold would allow for the exposed electronics cable to be placed inside and a new protective plastic casing to be molded over the cable assembly. This results in significant cost savings as well as reduced lead times when compared to replacing the damaged cable assembly. Through this research, SLS processing parameters for building with CF-PPS were developed and the electronics cable assembly repair mold was fabricated. After construction, the mold was shipped to our sponsor and put through a series of tests to evaluate its performance. The fabricated quality of the mold and initial testing results showed promise for the use of CF-PPS in future tooling applications.

Experimental Methods and Procedures

Parameter Development

The first step when introducing a novel material to the SLS process is parameter development. Parameter development refers to identifying the appropriate processing window and set point for each of the important variables in the SLS process. These variables include laser power, laser scan speed, layer thickness, hatch spacing, and powder bed preheat temperature. If any of these variables are outside of the necessary processing window, the novel material will not successfully build in SLS. The forthcoming paragraphs will introduce a proven method for parameter development for a novel material to be used in SLS.

The work outlined in this paper was performed on the Laser Additive Manufacturing Pilot System (LAMPS) at the University of Texas at Austin. LAMPS is a research grade SLS machine capable of processing thermoplastic powders with melting temperatures approaching 400 ℃. This is made possible by an insulated upper build chamber that houses three quartz lamps directly over the powder bed to preheat to the desired temperature. Two of the quartz lamps are directed at the powder bed, and the remaining lamp is pointed at the powder drop to help preheat the new powder layer prior to spreading. Preheating the powder in the SLS process is important in maintaining a uniform powder bed temperature to help fight part curling during the build process. LAMPS is also outfitted with two thermal cameras and a visual camera to provide real time monitoring of the build taking place. The data generated from the two thermal cameras is used to monitor and control the thermal environment during operation and also for research purposes to help characterize the SLS process. The visual camera can be monitored by the operator to have eyes inside of the build chamber and to ensure everything looks as it should throughout the SLS build process. On the next page, Figure 1 shows a CAD rendering of the LAMPS research SLS machine at UT Austin.

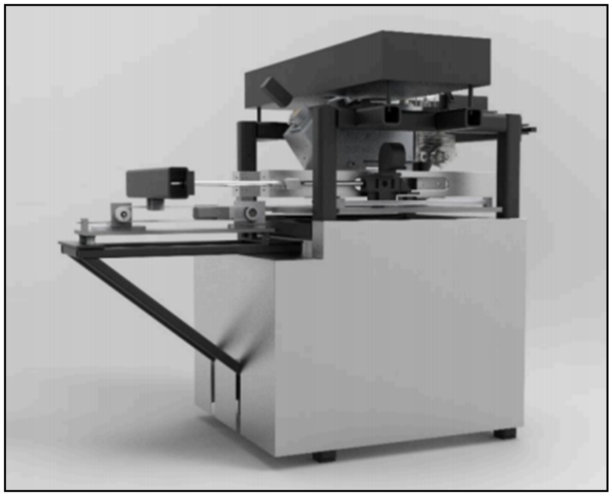

Figure 1: CAD rendering of the LAMPS SLS machine

LAMPS utilizes a 40 W CO_2 laser and a galvo mirror system to scan two-dimensional cross sections of the powder bed. The input power and scan speed of the laser on the powder bed directly correlates to the energy input into the material. When selecting which laser power to use in LAMPS, a percentage of the total power is the input. In theory, 50% power would result in 20 W of power being output from the laser and into the powder bed. In practice, however, this is not the case as there are several factors that reduce the power of the laser. These factors include degradation of the laser over time, inefficiencies in the optic track of the laser, and a contaminated laser window inside the build chamber. To account for this, the laser is tested before and after each build to quantify the laser power delivered to the powder bed during the build. This is important because during longer duration builds, the laser window can become contaminated with powder or off-gassing from the material and reduce the laser power seen by the powder bed. This issue is somewhat mitigated by flowing nitrogen over the window but cannot be completely eliminated. Figure 2 shows the laser power curve obtained before the tensile bar builds from this study. An after measurement is not presented as no contamination was detected due to the short duration of the builds.

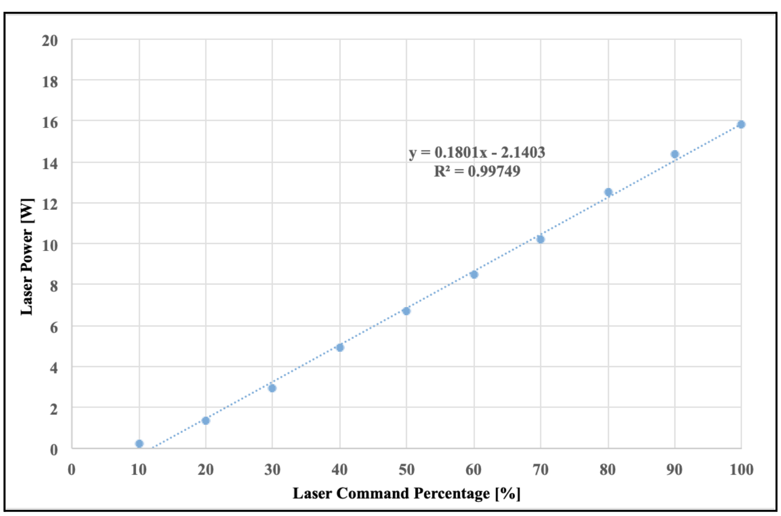

Figure 2: Measured laser power at build surface

This research utilized a modified version of the processing parameter development procedure laid out by Snarr et al. [5]. The modified approach contained the following steps:

1. Verify the material's powder particle size is suitable for the SLS process
2. Identify sintering window for the material
3. Use visual inspection to determine powder bed preheat temperature
4. Sinter energy density grid to identify laser scanning parameters
5. Fabricate tensile bars to quantify part strength and geometrical accuracy for parameters

A modified version of the parameter development steps was used because the end goal of this research was not to optimize parameters for strength or geometrical accuracy, but to fabricate a functioning repair mold for the sponsor.

The first two steps in the parameter development process involved characterization of the CF-PPS powder to determine its suitability for the SLS process. This included checking the average mean diameter and sphericity of the powder particles along with identification of the material's glass transition and melting temperatures. A Scanning Electron Microscope (SEM) was used to image the powder and verify the powder was composed of high sphericity particles, which facilitate smooth spreading during the SLS process. Collected images were then put through an ImageJ software analysis where particle cross-sectional area measurements were recorded for representative samples of the powder. The average particle diameter and a particle diameter distribution for the CF-PPS powder were then calculated from this data. The particles were sufficiently spherical and their mean particle diameter was within the desired range for use in the SLS process (approximately 30 - 80 um). Once this analysis was complete, the powder was then tested using a Differential Scanning Calorimetry (DSC) machine. The DSC machine runs the powder through a heating and cooling cycle in order to identify the material's glass transition and melting temperatures. The region between these two temperatures is known as the material's sintering window. It is critical to the SLS process as this is the temperature range to which the material is preheated before it is sintered. The DSC process clearly identified the desired temperatures and subsequently the sintering window. At the request of our sponsor, none of the results or information collected in these steps is presented due to the proprietary nature of the material's composition.

Once the powder was deemed suitable for the SLS process and a sintering window identified, the material was put into the LAMPS machine to identify the preheat temperature. The shimmer test is a commonly used method to identify this set point. A uniform layer of powder is spread in the machine and then the preheat temperature is slowly increased until the powder bed begins to slightly shimmer. Once the powder begins to shimmer, melting has started to occur and the upper limit of the powder preheat has been reached. The preheat temperature is then set several degrees below this upper limit in order to require minimal energy input from the laser to melt the material during the sintering phase.

The next step in the parameter development process was to utilize a parameter grid to identify laser parameters for building in the CF-PPS material. The standard volumetric energy density equation, commonly used in additive manufacturing applications with continuous wave lasers, is presented below in Equation 1. This equation lumps the standard laser and machine parameters used in the sintering processes together to create a single value representing the total volumetric energy density (E) applied to the material. The variables and their units used in the equation are as follows: P for laser power in watts, v for scan speed in mm/s, h for hatch spacing in mm, and z for layer thickness in mm [6].

$$E = \frac{P}{v * h * z} \quad [\frac{J}{mm^3}] \qquad \text{EQ. 1 [6]}$$

Several parameters had already been set based on the laser spot size or the machine itself. For example, the layer thickness for the SLS machine was .1 mm (100 um) and the hatch spacing was selected as .3 mm (300 um) based on the laser spot size of the machine (approximately .5 mm or 500 um) and standard procedures. In order to identify the two remaining laser parameters, laser power and scan speed, parameter grids were used. These grids are used as a visual inspection tool to look for complete sintering of the powder bed and narrow down a parameter range. The color of the sintered material and slight sinking of the material into the powder bed are indicators used to find the most promising parameter set. A few different parameter grids were run with varying ranges of energy densities. The final sintered parameter grid is shown on the next page in Figure 3. In this grid, each row has a different laser power. Starting from the top row with a command of 50% laser power (6.9 W), each row increased by 10% laser power. The bottom row had a laser power command of 90%, resulting in 14.1 W of laser power. Each vertical column had a different laser scan speed. Starting from the left with a scan speed of 1500 mm/s, each row decreased by 200 mm/s leaving the far right row with a scan speed of 700 mm/s. The bracketed square identifies the selected parameter set to move forward with. It was chosen because it showed signs of achieving full melting (dark color change and slight sinking into the powder bed) with no loss of geometrical accuracy from overmelting or degradation of the powder. This final set of selected parameters was used for the remainder of the experiments to fabricate tensile bars and the electronics mold. The final parameters are presented on the next page in Table 1.

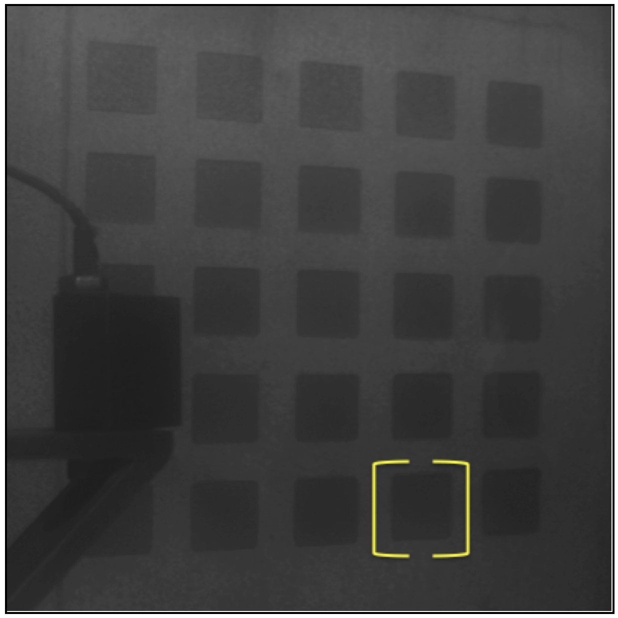

Figure 3: Parameter grid used to identify final scanning parameters

Table 1: Final parameters used for tensile bar and mold fabrication

Laser Power	Scan Speed	Hatch Spacing	Layer Thickness	Laser Spot Size	Preheat Temperature
14.1 W	900 mm/s	300 um	100 um	500 um	280 °C

With the final parameter set identified, the last step in the parameter development process was to fabricate several tensile bars in order to quantify the tensile strength, compression strength, and geometrical accuracy for the parameters. Two builds, each containing seven standard ASTM Type IV tensile bars, were completed to construct a total of 14 tensile bars. On the next page Figure 4 shows the sintered tensile bars during the build process. The testing procedures used to collect data for these tensile bars are described in the following sections. This study did not seek to optimize for strength or geometrical accuracy, but instead fabricated the tensile specimen to quickly quantify some of these values as a base case for the mold fabrication.

Once the tensile specimen had been fabricated, they were taken out of the build chamber and the parts were inspected to ensure they were fairly dense and had reasonable geometrical accuracy. The as-built tensile bars can be seen on the next page in Figure 5. They were deemed acceptable and allowed the study to continue towards its main goal, fabrication of the electronics repair mold. The same set of parameters were then used to build the full-scale repair mold. The outcome from the fabrication of the mold is presented in the subsequent results section.

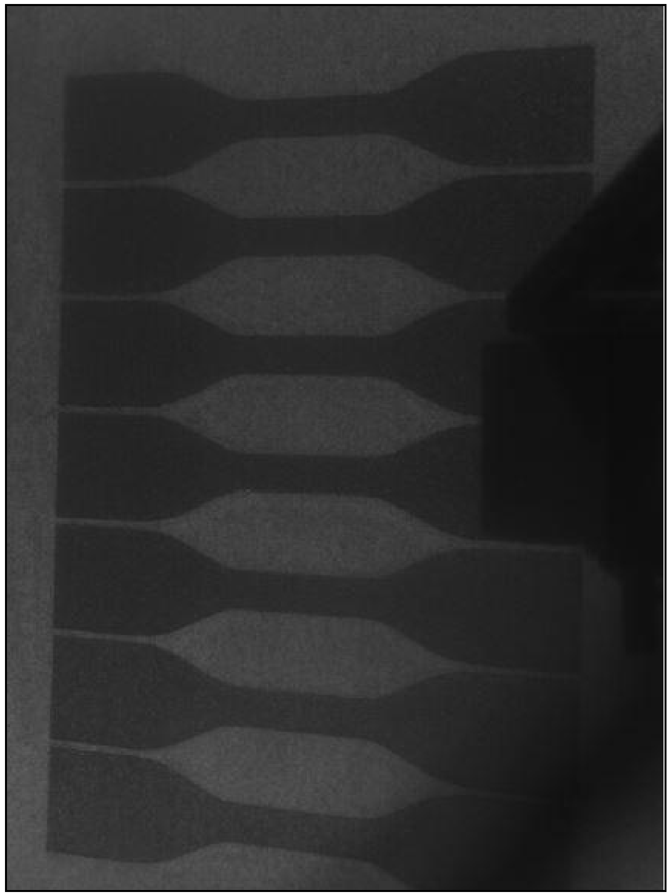

Figure 4: Sintered tensile bars in the build chamber

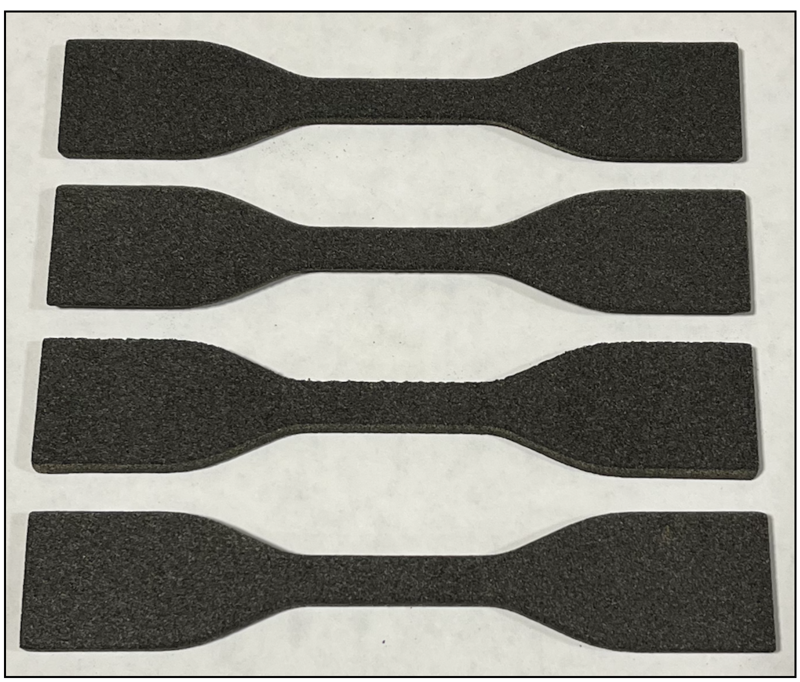

Figure 5: As-built tensile bars

Testing Procedures

Several tests were performed on samples fabricated from the novel CF-PPS material to begin validation for the use of the material for the repair mold application. These tests include tensile testing, geometric accuracy measurements, and compression testing. The results of these tests will provide insight into whether this novel material is a candidate for the desired application.

Tensile testing was performed on fabricated CF-PPS tensile specimens in accordance with ASTM D638: Standard Test Method for Tensile Properties of Plastics [7]. These tests were performed on an Instron 3345 with a 5 kN load cell. ASTM Type IV tensile specimens were fabricated to complete the testing. Figure 6 shows the nominal dimensions for a Type IV tensile specimen. A total of 5 tensile specimens were tested at a crosshead velocity of 5 mm/min as required by the standard.

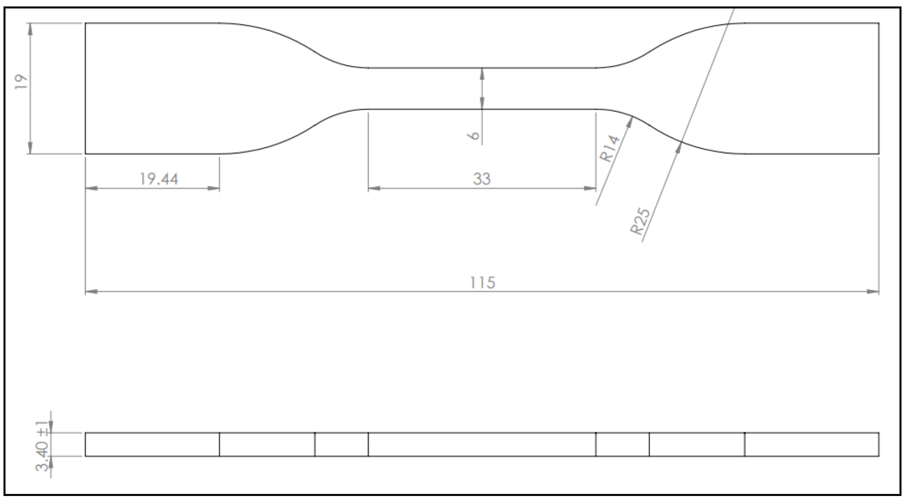

Figure 6: ASTM Type IV Tensile Specimen (units in mm)

To test the geometrical accuracy achieved with the selected parameter set, the fabricated tensile specimens were measured at three locations using digital calipers and compared to the nominal dimension of the tensile specimen. Three measurements were taken to quantify the accuracy of the tensile specimens in the X and Y directions of the build. These measurements include the overall length (LO), the overall width (WO), and the width of the narrow section (W). The overall length tests the accuracy of the print in the x-axis, while the overall width and width of the narrow section reveal accuracy information of the y-axis.

The final test that was performed was a standard compression test on an MTS Criterion Model 45 with a 100 kN load cell. This test was performed to begin understanding how the CF-PPS mold would handle the applied compression force when being used to repair the electronic cable assemblies. In use, the mold will be at an elevated temperature while the compression is applied, however, timely access to a machine with this capability was not accessible. To perform the compression test, a thin rectangular cross section of the CF-PPS was placed between the two compression heads and the applied force was set to a max of 95 kN. The crosshead was moved at a rate of .01 mm/s.

<u>Results</u>

Tensile Testing Results

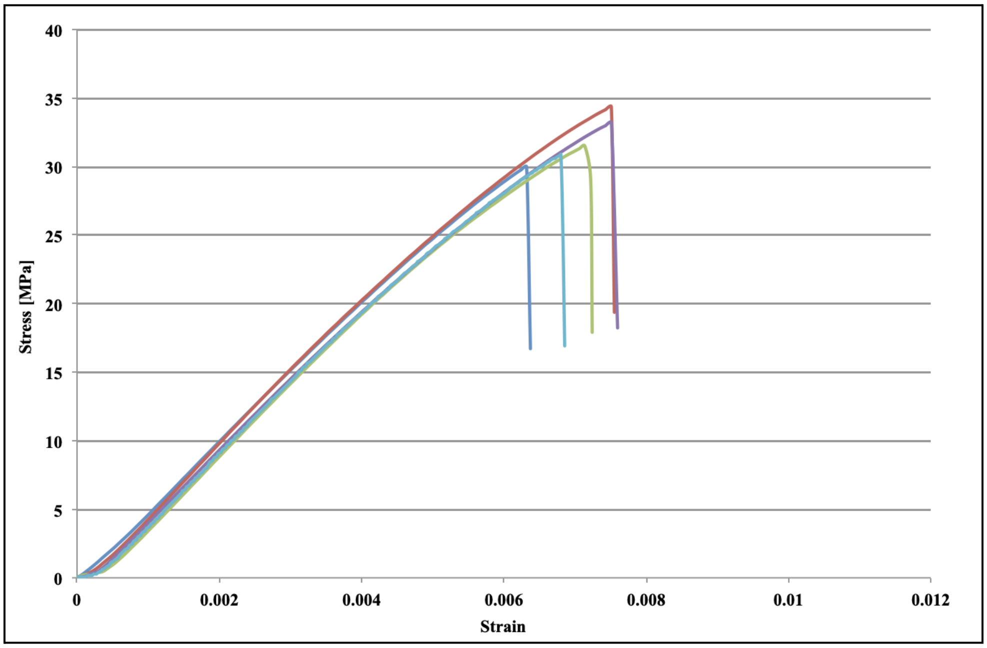

Figure 7: Stress-Strain curve for CF-PPS fabricated via SLS

The tensile test results provided several insights into CF-PPS parts made using the SLS process. First, the bars demonstrated extremely brittle behavior having little to no ductility. No visible necking of the bars occurred during the tensile tests. Second, the average ultimate tensile strength of the CF-PPS was measured to be just under 32 MPa. This was much lower than expected for CF-PPS (previously tested PPS parts averaged near 43 MPa) but this shortcoming can be attributed to several things. The scanning parameters developed in this study were not optimized for strength in any way. Further exploration of scanning parameters could increase the strength. Also, when breaking out the bars from the builds, the powder bed was very loose, showing no signs of being near the material's melting point. This indicates the preheat temperature could be significantly increased in the future which plays a critical role in creating fully dense and stronger parts. Furthermore, the tensile bars had slight shifting in the Y direction which could potentially cause misalignment of the tensile testing grips, introducing a bending moment during the tensile test and ultimately reducing the ultimate tensile strength. Much higher strengths could be achieved in the future by addressing some of these key points. One additional insight taken from the stress-strain curve was the average modulus of elasticity of the CF-PPS samples, which was calculated to be 4.66 GPa. The carbon fiber reinforcement significantly stiffens the polymer resulting in an increased modulus of elasticity when compared to previously built PPS parts with a modulus of approximately 2.83 GPa.

Geometrical Accuracy Results

The results from the geometrical accuracy tests are housed in Table 2. The nominal dimensions for the overall length, overall width, and width of the narrow section are 115 mm, 19 mm, and 6 mm, respectively for the ASTM Type IV tensile specimen. The measurements from the four tensile specimens were averaged, and then the percentage error from nominal was calculated.

Table 2: Results from geometrical accuracy testing

	Overall Length (LO)	**Overall Width (WO)**	**Width of Narrow Section (W)**
Nominal [mm]	115	19	6
Average [mm]	112.09	21.77	7.15
Standard Deviation [mm]	0.17	0.11	0.16
Percent Error [%]	2.53	14.57	19.08

It can be observed in the results of the geometrical accuracy testing that the tensile specimens were much closer to the nominal dimension in the X direction than they were in the Y direction. This can be directly attributed to shifting in the Y direction that was observed in the final tensile specimens. If a new layer is shifted slightly compared to the layer below it, this will cause oversized dimensions in the Y direction. This phenomenon was observed in the overall width, and width of the narrow section, the two dimensions controlled by the Y direction accuracy. This is the same phenomenon that was discussed previously that led to lower tensile strengths than previously tested samples. The results show that further optimization is needed in both the X and Y direction to allow for manufacturing of tight tolerances.

Compression Test Results

The compression test was a quick look at how the CF-PPS material would hold up when subjected to a compression force similar to how the electronics mold will be used. The electronics mold will be clamped together with a force of just below 9 kN. With a surface area of approximately 10,000 square millimeters, the mold will only be subjected to about 0.9 MPa of stress. In the compression test, the CF-PPS part was loaded up to 95 kN corresponding to a stress of around 190 MPa in the part tested. The part survived the compression and showed no signs of cracking or failure. There was some deformation of the part but most of this is believed to be due to densification from the part's porosity. This test provided evidence that the electronics mold will be able to handle the compression force even at elevated temperatures as the CF-PPS piece showed no signs of failure at a stress over 200 times the stress it will be subjected to during operation.

Mold Application Prototyping

Figure 8: Completed electronics mold

The development of the processing parameters detailed throughout this paper were all in an effort to fabricate the electronics mold that the sponsor desired. Figure 8 shows the finished mold that was built using the previously determined parameter set. The mold turned out very good with sharp geometrical accuracy and no issues with the shifting that the tensile bars had. The only issue with the mold was the male mating pegs, seen in the top right corner of the figure, were oversized. This was contributed to a file issue and they were subsequently sanded down to fit into the female counterpart. The mold was packaged and shipped to the sponsor for it to undergo testing for use in the field. Early results from this testing are promising and validate the potential use of additively manufactured CF-PPS for a multitude of tooling applications.

Conclusion

The purpose of this research study was to develop selective laser sintering parameters for a novel CF-PPS material in order to fabricate a repair mold for an electronics cable assembly. In order to develop parameters, first, a series of tests were performed to determine the suitability of the material for the SLS process. Next, important scanning parameters were identified using a shimmer test and parameter grids. Once parameters were selected, tensile bars were fabricated in order to quantify several performance aspects of the material. Finally, the repair mold was built using the identified parameters and shipped to our sponsor to undergo further testing. The following results summarize the findings of this research:

- A set of parameters were developed for fabricating CF-PPS parts utilizing the selective laser sintering process (Summarized in Table 1)

- Several properties of the CF-PPS parts were quantified:
    - Ultimate tensile strength - 32 MPa
    - Modulus of elasticity - 4.66 GPa
    - Geometrical accuracy summarized in Table 2

- The CF-PPS material withstood compression stresses 200 times larger than the expected operating conditions of the mold

- A usable repair mold was fabricated and shipped to the sponsor for further testing

References

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