Material Characterization and Reduction of Thermal Deformation of Continuous Carbon Fiber Reinforced PEEK Parts Produced by Open Environment Fused Filament Fabrication

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Introduction

This report was produced in collaboration with the Connecticut Center for Advanced Technology (CCAT) using an Ingersoll robotic arm designed to print Continuous Carbon Fiber (CCF) Reinforced High Temperature Polymer (PEEK) parts by Fused Filament Fabrication (FFF) in an open-air environment. Manufacturing process parameters such as line width, nozzle diameter and geometry, and temperatures of the bed and nozzle were refined to enable effective extrusion and deposition of the composite material. Given PEEKs high extrusion temperature (450 ℃) compared to ambient air, heated build chambers are conventionally used to prevent the development of thermal gradients in the part during the print process which lead to thermal deformation. Using a heated build chamber to maintain a uniform ambient temperature is not applicable to the Ingersoll robotic arm's large build volume and ambient air operating temperature.

The correlation of maintaining an increased uniform ambient temperature during the print process with elimination of thermal deformation was confirmed via existing mathematical models, and a transient Ansys FEA thermal and mechanical simulation using an element kill/alive method to simulate the print process. Plans for an open environment infrared radiation heating system were developed to maintain constant part surface temperature during the print process. This system was designed to be integrated with an existing IR thermal camera feedback-based control system. The effects of varying fiber volume fraction on the microstructure and tensile strength of CCF Reinforced PEEK parts produced by FFF were also evaluated.

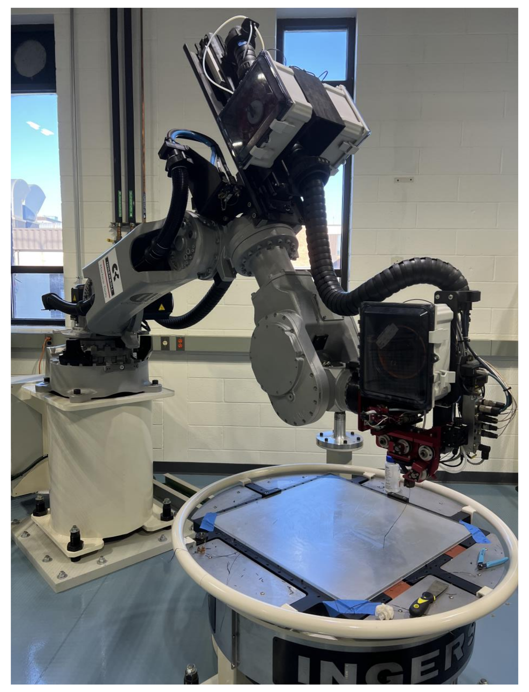

***Figure 1:** Ingersoll MasterPrint – 6 axis Continuous Fiber Reinforced High Temperature Polymer 3D Printer.*

Problem Validation

Warping is a major problem in the context of Fused Filament Fabrication 3D printing, especially on large prints that are exposed to the ambient air. Parts thermally deform when a temperature gradient develops, causing different sections of the part to expand and contract as a result of the coefficient of thermal expansion of the material. These expansions and contractions induce a bending moment which overcomes the force of the parts adhesion to the print bed. Thermal deflection from an FFF process can be estimated as a function of several process parameters such as part geometry and ambient air temperature using the analytical model of *Armilotta et al*, as seen below in Figure 2. Deflection significantly decreases approaching the materials glass transition temperature.

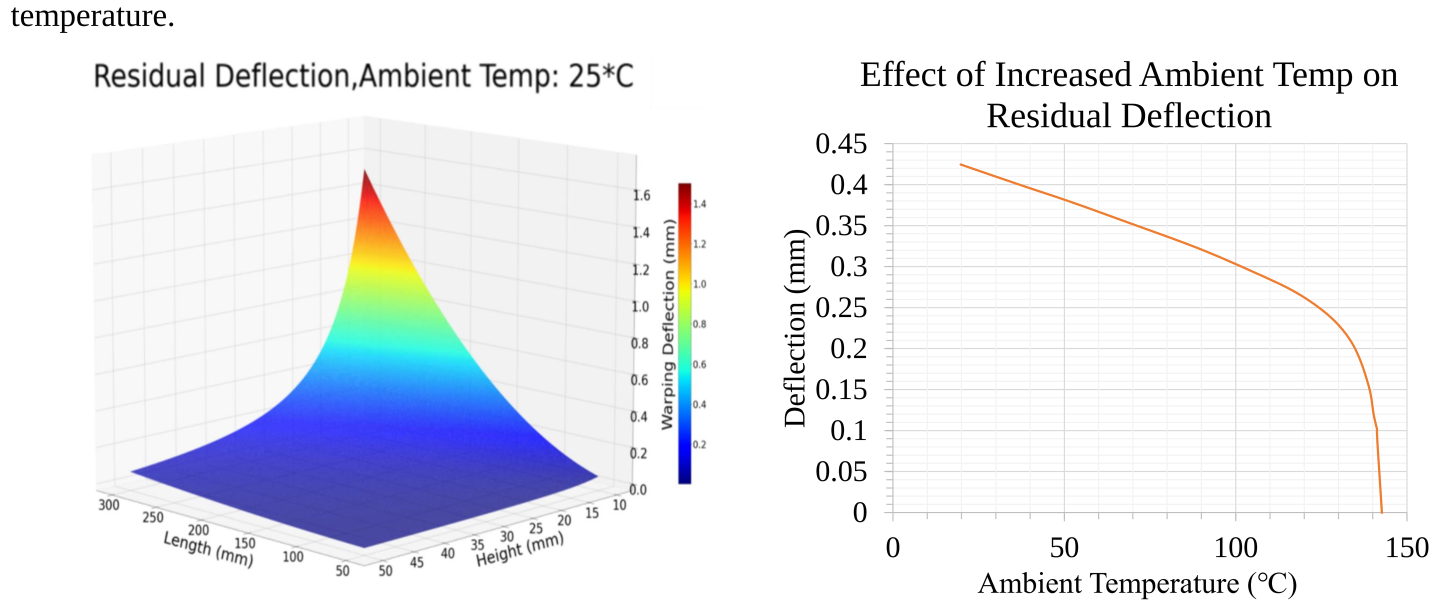

***Figure 2:** Warping model of Armillotta et al: Residual deflection due to warpage for a flat rectangular plate as a function of: Thermal properties, chamber (ambient temp), layer height, and geometry. (Left) part geometry is an independent variable. (Right) Ambient temperature is an independent variable.*

A warp-specimen as seen if figure 3 was designed to exaggerate the effects of warping to allow measurement. Note the shredded fibers. Longer geometries create a longer moment arm to induce a thermal bending moment. Thicker parts have a higher bending-stiffness to resist the warping force. Inversely, thin geometries are more flexible and prone to exhibiting thermal deformation. Therefore, a long, thin rectangular shape was chosen as the warp-specimen geometry.

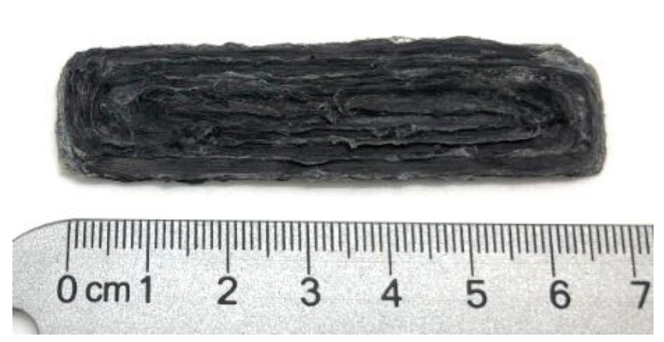

***Figure 3:** Final print result of warp-specimen sample geometry*

Process Optimization

G-code was generated for each test-specimen shape using a modular, parametric Python algorithm that allowed for adjustments to line-width and part geometry. Initially using CCF/PEI (Ultem 1100) the dogbone shaped toolpath with sharp turns was possible. Vision Miner nano polymer adhesive is helpful for PEEK bed adhesion.

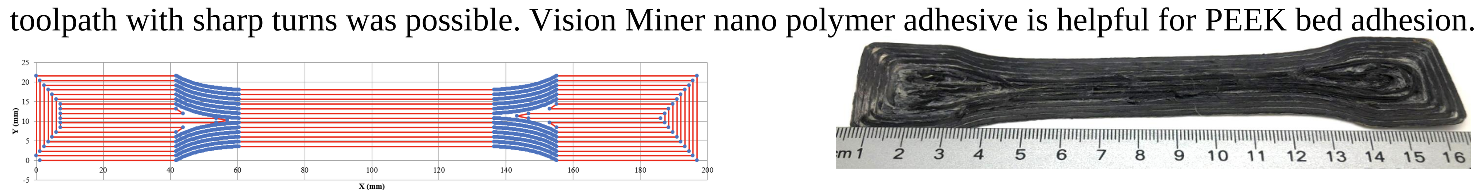

***Figure 4:** Toolpath of tensile-test specimen created with Python (left) and final print result with shredded fibers (right)*

A barbell specimen toolpath was used to avoid 90º turns and prevent fiber shredding. Other changes made that enabled effective printing:

- Increase of layer height from .33 to .45 mm
- Increase of extrusion temperature from 415 to 450 °C
- Increase of bed temperature from 100 to 150 °C
- Increase of nozzle diameter from 1.2 to 2.0 mm
- Adjustment of nozzle chamfer to reduce fiber shredding

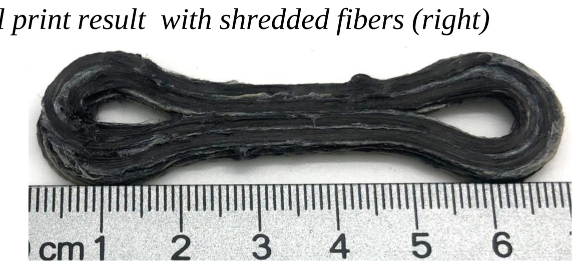

***Figure 5:** Final print results of barbell sample specimen geometry*

Ansys Print Simulation

The purpose of using ANSYS FEA simulation is to print using the optimum process material heating temperature and ambient temperature, with minimal control of the smallest temperature differences in mechanics, to help design a simulated heating device.

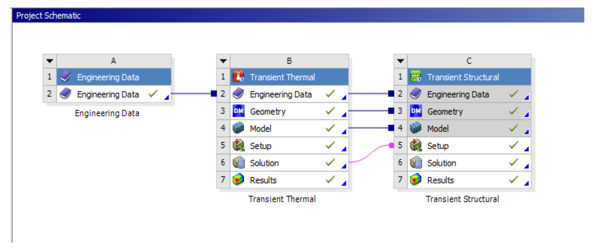

***Figure 6:** FEA simulation schematic*

Process of using ANSYS Thermal Simulation:

1. Material data of CCF/PEEK obtained through experiments and research is entered into the Engineering Data Source in Ansys.
2. Create a Transient Thermal module in the Workbench, import the geometric model created in SOLIIDWORKS, and set the initial temperature and boundary conditions.
3. Utilize *Element Kill and Alive* for contact elements using the APDL command, realize the process of simulating the CCF/PEEK material as it is gradually extruded from the printer nozzle, and adjust the boundary conditions of each element over time through the APDL command, thereby improving the accuracy of the simulation.

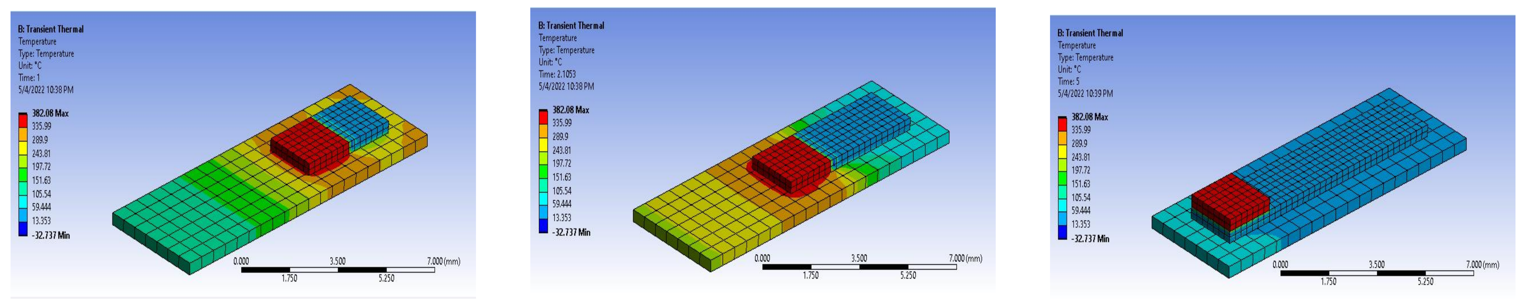

***Figure 7:** Using Kill and Alive command to simulate printing process*

4. Calculate the temperature change of each node in the model over time through the Transient Thermal module and import the result into the newly created Transient Structural module as its set up.
5. Perform operations through the Transient Structural module to calculate the Total Deformation, Equivalent Stress, and Equivalent Elastic Strain of the model.
6. Each model performed five sets of simulations under different ambient temperatures to obtain the influence of different ambient temperatures on thermal deformation.

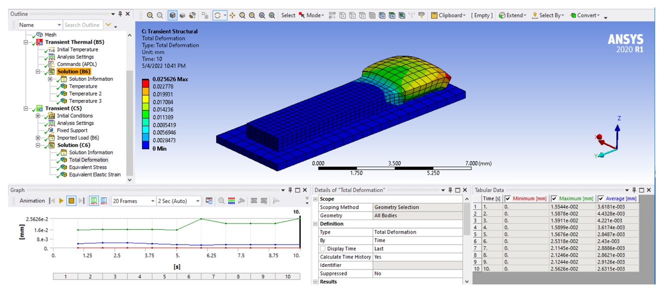

***Figure 8:** FEA model simulating exaggerated effects of thermal deformation*

Thermal Imaging Analysis

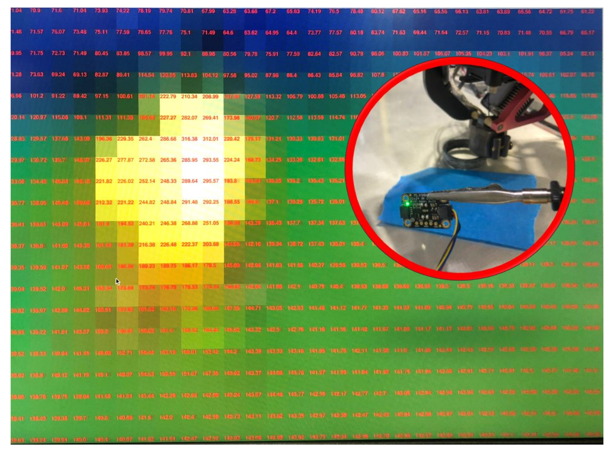

***Figure 9:** Thermal Image of Extruder at Beginning of Print*

An infrared thermal camera (Adafruit MLX90640) was used to image the print process of a single wall CCF/PEEK cylinder to validate the cameras efficacy for adaptation in a solution. An initial thermal image on the left (Figure 9) shows the part in the early stages of its creation before it could develop a significant thermal gradient that could be identified visually.

Tape was added in front of the part as it was believed the surface of the glass bed was reflecting IR radiation and giving false readings. The cameras resolution (32x24 pixels) also proved insufficient to capture the smaller parts temperature. Interestingly, the bed also read at 130 rather than 150 °C as it was set.

To the right in Figure 10, a thermal image later in the print can be observed. The print is now significantly larger, and its thermal gradient is capable of being detected by the camera. Its gradient was less drastic than expected for its height with its minimum temperature values at about 95 ℃. However, this value still significantly differs from the bed temperature and the extrusion temperature of 450 ℃. For future image processing it is intended to neglect any pixels that are at the bed temperature or approaching extrusion temperature.

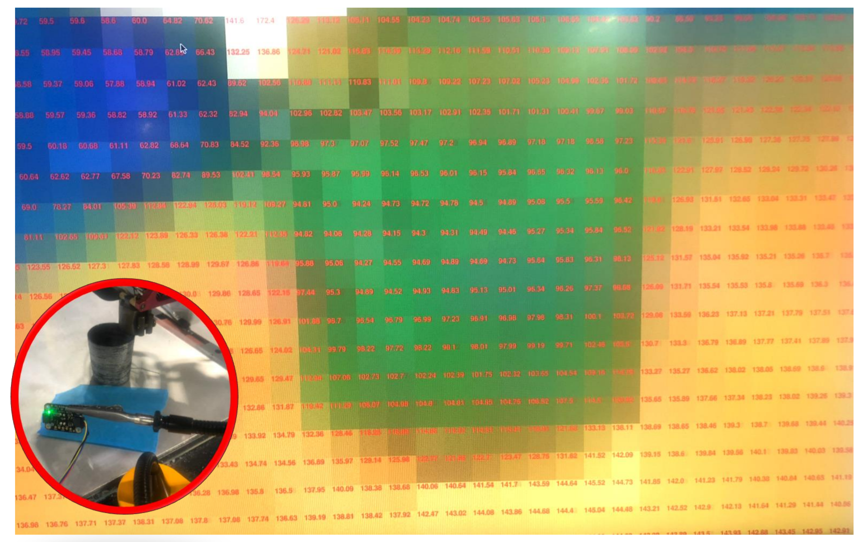

***Figure 10:** Thermal Image of Developing Temperature Gradient*

Acknowledgements

- Sam Greenbank, CCAT
- Dr. Wout De Backer, University of South Carolina
- The Connecticut Center for Advanced Technology
- Christina Alcaro, Trinity College
- NASA CT Space Grant Consortium
- Suprem, CCF/PEEK Filament Manufacturer
- Digesh Chikratar, Trinity College
- Dr. Timothy Curran, Trinity College

Material Characterization

Dumbbell specimens (as seen in Figure 5) were subjected to testing in Trinity's Instron tensile-tester to determine the yield strength of the CCF/PEEK material. The yield strength was measured upwards of 778 Mpa as seen in Figure 11.

This tensile strength is the third known highest in the world recorded for an AM continuous fiber reinforced composite with Tekinalp et al. in second with 800 MPa and Werken et al. in first with 1134.3 MPa prior to post processing.

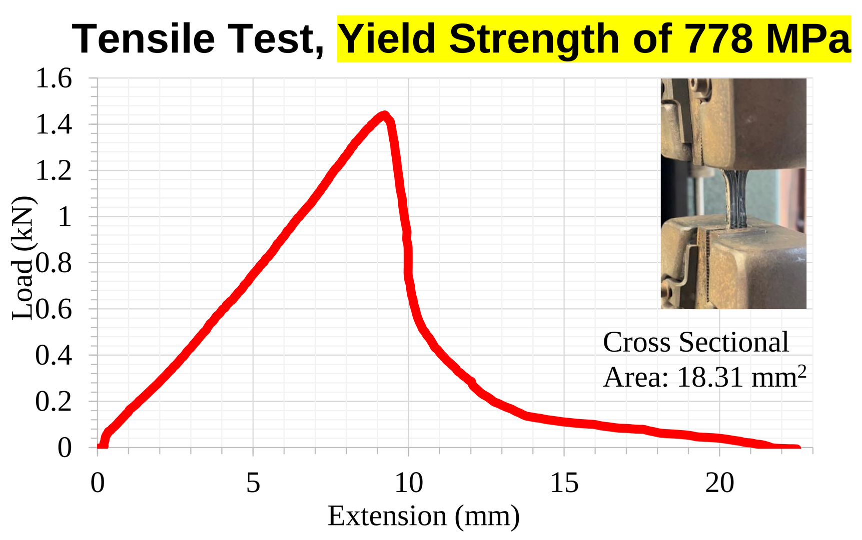

***Figure 11:** CCF/PEEK tensile test conducted with Instron-5500R*

It is important to note the circumstances of this test as it is it believed the true tensile strength of this material is significantly higher. This test was the third test on the same specimen. Additionally, the specimen failed in the curved end due to stress concentrations from the clamps rather than in the gage.

Microscopy was conducted on samples of CCF/PEEK to evaluate the microstructure of the printed material. Figure 12 (Left) depicts the cross section of two beads with fiber running transverse to the viewer. Visible is the bonding between the two beads. Figure 12 (right) depicts a cross section of the gage of the dumbbell test specimen. Based on the complete saturation between layers and passes and impregnation of the matrix material into the fiber it can be predicted that this specimen has excellent interlaminar shear strength.

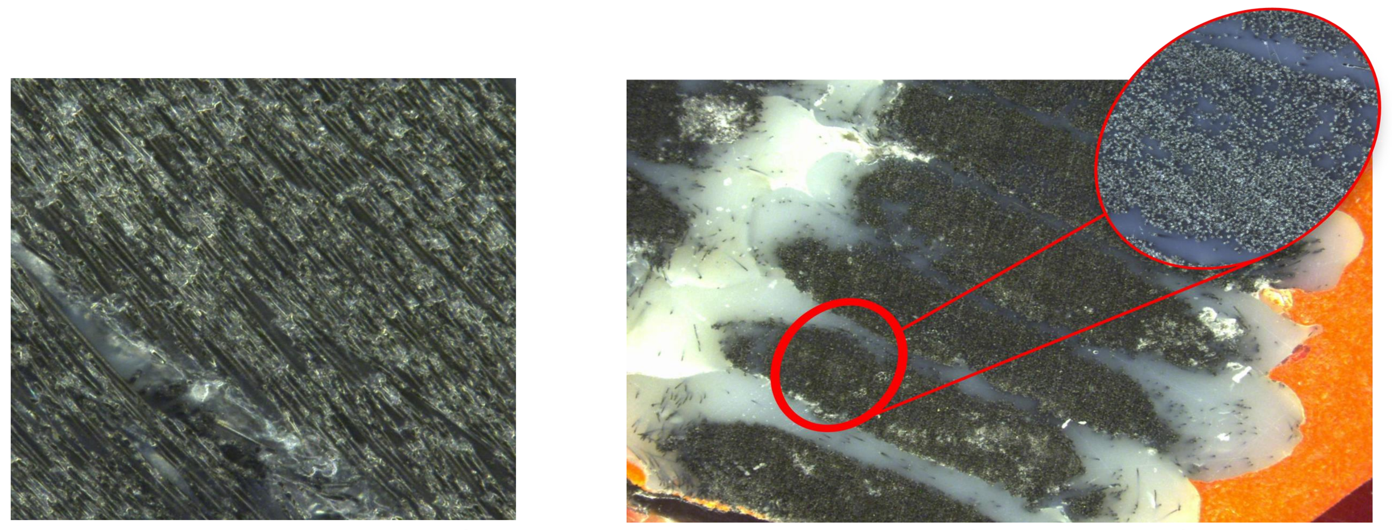

***Figure 12:** SEM Microscopy image of CCF/PEEK material taken transverse to fiber direction (left) and longitudinal to fiber direction with multiple beads (right)*

Small samples of CCF/PEEK were subjected to infrared-spectroscopy to determine the wavelength of IR light which is most absorbed into the material and transferred into heat. This data was used to determine how much power to provide the ceramic heater to ensure emission wavelengths are in the optimal range. Samples were also subjected to SEM microscopy to determine the fiber/matrix ratio in each sample.

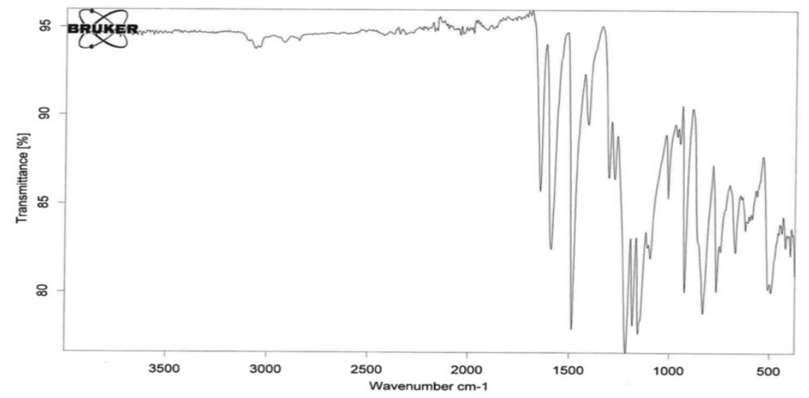

***Figure 13:** Infrared-Spectroscopy results of CCF/PEEK sample conducted on the 'Bruker Corporation' Alpha Fournier- Transform Infrared Spectrometer (FT-IR)*

Prototype Solution

The system is designed to minimize the thermal gradient in the CCF/PEEK part as it prints, thus reducing thermal deformation. The system consists of an array of infrared ceramic heaters controlled by a Raspberry Pi 4B interfaced with a thermal camera. A solid-state relay was also implemented such that a 3.3V signal from the Raspberry Pi triggers a switch to open and allow the 120V voltage source to power the ceramic heater. This provides the ability to turn the heater on/off quickly based on feedback from the camera. The code is designed such that the temperature reading from the thermal camera is processed through the Raspberry Pi to determine whether the ceramic heater should be powered. The system is designed to keep the printing part just below the glass transition temperature (Tg) of PEEK at 143º Celsius. Temperature readings below 140º Celsius trigger the ceramic heater to turn on and a temperature reading at 140º Celsius triggers the ceramic heater to turn off. This feedback loop minimizes the thermal gradient in the part as it is printed and decreases thermal deformation.

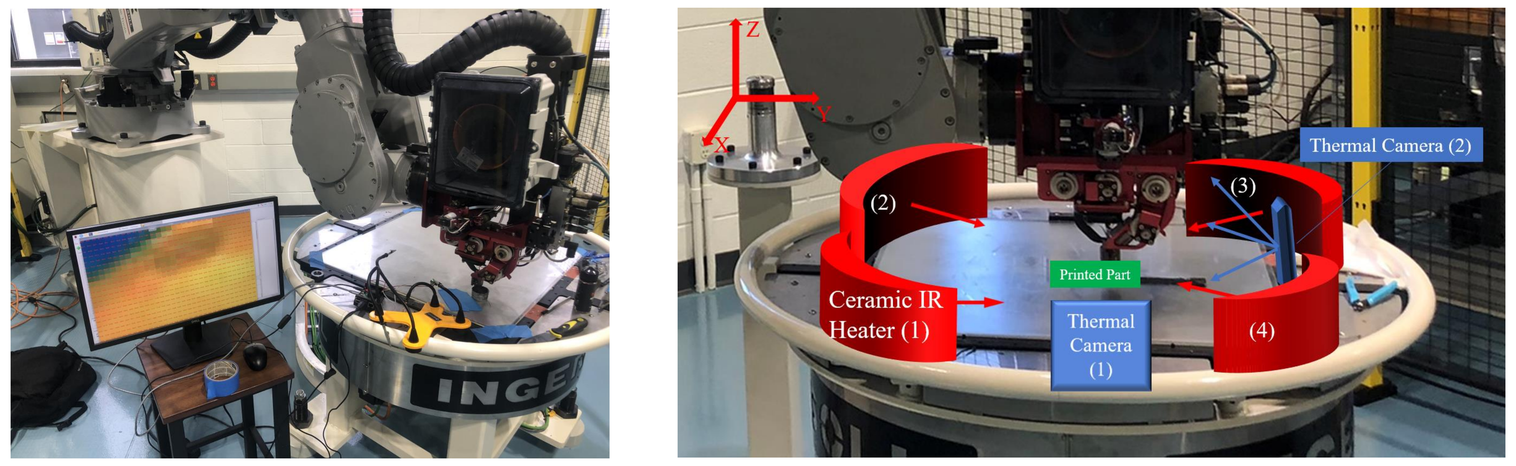

***Figure 14:** (Left) Thermal camera output displayed while printing CCF/PEEK. (Right) Design of thermal system to minimize warping*