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Multi-Scale Analysis of Multifunctional Composites and 3D Printed Polymers

Daniel Branden Perez
University of Miami, umfan92@gmail.com

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MULTI-SCALE ANALYSIS OF MULTIFUNCTIONAL COMPOSITES AND 3D PRINTED POLYMERS

By

Daniel Branden Perez

A DISSERTATION

Submitted to the Faculty of the University of Miami in partial fulfillment of the requirements for the degree of Doctor of Philosophy

Coral Gables, Florida

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MULTI-SCALE ANALYSIS OF MULTIFUNCTIONAL COMPOSITES AND 3D PRINTED POLYMERS

Daniel Branden Perez

Approved:

________________         _________________
Ryan L. Karkkainen, Ph.D.  Victoria L. Coverstone, Ph.D.
Assistant Professor of Mechanical Engineering  Professor and Chair of Mechanical Engineering

________________        _________________
Emrah Celik, Ph.D.   Guillermo Prado, Ph.D.
Assistant Professor of Mechanical Engineering  Dean of the Graduate School

________________
Edward A. Dauer, M.D.
Research Associate Professor of Biomedical Engineering
Methods of multi-scale analysis through combined physical testing and computational FEM were used to investigate structural battery composites and 3D printed polymers. Multifunctional composite structural batteries are materials capable of storing electrical energy while providing structural rigidity. Two battery cell configurations were considered: a carbon paper based cell with copper and aluminum foils, and a woven carbon fiber-based battery with nickel and iron deposition coatings. Flexural simulations were performed through simulated and physical three-point bend testing.

Unidirectional carbon fiber layers in the place of the carbon paper can lead to up to a 233.73 GPa stiffness, significantly greater than the original 13.79 GPa. Honeycomb core can be used to retain carbon paper layers with additional thickness but increased flexural rigidity. Micro-scale electrical current distribution analysis of carbon fiber reinforced composites was performed to investigate improving the overall carbon fiber conductivity. The conductivity of the epoxy matrix conductivity is the most dominant driving force for overall resistance/resistivity of carbon fiber composites. Full-scale panel level simulations assessed the stress concentrations created by changes in panel design can be mitigated with stiff gasket material that reduces sharp panel curvature. Reinforced Cu-Al prototypes were tested in three-point bending; metal foils created a barrier preventing resin flow and left voids and defects resulting in large delaminations.
For accurate strain measurements, a custom digital image correlation (DIC) procedure was created using open source software Digital Image Correlation Engine (DICe). A high-resolution camera captures images that, after processing, provide 2D strain fields and a virtual strain gauge that can then be used to generate stress-strain curves and calculate material stiffness. Verification of this technique was performed with 6061 aluminum specimens of known stiffness and resulted in an average error of 2.01% from the literal stiffness value.

Multi-scale analysis methods were applied to FDM 3D printed polymers to understand and improve the mechanical behavior. Tensile testing of orthogonal printing orientations showed significant differences in the failure characteristics and tensile strength of printed polymers. Extrusion temperatures, 180°C, 195°C, and 220°C were used to print specimens tested in tension pulling apart the interlayer interfaces to study the effect of temperature on the interface strength. Increasing extrusion temperatures creates a trade-off between achieving adequate interlayer bonding without degradation of the polymer.

A custom 3D printed specimen and tensile specimen preparation methodology was created to create a consistent cross-section to create a closely matching computational model. The results of the tensile tests were used to parameterize the interface strength using the models. The PLA interface strength was determined to be 33.75 MPa through FEM modeling of matching geometry, compared to the 43.57 MPa filament strength. Additional models were generated which had reduced or eliminated inter-extrusion gaps to determine the potential mechanical improvements. Eliminating gaps can lead to up to a 16.12% improvement in stiffness and a 19.8% improvement in strength.
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Chapter 1 Introduction

1.1 Multi-Scale Analysis of Materials

Multi-scale analysis is the process of using information obtained from the analysis of features at different length scales of the microstructure within a material and using this information to inform continuum-scale material system development. Multi-scale analysis can combine physical material testing with finite element method (FEM) based simulations that directly represents microstructure. Physical testing provides a direct measurement of the continuum-scale behavior, along with microscopic inspection to identify key features of a given microstructure. Computational FEM is utilized to directly model these key features and their effects on higher scale behaviors.

Two material systems were evaluated in this work: multifunctional structural battery composites and 3D printed polymers. Details of the microstructures are explicitly represented for each of these material systems. The principles of multi-scale analysis were applied to evaluate the flexural performance of structural battery laminates, discussed in Chapters 2 – 5, and the tensile properties of FDM 3D printed polymers, discussed in Chapters 6 - 9.

1.2 Structural Battery Composites Overview

Energy storage requirements are increasing in industries such as electric and hybrid automobiles, aircraft, spacecraft, and consumer electronics. As energy demands increase, additional batteries must be used to meet the demands. However, conventional batteries are often heavy and large, reducing available payload capacity. A structural battery composite is a material system that can store and deliver electrical energy while being built into the structure of the system. This composite would replace existing structural
components, providing additional electrical energy without increasing weight or reducing available space. There are a wide range of applications for this technology where weight and energy are prime commodities such as aircraft, orbital systems, and automobiles. The additional electrical energy could power onboard electronics, increase range, and increase efficiency. This research investigates performance and tradeoffs when incorporating structural composite materials with solid state battery technology. Computational FEM electro-mechanical simulation is used to investigate mechanical properties of multiple configurations of structural battery composites and reinforcing solid state battery technology with carbon and glass fiber composites. Multiple review articles have examined the various multifunctional structural battery technologies investigated [1–3]. Lithium ion and elastic polymers have been used to produce structural batteries that can provide tunable mechanical properties [4]. Solid lithium polymer electrolytes may potentially be incorporated into structural batteries for their high ionic conductivities along with mechanical performance [5, 6]. In addition, thermal analyses of a lithium-ion type cell integrated with honeycomb or truss core determined the viability of the technology in space applications [7].

Previous research by one of the authors has shown promise in using carbon fiber paper as an electrically conductive reinforcing layer in solid state batteries and hybrid supercapacitors [8].

The use of mediator molecules has been shown to improve ionic conductivity and permitted the use of solid-state materials as a battery [9]. While the carbon paper is suitable for electrical performance, it provides relatively lower structural capability.
Volvo, a major automobile manufacturer, has considered incorporating structural batteries into their vehicles to reduce weight and extend range [10]. Additional studies have explored electrolytic resins combined with carbon fiber anodes with a metal substrate as a current collector [11, 12], however, the use of metallic electrodes with carbon fiber current collectors was not explored. In addition, past research has focused on structural electrolytes either in gel or solid form as a method to increase mechanical properties while maintaining ionic conductivity [13–15], where the structural battery composed of carbon fiber electrodes, a polymer matrix, and a separator was tested experimentally.

Previous works by the authors have presented the effective use of FEM-based micromechanical analysis in predicting stiffness and strength of unidirectional and woven textile composites. These works have examined 2D and 3D composites under a variety of failure modes that are not readily approachable through closed-form analytical methods. Dedicated analysis of direct detailed microstructural representations has shown to be effective for strength prediction, including multiaxial loading, and dynamic impact loading [16–17].

The FEM simulations focused on the effect of different materials and orientations of the carbon layers in the solid-state battery unit cell. The flexural stiffness and strength of the structural battery laminate are the primary focus, as this material will used to replace body panels. Specifically, the structural battery will be employed in a 1U CubeSat by replacing the aluminum chassis to store electrical energy to power onboard electronics. FEM simulations were carried out with Abaqus/CAE™ and COMSOL Multiphysics® to virtually model the various materials and configurations to analyze flexural stiffness and strength at the laminate level, as well as preliminary. In addition, CubeSat panel scale
simulations were done to assess the mechanical behavior and the impact of reinforcing the battery with woven carbon fiber materials as well as incorporating unidirectional carbon fiber layers interstitially between battery layers. By combining the solid-state battery technology with high stiffness/strength carbon fiber composites, the mechanical performance is improved while retaining the electrical storage capability. The solid-state battery unit cell is a material capable of storing electrical energy without containing any liquid electrolyte, however the mechanical performance must be improved. The focus of this study is to investigate reinforcing the solid-state battery with high stiffness/strength composites such as woven carbon fiber and glass fiber composites.

In addition to the panel level structural simulations, a micromechanics approach was utilized to analyze the electrical performance of carbon fiber reinforce polymer composites. Typical epoxies used as a matrix for carbon fiber composites offer no electrical conductivity. Conductive epoxies can be substituted to improve the overall electrical performance of these composites. The micromechanics analysis aims to parameterize the epoxy conductivities to determine whether it is possible to incorporate carbon fiber composites as a multifunctional material being part of the structural reinforcement as well as active battery material.

Physical and virtual three-point bend tests were completed on these material systems. Prototype panels were fabricated and tested in a three-point bend test configuration until ultimate failure. The damage initiation and propagation were analyzed through qualitative and quantitative analyses. Electrical testing and characterization were performed on prototype cells to assess metrics such as energy density and power density.
Two battery material systems have been evaluated mechanically in this study. The first battery cell consists of thin copper and aluminum metal foil electrodes surrounding carbon paper current collectors, which will be referred to as the Cu-Al foil battery in this paper.

![Schematic of layers in Cu-Al battery](image)

Figure 1. (a) Battery cell schematic for the Cu-Al unit cell using metal foils. (b) Schematic of carbon fiber weave Ni-Fe battery cell.

A schematic of the layers is shown in Figure 1 (a). The carbon paper is treated with mediator molecules which have been shown to improve ionic conductivity in solid state batteries [8, 9]. Carbon paper is a fabric consisting of discontinuous, randomly oriented carbon fibers. While being an excellent electrical conductor, it is a structurally weak material. The copper anode and aluminum cathodes are thin 15-micron foil and offer superior ionic conductivity over solely carbon fiber electrodes. The carbon fiber reinforcement layers provide structural support as well as serving as current collectors. The dielectric layer is the separator between the electrodes.

The second battery system consists of woven carbon fiber textile electrodes on either side of a PVA polymer separator layer. Nickel and iron metals are deposited onto the surfaces of carbon fiber fabrics through electrodeposition to aid with conductivity, and therefore will be referred to as the Ni-Fe battery. This battery cell is shown in Figure 1 (b).
Incorporating carbon fiber fabric as part of the battery cells is preferred due to the superior mechanical properties of carbon fiber reinforced polymer (CFRP) composites, without the need for additional reinforcement layers. In the case of the Cu-Al metal foil battery cells, woven CFRP layers can be added surrounding the cells to provide necessary additional structural reinforcement.

Estimated energy and power requirements were based on a 1U CubeSat application. Employing a structural battery composite in the chassis of a CubeSat can augment the onboard battery cells. It was determined that based on the expected energy and power density of the battery unit cells, seven layers would be necessary for a small CubeSat application as a prototype test of the structural battery composite. Seven layers were used for all panel scale simulations for both battery chemistries to keep the mechanical results consistent. Structural materials substitutions and composite reinforcements were investigated to achieve a fully multi-functional composite laminate.

1.3 FDM 3D Printed Polymers Overview

Fused deposition modeling (FDM) is a process of additive manufacturing of polymers by extruding molten filament onto previously deposited layers until the part is completed. Due to the relatively low-cost components required, and the widespread access, this process allows for cost efficient rapid prototyping of complex geometries. Recent developments have combined FDM polymer printing with unidirectional fiber-reinforced composites to produce fully printed composites. However, there are limitations such as generally low strength parts, and low dimensional accuracy. In addition, the FDM process,
further described in Chapter 6, creates unique microstructures that result in significant full-scale mechanical performance reductions.

Characterization of the micro-scale morphologies and their effects on the strength of the final printed parts was conducted through physical testing and characterization combined with computational FEM simulations. Initial full-scale analysis of parts printed in orthogonal orientations revealed the inherent anisotropy of parts due to the discontinuous nature of the layers bonded by weaker interfaces. Further investigation of these weak interfaces was accomplished through combined physical and computational analyses. A custom tensile specimen design and preparation procedure was created to create a uniform printed cross-section. This uniform cross-section is vital to isolate the effects of the interface from defects and other features created during the imperfect printing process. Determination of the strength of the interlayer interfaces was accomplished through modeling of the microstructures and matching the models to the physical tests. The interface strength was then applied to new models investigating the potential improvements gained from improving the microstructures.
Chapter 2 Flexural Analysis Approach

2.1 FEM Flexural Mechanics Methodology

2.1.1 Laminate Flexural Stiffness Determination

The flexural stiffness of each laminate is computed using the deflection at the midpoint, the load applied, and the geometry. The equation used to calculate the flexural stiffness is

\[
E_f = \frac{L^3}{48I} \left( \frac{dF}{dv} \right) = \frac{L^3F}{4wh^3v} \tag{1}
\]

where \(L\) is the length of the span of the specimen, \(I\) is the area moment of inertia of the profile about the mid-plane, \(dF/dv\) is the slope of the load-deflection curve, \(F\) is the load applied, \(w\) and \(h\) are the width and height of the specimen, and \(v\) is the deflection at the mid-point under load. The results of each simulation provide the deflection under the specified loading. The deflections are assumed to be linear due to the small loading applied.

Figure 2. Laminate test specimen mesh.
2.1.2 Laminate Flexural Strength Determination

The flexural strength is calculated by a first element failure criterion. This simplification is a preliminary test to assess each material and configuration, though future analyses will incorporate more sophisticated methods such as non-linear geometry, element deletion, and other failure criteria. For this study, the strength was defined as the point at which one element reached its tensile yield stress. Each material was analyzed to find the element with maximum stress and interpolated linearly to determine the maximum load that can be applied to the laminate. For each model, the maximum loads for every material were compared to find the lowest load at which a material’s first element reached yield. This load was identified as the failure load. The load was used to calculate the flexural strength using the equation below.

\[ \sigma = \frac{3FL}{2wh^2} \]  

(2)

2.1.3 Computational Laminate Bend Test Mesh Validation

To assess the validity of the FEM model as well as the calculation methods, a validation study was conducted through analytical solutions as well as Classical Laminate Theory (CLT). The FEM model was altered to a homogeneous isotropic material (in this case, steel) and the simulations were resubmitted for solution. No changes were made to the mesh of the model or any other parameters other than the elastic material properties. The results of the steel benchmarking test were compared to the analytical solution using beam theory. The steel properties used were as follows: E=200 GPa, v=0.3, and \( \sigma_y = 250 \) MPa.
The beam theory for maximum deflection, $\delta_{\text{max}}$, and the axial stress, $\sigma_x$, a distance $y$ away from the mid-plane are shown below.

\[ \delta_{\text{max}} = \frac{PL^3}{48EI} \]  

(3)

\[ \sigma_x = \frac{My}{I} \quad \text{where} \quad M = \frac{PL}{4} \]  

(4)

This validation test was implemented to identify large faults in the FEM model or calculation methods used to analyze the laminates being studied. The analytical solution combined with CLT provided a basis to which the results were compared to validate both the FEM model and calculation methods.

2.2 Classical Lamination Theory Overview

Classical Lamination Theory (CLT) was also used to determine the mechanical performance of the structural battery laminates. CLT is an analytical method to determine the mechanical properties of a laminate consisting of unidirectional fiber layers of known properties [18]. CLT relates the strains, $\varepsilon$, and curvatures, $\kappa$, to the in-plane loads, $N$, and bending moments, $M$ [18].
Equation (5) shows the matrices A, B, and D denote the relationship between the applied forces and resulting deformations. The D matrix is the bending stiffness matrix which relates the applied moments to the curvatures. The bending stiffness matrices (D) from CLT were calculated for each of the laminates to support the FEM results and further illustrate performance differences of several configurations. The $D_{11}$ and $D_{22}$ terms are of most importance for a bending analysis because they relate the curvature of the laminate to the applied moments, according to

$$\begin{bmatrix} N \\ M \end{bmatrix} = \begin{bmatrix} A & B \\ B & D \end{bmatrix} \begin{bmatrix} \varepsilon \\ \kappa \end{bmatrix}$$

(5)

For laminates symmetric about the mid-plane, the Bending-Extension Coupling Matrices (B) are zero, and therefore

$$\begin{bmatrix} M_x \\ M_y \\ M_{xy} \end{bmatrix} = \begin{bmatrix} D_{11} & D_{12} & D_{16} \\ D_{12} & D_{22} & D_{26} \\ D_{16} & D_{26} & D_{66} \end{bmatrix} \begin{bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{bmatrix}$$

(6)

In this analysis, the most important values are the bending stiffnesses, $D_{11}$ and $D_{22}$. The bending stiffnesses relate the curvatures in the x and y directions due to the moments applied in the x and y directions, respectively. The CLT analysis also serves as a validation.
for the FEM model to ensure that the model is accurate. A direct comparison to the FEM simulations can be made by calculation of the flexural stiffness, $E_{fs}$, which can be determined from the inverse of equation (5) to represent the strains in terms of the applied loads [19].

$$\begin{bmatrix} \varepsilon \\ \kappa \end{bmatrix} = \begin{bmatrix} a & b \\ c & d \end{bmatrix} \begin{bmatrix} N \\ M \end{bmatrix}$$  \hspace{1cm} (7)

From equation (7), matrices [a], [b], and [d] are the extensional compliance matrix, coupling compliance matrix, and bending compliance matrix respectively [19]. The first term of the bending compliance matrix is used to calculate the effective laminate flexural stiffness.

$$E'_{fx} = \frac{12}{t^3d_{11}}$$  \hspace{1cm} (8)

In this equation, the thickness, $t$, is to the third power. This is the reason that the flexural stiffness can be deceptively low for thicker laminates, even if they are capable of withstanding larger flexural loads. The flexural stiffness is normalized by the thickness; therefore, design changes which increase the laminate thickness can spuriously decrease the modulus as traditionally defined, even when the resistance to deflection for a given load is stiffer. Because of this, it may be beneficial in certain circumstances to characterize a laminate based on the $D_{11}$ terms from the bending stiffness matrix, rather than the normalized flexural stiffness property.
Chapter 3 Solid-State Battery Unit Cell FEM Mechanical Modeling

3.1 Flexural Rigidity and Strength Improvements to SB Unit Cell through Carbon Fiber Material Substitution

The flexural performance of the laminate was analyzed for multiple materials in several configurations. The FEM models consisted of eight plies of the unit cell (shown in Figure 1) and tested in a three-point bend test. The total thickness of the 8-ply laminate is 2mm. Each structural battery ply contains two carbon layers, therefore there are 16 layers that could be altered individually. In order to improve the mechanical performance of the laminate containing carbon paper, a 3.175mm honeycomb layer was inserted at the center of the 8-ply beam specimen. Therefore, the thickness of the carbon paper/honeycomb test specimen is 5.175mm. For this study, three main configurations were examined: all carbon layers unidirectional, and two cross-ply orientations. The two cross-ply orientations are both symmetric to eliminate any shear-extension or bending-extension coupling. The two fiber configurations are represented below in Table 2.

The layups were chosen due to distribute properties in both the 0° and 90° directions in anticipation of future efforts in plate analysis, though it will perform better in bending in the 0° direction (as the outermost layer is in this direction).
Table 1. Constituent material properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_1$ (GPa)</th>
<th>$E_2$</th>
<th>$E_3$</th>
<th>$v_{12}$</th>
<th>$v_{13}$</th>
<th>$v_{23}$</th>
<th>$G_{12}$ (GPa)</th>
<th>$G_{13}$</th>
<th>$G_{23}$</th>
<th>$\rho$ (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Paper</td>
<td>0.50</td>
<td>-</td>
<td>-</td>
<td>0.25</td>
<td>-</td>
<td>-</td>
<td>0.20</td>
<td>-</td>
<td>-</td>
<td>0.45</td>
</tr>
<tr>
<td>Copper</td>
<td>117.00</td>
<td>-</td>
<td>-</td>
<td>0.34</td>
<td>-</td>
<td>-</td>
<td>45.00</td>
<td>-</td>
<td>-</td>
<td>8.96</td>
</tr>
<tr>
<td>Aluminum</td>
<td>69.00</td>
<td>-</td>
<td>-</td>
<td>0.33</td>
<td>-</td>
<td>-</td>
<td>24.00</td>
<td>-</td>
<td>-</td>
<td>2.70</td>
</tr>
<tr>
<td>Resin</td>
<td>3.30</td>
<td>-</td>
<td>-</td>
<td>0.35</td>
<td>-</td>
<td>-</td>
<td>1.20</td>
<td>-</td>
<td>-</td>
<td>1.25</td>
</tr>
<tr>
<td>Honeycomb</td>
<td>3.03</td>
<td>0.69</td>
<td>6.69</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.01</td>
<td>0.09</td>
<td>0.37</td>
<td>0.20</td>
</tr>
<tr>
<td>AS4</td>
<td>138.00</td>
<td>9.00</td>
<td>9.00</td>
<td>0.30</td>
<td>0.30</td>
<td>0.34</td>
<td>6.90</td>
<td>6.90</td>
<td>1.70</td>
<td>1.79</td>
</tr>
<tr>
<td>IM7</td>
<td>141.00</td>
<td>9.05</td>
<td>9.05</td>
<td>0.34</td>
<td>0.34</td>
<td>0.64</td>
<td>6.34</td>
<td>6.34</td>
<td>2.94</td>
<td>1.78</td>
</tr>
<tr>
<td>P100</td>
<td>468.90</td>
<td>6.20</td>
<td>6.20</td>
<td>0.31</td>
<td>0.35</td>
<td>0.35</td>
<td>5.58</td>
<td>5.58</td>
<td>1.17</td>
<td>1.80</td>
</tr>
</tbody>
</table>

Table 2. Laminate fiber layup configurations examined.

<table>
<thead>
<tr>
<th>Fiber Configuration 1</th>
<th>[0/90]$_{4S}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber Configuration 2</td>
<td>[0/0/90/90]$_{2S}$</td>
</tr>
<tr>
<td>Unidirectional Configuration</td>
<td>[0]$_{16T}$</td>
</tr>
</tbody>
</table>

Ultimately, the structural battery can be altered and adjusted for each specific application or purpose, as with traditional fiber-reinforced composites. Although only three possible configurations (Table 2) were examined in this study, there are many other possibilities that can be manufactured.

Determining the flexural stiffnesses of the composite plies was carried out through computational FEM. The flexural stiffness of the laminate is calculated by measuring the deflection at the center of a test specimen loaded under a typical three-point bend test. The test specimen dimensions were 100mm × 10mm × 2mm. The thickness of the structural battery unit cell is 250 microns; therefore, the test specimen is composed of eight plies in a laminate. The materials were defined by imposing partitions in the model. This
configuration assumes no delamination occurs within the laminates and is a reasonable assumption for the degree of loading examined. The eight plies can be arranged in a parallel (cathode to cathode; anode to anode) or series (cathode to anode) configuration. These simulations were done in parallel configuration. The elements defined in the model were 8 node linear brick elements. Due to the thin layers of carbon and aluminum foil, the mesh density increased in order to obtain at least three elements across the thin layers. For simplicity, the load was applied by partitioning a thin section along the top face and imposing a pressure load on that surface. The deflection at the mid-point and the maximum stresses in each material are used to compute the flexural stiffnesses and strengths.

Several materials and configurations were tested in the three-point bend test arrangement through FEM. The deformed shape of the specimen is shown in Figure 4, and beside is a cut of the cross-section showing the stress variations due to the layers of different materials. The stiffer materials such as the carbon fiber layers carry more stress than the surrounding layers. Table 3 contains the maximum deflections, flexural stiffnesses, and flexural strengths of the laminates for each of the simulations performed.
Table 3. Flexural properties of FEM material substitution analysis.

<table>
<thead>
<tr>
<th></th>
<th>Max Deflection (mm)</th>
<th>Flexural Stiffness (GPa)</th>
<th>Flexural Strength (MPa)</th>
<th>Specific Flexural Strength (kN·m/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(\delta_1)</td>
<td>(\delta_2)</td>
<td>(E_1)</td>
<td>(E_2)</td>
</tr>
<tr>
<td>Carbon Paper (CP)</td>
<td>5.56</td>
<td>5.56</td>
<td>13.79</td>
<td>13.79</td>
</tr>
<tr>
<td>CP/Honeycomb</td>
<td>0.57</td>
<td>0.57</td>
<td>7.77</td>
<td>7.77</td>
</tr>
<tr>
<td>AS4 Unidirectional</td>
<td>0.69</td>
<td>3.63</td>
<td>111.82</td>
<td>21.12</td>
</tr>
<tr>
<td>AS4 Config 1</td>
<td>1.01</td>
<td>1.32</td>
<td>75.58</td>
<td>58.02</td>
</tr>
<tr>
<td>AS4 Config 2</td>
<td>0.92</td>
<td>1.54</td>
<td>83.62</td>
<td>49.80</td>
</tr>
<tr>
<td>P100 Unidirectional</td>
<td>0.23</td>
<td>3.97</td>
<td>335.55</td>
<td>19.31</td>
</tr>
<tr>
<td>P100 Config 1</td>
<td>0.37</td>
<td>0.52</td>
<td>207.25</td>
<td>148.0/4</td>
</tr>
<tr>
<td>P100 Config 2</td>
<td>0.33</td>
<td>0.64</td>
<td>233.73</td>
<td>119.7/7</td>
</tr>
<tr>
<td>Steel</td>
<td>0.37</td>
<td>207.19</td>
<td>258.46</td>
<td>32.92</td>
</tr>
<tr>
<td>Steel (Analytical)</td>
<td>0.38</td>
<td>200.00</td>
<td>250.00</td>
<td>31.85</td>
</tr>
</tbody>
</table>

Each configuration was simulated along the 0° and 90° directions due to the orthotropic materials, designated in the table with the numbers 1 and 2, respectively. The isotropic models only have one value for stiffness and strength.

The laminates containing carbon paper for the carbon layers were weakest, however the addition of honeycomb core materials significantly reduced the deflection and increased the strength. The stiffness decreased with the addition of honeycomb core because of the increased cross-sectional area, however the laminate experienced less deflection comparable to the models with the stiffer carbon fiber composites. The laminate containing unidirectional carbon fibers exhibited high stiffness and strength values along the fiber direction, but as expected, showed weak performance in the transverse direction. The cross-ply laminates were able to achieve respectable performance in either direction.
The values for the steel benchmarking test are also shown in the table. The values calculated from the FEM model were within a reasonable margin of the analytically calculated values. Steel can be used to compare the laminate to a common engineering material. The P100 carbon fiber exhibited a larger stiffness than steel. Although none of the laminates exhibited a larger strength than steel directly, several of the laminates showed larger specific strengths than steel.

The bending stiffness matrix, \([D]\), was calculated for each of the configurations. From Table 3, the stiffness of the laminate with carbon paper and honeycomb is seemingly lower than the laminate with carbon paper and no honeycomb, however, as shown in Figure 5, the \(D_{11}\) and \(D_{22}\) terms are larger in the carbon paper/honeycomb laminate. The difference is an artifact due to the additional thickness of the honeycomb. The equation for the modulus relies on the area moment of inertia, which is a function of the cube of the thickness. While the stiffness decreased, the honeycomb layer significantly reduced the deflection during the three-point bend test. For comparison, the \(D\) matrices for the AS4 Config 1 and unidirectional configurations are shown.
Figure 5. Flexural stiffness matrices (D) for the laminates with carbon paper (CP), carbon paper with honeycomb (CP/HC), AS4 Config 1, and AS4 Unidirectional. All units are in GPa-mm³.

\[
[D]_{\text{CP}} = \begin{bmatrix} 9.43 & 3.14 & 0 \\ 3.14 & 9.43 & 0 \\ 0 & 0 & 3.12 \end{bmatrix}, \quad [D]_{\text{CP/HC}} = \begin{bmatrix} 128.9 & 40.39 & 0 \\ 40.39 & 122.6 & 0 \\ 0 & 0 & 39.94 \end{bmatrix},
\]
\[
[D]_{\text{AS4 C1}} = \begin{bmatrix} 50.71 & 4.38 & 0 \\ 4.38 & 38.56 & 0 \\ 0 & 0 & 6.34 \end{bmatrix}, \quad [D]_{\text{AS4 Uni}} = \begin{bmatrix} 75.75 & 4.38 & 0 \\ 4.38 & 13.52 & 0 \\ 0 & 0 & 6.34 \end{bmatrix}.
\]

3.2 Solid State Battery Unit Cell Homogenization

The orthotropic material properties were determined for the solid-state battery layers. Specifically, the moduli of elasticity in each direction (\(E_x, E_y, E_z\)), the Poisson’s ratios for each orientation (\(\nu_{xy}, \nu_{yz}, \nu_{xz}\)), and the shear moduli in each orientation (\(G_{xy}, G_{yz}, G_{xz}\)) were determined. These material properties could then be used in a large-scale panel model to represent several layers of the solid-state battery without the added mesh complexity. Each material in the solid-state battery laminate is isotropic, therefore it is transversely isotropic, and the in-plane elastic moduli are equal in the \(x\) and \(y\) directions. Composite laminate theory was used to determine the in-plane elastic moduli (\(E_x\) and \(E_y\)), as well as the in-plane shear modulus (\(G_{xy}\)) and Poisson’s ratio (\(\nu_{xy}\)). However, the transverse properties were determined through simulations on one small block of a single unit cell of the solid-state battery. The transverse shear moduli are equal (\(G_{23} = G_{13}\)) because the material is transversely isotropic. The shear moduli were determined by loading the unit cell in a shear configuration. The shear modulus is the ratio between the shear stress experience by the material and the shear strain it induces. The shear strain was prescribed in the computational model and the simulation solved for the reaction force generated by the addition of the strain. In a similar manner, the Poisson’s ratio was
computed through the computational models. An in-plane strain was prescribed, and the transverse strain was determined through the simulation. The orthotropic material properties for the solid-state battery material are shown in Table 4.

Table 4. Solid state battery homogenized orthotropic properties.

<table>
<thead>
<tr>
<th>SSB Material Properties</th>
<th>E₁, E₂ (GPa)</th>
<th>E₃ (GPa)</th>
<th>G₁₂ (GPa)</th>
<th>G₂₃, G₁₃ (GPa)</th>
<th>ν₁₂</th>
<th>ν₂₃, ν₁₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.32</td>
<td>0.789</td>
<td>4.1</td>
<td>0.227</td>
<td>0.333</td>
<td>0.251</td>
<td></td>
</tr>
</tbody>
</table>

### 3.3 Mechanical Effects of Composite Structural Battery Panel Edge Condition

Structural carbon fiber reinforcement will be used to improve the mechanical properties for the laminate. From previous energy requirements calculations, it was determined that 7 layers of battery material would be required in each face panel on the CubeSat. The battery layers will be at the center of the laminate surrounded by 2 layers of carbon fiber reinforcement on either side. The carbon fiber reinforcement layers selected are a 2x2 twill weave 3k carbon fiber pre-preg. These layers will protect the battery material and provide significant improvement in mechanical properties of the composite laminate.

Figure 6. Side view representation of structural battery panels with carbon fiber reinforcement without gasket (left), with gasket (middle), and complete solid-state battery edge-to-edge (right).
These panels are fabricated using a vacuum bag process that compresses the edges of the panels under the vacuum bag. Ideally, the edges would be kept straight and constant thickness, however initial prototypes have crimped edges as shown in the left image in Figure 6. The three possible fabrication results are shown in the figure. If the battery layers do not extend to the edge of the material, the carbon fiber reinforcement layers will compress together at the edges. This configuration will cause a decrease in flexural stiffness and strength due to the decrease in thickness as well as the stress concentrations in the curvature. For this reason, the other two cases are considered. The battery layers can extend to the edges such that the laminate is a consistent thickness, however this will expose the electrodes of the battery cells which could cause shorting. It would be preferred to shorten the battery layers slightly and add a gasket material to the edges of the panel. This would keep a consistent thickness while protecting and sealing the battery cells within the panel. Each of these cases were simulated to calculate the stiffness and strength of the panel.

Figure 7. COMSOL model of structural battery laminate without gasket showing stress concentrations at corners on either end.
The material selection for the gasket was parameterized in this study. Compliant materials such as silicone are excellent sealants, however these will reduce the flexural stiffness and overall mechanical performance. A parametric study of the gasket material was conducted to analyze the effects the gasket has on the structural battery panel.

The flexural stiffness as well as the weight specific flexural stiffness were compared with 7075 aluminum. The stiffness and strengths analyses were calculated utilizing equation (1) for the stiffness and equation (2) for the strength. The strength analysis was through a first element failure approach to provide a conservative strength estimate.

Table 5. Flexural results of fabrication method study with aluminum comparison.

<table>
<thead>
<tr>
<th></th>
<th>Flexural Stiffness (GPa)</th>
<th>Specific Flexural Stiffness (kN·m/kg)</th>
<th>Flexural Strength (MPa)</th>
<th>Specific Flexural Strength (kN·m/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Gasket</td>
<td>20.43</td>
<td>13.89</td>
<td>92.63</td>
<td>62.97</td>
</tr>
<tr>
<td>Epoxy Gasket</td>
<td>23.36</td>
<td>16.25</td>
<td>92.66</td>
<td>64.44</td>
</tr>
<tr>
<td>Full Battery</td>
<td>23.00</td>
<td>16.41</td>
<td>92.49</td>
<td>65.97</td>
</tr>
<tr>
<td>6061 Al.</td>
<td>68.90</td>
<td>25.52</td>
<td>276.00</td>
<td>102.22</td>
</tr>
<tr>
<td>7075 Al.</td>
<td>68.90</td>
<td>24.52</td>
<td>400.00</td>
<td>142.35</td>
</tr>
</tbody>
</table>

The stiffnesses and strengths of the different structural battery panels and listed in Table 5. The models with the gasket and complete solid-state battery edge-to-edge performed quite similarly. However, the model without a gasket and sharp curves suffered with respect to the stiffness. This is due to the reduced thickness of the panel at the edges as well as the stress concentrations at the corners. The stress concentrations are shown in the figure; the
bright red color indicates greater magnitude of stress. These results prove that it is important to ensure constant panel thickness to fully take advantage of the carbon fiber support.

3.4 Solid-State Battery Cells Reinforced by Unidirectional Composite Layers

In addition, interstitial composite layers of carbon fiber or glass fiber composites can be inserted between each of the battery layers. These additional reinforcement layers will serve as insulation between cells as well as further structural reinforcement. In order to minimize the thickness of the laminate, thin unidirectional carbon fiber layers or plain weave glass fiber fabrics were chosen. Two thicknesses of the unidirectional carbon fiber layers were considered as well: 0.10mm and 0.15mm. The layup chosen for this study is a [0/90/0/90/0/90] cross-ply layup. This laminate orientation is balanced but not symmetric, since there are equal 0° and 90° layers, but they are not symmetric about the mid-plane.

![Figure 8. Structural battery laminate layup with unidirectional interstitial layers.](image)

The results of the interstitial materials assessment are listed in Table 6. The exterior layers remained carbon fiber 2x2 twill weave and only the interstitial layers were modified. Though the unidirectional carbon fiber layers are stiffer, three of the unidirectional layers were orthogonal to the loading direction because of the cross-ply configuration. The panel
with plain weave glass fiber was not as stiff or strong as the panel with unidirectional carbon fiber, however they glass fiber might provide better insulation. The $D_{11}$ terms from CLT are also listed in the table, as well as the specific $D_{11}$, which is divided by the density. The values for 7075 aluminum and steel are shown for comparison.

Table 6. Flexural results of carbon vs. glass interstitial layers with aluminum and steel comparisons.

<table>
<thead>
<tr>
<th></th>
<th>Flexural Stiffness (GPa)</th>
<th>Specific Flexural Stiffness (MN·m/kg)</th>
<th>Flexural Strength (MPa)</th>
<th>Specific Flexural Strength (kN·m/kg)</th>
<th>$D_{11}$ (GPa·mm$^3$)</th>
<th>Specific $D_{11}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unidirectional CF Interstitial</td>
<td>39.51</td>
<td>28.84</td>
<td>165.38</td>
<td>120.71</td>
<td>147.00</td>
<td>107.30</td>
</tr>
<tr>
<td>Plain Weave Glass Fiber Interstitial</td>
<td>37.65</td>
<td>26.24</td>
<td>162.89</td>
<td>113.51</td>
<td>136.90</td>
<td>95.40</td>
</tr>
<tr>
<td>7075 Aluminum</td>
<td>68.90</td>
<td>25.52</td>
<td>400.00</td>
<td>142.35</td>
<td>242.20</td>
<td>86.19</td>
</tr>
<tr>
<td>Steel</td>
<td>200.00</td>
<td>25.64</td>
<td>350.00</td>
<td>44.87</td>
<td>686.60</td>
<td>87.46</td>
</tr>
</tbody>
</table>

Finally, the gasket parametrization study results are shown in Table 7. The stiffnesses of the gasket materials were varied from 0.05GPa to 10GPa to cover a range of possible gasket materials. Two different thicknesses of unidirectional carbon fiber layers were used in this analysis. For a very compliant materials such as silicone, there is a large decrease in stiffness. The panel stiffness varies only slightly between the stiffer gasket materials.
Table 7. Gasket material parameterization study results with different carbon fiber interstitial thicknesses.

<table>
<thead>
<tr>
<th>0.10mm Unidirectional CF Layers</th>
<th>Flexural Stiffness (GPa)</th>
<th>Specific Flexural Stiffness (kN·m/kg)</th>
<th>Flexural Strength (MPa)</th>
<th>Specific Flexural Strength (kN·m/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05GPa (Silicone)</td>
<td>21.20</td>
<td>15.14</td>
<td>95.63</td>
<td>68.30</td>
</tr>
<tr>
<td>1GPa</td>
<td>24.42</td>
<td>17.44</td>
<td>95.63</td>
<td>68.30</td>
</tr>
<tr>
<td>3.3GPa (epoxy)</td>
<td>24.65</td>
<td>17.61</td>
<td>95.65</td>
<td>68.31</td>
</tr>
<tr>
<td>10GPa</td>
<td>24.76</td>
<td>17.68</td>
<td>95.64</td>
<td>68.30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>0.15mm Unidirectional CF Layers</th>
<th>Flexural Stiffness (GPa)</th>
<th>Specific Flexural Stiffness (kN·m/kg)</th>
<th>Flexural Strength (MPa)</th>
<th>Specific Flexural Strength (kN·m/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05GPa (Silicone)</td>
<td>20.99</td>
<td>15.14</td>
<td>88.82</td>
<td>64.08</td>
</tr>
<tr>
<td>1GPa</td>
<td>25.06</td>
<td>18.08</td>
<td>88.82</td>
<td>64.08</td>
</tr>
<tr>
<td>3.3GPa (epoxy)</td>
<td>25.37</td>
<td>18.30</td>
<td>88.82</td>
<td>64.08</td>
</tr>
<tr>
<td>10GPa</td>
<td>25.52</td>
<td>18.41</td>
<td>88.83</td>
<td>64.09</td>
</tr>
</tbody>
</table>

3.5 Conclusions

The structural battery composites were modeled in FEM software to virtually test substitution of several carbon fiber materials and fiber orientations. Each model was identically tested in a three-point bend configuration to determine the maximum deflection under loading. The deflection was used to calculate the stiffness and strengths of each of the individual models. Laminates which included a carbon fiber reinforced polymer were superior to the carbon paper laminates. There exists a trade-off between electrical and structural performance. Incorporating carbon fiber composites into the structural battery is the largest concern. The refinement of the FEM model will include incorporating more sophisticated damage criteria and methods such as element deletion and nonlinear geometry.
The laminate level mechanical properties were also calculated with the addition of various reinforcement materials, including 2x2 twill weave carbon fiber/epoxy pre-preg, unidirectional carbon fiber interstitial layers between battery cells, and glass fiber interstitial layers. The fabrication methods were also taken into consideration. The effects of panel with a crimped edge as well as a gasket to keep constant thickness. The results show that it is possible to achieve weight specific stiffnesses and strengths comparable to aluminum and steel. Further mechanical improvements to the solid-state battery unit cell can be made by replacing the weak carbon paper with unidirectional continuous carbon fiber layers. The limiting factor of these layers in this application is the low electrical conductivity driven by the epoxy matrix. The micromechanics analysis shows a significant improvement over current carbon fiber/epoxy materials by utilizing conductive epoxies as the matrix. These improvements could lead to a multifunctional solid-state battery capable of bearing mechanical loads without extensive reinforcement. These developments would further reduce the weight of the structural battery as well as increase the energy density due to the elimination of some or all of the reinforcement layers. Future works include multi-physics modeling of the interaction between mechanical loading/deformation and electrical performance.
Chapter 4 Structural Battery Laminate Strength and Failure Analysis through FEM and Prototype Mechanical Testing

4.1 Prototype Structural Battery Panel Flexural Testing

The metal foil battery panel was fabricated by hand using a wet layup and vacuum bagging. The layers were coated with a thin layer of epoxy and assembled into the battery cells as shown in Figure 9. Seven battery unit cells were assembled in a laminate and placed under vacuum for excess epoxy outflow. The battery cells compose the center core of the laminate, surrounded by woven CFRP layers. Two layers of CFRP were added surrounding the center battery core, for a total of 4 layers on the panel. Due to the non-permeability of the metal foils, excess resin outflow was limited. The panels were cured in an oven while vacuum was applied at temperatures corresponding to the manufacturer recommendations. The three-point bend tests were accomplished using an Instron test frame with a 1kN load cell. Force and displacement results were exported for processing from the Instron software. Bending loads were applied until complete failure occurred. Analysis of the failed specimens and the force/displacement data.

Figure 9. Photograph of prototype panel consisting of seven Cu-Al cells surrounded by woven 2x2 twill weave carbon fiber composite.
Analysis of the test specimens was accomplished to assess the dimensional stability (avoidance of potential electrical shorts between layers) as well as the presence of defects from the hand layup process. The impermeability of the foils did not allow the vacuum to reach to the internal regions of the cell, thus the air bubbles remained in the center of the laminate. The epoxy was not able to flow, and excess epoxy remained between the layers. Analysis of the cross-section of the laminate was conducted using a digital optical microscope as shown in Figure 10. The non-uniformity of the layers and the voids are apparent. These factors play a large role in the mechanical performance and will be improved on subsequent prototype samples. In addition, the epoxy was not adequately able to adhere to the smooth surfaces of the copper and aluminum foils.

![Micrograph of cross-section of prototype Cu-Fe panel. Carbon fiber can be seen towards the top of the image. Metal foils and carbon paper/epoxy layers towards the bottom of the image.](image)

Figure 10. Micrograph of cross-section of prototype Cu-Fe panel. Carbon fiber can be seen towards the top of the image. Metal foils and carbon paper/epoxy layers towards the bottom of the image.

Multiple cracks and delaminations formed throughout the tests and can be observed in the force/displacement graphs. Figure 11 shows the progression of damage one sample
underwent during the test. The brittle carbon paper/epoxy composite that makes up the center of the laminate cracked through several layers, leading to significant drop in loading. These cracks also caused localized delamination between the metal foils and adjacent layers. Upon further loading, the delamination grew until the length of the span of the specimen was completely debonded, leading to severe reduction in load capacity. These failure modes are heavily influenced by existing defects within the laminate, however, provide valuable insight into the performance of the structural battery laminate, as well as identify future challenges. Such delamination is not only detrimental to the structural performance of the panel, but it is especially damaging for the electrical performance of the cells that make up the center of the panel. Further work is required to observe the electrical effects due to cell damage, as well as what safety and hazards exists if these failures were to occur on a fully charged cell. Such studies will be conducted at a future date as part of this project.

Figure 11. Progressive damage due to bending loading in one of the Cu-Al laminate specimens.
The specimens fail in a brittle manner, with rapid reductions in load when a fracture occurs. Figure 12 shows the load vs. displacement curves for the tested specimens. Partial fracture occurred in the laminate, allowing some reloading to take place before ultimate failure. Variability of samples was large due to the non-uniformity of the panel that was fabricated. This non-uniformity is due to the imperfect hand layup process resulting in voids and air pockets that remained in the laminate. Future prototypes will be created with an improved fabrication method for further analysis and testing. Table 8 reports the mechanical properties of the specimens. The average stiffness of the panel was 13.70 GPa, and the average strength was 96.37 MPa. Overall peak load average was 242.99 N. This structural battery composite is capable of withstanding the loads present in a CubeSat, and further improvements will be made to increase the mechanical performance of this composite for additional applications.
Table 8. Flexural properties of Cu-Al three-point bend tests.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Flexural Stiffness (GPa)</th>
<th>Flexural Strength (MPa)</th>
<th>Force/Displacement (N/mm)</th>
<th>Peak Load (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen 1</td>
<td>15.09</td>
<td>108.55</td>
<td>139.01</td>
<td>273.72</td>
</tr>
<tr>
<td>Specimen 2</td>
<td>10.58</td>
<td>48.04</td>
<td>94.74</td>
<td>121.14</td>
</tr>
<tr>
<td>Specimen 3</td>
<td>15.43</td>
<td>132.50</td>
<td>143.99</td>
<td>334.10</td>
</tr>
<tr>
<td>Average</td>
<td>13.70</td>
<td>96.37</td>
<td>125.91</td>
<td>242.99</td>
</tr>
</tbody>
</table>

4.2 Structural Battery Interface Damage and Delamination Modeling

In order to be able to understand and later predict the behavior of these composites under mechanical loading, computational FEM using Abaqus/CAE software was utilized to model damage of these structural battery composites due to bending. Damage modeling within the FEM software consists of linear elastic loading, damage initiation, and damage evolution.

Modeling of damage, particularly brittle fracture, is difficult due to the instability and speed of the fracture. For this reason, the dynamic implicit solver was used to aid convergence and minimal viscous damping to help convergence large dynamic events. Two main failure modes were considered in these models: layer delaminations, and material damage. To model delamination between layers, cohesive surfaces were used at the interfaces. The cohesive surfaces undergo stiffness degradation after a damage initiation criterion is met, which represents progressive damage at the interface between two layers. In addition, material damage must also be considered. Ductile damage models were used to describe the damage behavior of the metal foils. The carbon paper/epoxy composite exhibits a brittle behavior. The ductile damage model was used with very low...
fracture strain set so that plasticity would not occur in these layers. The Hashin damage model was used for the carbon fiber layers, which is able to represent damage in compression, tension and shear individually, as well as in the fiber or matrix direction. The carbon fiber material properties were determined through previous mechanical testing of the carbon fiber.

![Figure 13. Schematic showing the Ni-Fe battery and the inclusion of cohesive surfaces between the layers.](image)

Figure 13. Schematic showing the Ni-Fe battery and the inclusion of cohesive surfaces between the layers.

![Figure 14. Quarter symmetry computational model, including mesh refinement at mid-span.](image)

Figure 14. Quarter symmetry computational model, including mesh refinement at mid-span.

These laminates consist of thin layers such as in the case of metal foils. Thin layers pose a challenge to generate a model that possesses a refined enough mesh without increasing the
number of elements to the point of excessively large computational times. Strategies to reduce overall mesh size were employed for these models. Quarter symmetry was used rather than modeling the full laminate, as shown in Figure 14. In addition, finer mesh sizes were used at the mid-span to capture the material damage and degradation completely, while leaving a coarse mesh towards the edges of the span. These techniques aided the computational speed and could be used to simulate many iterations and configurations efficiently and at a fraction of the cost and time that it would take to fabricate and test many redesigns and iterations.

The computational FEA models simulated two structural battery material configurations. The left image in Figure 15 shows the stress contours of the laminate in the axial direction. The main load bearing element in the panel is clearly the outer woven CFRP layers. The center layers act as a mechanical separator such as honeycomb core materials. As the outer layers reach damage initiation and stiffness degradation occurs, the center layers begin to deform. Delaminations occur through the cohesive surfaces, as shown in the right image of Figure 15, however the degree of debonding did not reach the same complete debonding that occurred in the test specimens.

Figure 15. Axial stress contours due to bending loading (left), and cohesive damage in one of the interfaces (right).
Figure 16. Load vs. displacement of the prototype specimens and computational model. Model captured overall failure behavior but was over-stiff due to defects and voids in prototype specimens that are unaccounted for in the model.

Figure 17. Load vs. displacement of the Cu-Al panel and Ni-Fe panel simulations.

The model predicts a larger stiffness than the prototyped tests. A likely cause of the reduction of stiffness in the tested samples is the prevalence of air voids within the battery layers. These voids offer no resistance to loading and therefore reduce the overall stiffness.
Further materials testing is required to accurately determine the material properties of each individual layer within the laminate. The damage behavior of the models is similar to the behavior of the tested specimens. Initial cracks form and results in a load reduction, followed by reloading as a decreased stiffness. Reloading continues until new cracks or delaminations grow and this continues until the panel is completely fractured.

Table 9. Flexural properties comparison between bend tests and models.

<table>
<thead>
<tr>
<th></th>
<th>Flexural Stiffness (GPa)</th>
<th>Flexural Strength (MPa)</th>
<th>Force/Displacement (N/mm)</th>
<th>Peak Load (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-Al Tests</td>
<td>15.26</td>
<td>120.53</td>
<td>141.50</td>
<td>303.91</td>
</tr>
<tr>
<td>Cu-Al Model</td>
<td>19.79</td>
<td>162.38</td>
<td>183.24</td>
<td>409.30</td>
</tr>
<tr>
<td>Ni-Fe Model</td>
<td>48.09</td>
<td>482.91</td>
<td>445.23</td>
<td>1221.91</td>
</tr>
<tr>
<td>Aluminum</td>
<td>69.00</td>
<td>310.00</td>
<td>102.22</td>
<td>229.63</td>
</tr>
</tbody>
</table>

The Ni-Fe carbon fiber battery material system was modeled utilizing the same FEM procedure. In Table 9, the average for the testes specimens does not include Specimen 2, as it is considered an outlier. As expected, the flexural stiffness and flexural strength of this material is far superior to the metal foil battery system. Failure of the Cu-Al foil battery panels occurs slower due to several cracking and reloading sections, while with the Ni-Fe carbon fiber batteries, failure is more abrupt. An aluminum specimen with the same width and span with an equal mass of the Cu-Al samples would be approximately 3.33mm. The force/displacement and peak load were calculated using the same equations and the properties of aluminum and added to the table. Although the samples are much thicker, the usable stiffness and strength are greater than aluminum when accounting for density.
4.3 Conclusions

Computational FEM was used to investigate multifunctional composite structural batteries and assess their structural performance. Two structural battery chemistries were analyzed: the metal foil Cu-Al battery, and the carbon fiber Ni-Fe battery. A flexural three-point bend test was used to investigate the ability of the structural battery laminates to resist bending loads. Multiple configurations and possibilities were simulated using Abaqus/CAE and COMSOL software. Textile and unidirectional composites were considered as reinforcements to improve the mechanical performance of the Cu-Al battery system. The carbon fiber-based Ni-Fe battery system outperformed the Cu-Al battery both in stiffness and strength.

The Cu-Al battery model showed a larger stiffness and strength than the tests, due to unexpected extensive delaminations spanning the length of the samples that were not fully represented in the model. It was learned that these arise from fabrication issues regarding resin flow obstruction from interstitial metal foil layers. The layer by layer nature of the progressive damage behavior in the model was consistent with the tests. The primary damage in the model was due to carbon fiber fracture, and the delaminations contributed to a lesser degree. Without explicitly testing bonding strength between layers for foil battery, it is difficult to match the behavior through the computational simulations. The models were still able to represent the effect of material changes or layer thickness changes, and later prototypes were to be fabricated to avoid layer debonding.
Chapter 5 Electrical Prototyping and Characterization

5.1 Carbon Fiber Composite Electrical Conductivity Micromechanics Modeling

In addition to the macroscopic laminate analyses, in a structural battery context it is important to predict the interactions between structural deformation and electrical performance. A structural battery will undergo bending, tension and compression, and possibly damage. Understanding the interaction between the electrical properties and each type of mechanical strain is important to be able to successfully utilize the composite in various applications. A significant area of interest is the electrical response to bending, and if there is a point at which the electrodes will compress the dielectric layer significantly to the point where a short occurs. To gain a better understanding of the electro-mechanical coupling, the micromechanics were analyzed beginning at the individual carbon fiber level. Though it is still an ongoing study, the results obtained will be incorporated into macro scale models to understand the interaction in the laminate.

Several models were created for the micromechanics simulation to perform coupled electrical-structural-thermal analyses. The primary focus of the micromechanics investigation was electrical response to mechanical loading of carbon fiber. The simplest model consisted of the representative volume element for carbon fiber, i.e. a single carbon fiber inside epoxy matrix. For the electro-mechanical simulations, thermal-electrical-structural elements were used to mesh the geometry. A variety of loads can be applied to the unit cell while simultaneously imposing voltage boundary conditions. Primarily, a voltage boundary condition was applied to the faces at the top (positive y direction) and bottom (negative y direction) surfaces. A positive voltage was applied to the top surface and a zero-voltage condition for the ground on the bottom surface. These
boundary conditions imposed a current flow transverse to the fiber as it would occur in the structural battery composite. The electrical current distribution can be analyzed throughout the fiber array. Several types of mechanical loadings were applied to the unit cell such as tension, compression, bending and torsion. The current distribution was analyzed to determine if any significant differences could be observed.

Figure 18. Carbon fiber micromechanics test specimen (left) and mesh (right).

Figure 19. Micromechanics 3x3 fiber array (left) and 5x5 fiber array (right).
This model was expanded to include a matrix of 3×3 and 5×5 carbon fibers to observe the interactions between fibers. For simplicity, the fibers were assembled through multiplication of the representative volume element into grids. This kept the spacing of the carbon fibers equal, which is acceptable for the purposes of this study. These configurations are shown in Figure 19. The voltage boundary conditions were again applied to the surfaces that are transverse to the fibers. The mechanical loadings were also applied to analyze the response of these systems.

Figure 20. Electrical current density distribution of 3x3 fiber array and 5x5 fiber array.

Preliminary micromechanics simulations have been carried out to examine the electro-mechanical performance of the carbon fiber materials. The figures show several different simulations, included the single unit cell, a 3×3 matrix of fibers, and a 5×5 matrix of fibers. The electrical current per unit area (ECD) is shown in the figures. The 3V voltage was applied transverse to the fiber. The current clearly flows through the far more conductive carbon than the epoxy. The current flows directly from one fiber to the next through the shortest paths. As the deformation occurs, the gaps between fibers are adjusted and
therefore the electrical conductivity of the carbon fiber layers are altered. This work is ongoing, and the continuation of this investigation will continue to explore the electrical response of carbon fiber materials in response to various modes of mechanical loading, as well as the coupled electrical-structural interaction at the laminate level.

In addition to the structural battery panel level simulations, carbon fiber composites were analyzed using a micromechanics approach to assess the electrical performance of carbon fibers with different epoxies. The solid-state battery unit cell (Figure 1) utilizes carbon paper, which is a poor structural material. Carbon fiber would significantly improve the mechanical performance of the solid-state battery composite; however, it is important to avoid sacrificing electrical performance of the cell. A high conductivity material is required for this layer. Typically, epoxies such as those used in carbon fiber composites exhibit very low electrical conductivities, however certain epoxies have been specifically created for high electrical conductivity. Ideally, carbon fiber/epoxy composites could be incorporated directly into the solid-state battery unit cell in the place of carbon paper. The carbon paper is very weak and is the failure point of the battery laminate. It will greatly improve the overall structural battery performance to utilize a high stiffness/strength material that also has a high electrical conductivity. High conductivity epoxies were examined to determine if a carbon fiber substitute could function in the place of carbon paper without sacrificing battery performance.

The micromechanics study simulates various epoxy properties, as well as the effects of a high conductivity interface surrounding the carbon fibers. This allows a homogenized conductivity property can to be determined for the carbon fiber/epoxy system to be compared to other materials. This study will provide an understanding of the electrical
performance of carbon fiber/epoxy composites, and whether the adjustment of several parameters can produce an acceptable material to be used within the solid-state battery unit cell.

Various epoxy conductivities, fiber-matrix interface conductivities, and geometries were simulated to characterize the carbon fiber/epoxy composites electrical properties. Three epoxy conductivities were parameterized: a typical insulative epoxy, a conductive silver-filled epoxy, and an intermediate conductivity epoxy. In addition, an interface layer between the fiber and matrix was analyzed simulating a coated fiber such as with carbon nanotubes or other coatings. The conductivity of the interface was set to be a multiple of 5, 10, or 20 times greater than the carbon fiber conductivity.

Table 10. Epoxy conductivities/resistivities simulated in micromechanics study.

<table>
<thead>
<tr>
<th>Epoxy Type</th>
<th>Conductivity (S/cm)</th>
<th>Resistivity (Ω·cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Typical Insulative Epoxy</td>
<td>1.47E-15</td>
<td>6.80E+14</td>
</tr>
<tr>
<td>Intermediate Conductivity Epoxy</td>
<td>1.00E-10</td>
<td>1.00E+10</td>
</tr>
<tr>
<td>Silver-filled Conductive Epoxy</td>
<td>5.00E+02</td>
<td>2.00E-03</td>
</tr>
<tr>
<td>Copper Conductivity</td>
<td>5.95E+05</td>
<td>1.68E-06</td>
</tr>
</tbody>
</table>
Figure 21. Micromechanics two carbon fiber model with thin interface around fibers within epoxy matrix.

A two-fiber model was chosen to represent the current flow between individual carbon fibers. The two-fiber model allows for varying factors such as fiber gap distance and other geometric variations while reducing the model complexity. The model could be extended to include additional fibers in an array, however the mesh complexity and computational time increase. Simulations were carried out with Abaqus CAE using coupled structural-thermal-electrical linear quadratic elements. Voltage boundary conditions are added to the top and bottom of the two-fiber model. The top voltage is prescribed to be 4V and the bottom surface 0V. The voltage potential difference imposes a current through the model transverse to the fibers. The simulation solves for the electrical current density (ECD) through the material. The model can be considered as an electrical circuit with a voltage, a resistance, and a current. The electrical current flowing through the system can be found through the ECD, and the resistance can be found using Ohm’s law.
After computing the resistance, the resistivity of the system can be found as a homogenized material property for the system. The equation for determining the resistivity is

$$\rho = \frac{RA}{L}$$

where \(\rho\) is the resistivity, \(R\) is resistance, \(A\) is the cross-sectional area, and \(L\) is the length. The resistivity is the inverse of the conductivity, and therefore the conductivity of the carbon fiber system can be determined and compared through multiple material substitutions and geometric variations. The conductivity of the carbon fiber/epoxy composite can be parameterized to describe the electrical performance of the material.

![Figure 22. Micromechanics model showing electrical current density through two fiber model.](image)

The ECD distribution is shown in the Figure 22. A greater magnitude of current flows through the carbon fibers than the surrounding epoxy, as expected. The effects of the
interfaces are shown in Table 11. The interface increases the total conductivity slightly, but the epoxy had the greatest impact on overall conductivity. A conductive epoxy could yield a suitable carbon fiber/epoxy composite that could function as a structural reinforcement as well as part of the energy storage system in the structural battery composite. The conductive interface improved overall performance slightly, however it increased the maximum electrical current density, which could create hot spots within the material.

Table 11. Micromechanics analysis of ECD due to various epoxy conductivities.

<table>
<thead>
<tr>
<th></th>
<th>Max ECD (A/m²)</th>
<th>Conductivity (S/m)</th>
<th>Resistivity (Ω·m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Insulative Epoxy</td>
<td>2.06E-04</td>
<td>2.78E-12</td>
<td>3.60E+11</td>
</tr>
<tr>
<td>Insulative Epoxy - 5X Interface</td>
<td>1.39E-03</td>
<td>3.31E-12</td>
<td>3.02E+11</td>
</tr>
<tr>
<td>Intermediate Conductivity Epoxy</td>
<td>3.19E-01</td>
<td>5.04E-08</td>
<td>1.98E+07</td>
</tr>
<tr>
<td>5X Conductivity Interface - Intermediate Conductivity Epoxy</td>
<td>1.06E+00</td>
<td>6.10E-08</td>
<td>1.64E+07</td>
</tr>
<tr>
<td>10 Conductivity Interface - Intermediate Conductivity Epoxy</td>
<td>1.97E+00</td>
<td>6.72E-08</td>
<td>1.49E+07</td>
</tr>
<tr>
<td>20X Conductivity Interface - Intermediate Conductivity Epoxy</td>
<td>3.52E+00</td>
<td>7.29E-08</td>
<td>1.37E+07</td>
</tr>
<tr>
<td>High Conductivity Epoxy</td>
<td>2.26E+11</td>
<td>6.23E+04</td>
<td>1.61E-05</td>
</tr>
<tr>
<td>High Conductivity Epoxy - 5X Interface</td>
<td>9.62E+11</td>
<td>6.85E+04</td>
<td>1.46E-05</td>
</tr>
<tr>
<td>Copper</td>
<td></td>
<td>5.95E+07</td>
<td>1.68E-08</td>
</tr>
</tbody>
</table>

This study used a two-fiber system under the assumption that the carbon fiber spacing is equal and repeats throughout the material, however this does not happen in actual carbon fiber materials. A continuation of this study involving various fiber gaps, fiber contacts,
voids, misalignment, etc. is the subject of ongoing research, and will yield a more complete assessment of the electrical behavior of carbon fiber/epoxy composites.

Figure 23. FEM ECD contour plots of the epoxy conductivity parameterization study.

Figure 24. Graph of resistance of two fiber model for different epoxy conductivities with and without interface.
Table 11 shows the results of the epoxy conductivity micromechanics study. In addition, the conductivity and resistivity of copper is included for comparison. The results vary widely due to the wide range of epoxy conductivities. These results show that the electrical conductivity of the carbon fiber/epoxy composite is highly dependent on the conductivity of the epoxy. Further research is being done to include the effects of different fiber gap distances, missing fibers, voids, and other geometric variations in the carbon fiber micromechanics. These additions will lead to a greater understanding of the more realistic characteristics of carbon fiber composites.

5.2 Battery Cell Prototype Electrical Characterization

Several structural battery specimens have been fabricated for prototyping and testing purposes. These samples consist of the solid-state battery material reinforced with 2x2 twill weave carbon fiber pre-preg. The preliminary tests were conducted to ensure the solid-state battery material could undergo the carbon fiber composite fabrication process: vacuum bagging, oven cycle, etc. Cyclic voltammetry (CV) and computed tomography (CT) was used to view the battery cells inside the carbon fiber reinforcement after fabrication. These prototypes prove the feasibility of the structural battery composite. The CV test has been commonly used to determine the performance of supercapacitors. During a CV test, the voltage is applied to the device and the current accepted and delivered by the device is recorded. The voltage is varied through a specified range and several cycles are repeated. For these tests, the voltage was varied from 0V to 1.5V. The voltage begins at 0V and is linearly increased until 1.5V and then back to 0V. The rate at which the voltage is changed
is called the scan rate, measured in millivolts per second. Each sample was tested using four scan rates: 5mV/s, 10mV/s, 25mV/s, and 50mV/s.

Figure 25. Cyclic voltammetry linear voltage cycles from 0V to 1.5V using a 50mV/s scan rate.

As the voltage applied to the cell increases, the cell begins to accept charge. Current flows from the test equipment to the device. For a slower scan rate, the cell will have more time to accept charge, as well as deliver the charge on the second half of the cycle. Faster scan rates determine how well the cell is able to accept and deliver charge quickly. Supercapacitors are able to charge and discharge very quickly, whereas other types of battery such as lithium-ion batteries are not capable of charging or discharging as quickly. Testing with a wide range of scan rates ensures the full capability of the device has been characterized. A faster scan rate does not allow the device to accept or deliver as much charge as it is capable of doing at a slower rate.
The CV test is an overall test of the performance of the capacitor. Several values are calculated from these tests, such as power density, energy density, and capacitance. The power density is the ability for the device to deliver energy as quickly as possible, in other words, be able to draw high current from the device. Energy density is the measure of how much electrical energy can be stored in the device. A high energy density means a small amount of this material will be able to store a large amount of electrical energy. This is critical for space and lightweight applications. In this case, the power and energy densities were calculated per unit area, however they can also be calculated per unit volume or per unit mass.
The results of the CV tests are shown in Figure 27 and 28. The voltage is shown on the horizontal axis and the current accepted or delivered by the device is on the vertical axis. The area under the curve signifies the power delivered by the device. The energy is
calculated by the multiplying the power by the discharge time. The optimal graph shape is a steep incline and steady current until the voltages reaches 1.5V, followed by a sharp decline and steady current until it reaches 0V. This results with a more rectangular graph with a large area under the curves. The figure below shows the various test cycles for one device with the four scan rates. The effect of the scan rate and the time delay is apparent in this figure. The largest graph is the 50mV/s graph and the areas decrease with decreasing scan rate. The faster scan rate increases and decreases the voltage quickly, therefore the cell accepts and delivers larger currents. The slow rise in current at the beginning of the cycle (left side of graph) is caused by the internal resistance of the cell, including the electrodes and contacts. The graph in Figure 28 show the four samples created for this batch under the same voltage rate of 5mV/s. This shows the variability throughout the different samples. Overall, all devices successfully endured the composite fabrication process. Sample 3 outperformed the other devices for each of the tests, proving that repeatability will be an area of investigation going forward. Currently, these prototypes are fabricated using hand layup techniques to determine feasibility and preliminary performance results, therefore some degree of variability is expected.

Table 12. Power densities for the two samples for each of the scan rates.

<table>
<thead>
<tr>
<th></th>
<th>Power Density (W/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5 mV/s</td>
</tr>
<tr>
<td>Sample 2</td>
<td>2.68E-03</td>
</tr>
<tr>
<td>Sample 3</td>
<td>3.76E-03</td>
</tr>
</tbody>
</table>
Table 13. Energy densities for the two samples for each of the scan rates.

<table>
<thead>
<tr>
<th>Energy Density (W·hr/cm²)</th>
<th>5 mV/s</th>
<th>10 mV/s</th>
<th>25 mV/s</th>
<th>50 mV/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 2</td>
<td>2.24E-04</td>
<td>1.84E-04</td>
<td>1.16E-04</td>
<td>6.48E-05</td>
</tr>
<tr>
<td>Sample 3</td>
<td>3.13E-04</td>
<td>2.70E-04</td>
<td>1.84E-04</td>
<td>1.16E-04</td>
</tr>
</tbody>
</table>

The above tables show the power and energy densities for Sample 2 and Sample 3. Faster scan rates produce larger power densities due to the larger currents delivered under a short amount of time. The slower scan rates ensured that the cells had sufficient time to fully charge, therefore the largest energy densities were recorded for the slower scan rates. The best performing device was Sample 3, with a power density of 1.39E-2 W/cm² and an energy density of 3.13E-4 W·hr/cm².

The prototypes prove the feasibility of the structural battery composite. The battery cells were able to withstand the mechanical loads and thermal stresses due to the carbon fiber pre-preg process. The hand layup procedure induced variability throughout the samples, therefore future areas of focus will be to develop a consistent fabrication method. Work is now being done to optimize the chemistry of the battery cells to further improve the power and energy densities. Alternative composite fabrication methods that eliminate the necessity of an oven cycle can be incorporated to reduce the thermal stresses. This will be a subject of future studies.
5.3 Conclusions

The coupled electrical-structural behavior of carbon fiber examined through a micromechanics simulation of a single fiber, a $3\times3$ matrix, and a $5\times5$ matrix of fibers under applied voltages and loads. This is an ongoing study to observe any changes in electrical performance due to mechanical loadings. The results of the stiffness and strength simulations show that the structural battery composite is a viable concept. The limiting factor of these layers in this application is the low electrical conductivity driven by the epoxy matrix. The micromechanics analysis shows a significant improvement over current carbon fiber/epoxy materials by utilizing conductive epoxies as the matrix. These improvements could lead to a multifunctional solid-state battery capable of bearing mechanical loads without extensive reinforcement. These developments would further reduce the weight of the structural battery as well as increase the energy density due to the elimination of some or all of the reinforcement layers.

Prototyping of the structural battery panels proves the feasibility of incorporating structural carbon fiber reinforcement while maintaining electrical energy storage abilities. Process control and improvement will be necessary to ensure consistent performance across many samples; however, the results have shown that the concept is viable and can produce a material capable of withstanding structural loads while providing electrical energy storage.
Chapter 6 Additive Manufacturing Process Induced Microstructures and Properties

6.1 Fused Deposition Modeling 3D Printing Process Overview

Additive manufacturing methods have quickly become popular and have increasingly been used in serious engineering applications in industries such as aerospace, automotive, medical, consumer, and others. NASA has researched using this technology in the International Space Station, as well as using low-cost materials and open-source hardware [20, 21]. FDM 3D printing has been shown to be successful in printed surgical instruments for space missions [22]. This technology has been used in medical applications to print prostheses [23, 24]. Researchers in Russia are developing methods to recycle biomass material to be used in a 3D printer [25]. FDM 3D printing has been used to print composites by combining a regular plastic extrusion nozzle and a carbon fiber spool to print complex composite parts that are not possible through traditional methods [26].

Fused Deposition Modeling (FDM) is a method of additive manufacturing where a filament is pushed through a heated nozzle where it melts and is deposited in a predetermined path [27]. A schematic of this process is shown in Figure 29 (a). The path is generated by a program commonly called a slicer which generates a gcode file of instructions for the printer to follow. The gcode consists of many individual slices (or layers) of the part that is to be printed. Each layer consists of perimeters and infill. The infill is usually a grid or linear pattern and the density of the infill can be increase for higher strength (longer print duration) or decreased for shorter print duration (lower strength). Figure 30 shows examples of different infill densities of the same part. Due to the layer-by-layer approach, there is an inherent anisotropy of the final parts. In addition,
the interlayer bonding creates a weakness that makes 3D printed parts weaker than if they were created through traditional methods such as injection molding. The layers create corners at the interface between layers as shown in Figure 29 (b) and these corners create stress concentrations. It is necessary to analyze the characteristics of the FDM process to understand the mechanisms responsible for the performance and to research possible improvements.

Figure 29. (a) Schematic showing the FDM 3D printing process [28]. (b) 2D Illustration of FDM layers with stress concentrations [27].

Figure 30. Illustration showing the infill density from less dense (left) to denser (right) [27].
The purpose of this study is to examine and understand the anisotropy associated with the mesoscale structures created through FDM 3D printing of plastics. Typically, thermoplastic polymers makeup the majority of materials used in the FDM process. This study aims to investigate the effect several factors have on the mechanical properties of FDM 3D printed parts, such as print orientation with respect to the z axis (layers), print configuration (i.e. the slicing of the part regarding infill percentage vs perimeters and orientation of infill structures), and the effect of extrusion temperature on the strength and microscopic geometry of the interlayer adhesion. The material used for this study is polylactic acid (PLA). PLA is a biodegradable thermoplastic made from sugars such as corn, sugarcane, and other renewable resources [29]. It has a glass transition temperature of 60°C, which makes printing challenging at times because it much cool below this temperature before the nozzle reaches the same point again in the next layer. Warping and distortions can occur if it is not allowed to cool enough during printing. Tensile tests will be done using the ASTM D638 for polymers. The tensile samples will be printed, tested until failure, and then analyzed under the scanning electron microscope. The SEM is useful to view the fracture surfaces and the layer characteristics to understand the mechanisms present and the intricacies involved with 3D printing.

The testing method consisted of tensile testing 3D printing dog-bone samples. The samples were printed according to the ASTM D638 Type IV sample dimensions which consists of a 25mm gauge length and a 6mm width of reduced section. The samples were printed at several orientations. The print orientation can alter the properties significantly due to the inherent anisotropy of the FDM process. The samples were then tested in an Instron material test machine using a displacement-controlled method until complete
failure of the plastic occurred. The maximum load was of primary concern for each of the samples, as well as the extension to failure. A proper sized extensometer was not available to measure the strain; therefore, the machine extension is used instead. Due to the relatively low loads necessary because the samples are plastic, the compliance of the testing machine was neglected. Maximum load and extension to failure are sufficient metrics to characterize the performance of these tests. Maximum load is a measure of the strength of the sample, while extension to failure will provide an understanding of the ductility of the sample.

6.2 Effects of the Printing Orientation on the Strength of FDM 3D Printed PLA

The ASTM D638 dog-bone samples were printed in three variations of print orientations and print parameters. To test the interlayer adhesion properties, upright samples were printed so that the tension pulled the layers apart. The sample called Axial are printed with extra perimeter lines so that the gauge section of those samples consisted of only print lines that were along the tension axis. This print configuration tests the strength and ductility of the plastic. Finally, the third configuration was printed with grid pattern infill at 45° angles to the tension. Infill is utilized often in 3D printing to save time and material while providing a rigid internal structure within the part. The orientation of the infill was not varied during these tests, however varying the infill orientation will likely influence the properties of the part. The three variations are shown in Figure 31 which also shows the orientation of the layers.
Figure 31. Overview of print orientations tested in this study. (a) 45-degree infill pattern with one perimeter. (b) Transverse layer sample printed upright. (c) Axial sample printed with many perimeters so that gauge section is all in line with tension.

The results of the tensile tests varied largely. The response of the 3D print can vary from a ductile material to that of a brittle material. Figure 32 shows a picture of two samples of the same material printed in different orientations. The top sample was printed with the layers that are transverse to the tension and the sample shown below was printed in the axial configuration with the print lines parallel to the applied tension.

Figure 32. Transverse sample (top) shows brittle fracture and Axial sample (bottom) shows ductile fracture.

The average max load and average extension to failure for the 45°, Axial, and Transverse samples are listed in Table 14. The Axial samples are the strongest because the plastic lines are continuous and therefore there is no interlayer bonding that can break prematurely.
Because the layers are all printed parallel to the tension, there are no stress concentrations in that axis. The Transverse samples were weaker than the Axial samples because the interlayer adhesion strength is weaker and the stress concentrations due to the interface between layers creates crack initiation points.

Table 14. Average max load and average extension to failure of orientation study.

<table>
<thead>
<tr>
<th></th>
<th>Average Max Load (N)</th>
<th>Average Extension to Failure (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>45° Infill</td>
<td>768.97</td>
<td>1.61</td>
</tr>
<tr>
<td>Axial</td>
<td>1600.70</td>
<td>1.76</td>
</tr>
<tr>
<td>Transverse</td>
<td>1174.54</td>
<td>1.59</td>
</tr>
</tbody>
</table>

Figure 33 shows the microscopy of the fracture surfaces of the three print orientations. In image (a), the infill grid structure is seen. The sample broke at the crossing of subsequent infill layers. There are two failure mechanisms visible in this image. The green arrows show voids and the green arrows show gaps between print extrusions.

Figure 33. (a) SEM image of 45-degree sample fracture. Orange arrows show plastic fracture and green arrows show interlayer debonding. (b) SEM Image of Axial sample failure. (c) SEM image of Transverse sample fracture. Orange arrows show voids and green arrows show gaps between print extrusions.
point to the debonding between the print line that has fractured and the one above it, and the orange arrows point to fracture of the plastic. In the second image, (b), the axial print “fibers” are shown. The strands debonded from adjacent strands, however the continuous nature of the plastic withstood more load and larger elongation before breaking. This was the strongest and most ductile configuration tested. The third image in Figure 33 shows the fracture surface of the Transverse orientation part. The orange arrows point to voids that are created during printing, and the green arrows point to the channel between print lines where the plastic material does not fully bond to the underlying layer as it’s laid. The fracture surface exhibits clear failure and most of the fracture was caused by debonding between layers except for occasional breakage within a single layer.

6.3 Effects of the Extrusion Temperature on the Strength of FDM 3D Printed PLA

The interlayer adhesion strength can be altered by changing several settings of the printing process. For this study, the effect of print temperature on the adhesion strength was examined. To do so, the transverse printing orientation from Figure 33 was used. The upright sample was printed at three temperatures: 180°C, 195°C, and 220°C. It is anticipated that the hotter temperature would lead to a better “weld” between subsequent layers and reduce the stress concentration shown in Figure 29 and increase the maximum tensile load. However, the dimensional stability of the final part might suffer due to the increased time required before the material has cooled below its glass transition temperature of 60°C. The lower temperature represents the lowest reasonable temperature before the material becomes too difficult to extrude through the nozzle and causes feed
restrictions. These temperatures characterize the full range of temperatures that can be used to print this material.

SEM was conducted on the layers and the fracture surfaces. It is important to analyze the features of the printed parts and the fracture surfaces to understand the mechanisms responsible for fracture. The maximum load and the extension to failure only provide a portion of the information available. Analysis of the microstructure and the surfaces yield a large amount of additional information about the behavior of these materials and lead to a greater understanding of the FDM 3D printing process.

The extrusion temperature has a large effect on the overall print quality. Even at low magnification, an effect on the dimensional stability can be observed. Figure 34 and Figure 35 show the layers at 50x and 200x magnification. In the 50x images, the layers of the 180°C sample all have constant thickness, while the 220°C sample shows irregular layer interfaces and inconsistent thicknesses of layers. The dimensional stability begins to suffer at higher temperatures. At high magnifications, the interface between layers are very different among the samples. Figure 36 shows the interface between two layers for each of the samples. The interface of the 180°C sample is jagged and appears to have incomplete bonding. The interface of the 195°C sample has a nice clean bond with a short radius, while the interface of the 220°C is well bonded with a smooth large radius.
Although it was expected that the higher extrusion temperatures would yield a stronger part, Table 15 shows the average maximum load and the average extension to failure for
the samples. Four samples were tested for each case. The 195°C samples yielded a stronger part with more ductility. However, the higher temperature of the 220°C samples seems to have embrittled the parts or the plastic and the maximum load as well as the extension to failure suffered. Figure 37 shows a graph of the average maximum loads with a polynomial fit line. The optimal temperature according to the polynomial fit line is 194.68°C, however more samples and more temperatures would have to be tested before statistical significance is reached.

Table 15. Average max load and average extension to failure of extrusion temperature test samples.

<table>
<thead>
<tr>
<th>Extrusion Temperature</th>
<th>Average Max Load (N)</th>
<th>Average Extension to Failure (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>180°C</td>
<td>1104.37</td>
<td>1.40</td>
</tr>
<tr>
<td>195°C</td>
<td>1170.21</td>
<td>1.38</td>
</tr>
<tr>
<td>220°C</td>
<td>979.48</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Figure 37. Graph of the average max loads with respect to print temperature.
Figure 38. Fracture surfaces magnified to 30x of (a) 180°C sample, (b) 195°C sample, and (c) 220°C sample.

Figure 39. Fracture surfaces magnified to 200x of (a) 180°C sample, (b) 195°C sample, and (c) 220°C sample.

Figure 40. Fracture surfaces magnified to 1000x of (a) 180°C sample, (b) 195°C sample, and (c) 220°C sample.
It is important to understand the mechanisms responsible for failure. Scanning electron microscopy is a valuable tool to be able to analyze the fracture surfaces to identify the characteristics and, if possible, the causes of failure. Figure 38 contains three images of the fracture surfaces of the three temperature cases. The image shows the bottom left corner of the sample. The top of the images shows the center of the cross-section of the part and the infill can be seen. The sample printed at 180°C and the 220°C have very smooth and flat fracture surfaces. It can be seen how the layers debonded and separated. The 195°C sample contained many voids, and the surface was slightly rougher. At higher magnifications, Figure 39 and Figure 40, the differences in surface roughness and features are clear. The surface roughness of the 195°C shows that there was excellent bonding between layers, but the materials was still ductile enough to deform plastically.

It is surprising that the higher temperature transverse parts were weaker. In fact, they became brittle with little yielding before fracture. The graphs of the various samples can be found in Appendix B. It is possible that the higher temperature is causing the plastic to degrade at the nanoscale. Thermal degradation is a common result of overheating polymers. This can occur in several ways including depolymerization, random chain scission, side-group elimination, and oxidation of the polymer [30]. Researchers have identified that PLA undergoes depolymerization in a temperature range of 180°C to 240°C. The polymer chains are degrading when the plastic is extruded at excessively high temperatures leading to a reduction in ductility and strength.

While these results reveal an interesting trend, the sample size was not large enough to declare statistical validity. Four samples were tested per configuration/variation. These tests would have to be repeated many more times before a more meaningful result can be
determined. Although variations were kept to a minimum, there are factors that were uncontrollable. Such factors include environmental conditions surrounding printer, filament spool variations from the manufacturer, moisture content of the filament, random defects introduced during printing, etc. Increasing the sample size of the test specimens and randomizing the order under which they were printed (to ensure that filament variations on the spool aren’t changing the results) will improve the certainty and validity of the results.

There are several challenges associated with imaging plastic materials. PLA has a very low glass transition temperature and begins to soften easily under the electron beam. It is important to lower the accelerating voltage enough to prevent distorting the material. In addition, because of plastics low conductivity, charging can occur frequently. This requires gold sputter coating to dissipate the electrons away from the surface being examined. These careful considerations make it possible to image these materials.

6.4 Conclusions

FDM 3D printing has quickly become a popular prototyping and fast method of fabricating useful parts. Due to the layer-by-layer printing method, the parts are anisotropic and can suffer from poor strength in the axis transverse to the layers. Tensile testing was carried out on several 3D printing dog-bone samples printed in a variety of orientations and configurations. The Axial parts were strongest due to their in-plane print lines that were continuous fibrous strands. The parts with transverse layers and largely made up of grid-like infill suffered in strength and ductility. Scanning electron microscopy is useful in analyzing several aspects of 3D printed parts. The radii and quality of the layer interfaces with respect to the different print temperatures shows the better bonding with increased
print temperature. Although the bonding was better on the higher temperature samples, the polymer had degraded due to the high temperatures and caused the part to become brittle and fail sooner. There exists an optimal extrusion temperature to maximize the bonding between layers while minimizing the thermal degradation of the polymer.
Chapter 7 2D Digital Image Correlation (DIC) Tensile Testing Methodology

7.1 DIC Overview

Tensile testing requires accurate load and strain measurements of the specimen. Load measurements are acquired from a calibrated load cell and recorded by the Instron test frame. The accuracy of the modulus calculation depends on recording accurate and precise strain data. Traditionally, strain measurements have been taken with strain gauges or extensometers. Strain gauges are thin sensors that are adhered to the tensile specimen surface and stretch with the specimen. These can be difficult to properly adhere and are disposed after a single use. Extensometers are devices that clip onto the specimen at two points and measure the displacement between the points. Extensometers can be unreliable, and the blades may slip on the surface of the tensile specimen and suddenly shift the data erroneously. In addition, the ASTM E111 standard for Young’s Modulus testing outlines using multiple extensometers and averaging the results, as well as the potential need to correct systematic errors from extensometer data [31]. These methods can provide accurate results, but are prone to errors and limitations.

Digital image correlation (DIC) is a non-contact method of determining strain fields from processing a series of specimen images [32]. DIC algorithms process images to determine relative displacements of marked points between successive images. In some cases, DIC can track geometric features, in other cases a pattern of dots is manually spray coated on a part or specimen. There are several advantages that DIC has over other methods. Unlike glue-on strain gauges or clip-on extensometers, DIC strain analysis is non-contact and does not risk modifying the test results [33]. In addition, DIC produces a full strain field rather than a singular strain measurement [33]. All strain components can
be extracted from a single DIC pattern. The strain field can also reveal more information about the material, such as possible inhomogeneity, stress concentrations, or defects. In more advanced setups with multiple cameras, a 3D strain field can be analyzed.

One major disadvantage until recently has been a prohibitively high cost to purchase and set up a DIC testing system. Open-source software has been developed and made publicly available, such as the Matlab-based Ncorr v1.2 2D software [34]. Researchers at Sandia National Laboratory developed a standalone program, Digital Image Correlation Engine (DICe), capable of subset-based full-field analysis, and feature displacement tracking [35]. This software was chosen to analyze the strain data for the tests outlined in later sections of this paper. The subset-based full-field divides the images into many subsets and computes the displacement of each subset from image to image. This method was used for 2D strain field and average strain analysis. The second method, feature tracking, does not require a speckle pattern and is used to track regions of interests (ROIs) that have been traced out by the user. This method was utilized in later sections where a speckle pattern could not be applied.

7.2 Experimental 2D DIC Test Setup

The experimental test setup acquires images of the specimen throughout the test. Speckle pattern displacements are processed frame-to-frame and combined with the load data from the test frame. For a typical 2D tensile-test of a flat dog-bone shaped specimen, a single camera is needed to record the image data. A high-resolution USB camera was used for the image acquisition. The camera must be pointed directly at the specimen perpendicular to the flat test surface. It is also important to level the camera such that the vertical axis of the test frame matches with the vertical axis of the image frames.
The data was collected in video form with the framerate controlled to a constant 4 frames per second. In addition, the Instron tensile test method was controlled to record the load data at matched sampling times. The image frame during which failure occurs can also be easily identified to mark the failure point for the strain data.

Figure 41. Experimental tensile test setup with USB camera video capture for DIC strain measurement.

Figure 42. Monochrome still frame of tensile test (left) and close-up view of speckle pattern with visible layer lines (right).
The speckle pattern quality is important to achieving accurate pattern recognition by the software. Spray paint cans create a fine mist of paint that covers the surface evenly, however this can create a random speckle pattern with light coverage. The speckle pattern was applied by spraying a fine mist of spray paint downward from approximately 2 feet above the specimens. This method creates a fine speckle pattern randomly distributed throughout the surface. The horizontal striations seen in Figure 42 are present due to the paint falling into the crevices of the 3D printed surface. Pattern quality analysis ensured the strain analysis was not negatively impacted.

### 7.3 Strain Data Processing and Analysis using DICe Image Analysis

After testing, the videos must be split into image frames. Open-source software FFmpeg was used to convert the videos. The sequential image frames were then imported into DICe software. The first image is chosen as a reference state upon which to base the strain measurements. The remaining images are loaded as the deformed images. The region of interest was selected as the center of the tensile specimen, excluding portions near the test frame grips where stress concentrations arise. For strain fields, the analysis mode was set to “subset-based full-field.” This mode divides the pictures into many overlapping subsets and tracks the displacement of each subset for each frame.

For this procedure, the value of interest is the strain in the vertical direction. There is a virtual strain gauge feature that will be used to determine the average strain in the region of interest. The gauge size determines the distance of the subsets used to calculate strain. If a more precise strain field was the intent, a smaller gauge size would be beneficial. In this case, the larger gauge size reduces the error and averages the strain over a longer
distance. The DICe software outputs an exodus file that can be read by Paraview, an open-source data visualization and processing software.

![4-Extrusion Specimen 1](image)

Figure 43. Average strain in the region of interest of a 4-extrusion specimen during a tensile test. The gray band shows the inter-quartile range of the strain for all subsets.

The data was imported into Paraview where the only the VSG_STRAIN_YY variable is selected. This imports the virtual strain gauge data in the y direction. This dataset is composed of a 2D strain field for each of the image frames. The strain data must be averaged for each frame before creating the stress vs strain curve. A filter in Paraview called Plot Data over Time was used to average the data for each time step. Figure 43 shows the linear strain increase during the elastic portion of the test, then the rapid strain increase when plasticity occurs. The data were converted to spreadsheet view and exported. In a spreadsheet editor, the data were processed and synchronized with the load/stress data from the Instron.
7.4 DIC Strain Measurement Verification

The procedure outlined in the previous section was tested and validated with a known material to determine if the procedure is sufficiently accurate. Flat 6061 aluminum dog-bone specimens were tested in uniaxial tension. The speckle patterns were applied using the same methodology as well to ensure consistency between tests. The strain measurements were processed with DICe software and the load/stress data were collected by the Instron load cell. The average stiffness of the aluminum specimens was calculated and the values were compared to the 68.9 GPa literature value [36].

Table 16. Stiffnesses of aluminum DIC validation specimens and percent error from literature aluminum stiffness value.

<table>
<thead>
<tr>
<th></th>
<th>Stiffness (GPa)</th>
<th>Percent Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen 1</td>
<td>67.16</td>
<td>-2.67</td>
</tr>
<tr>
<td>Specimen 2</td>
<td>67.63</td>
<td>-1.99</td>
</tr>
<tr>
<td>Specimen 3</td>
<td>68.75</td>
<td>-0.36</td>
</tr>
<tr>
<td>Specimen 4</td>
<td>66.91</td>
<td>-3.03</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>67.61</strong></td>
<td><strong>-2.01</strong></td>
</tr>
</tbody>
</table>

The table above shows the results of the aluminum tensile validation tests. The average stiffness was 67.61 GPa which corresponds to an average error of about -2.01%. These results demonstrate that the 2D DIC methodology is accurate for tensile strain analysis.
Chapter 8 FDM Tensile Testing and Characterization

8.1 FDM Interlayer Interface Strength Overview

The weakest orientation of a 3D printed polymer is along the \( z \) direction, perpendicular to the build plate. The FDM layer-by-layer deposition approach relies on the last layer cooling before the new material is added. This creates weak bonding when the molten polymer is deposited onto the previous layer that has already cooled and partially solidified. This study aims to characterize the stiffness and strength properties along the \( z \) direction of 3D printing PLA. Previous research on the interlayer strength of 3D printed PLA investigated the effect of layer height on the interlayer fracture energy [37]. The research showed that the interlayer fracture energy is dependent on layer height, however this can be due to geometric differences rather than a change in bond strength. This research aims to combine tensile tests of 3D printed PLA specimens with FEM computational analysis to characterize the interface strength.

Tensile specimens were printed in an upright orientation such that the tension is normal to the planar interfaces. The specimens were subjected to displacement controlled uniaxial tension until failure. The schematic in the figure shows the direction of the applied tension, aimed to pull the interfaces apart. The strength of the 3D printed material in this orientation is entirely dependent on the interface strength.
Figure 44. Tensile direction schematic showing interfaces (dashed lines) in uniaxial tension.

The stiffness and strength of the 3D printed PLA were compared to the bulk PLA properties and the mechanical performance reductions were assessed.

8.2 Tensile Specimen Printing and Preparation

Tensile tests were designed in parallel with computational models to determine the interface strength of the 3D printed PLA. A specially designed tensile specimen and preparation procedure was developed to isolate the extrusion microstructure and create a uniform extrusion that can be easily modeling in computational FEM software.

In previous tests described in Chapter 6, the tensile tests pulling apart the interfaces were tested as-printed, with no processing printing. This created a non-uniform cross-section due to the so-called “seam” that is caused by the starting and stopping of the perimeter. This creates a small molten zone that is inconsistent with the general material microstructure. In addition, accelerations at the sharp corners of the previous specimen design introduce rounded corners if the rigidity of the machine is insufficient. A new specimen design was required to create a consistent cross-section to create an accurate representative computational model. The FEM studies are described in the next chapter.
In order to isolate the effect of microstructural features such as interlayer gap size on the ultimate property of the printed material, it is necessary to achieve a uniform microstructure in the specimen. For this reason, careful specimen preparation was done to avoid non-uniform or inconsistent geometries. A specialized slotted shape shown in Figure 45 was designed to be printed smoothly with as little acceleration as possible on the flat surfaces and no stopping in the specimen area. Flat sections were later be cut out of this slotted shape to fabricate the dog-bone tensile specimens. The 3D printer nozzle size used was 0.4 mm diameter and the layer height was 0.2 mm. Tuning of the machine parameters and filament diameter was done to ensure the most consistent microstructure possible. The simple slot design was directly imported into slicing software with 0% infill selected and the specified number of walls, or perimeters. Two microstructures were tested in this study: 3 perimeter extrusions, and 4 perimeter extrusions. These will be called the 3-extrusion and 4-extrusion models. The right image in Figure 45 shows an example of the 4-extrusion microstructure slicing preview.

Figure 45. Custom slot design (left) and close-up view of 4-extrusion configuration layer preview showing the nozzle position where the layer seam occurs (right).
The layer seam is the visible point at which the printer nozzle begins and ends the perimeter of each layer. This seam was controlled in the Cura slicing software and placed on the curved section. This eliminates the overlap melt zone that the seam creates from the test area of the dog-bones. The purpose of this is to create the most uniform cross-section that could be modeled in FEM software.

From each slotted print, the two flat sections will each be cut out to create a tensile specimen. Cutting creates sharp flat edges and significantly reduces the matching computational model. The interface strength is then isolated from additional microstructures or edge effects that are not representative of the general interlayer bonding. The printed slot specimens were placed into a CO₂ laser cutter to cut out the flat sections. The stages of the laser cutting process is shown in Figure 46. The first pass removes the excess and to separate individual test specimens. Multiple faster low-power passes are preferred, rather than a single high-powered laser pass to cut the shape. Due to the low glass transition temperature of PLA, high power laser cutting leaves a large melt zone, and changes the microstructure around the edges of the test specimen. Faster low-power cuts result in the cleanest cut with as small a melt zone as possible.
Figure 46. FDM tensile dog-bone specimen in various stages of the laser cutting preparation.

A small melt zone was still present and was removed by light sanding and scraping with a blade. This procedure isolates the extrusions and creates square edges that can be discretely modeled. Microscopy and model generation are described in the next chapter. After laser cutting, the speckle patterns were applied to the specimens using the methods outlined in the previous chapter.

8.3 PLA Filament Material Properties

Filament properties were directly measured to be used in Abaqus to then investigate the effect of varying geometry on overall part stiffness and strength. Filament properties must be measured directly to ensure the properties used in the model match the property of the filament printed specimens. Note that mechanical properties of PLA can vary greatly depending on the molecular weight, so parity must be ensured. A filament may also have a slightly different microstructure from a bulk polymer if the filament spinning process causes some alignment or bias in the microstructure. Material properties can also potentially vary between manufacturers and filament batches. Therefore, it is beneficial to perform tensile tests on the filament used to produce the tensile dog-bone specimens. The
results from the filament material tensile tests will be imported into the Abaqus FEM models in the next section.

DICe software was again used to measure strain, however, due to the thin diameter of the filament, application of a speckle pattern was not effective. Instead, two black pieces of tape were applied to the filament as markings for one-dimensional feature tracking in DICe. Feature tracking recognizes and measures the displacements of regions of interest selected in the reference frame. It tracks the displacement for each frame of the analysis. The vertical displacements of each feature are exported from the DICe software. Strain measurements are then determined from the relative displacements. This provides a 1D strain measurement that is suitable for stiffness determinations. The table shows the average stiffness and ultimate tensile strength of the filament tensile tests.
Table 17. PLA filament stiffness and ultimate tensile strength results from tensile tests and bulk PLA properties [38].

<table>
<thead>
<tr>
<th></th>
<th>Stiffness (GPa)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filament Tension Tests</td>
<td>3.57</td>
<td>43.57</td>
</tr>
<tr>
<td>Bulk PLA</td>
<td>3.50</td>
<td>73.00</td>
</tr>
</tbody>
</table>

Abaqus requires the material properties be in true stress rather than engineering stress. The yield stress will be at the point that the necking begins and the load begins to decrease. The stress at this point is 43.57MPa before the diameter decreases. Using photos that were acquired during the DIC video acquisition, digital diameter measurements were taken to determine the stress in this smaller diameter region.

Figure 48. Filament after undergoing large plastic deformation.

After the filament undergoes plastic deformation and the large change in diameter occurs, it continues to extend and draw more material as it deforms. The large decrease in diameter can be seen in the figure. The feature-based strain measurements are only reliable up until
this yield point. After the necking occurs near the stress concentration of the grips, the strain measurements are no longer valid since the load drops and the strain between the markings reduces.

![PLA Filament - Load vs Time](image)

Figure 49. Load over time of filament tension test showing extreme elongation and plasticity.

Through digital measurements, the diameter reduces to a constant 1.04 mm as it continually lengthens. The load after the diameter reduces to this value remains constant as shown in Figure 49. The true stress in this decreased diameter region can be determined by dividing the load by the reduced area. This true stress value is 96.53MPa. The filament transitions from linear elastic to plastic until it reaches this maximum stress.
Figure 50. PLA trilinear material law developed for Abaqus simulations.

The trilinear stress model shown in Figure 50 was developed to represent the filament material in Abaqus. The first linear portion has a slope corresponding to the filament stiffness, 3.57 GPa. The next linear portion reaches the maximum true stress and 5% strain, near the literature reported elongation at break values. At this point, the material becomes perfectly plastic and continues to extend with no additional stress increase, matching the behavior of the filament tension tests. This material law was imported into Abaqus and used as the material property for the FDM models.

### 8.4 FDM Tensile Test Results

The average stiffnesses for each of the two specimen types are shown in the table along with the average strength and strain to failure. The results show that the 4-extrusion model exhibited a greater stiffness than the 3-extrusion model, however the strengths were similar.
Table 18. Average stiffness and strength of FDM tensile tests.

<table>
<thead>
<tr>
<th></th>
<th>Average Stiffness (GPa)</th>
<th>Average Strength (MPa)</th>
<th>Average Strain to Failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Extrusion</td>
<td>2.38</td>
<td>22.82</td>
<td>1.10</td>
</tr>
<tr>
<td>4-Extrusion</td>
<td>2.45</td>
<td>22.76</td>
<td>1.02</td>
</tr>
</tbody>
</table>

The material properties of the bulk stiffness were analyzed in the previous section. The stiffness of the filament PLA is 3.57 GPa and the ultimate tensile strength is 43.57 MPa. From filament to printed specimen, there was an average of a 32.4% reduction in material stiffness. This can be attributed to a variety of factors, including degradation of the polymer during printing. This effect was studied in section 6.3, however stiffness was not analyzed in relation to temperature. The strength is also much lower than the bulk PLA strength, exhibiting a 47.8% reduction. There are many potential causes at the microstructural level for this low strength. The strength of the bond between subsequent layers and between adjacent extrusions is much weaker than the bulk polymer. Gaps and voids between the extrusions can cause significant stress concentrations. Further investigation into the microstructure will provide a better understanding of the causes of mechanical performance reduction and lead to potential printing improvements.
Figure 51. Post-failure still frame showing clean interface failure occurring between layers.

The figure above shows an image of the tensile sample just after fracture has occurred. These specimens fail in brittle fashion and no plastic deformation can be observed. Fracture occurred through a single layer, in a plane perpendicular to the tension direction. The extrusions on either side of the broken specimen appeared to be intact, i.e. the interface between print lines has failed. The average strain at failure, or elongation at break, for these specimens was much lower than the bulk PLA value. Reported values of elongation at break for bulk PLA range from 2.5 to 6% [39]. The failure occurred before any significant plastic deformation was able to progress. This is due to the comparatively weaker interface bond between layers. The strength of the interface is the limiting factor that is primarily responsible for failure.
The figures show the failure morphology of the tensile specimens. Figure 52 shows the intact extrusions with the damage occurring where adjacent layers meets. This suggests an interface strength that is lower than the ultimate tensile strength of the bulk PLA material. Identifying and improving this interface strength can result in significant improvements in the mechanical performance of FDM 3D printed polymers.
Chapter 9 FDM Microstructural Analysis and Interface Strength Parameterization

9.1 FDM Interface Modeling Overview

In combination with physical testing, computational FEM modeling of the microstructures in these FDM printed parts will illuminate the mechanism leading to the demonstrated property reductions seen in Chapter 8. The models were created in Abaqus/CAE to replicate the extrusion geometries and the interface bonding that occurs in the specimens. Validity of the model depends on parity with the physical geometry of the test specimens. Careful considerations were given to the design and processing of the 3D printed tensile specimens to create a uniform cross-section that could be modeled efficiently and accurately. In addition to correctly matching geometry, the material properties must be correctly determined from the original filament to determine the bulk PLA properties. Filament tension tests were carried out to ascertain the stress vs strain curve to then be imported into Abaqus.

Modeling was used to determine the interface strength between printed filament lines by employing bulk properties and varying the interface strength to match the failure of the printed specimens. The consistency of interface failure strength was evaluated at two points. The geometry of the printed simulated specimens was then varies to evaluate the effects of print geometry on overall stiffness and strength. Modeling interface strength was achieved by defining surface-based cohesive behavior between the layers and extrusion of the model. Zero-thickness cohesive surfaces are designed to represent interface failure governed by a traction separation law set by the user. The strength of these surfaces was varied until the simulations matched the tensile tests. The procedure was repeated with both microstructures and the results compared. The resulting interface
strength was imported into various models with improved geometry to predict the possible improvements to the overall printed material performance.

9.2 Extrusion Microscopy and FEM Model Generation

In order to accurately represent the behavior of these 3D printed materials, it is important to understand the microstructure created during the FDM process. The printer was tuned to reduce defects or voids, but gaps still occur between extrusions laid side by side. After polishing, the cross-sections of the tensile specimens were viewed under a digital microscope. Figure 46 shows the laser cutting process. The material removed in the last step of the process, near the gauge section of the tensile specimen, shares the exact microstructure of the tested material. These were set in epoxy and polished on a Struers LaboForce-100 polishing machine. The polishing reveals the gaps and allows for accurate measurements and characterization of the microstructures.

The printed specimens tested in this study had significant gaps between extrusions, as is common in all such printed materials. The gaps showed a slight misalignment and asymmetry. The computational models were created to match this geometry as closely as possible.
Figure 53. Digital microscope images of 3-extrusion (left) and 4-extrusion (right) polished specimen cross-sections.

Figure 54. Digital image measurements (yellow boundary) of gaps to record average dimensions and areas.

Digital measurement software was used to carefully trace each gap of select microscope images. The 3-extrusion specimen had two distinct columns of gaps of different dimensions. The average length and width of the gaps were matched in the model and the
radii were adjusted until the model gap area matched the specimen average gap area. The same procedure was repeated for the 4-extrusion specimen and the resulting models are shown in Figure 55 and Figure 56.

Figure 55. Matching 3-extrusion model geometry recreated in Abaqus FEM software.

Figure 56. Matching 4-extrusion model recreated in Abaqus software.
Boundary conditions were designed to match the physical tests. The top surfaces were assigned a displacement boundary condition in the vertical direction in tension and the bottom surfaces were held in the vertical direction. Both surfaces were allowed to contract in the transverse directions to allow for the Poisson effect.

In the physical tests, failure occurs at the interface between layers. This behavior was represented in the models through use of cohesive surfaces. The horizontal surfaces between the extrusions were defined as cohesive surfaces that are able to break at a specified nominal stress. This nominal stress represents the interface strength of the 3D printed material. The nominal stress of the cohesive surface property was parameterized until the strength of the overall models matched the tensile tests results. The strength of the model is the maximum tensile load divided by the overall cross-sectional area of the model, as it would be measured by calipers during a physical test. The average stiffness of the models was compared to the physical tests.
Table 19. Comparison of tensile test stiffness vs. model stiffness.

<table>
<thead>
<tr>
<th></th>
<th>Tensile Tests (GPa)</th>
<th>FEM Model (GPa)</th>
<th>Percent Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Extrusion</td>
<td>2.38</td>
<td>2.59</td>
<td>8.65</td>
</tr>
<tr>
<td>4-Extrusion</td>
<td>2.45</td>
<td>2.55</td>
<td>4.21</td>
</tr>
</tbody>
</table>

Using the updated filament material properties, the models predicted a higher stiffness for each model. The physical specimens contain defects and internal voids (essentially trapped air within printed filament lines) not currently accounted for by the computational models.

9.3 FDM Interface Strength Determination through FEM Microstructural Modeling

The interface strength is an unknown material property that can be determined by the computational models. The cohesive surfaces are governed by the maximum nominal stresses in the normal and shear directions. The interface strength of the 3D printed PLA specimens was parameterized by varying the maximum nominal stress in the normal direction until the model strength matched the tensile tests. The data are shown in Tables 20 and 21. Both parameterization studies resulted in nearly identical interface strengths, showing close agreement.
Table 20. Interface strength parameterization of 3-extrusion model.

<table>
<thead>
<tr>
<th>Cohesive Interface Strength (MPa)</th>
<th>Model Strength (MPa)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>20.60</td>
<td>-9.70</td>
</tr>
<tr>
<td>32</td>
<td>21.88</td>
<td>-4.12</td>
</tr>
<tr>
<td>33</td>
<td>22.27</td>
<td>-2.39</td>
</tr>
<tr>
<td>33.5</td>
<td>22.88</td>
<td>0.27</td>
</tr>
<tr>
<td>34</td>
<td>23.06</td>
<td>1.06</td>
</tr>
<tr>
<td>36</td>
<td>24.37</td>
<td>6.78</td>
</tr>
</tbody>
</table>

Table 21. Interface strength parameterization of 4-extrusion model.

<table>
<thead>
<tr>
<th>Cohesive Interface Strength (MPa)</th>
<th>Model Strength (MPa)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>20.27</td>
<td>-10.95</td>
</tr>
<tr>
<td>32</td>
<td>21.56</td>
<td>-5.26</td>
</tr>
<tr>
<td>34</td>
<td>22.73</td>
<td>-0.16</td>
</tr>
<tr>
<td>36</td>
<td>23.58</td>
<td>3.58</td>
</tr>
</tbody>
</table>

The average interface strength for this material was determined to be 33.75 MPa. Earlier assessments of the brittle failure morphology showed evidence that the failure occurs at a stress below the ultimate tensile strength, due to lack of any observed distortion or plastic deformation on the failure surface. This suggests that the earlier assessment is correct and the interface strength is not strong enough to cause the surrounding PLA to reach plastic deformation. This interface strength was then used in the remaining models to investigate the potential stiffness and strength changes for FDM 3D printed parts.
In addition to the weaker interface strength, the microstructure creates many stress concentrations. The rounded edges of the extrusions meet at sharp corners and raise the stress substantially over the surrounding material. The stress at the edges of the interfaces was greater than 1.6x the stress at the center. Reducing or eliminating these inner voids can increase the strength substantially.
The stress contour in Figure 59 shows the stress halfway through the failure of the model. The failure initiated on right extrusion because the gap geometry creates a larger stress concentration. The right interface has fully debonded in the figure and the failure now is travelling through the middle extrusion.

9.4 Stiffness and Strength Improvement Modeling of FDM Materials

The interface determined in the previous section was used to investigate the potential improvements that can be achieved by reducing or eliminating the gaps between the extrusions. The model in the Figure 60 was generated to create a symmetric gap to reduce computational complexity and to be able to use as a reference for the improved models. The width and height of the gaps were kept consistent and the radii of the rounded corners were adjusted until the gap areas matched.
The symmetric model is an idealized case that corrects the misaligned extrusions caused by printer motion precision. The symmetric model is the assumed geometry created by a printer with increased precision. Reducing the gap size will have the most significant effect on the mechanical properties of this microstructure. The improved gap model has gaps scaled down by a factor of 4. The resulting gap area is 15.8 times smaller than the symmetric model. This improved model is shown in the next figure. The interfaces have now been lengthened and will undergo larger tensile loads.
In addition to the improved model, a model with no gaps or voids was created to show the perfect inner bonding while retaining the outer rounded edges that can create stress concentrations. Eliminating the gaps will increase the interface area and avoid the
additional internal stress concentrations. This may be difficult to achieve in practice, but this model serves as an upper limit of what can be achieved by improving the internal geometry.

The results of the study are shown in the following tables. The improvement values in the tables are based on the relative improvement from the matching model, which is the baseline configuration representing the real-world geometry. The symmetric models were similar to the matching model, with slight improvement in the 3-extrusion model. Reduction in gap size led to a 7.65% and 10.67% increase in stiffness for the 3-extrusion and 4-extrusion models, respectively.

Table 22. Stiffness and strength improvements for 3-extrusion models.

<table>
<thead>
<tr>
<th></th>
<th>Stiffness (GPa)</th>
<th>Improvement (%)</th>
<th>Strength (MPa)</th>
<th>Improvement (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matching (Baseline)</td>
<td>2.59</td>
<td>-</td>
<td>22.88</td>
<td>-</td>
</tr>
<tr>
<td>Symmetric</td>
<td>2.67</td>
<td>3.28</td>
<td>22.59</td>
<td>-1.29</td>
</tr>
<tr>
<td>Improved</td>
<td>2.79</td>
<td>7.65</td>
<td>24.96</td>
<td>9.09</td>
</tr>
<tr>
<td>Perfect - No Gap</td>
<td>2.88</td>
<td>11.21</td>
<td>25.86</td>
<td>13.01</td>
</tr>
</tbody>
</table>

Table 23. Stiffness and strength improvements for 4-extrusion models.

<table>
<thead>
<tr>
<th></th>
<th>Stiffness (GPa)</th>
<th>Improvement (%)</th>
<th>Strength (MPa)</th>
<th>Improvement (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matching (Baseline)</td>
<td>2.55</td>
<td>-</td>
<td>22.73</td>
<td>-</td>
</tr>
<tr>
<td>Symmetric</td>
<td>2.56</td>
<td>0.20</td>
<td>22.56</td>
<td>-0.75</td>
</tr>
<tr>
<td>Improved</td>
<td>2.82</td>
<td>10.67</td>
<td>25.45</td>
<td>11.97</td>
</tr>
<tr>
<td>Perfect - No Gap</td>
<td>2.93</td>
<td>14.98</td>
<td>26.91</td>
<td>18.39</td>
</tr>
</tbody>
</table>

The interface strength was consistent between microstructures in section 9.3, however, in the improvement models, the 4-extrusion microstructure showed significantly larger improvements over the 3-extrusion model. This suggests a more inefficient printing
geometry with increased walls/perimeters. To study this effect further, a 5-extrusion model was created and analyzed matching the model geometry of the 3 and 4-extrusion models. Identical improvement models from Figures 60 – 62 were created for the 5-extrusion study. These results are tabulated below.

Table 24. Stiffness and strength improvements for 5-extrusion models.

<table>
<thead>
<tr>
<th></th>
<th>Stiffness (GPa)</th>
<th>Improvement (%)</th>
<th>Strength (MPa)</th>
<th>Improvement (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matching (Baseline)</td>
<td>2.55</td>
<td>-</td>
<td>22.93</td>
<td>-</td>
</tr>
<tr>
<td>Symmetric</td>
<td>2.58</td>
<td>1.18</td>
<td>22.82</td>
<td>0.40</td>
</tr>
<tr>
<td>Improved</td>
<td>2.93</td>
<td>14.94</td>
<td>26.47</td>
<td>16.45</td>
</tr>
<tr>
<td>Perfect - No Gap</td>
<td>2.96</td>
<td>16.12</td>
<td>27.23</td>
<td>19.80</td>
</tr>
</tbody>
</table>

The results show a maximum increase of 16.12% in stiffness and 19.80% in strength over the matching geometry models. The maximum stiffness is 2.96 GPa, corresponding to a 17% decrease from the 3.57 GPa filament stiffness. The reduction is due to the external rounded portions of the layers created during deposition. Even if the internal gaps are removed, improving the external rounded contours can improve the stiffness of the 3D printed parts significantly. The strength showed a 19.80% improvement over the baseline matching model. The strength is approaching the interface strength of 33.75 MPa, however the sharp corners create stress concentrations that cause premature failure.
Figure 63. Stress contour of 5-extrusion perfect model showing the stress concentrations forming due to the external rounded edges meeting at the sharp interface.

The improvement increases with increasing side-by-side extrusions. This trend is tabulated in Table 25. The 3-extrusion model suffered the largest mechanical property reduction because of the ratio of the stress concentration to the area of the interface. The larger area interfaces of the 5-extrusion model do not impact the overall strength as severely because of the relatively small area of the stress concentrations compared to the wide intact interface.

Table 25. Maximum stiffness and strength improvement from matching geometry model to no gap model.

<table>
<thead>
<tr>
<th></th>
<th>Maximum Stiffness Improvement %</th>
<th>Maximum Strength Improvement %</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Extrusion</td>
<td>11.21</td>
<td>13.01</td>
</tr>
<tr>
<td>4-Extrusion</td>
<td>14.98</td>
<td>18.39</td>
</tr>
<tr>
<td>5-Extrusion</td>
<td>16.12</td>
<td>19.80</td>
</tr>
</tbody>
</table>

This effect seems to converge to about 20% reduction in strength from the filament strength. Increasing the amount of extrusions may lead to small improvements, however
the most significant improvement would be achieved by eliminating the stress concentrations.

9.5 Multi-Scale Analysis of 3D Printed Polymers Conclusions

FDM parameterization studies were done to characterize the mechanical behavior of 3D printed parts. The relationship between fabrication process, resulting microstructure, and consequent mechanical properties has been investigated through experiment and simulation. For printed PLA polymers, extrusion temperatures as high as 220°C can improve the radius of the interface, thereby reducing the stress concentration that can lead to premature failure. Conversely, higher extrusion temperatures lead to degradation and depolymerization of the material. This resulted in experimentally measured reduced strength as well as reduced ductility. The results of the study demonstrate the multifaceted nature of additive manufacturing that depends on the careful consideration of multiple variables. The layer-by-layer printing approach induces anisotropy to the final product because there are inter-layer interfaces that are weaker than the bulk material. Scanning electron microscopy was employed to directly view the interface and failure morphologies. The morphology of the interface is impacted greatly by the extrusion temperature of the polymer.

Tensile tests of carefully prepared 3D printed PLA specimens were conducted to investigate the effect of unique printed microstructures on the stiffness, strength, and strain to failure characteristics. Specifically, the specimens were tested in an orientation perpendicular to the build plate such that it pulls the layer-to-layer interfaces apart. The average stiffness and strength of the tested 3D printed specimens are 242 GPa and 22.8 MPa, respectively. The interface between print layers created during layer-by-layer deposition is imperfectly bonded
and resulted in a 32.4% reduction in stiffness and 47.8% reduction in strength compared to the filament property.

Further investigation of this behavior was accomplished by recreating the microstructure of the specimens in Abaqus for computational simulations. The FEM analysis showed agreement with the physical tests, with an average overestimation of the stiffness by 6.43% likely due to void content in the printed specimens. Cohesive surfaces with modifiable strength parameters were used to represent the failure occurring along the interfaces. The models were used to perform a parametric study to determine the correct interface strength of the printed PLA material. The interface strength was determined to be 33.75 MPa, significantly below the 43.57 MPa filament strength. Layer to layer interface weakness can be attributed to both imperfect bonding as well as imperfect packing of round print lines. Cross-section microscopy showed large gaps between adjacent print extrusions, leading to stress concentrations that precipitate failure.

Utilizing the interface strength from the parametric studies, additional models were created to investigate the potential mechanical improvements associated with reducing or eliminating internal gaps between rounded print lines. Three by four, four by four, and five by four print line geometries were analyzed to ensure isolation of internal geometry effects from edge effects. Eliminating the inter-layer gaps while maintaining external rounded surface features improved the strength by 13.01%, 18.39%, and 19.80% for the 3x4, 4x4, and 5x4 extrusion models, respectively.

9.6 Future Work

The evaluation of FDM 3D printed materials focused on the outer walls/perimeters of a printed part. Typically, parts are printed with an internal cross-hatching structure called
infill. This reduces process time and final product weight. Example overlapping extrusions are shown in Figure 64 image A and the spaces between the infill are shown in image B. Fracture occurring in this region and the interaction between the infill pattern and surrounding perimeters can be modeled in FEM software to further understand these materials.

![Figure 64. SEM image of FDM 3D printed microstructures [40]. Image A shows voids in the cross-hatch infill pattern in a nylon specimen and image B shows the large gaps between infill walls.](image)

The inclusion of the infill pattern will lead to full-scale mechanical models to describe the behavior of a 3D printed part. Understanding the effects of changes to the printing parameters, such as layer height, nozzle diameter, extrusion width, can illuminate improved printing parameters for various geometries. Additionally, methods to reduce the rounded stress concentrations by adjusting printing parameters and extrusion nozzle shape. Post-processing heat treatment of the printed can be investigated to attempt to improve interface bonding as well as relieve residual stresses.

Recently, methods to interstitially add carbon fiber or other composites between traditional FDM printed layers have been developed to improve performance of printed
parts. The interactions between the stiff composite and the printed polymer matrix are not fully understood. Failure of these composites is dependent not only on the properties of the constituents, but also the strength of the bonds between the materials. Microstructural FEM modeling of the complex interactions in these printed composites will characterize the stress concentrations that can inform process improvements.
References


