3D PRINTING OF 316L STAINLESS STEEL AND ITS EFFECT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES

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3D PRINTING OF 316L STAINLESS STEEL AND ITS EFFECT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES

by

Penn Rawn

A thesis submitted in partial fulfillment of the requirements for the degree of

Masters of Science in Metallurgical Engineering and Mineral Processing

Montana Tech

2017
Abstract

Laser powder bed fusion or 3D printing is a potential candidate for net shape forming and manufacturing complex shapes. Understanding of how various parameters affect build quality is necessary. Specimens were made from 316L stainless steel at 0°, 30°, 60°, and 90° angles measured from the build plate. Three tensile and four fatigue specimens at each angle were produced. Fracture morphology investigation was performed to determine the fracture mode of specimens at each build angle. Microstructural analysis was performed on one of each orientation. The average grain size of the samples was marginally influenced by the build angle orientation. Tensile yield strength was the highest for 0° and decreased in the order of 60°, 30°, and 90° angles; all had higher yield strength than wrought. Unlike with the tensile results, the 60° had the highest fatigue strength followed by the 0°, then the 30°, and the 90° build angle had the lowest fatigue strength. Tensile specimens all failed predominantly by ductile fracture, with a few locations of brittle fracture suspected to be caused by delamination. Fatigue fracture always initiated at void space.

Keywords: Additive manufacturing, processing parameters, microstructure, mechanical properties, fatigue, fracture morphology
Dedication

I wish to thank Mom and Dad for all their support and love, as well as our cats and dogs for their infinite enthusiasm.
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Specimens were machined by Imperium. Bryce Abstetar, Steven Keckler, Luke Suttey, and Eddie Stugelmayer were my research partners. K.V Sudhakar was my advisor and Bruce Madigan was the PI. Gary Wyss provided SEM training. Ronda Coguill and Taylor Winsor provided tensile data. Jeff Braun designed the database.
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# List of Acronyms

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<th>Meaning</th>
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<td>EBM</td>
<td>Electron beam melting</td>
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<td>GED</td>
<td>Global energy density</td>
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<td>LENS</td>
<td>Laser enabled net shaping</td>
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<td>MPB</td>
<td>Melt pool boundary</td>
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<td>RBF</td>
<td>Rotating beam fatigue</td>
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<td>SEM</td>
<td>Scanning electron microscope</td>
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<tr>
<td>SLM</td>
<td>Selective laser melting</td>
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<tr>
<td>SLS</td>
<td>Selective laser sintering</td>
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<tr>
<td>UTS</td>
<td>Ultimate tensile strength</td>
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<td>YS</td>
<td>Yield strength</td>
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<td>µ-CT</td>
<td>Computed microtomography</td>
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1. Introduction

In the 1980s additive manufacturing became a widespread topic of interest to researchers and industry\(^1\). Additive manufacturing refers to any process for fabricating components by adding material, as opposed to subtractive manufacturing where material is removed to gain the desired shape. Several techniques including 3D printing techniques have been developed.

A discussion of laser welding basics follows. After comes a review of various methods for 3D printing of metals, common discontinuities, parameters, and plan for optimization. Finally, the methodology, results, discussion, and conclusions of the experiments will be presented.

1.1. Forms of 3D printing

Many different 3D printing and additive manufacturing techniques have been developed since companies began rapid prototyping. Newer techniques were developed with the goal of building fully functional components rather than prototypes\(^1\). Many techniques can only be used with specialized materials. For example, stereolithography requires the use of resins that cure in the presence of ultraviolet light. Electron beam melting requires highly conductive materials, limiting its use to metals\(^1\).

A few 3D printing techniques using metals include selective laser sintering (SLS), selective laser melting (SLM), electron beam melting (EBM), and laser enabled net shaping (LENS). All four methods listed are powder based \(^1\).

All methods other than LENS are powder bed techniques that follow the same basic process. A 3D drawing is generated in a CAD program. A second software program divides the drawing into several “slices” of a predetermined thickness. Powder is deposited in the build chamber and smoothed by a rake. An energy beam then scans the powder bed in the necessary
pattern to build the desired cross-section. The platform lowers by the predetermined layer thickness and the process continues until the component is complete \(^3\).\(^4\).

In SLS metal powder particles are usually either coated or mixed with polymer binders which help support the component\(^1\). Preheating the metal powder to just below the melting point\(^2\) allows for performance without a binder. A laser provides just enough energy for fusing or sintering of powder particles to begin. Post-processing is often needed to remove any residual binder and porosity. Polymer burn-out and liquid metal infiltration is a common post-process treatment\(^3\). By selecting an appropriate second phase, SLS can be used to fabricate micro-composites\(^3\).

Unlike SLS, SLM and EBM fully melt the powder to form a near-full density part. In SLM the powder is deposited and a high-power laser is used to melt the layer. The only difference between SLM and EBM is that EBM uses an electron beam under high vacuum instead of a laser in an inert gas. Near-full density is achieved by SLM, but the high heat input causes more loss of alloying elements, residual stress, and thermal distortion than found in SLS. A higher build rate and fully dense parts can be formed by EBM than SLM, but the surface finish is even worse than for SLM\(^1\),\(^2\).

Finally, LENS differs from SLS, SLM, and EBM in that it uses no powder bed. Instead, powder is deposited through a nozzle directly into the laser’s path. Both the nozzle and laser move in conjunction so that molten metal is deposited on the substrate as programmed. Metal can be deposited on preexisting components, making LENS usable for repair and coating applications\(^1\). Much larger components can be made than with the other processes. Unfortunately dimensional accuracy is low for LENS compared to SLS, SLM, and EBM\(^2\).
1.2. Laser welding

Laser 3D printing techniques are similar to laser welding; it could be said that the entire component is built by welding. The multiple thermal cycles involved in 3D printing prevent using laser welding models for the 3D printing process. However, background information on laser welding is beneficial to understanding the SLM process.

Compared to arc welding methods, laser welding has a much higher energy density which allows a high melt pool penetration and small base metal heat affected to zone to be achieved. However, the heat transfer efficiency is greatly reduced because metals have high optical reflectance. The high depth to width ratio of laser welding may also increase the chances of thermal cracking. In a shallow weld pool, thermal stress is distributed fairly evenly in all directions. Deep, narrow weld pools have very little thermal gradient near the bottom, but much higher thermal gradients along the sides parallel and perpendicular to the weld track. High thermal stresses pull directly on the fusion zone, leading to high chances of thermal cracking.

Formation of keyholes is a common phenomenon in laser welding. High temperatures where the laser strikes the surface induces a high metal vapor pressure on the molten metal that pushes material away. The “keyhole” formed has a high aspect ratio. A void is created which the molten material tries to refill after the laser passes. The high solidification rate does not always allow time for the keyhole to be refilled before solidification.

Despite the similarities, laser 3D printing is very different from laser welding. Powders have higher surface area than sheet or plate, so reflectance decreases efficiency even more during 3D printing. Remelting, multiple thermal cycles, and multiple melt pools all cause complications when modelling 3D printing. The entire component is heated during printing, so thermal profiles are completely different.
1.3. 3D Printing discontinuities

Several discontinuities specific to 3D printing have been identified\textsuperscript{1,3,8,9}. Some such discontinuities include microcracks, gas porosity, faceted voids\textsuperscript{3,10,11}, balling, and denudation zones\textsuperscript{9}. Certain discontinuities such as faceted voids are only expected at low heat input; other discontinuities such as balling and gas porosity are expected at high heat input.

A combination of stress state, constitutional supercooling from alloy segregation, and the keyhole effect contribute to microcracking. Bonding between the molten layer and the solid substrate restricts thermal contraction at the bottom of the melt pool. The unrestrained upper melt pool surface can contract almost freely, inducing a state of bending stress\textsuperscript{12}. Bending stress occurs when one surface is under compression and the opposite is under tension. Small cracks initiate and grow to absorb energy, reducing the overall stress in the component\textsuperscript{10,12}.

Constitutional undercooling occurs during solidification of solutions when solute is rejected from one phase and concentrated in another\textsuperscript{6,7}. Under rapid solidification, rejected solute concentrates near the liquid/solid interface, creating a brittle phase that may crack during or immediately following solidification\textsuperscript{6,7,10}. In addition the keyhole effect\textsuperscript{6,7} may also cause cracks to form if the keyhole refilled before solidification\textsuperscript{10}. Keyhole discontinuities may look like either thin faceted voids or wide cracks.

Most gaseous porosity in 3D printing is the result of gas entrapped between powder bed particles\textsuperscript{16}. Gas pores are often spherical because of hydrostatic pressure. Other possible sources are evaporation of alloying elements (common in welding)\textsuperscript{6,7} and atmospheric gasses dissolved in the molten metal which is later rejected during cooling. Porosity from dissolved gasses is more rare because an inert argon atmosphere is usually used\textsuperscript{1,3,17,18}, although at high temperatures even argon becomes slightly soluble\textsuperscript{19}. 
Faceted voids are thought to be caused by lack of fusion or incomplete melting and are often accompanied by unmelted particles\textsuperscript{3,13,14}. Faceted voids are generally worse than gaseous pores because faceted voids are larger and the irregular shape acts as a stress concentrator\textsuperscript{3,15}. Unmelted powder particles make it more difficult to measure porosity accurately\textsuperscript{10}.

Denudation zones are locations where material was lost due to spatter\textsuperscript{9}. A “significant backpressure”\textsuperscript{16} is present where the laser strikes\textsuperscript{16}. Additionally, boiling temperatures have been reached where the laser strikes\textsuperscript{9}. Surface tension varies greatly as a result of the high temperature gradient present during laser melting. Metal vapor pressure and the high surface tension gradient combined causes spatter, thus creating the denudation zones\textsuperscript{9}.

A surface tension effect called Marangoni convection causes balling\textsuperscript{3,9,14}. Marangoni convection results from differences in surface tension between the center and edge of the melt pool\textsuperscript{6,7}. Surface tension is typically higher at the relatively cool edge of melt pools than at the hotter center. Molten metal is pulled from the center towards the edges by the surface tension gradient\textsuperscript{6,7}. Material at the edges flows downward and towards the center. Finally, to fill the space, material flows up at the center. Under very high temperature gradients, Marangoni convection causes balling to occur. Balling increases when wettability between the melt and substrate is poor\textsuperscript{14}.

1.4. Parameters

Laser 3D printing involves several parameters. Laser power, travel speed, and hatch spacing are three of the most easily manipulated. The hatch pattern, which affects the thermal stress profile, is easy to manipulate but the effect is difficult to quantify. Layer-by-layer fabrication causes anisotropy, so build angle or component orientation becomes important.
Heat input is a function of several parameters. One definition of heat input is the energy density \( E \):

\[
E = \frac{P}{\nuht}
\]  

(1)

where \( P \) is the laser power in watts, \( \nu \) is the travel speed in mm/s, \( h \) is the hatch spacing in mm, and \( t \) is the layer thickness in mm. An alternative method of 3D printing has a laser blinking on and off instead of scanning continuously\textsuperscript{14}. In such cases the spot distance (distance between exposure locations) divided by exposure time is analogous to travel speed. Decreasing travel speed decreases porosity and increases microhardness up to a peak, but at too low of a travel speed microhardness decreases again. Microhardness always decreases as porosity increases\textsuperscript{14}.

Layer thickness was kept constant in the present study. The formula from Sun \textit{et al}\textsuperscript{10} was modified to form the global energy density (GED) defined as

\[
GED = \frac{P}{\nu h}
\]  

(2)

without the layer thickness. While the GED is important, each individual parameter used in the calculation may have its own effect on the resulting properties.

Laser power directly influences the GED. With all other parameters held constant, higher laser power results in more heat accumulation. Denudation and spatter may increase with laser power because a higher surface tension gradient forms\textsuperscript{9}. Boiling temperatures in the melt pool even occur with high enough laser power\textsuperscript{9}.

Increasing laser speed decreases laser/material interaction time and lowers the heat input. While increasing the laser speed decreases the overall build time, the loss of heat input may
cause faceted voids and unmelted particles\textsuperscript{3,10}. Alternatively, high travel speeds during laser welding are known to cause the formation of long, narrow, teardrop-shaped melt pools which act as stress concentrators\textsuperscript{6,7}. Depth of penetration also decreases as travel speed increases\textsuperscript{6,7}, which may lead to poor bonding between the current melt and previously melted layers.

Hatch spacing affects heat transfer between laser tracks. Higher hatch spacing means fewer scan lines and lower heat input. With high hatch spacing, less heat is available for transfer between melt tracks. Poor bonding or incomplete fusion between melt tracks becomes possible, resulting in porosity when hatch spacing is high. Low hatch spacing increases the overall build time and thermal stress\textsuperscript{10}. Excessively high travel speed and low hatch spacing causes vertical cracking\textsuperscript{10}.

Powder bed thickness is another parameter. Thick powder beds require more energy for complete melting than thin powder beds. More gas is present per layer and the gas has to travel through more material to escape the melt, so porosity becomes more likely\textsuperscript{3}. Powder bed thickness influences heat and mass transfer in the melt pool\textsuperscript{18}. Powder bed thickness was kept constant in this study.

Other parameters exist, but are neither used in heat input calculations nor modified in this study. One such parameter is the hatch pattern\textsuperscript{13}. Scan lines can be made in various patterns such as horizontal lines, vertical lines, or grids\textsuperscript{13}. The thermal stress profile changes depending on the pattern. Horizontal stripes or vertical stripes concentrate thermal stresses in one dimension; grid patterns balances thermal stresses in two directions.

Thermal stresses relieving strategies are available. One strategy is rescanning the component layers\textsuperscript{13,20}. Rapid cooling of the component and constraints to thermal expansion cause thermal stresses. Rescanning gives more time for metal to contract, relieving up to 8% of
thermal stress as measured by differences in curvature\textsuperscript{13}. Rescanning not only relieved thermal stress, but also significantly decreased porosity\textsuperscript{4,20}. Only a single rescan is beneficial for porosity; density drops again after multiple rescans\textsuperscript{4}. Disadvantages to rescanning are that it increases build time\textsuperscript{10,20} and the energy consumption.

Various scanning strategies are available to influence thermal stresses. Vertical stripes, horizontal stripes, and grid patterns all influence the thermal stress profile differently\textsuperscript{13}. Scan line length may be changed. Rather than scanning the entire line continuously, it is possible to scan a short length in the scanning direction, move down the part, and then scan again moving up the part. Figure 1 is a schematic drawing of two possible strategies for shorter scan vectors. Solid arrows are scan lines. The dotted arrow shows the laser travel path between scans.

![Figure 1: Scan pattern to investigate short scan vectors. Source: Kruth, Jean-Pierre, et al. “Assessing and comparing influencing factors of residual stresses in selective laser melting using a novel analysis method.” Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering](image)

Another strategy is to rotate the scan pattern after each layer\textsuperscript{3}. Each time the pattern rotates, the thermal stresses concentrate in a different direction. Balancing the thermal stress distribution helps prevent distortion. Choosing an appropriate angle of rotation allows the stresses to almost fully balance before repeated loading in the same direction.
1.5. Grain growth

Interactions between fluid flow, thermal gradient, and crystal properties control grain growth\textsuperscript{21,22,23}. Epitaxial grain growth, growth along the highest thermal gradient along the close pack direction\textsuperscript{6,21}, dominates under laminar flow conditions. Competitive growth prevents grains from growing further when the preferred growth direction is not in line with the thermal gradient\textsuperscript{21}. Fluid flow is often turbulent during welding\textsuperscript{24}, and by extension, during 3D printing.

Figure 2 shows the basic solidification modes and the relative level of constitutional supercooling at which they occur. Constitutional supercooling is related to the ratio of the thermal gradient (G) to the solidification rate (R), or the G/R ratio. Higher G/R ratios indicates more constitutional supercooling and increases the chances of either planar or cellular solidifications modes. Lower G/R ratios indicate an increased chance of dendritic solidification occurring\textsuperscript{6}. The solidification mode effects grain shape and may impart directional properties on the metal.

![Figure 2: Effect of constitutional supercooling on solidification mode: (a) planar; (b) cellular; (c) columnar dendritic; (d) equiaxed dendritic (S, L, and M denote solid, liquid, and mushy zone, respectively). Source: Kou, Sindo. \textit{Welding metallurgy}. Hoboken (New Jersey), John Wiley & Sons, 2003.](image-url)
Fluid flow, especially turbulent flow, complicates grain growth\textsuperscript{22}. Dendrite tips may break off and rotate in the fluid, cycling in and out of orientation for epitaxial growth and provide nucleation sites\textsuperscript{6,22,23}. Grain growth is further complicated in 3D printing because of heat accumulation between layers. Heat transfers through previous layers\textsuperscript{3}, so that more heat is absorbed by the component, the effective preheat temperature increases, and grains grow larger than in later layers. However, the cooling rate decreases on subsequent layers\textsuperscript{5} so that more time is available for grain growth. Misaligned dendrites indicate that the direction of the highest thermal gradient varies. Some variation may be expected because the scan pattern rotates after each layer.

Grain growth in SLM starts at the substrate below the melt pool\textsuperscript{9}. Grains initially grow via heterogeneous nucleation and epitaxial growth. Heterogeneous nucleation is grain nucleation at preexisting solid/liquid interfaces, rather than within the center of the melt pool as in homogenous nucleation. Heterogeneous nucleation is kinetically favored because the high energy of preexisting interfaces lowers the activation energy required for nucleation\textsuperscript{6,7}.

Several models have been used to predict grain growth behavior in SLM. Most are based on modifications of finite element method\textsuperscript{9,25}. Models used in laser welding such as the Monte Carlo\textsuperscript{24} simulation are a logical starting point, but the layered structure and repeated heating complicates such models\textsuperscript{5}.

### 1.6. Porosity measurement

Porosity can be measured as a ratio of void area to expected full density. Porosity measurements are usually made by density differences according to Archimedes' principle. In 3D printing the Archimedes method often underestimates the amount of porosity\textsuperscript{13}. Unmelted
particles have the same mass and volume as the bulk component, so voids containing unmelted particles read as if almost fully fused.

Another potential method to measure porosity is x-ray computed microtomography (µ-CT). In x-ray µ-CT, an x-ray source rotates around a specimen generating 2D images through the thickness of all sides. Superimposing the 2D images produces a single 3D image. Resolutions of less than 1 µm are theoretically possible\textsuperscript{26}. Area analysis was chosen here because cross-sections were already prepared for microscopy. Accurate porosity measurements require images of multiple representative regions. Observations suggest porosity varies periodically as does microstructure. Porosity is volumetric, so area analysis may not accurately represent the true porosity. Pores are assumed to be uniformly distributed and that the results will be the same no matter which axis the specimen was sectioned across. Neither assumption is necessarily true.

1.7. Optimization

As 3D printing is a young technology, optimization is a major challenge. Working, casting, and machining processes have been used for centuries and a corresponding amount of data on how to obtain desired properties already exists. Research into 3D printing started much more recently. More data is needed on how each build parameter affects mechanical properties and microstructure. Optimization can then be accomplished.

Various studies on parameter optimization have been reported in literature. One study attempted to decrease build time by increasing bed thickness\textsuperscript{18}. High travel speeds of 2000 to 4000 mm/s were used. The laser power was not specified and the actual travel speed used at each thickness was not specified, so the laser power cannot be determined. Greater than 99% density and higher tensile yield strength than wrought was achieved even at bed thicknesses of up to 150 µm\textsuperscript{18}. 
Sun et al\textsuperscript{10} attempted using higher laser power and scan speed to decrease build time. Using a laser power of 380 W with appropriate adjustments of travel speed and hatch spacing, they decreased build time by 72\%\textsuperscript{10}. More than 99\% density and higher microhardness were obtained at all builds.

Mechanical properties of interest include yield strength (YS), ultimate tensile strength (UTS), and Young’s modulus. Non-tensile properties of interest include fatigue strength and fatigue life\textsuperscript{1,3}. Microstructure is inherently linked to mechanical properties. Tensile and fatigue strength both depend on grain size and shape\textsuperscript{27,28}. Precipitate and phase distribution in multiphase alloys also affects mechanical properties.

Metallography provides a method for measuring porosity and microstructural features. In addition to determining porosity as a ratio, the size and shape of pores can also be measured by metallography. Correlating the microstructure to varying build parameters and mechanical properties is thus an important part of the optimization process.

The relationship between build angle and mechanical properties was examined. Specimens were built at four different angles with all other parameters held constant for tensile tests and fatigue tests. Metallography was performed on specimens from each angle. Eight more specimens were built at 90° from the build plate with varying laser power, hatch spacing, and travel speed for metallography. Following is a detailed procedure of the study, the results, a discussion, and conclusions based on the data.
2. Methodology

Selective laser melting was the specific 3D printing method used. Particles are completely melted and fused. The scan pattern was rotated 67° after each layer to accommodate for thermal stress distribution. The bed thickness and d_{10} particle size are both 20 µm.

2.1. Equipment used

Equipment and software used for this study include:

- EOS 290 3D printer
- Hitachi S4500 Field Transmission Scanning Electron Microscope (SEM)
- Leica DM750P Optical Microscope
- Fatigue Dynamics RBF 200 Fatigue Tester
- MTS Landmark Servohydraulic Universal Test Frame
- Leica Grain Expert software
- Minitab statistical software

Specimens were built at 0°, 30°, 60°, and 90° from the horizontal for tensile and fatigue testing. Additional specimens were built at varying laser power, travel speed, and hatch spacing to study the effect on microstructure. Figure 3 represents the build angle of each specimen.

![Figure 3: Build angle of specimens for mechanical testing](image)
2.2. Tensile testing

Wrought specimens were built from rolled bar stock and tested. Tensile testing was performed on three tensile rounds. Yield strength, ultimate tensile strength, and elastic modulus were determined. Fatigue tests on the printed samples followed. Four specimens of each orientation were used. Tensile strength values were used to determine starting stress range values for fatigue testing. Dimensions of tensile specimens are provided in Figure 4.29.

2.3. Fatigue testing

Rotating beam fatigue testing was carried out after tensile testing. Again wrought specimens were tested first. Fatigue tests on the wrought were performed using 50% of the ultimate tensile strength (UTS) as the starting point. Next the 90°, 60°, 30°, and 0° build angle
specimens were tested in order using half of the UTS. Fatigue specimen dimension are shown in Figure 5.

![Fatigue specimen dimensions](image)

Figure 5: Fatigue specimen dimensions. Schematic provided by Imperium

A rotating beam fatigue tester based on cantilever theory was used. A beam subjected to a point load experiences bending stress, where pure tension is exerted on one surface and pure compression on the opposite. By rotating the beam every point on the surface cycles through 100% pure compression ($-\sigma$) and 100 percent pure tension ($\sigma$).

By fixing one end and placing the load on the opposite end, bending stress is theoretically highest at the fixed end. In real components, stress concentrations exist along the entire length and the highest stress is difficult to predict. An annular notch with a constant radius of curvature cut into the beam resolves the issue. Bending stress is a cubic function with respect to diameter
and a linear function with respect to length. Having the thinnest cross-section in the center therefore increases the chances that the stress will be highest at the center. On the RBF 200 one end of the specimen is inserted into a rigid support. The other end is placed in a free-floating arm connected to a graduated lever and sliding weight. The lever arm is calibrated in inch-pounds.

2.4. Metallography

Metallography specimens were first polished on silica grit paper. Fine polishing used cloth with 5 µm and 1 µm alumina powder. A final polishing step using 0.25 µm diamond paste was performed. After polishing the samples were etched by immersion in Viella’s reagent. Optical microscopy was performed. A Leica DM750P optical microscope was used. Grain length and compactness data were generated using five images of each orientation and five images of each parameter set. Data was compiled in Minitab® analysis software and used to create histograms.

2.4.1. Orientation

Metallography was performed on tensile specimen remnants. Two cross-sections were taken for each orientation: an x-y cross-section (perpendicular to the loading axis) and a z cross-section (parallel to the loading direction). Because the properties of printed metals have been typically found to be anisotropic, both cross-sections were required to understand how microstructure develops. Figure 6 illustrates where each cross-section was taken.
2.4.2. GED

Additional specimens were built with varying laser power, travel speed, and hatch spacing to observe how changing the parameters affected microstructure. All specimens for this portion of the study were built at 90°. Only under the P-9 conditions, which were the original build parameters, were specimens built at varying orientations and used for mechanical testing. A two-level three-parameter factorial design matrix was used. One high and one low value were chosen for each of the three parameters. A specimen was built for each possible combination of parameters. Table I lists the label and parameters of each specimen. Metallography was performed as described above on the z-sections.
Table I: Build parameters

<table>
<thead>
<tr>
<th>Sample Label</th>
<th>Power (P)</th>
<th>Speed (v)</th>
<th>Spacing (h)</th>
<th>GED (P/vh)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-1</td>
<td>156</td>
<td>866.4</td>
<td>0.072</td>
<td>2.50</td>
</tr>
<tr>
<td>P-2</td>
<td>234</td>
<td>866.4</td>
<td>0.072</td>
<td>3.75</td>
</tr>
<tr>
<td>P-3</td>
<td>156</td>
<td>1299.6</td>
<td>0.072</td>
<td>1.67</td>
</tr>
<tr>
<td>P-4</td>
<td>234</td>
<td>1299.6</td>
<td>0.072</td>
<td>2.50</td>
</tr>
<tr>
<td>P-5</td>
<td>156</td>
<td>866.4</td>
<td>0.108</td>
<td>1.67</td>
</tr>
<tr>
<td>P-6</td>
<td>234</td>
<td>866.4</td>
<td>0.108</td>
<td>2.50</td>
</tr>
<tr>
<td>P-7</td>
<td>156</td>
<td>1299.6</td>
<td>0.108</td>
<td>1.11</td>
</tr>
<tr>
<td>P-8</td>
<td>234</td>
<td>1299.6</td>
<td>0.108</td>
<td>1.67</td>
</tr>
<tr>
<td>P-9</td>
<td>195</td>
<td>1083.0</td>
<td>0.090</td>
<td>2.00</td>
</tr>
</tbody>
</table>

Pairs of specimens where only one parameter changed were chosen for comparison as summarized in Table II. Discontinuities were so clearly evident in P-7 (lowest GED) that it was excluded from these comparisons.

Table II: Build parameters selected for comparison

<table>
<thead>
<tr>
<th>Parameter of Interest</th>
<th>Low Value</th>
<th>High Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>P-1</td>
<td>P-2</td>
</tr>
<tr>
<td></td>
<td>P-3</td>
<td>P-4</td>
</tr>
<tr>
<td></td>
<td>P-5</td>
<td>P-6</td>
</tr>
<tr>
<td>Travel Speed</td>
<td>P-1</td>
<td>P-3</td>
</tr>
<tr>
<td></td>
<td>P-2</td>
<td>P-4</td>
</tr>
<tr>
<td></td>
<td>P-6</td>
<td>P-8</td>
</tr>
<tr>
<td>Hatch Spacing</td>
<td>P-1</td>
<td>P-5</td>
</tr>
<tr>
<td></td>
<td>P-2</td>
<td>P-6</td>
</tr>
<tr>
<td></td>
<td>P-4</td>
<td>P-8</td>
</tr>
</tbody>
</table>

Images were taken on both the optical microscope and the scanning electron microscope (SEM). Based on qualitative observations of the optical microscope images a set of parameters giving a high GED and a set of parameters giving a low GED were chosen for further analysis. A cross section of each bar was imaged on an SEM. Microstructures were heterogeneous. Several images were taken for analysis.
An image analysis software package including the Leica Grain Master tool was used for grain size and shape measurement. Pore size and overall percent porosity were also measured. Overall porosity was measured as an area percent and assumed to be uniformly distributed. Porosity was analyzed using three SEM images of each parameter. Pore size was measured as the equivalent diameter of a circle with the same area as the pore. Shape was measured as compactness, defined by the equation

\[ C = \frac{4\pi A}{P_r^2} \]  

where \( P_r \) is the perimeter and \( A \) is the area. A perfect circle has a compactness of one and the compactness approaches zero as the shape becomes more irregular \(^\text{30}\).
3. Results

Tensile test results, fatigue test results, and micrographs follow with qualitative descriptions. Fractographs are also presented along with an analysis into the fracture mechanism and root cause of failure.

3.1. Microstructure

Microstructural analysis was performed on a cross-section taken from tensile bars of each specimen built using P-9. Specimens later built with varying hatch spacing, travel speed, and laser power were analyzed along the length for microstructure. An image of the wrought tensile bar was taken for reference along both the x-y plane and z-plane (Figure 7). Coarse, randomly oriented grains are in the x-y cross-section. In the z-direction grains are still coarse but grain boundaries are more aligned with each other. Twins, grains that are mirror images of each other, are present in both cross-sections.

Figure 7: Optical micrographs of the wrought a) x-y plane showing randomly oriented grains and b) z plane showing aligned grains
3.1.1. Orientation

Table III contains the mean, maximum, and minimum grain length and compactness for each build angle. All specimens built at varying orientations were built under P-9 conditions. Appendix A collects histograms of grain size and shape. All orientations had the same minimum grain length, which suggests the smallest grain size was below the detection limit of the imaging software. The largest maximum grain size and smallest mean grain size were in the 90° build angle. The smallest maximum grain length and largest mean grain length belonged to the 30° specimen. The 90° had the highest mean, the highest maximum, and the lowest minimum compactness. The 30° had the lowest mean compactness. The 0° had the lowest maximum compactness.

<table>
<thead>
<tr>
<th></th>
<th>Orientation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0°</td>
</tr>
<tr>
<td>Mean Length (µm)</td>
<td>1.469</td>
</tr>
<tr>
<td>Max Length (µm)</td>
<td>10.298</td>
</tr>
<tr>
<td>Min Length (µm)</td>
<td>0.233</td>
</tr>
<tr>
<td>Mean Compactness</td>
<td>0.485</td>
</tr>
<tr>
<td>Max Compactness</td>
<td>0.870</td>
</tr>
<tr>
<td>Min Compactness</td>
<td>0.111</td>
</tr>
</tbody>
</table>

Long dendrites are seen growing parallel to melt pool boundaries in Figure 8a. Most grains are fine cellular structures. Two small discontinuities here-on referred to as “islands” separated from the surrounding microstructure are present. Arrows surround very faint grain boundaries which confirm that the cellular structure is subgranular as has been previously reported. Voids and denudation zones were observed on the 0° build angle x-y cross-section as shown in Figure 8b.
Grain size and compactness data was generated from five different 1000X micrographs. The data was consolidated and used to generate histograms in Minitab 17 statistical software.

A discontinuity best described as an island separated from the surrounding metal is seen in the 0° build angle z-section in Figure 9. A bead of metal seems not to have melted, to have separated from the previously melted layer, and rotated 90°. Voids surround the border of the island.
More of the longer grains are observed in the micrograph of 30° build angle x-y-section (Figure 10a). Smaller cellular regions are present. Fine and coarse cellular structures are present, as opposed to in the 0° where grains were either fine cellular or dendritic. Figure 10b reveals balling. Dendrites warp around the edges and gaps are present between the ball and the rest of the surface.

Figure 10: Optical micrograph (a) showing fine and cellular structures and SEM image (b) of 30° x-y-section showing balling

Figure 11 displays a denudation zone in the 30° build angle z-section. Material at the edge of the cavity has a slight upward and inward slope. Nearby spattering discontinuities show as bright spots.
Figure 11: SEM image of 30° z-section containing a denudation zone

Figure 12a shows a microstructure almost identical to that seen in the 30°, as may be expected from the histograms reported in Appendix A. Some difference is noted in the shape of MPBs between the two specimens, but that may be due to how the specimen was cut. Figure 12b indicates an island on the 60° build angle x-y-section similar to that seen on the 0° z-section. Porosity is not present around this island, but faint lines which may be microcracks are present. A MPB crosses the island and disappears near the middle before reappearing on other side. The microstructure does not look like it has been rotated to the same angle as the island on the 0° z-section.

Figure 12: Optical micrograph (a) showing sharply curved MPBs and SEM image (b) showing an island in the 60° x-y-section
The 60° build angle z-section contains a faceted void. Some nearby spherical pores are also observed in Figure 13. The large void resembles a tear. An MPB intersects the tear and delaminates from the rest of the metal.

![Figure 13: SEM image of 60° z-section showing a void](image)

Even though the histogram in Figure 40d of Appendix A shows that the 90° build angle has the longest grains of all the x-y-sections, such long dendrites are not seen in Figure 14a. Nearly all grains are very fine cellular structures, with just a small region of dendrites. A site of incomplete fusion or possibly a keyhole was found on the 90° x-y-section (Figure 14b). Several smaller voids and pores are nearby.
Figure 14: Optical micrograph (a) showing a highly cellular microstructure and SEM image (b) showing a keyhole in the x-y-section.

Figure 15 is a ball found on the 90° build angle z-section. The surrounding microstructure seems to have somehow glazed over a part of the ball. Pores resembling casting blows are near the intersection between the ball and the plane of the cross-section.

3.1.2. GED

Metallography was performed on specimens of varying laser power, travel speed, and hatch spacing that determines GED. Metallography was used to screen potential candidates for future mechanical testing. Table IV contains the mean, minimum, and maximum grain length
and compactness of the various parameter sets. Histograms of the grain size and shape are shown in Appendix B.

Table IV: Statistics of changes in grain size and shape with GED

<table>
<thead>
<tr>
<th>Orientation</th>
<th>P-1</th>
<th>P-2</th>
<th>P-3</th>
<th>P-4</th>
<th>P-5</th>
<th>P-6</th>
<th>P-8</th>
<th>P-9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Length (µm)</td>
<td>1.327</td>
<td>1.283</td>
<td>1.200</td>
<td>1.213</td>
<td>1.198</td>
<td>1.566</td>
<td>1.188</td>
<td>1.201</td>
</tr>
<tr>
<td>Max Length (µm)</td>
<td>6.090</td>
<td>6.567</td>
<td>4.836</td>
<td>4.597</td>
<td>5.373</td>
<td>7.680</td>
<td>17.194</td>
<td>4.836</td>
</tr>
<tr>
<td>Min Length (µm)</td>
<td>0.239</td>
<td>0.299</td>
<td>0.239</td>
<td>0.239</td>
<td>0.239</td>
<td>0.233</td>
<td>0.239</td>
<td>0.299</td>
</tr>
<tr>
<td>Mean Compactness</td>
<td>0.608</td>
<td>0.657</td>
<td>0.664</td>
<td>0.669</td>
<td>0.657</td>
<td>0.503</td>
<td>0.657</td>
<td>0.664</td>
</tr>
<tr>
<td>Max Compactness</td>
<td>0.918</td>
<td>0.938</td>
<td>0.938</td>
<td>0.922</td>
<td>0.941</td>
<td>0.862</td>
<td>0.939</td>
<td>0.939</td>
</tr>
<tr>
<td>Min Compactness</td>
<td>0.177</td>
<td>0.221</td>
<td>0.195</td>
<td>0.238</td>
<td>0.225</td>
<td>0.145</td>
<td>0.133</td>
<td>0.234</td>
</tr>
</tbody>
</table>

Optical microscopy was performed to obtain a broad overview of the features present. Further analysis using an SEM was required because the microstructure was so fine. Several of the SEM images display discontinuities. Porosity measurements, shown in Table V, were made on the Leica software using SEM images. Cells where porosity was not observed are labeled “NO”.

Table V: Porosity measurements at different parameters

<table>
<thead>
<tr>
<th>Specimen</th>
<th>P-1</th>
<th>P-2</th>
<th>P-3</th>
<th>P-4</th>
<th>P-5</th>
<th>P-6</th>
<th>P-7</th>
<th>P-8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Pore Size (µm)</td>
<td>0.097</td>
<td>0.115</td>
<td>NO</td>
<td>0.052</td>
<td>0.178</td>
<td>NO</td>
<td>2.576</td>
<td>0.120</td>
</tr>
<tr>
<td>Porosity (area %)</td>
<td>1.802</td>
<td>14.667</td>
<td>NO</td>
<td>0.482</td>
<td>14.880</td>
<td>NO</td>
<td>0.361</td>
<td>1.923</td>
</tr>
</tbody>
</table>

Figure 16a is a micrograph of P-1. A complex microstructure containing melt pool boundaries (MPBs), columnar dendrites, and cellular features appears. Extremely fine, needlepoint-like features, thought to be pores, are present near grain boundaries. Large, discolored features are etching artifacts. A high magnification SEM image of the cellular
structures (Figure 16b) reveals nano-sized pores within the grains. Some pores contain small, bright particles believed to be nano-inclusions.

![Figure 16: Optical micrograph showing dendrites and cellular features(a) and SEM image showing porosity(b) in P-1: Power 156; Speed 866.4; Hatch 0.072](image)

Microstructure varied with position. Figure 17 demonstrates a different location on the cross-section with a different microstructure. Compared with the micrograph in Figure 16, more elongated grains are present. Elongated grains in different areas have mismatched orientations. The various regions repeat periodically throughout the specimen. Further analysis is done using images that reveal the most varied features within a specimen.

![Figure 17: Additional image of P-1 demonstrating variation of microstructure with location on specimen](image)
Other specimens had similar microstructures. Figure 18a is the microstructure of specimen P-2, which was built at the highest GED. Cellular structures in P-2 are slightly coarser than in P-1. Columnar dendritic grains look longer but thinner. More of the small, needlepoint-like features are present. Columnar dendritic grains were not all aligned in the same direction. A high pore density with pores of varying sizes is shown under the SEM (Figure 18b). Nano-inclusions were not observed under these conditions.

![Figure 18: Optical micrograph showing highly aligned dendrites (a) and SEM image showing microporosity (b) in P-2: Power 234; Speed 866.4; Hatch 0.072](image)

Figure 19a presents the microstructure of P-3. In contrast to Figure 16, columnar dendritic grains are short and thick. Fine cellular structures are present. Unlike in the previous two specimens, sharp cornered features are visible. Isolated pores were observed, but no widespread porosity or voids were seen on the SEM. Denudation zones were present in a few locations as shown in Figure 19b. Nano-inclusions were not observed.
Figure 19: Optical micrograph short, thick dendrites (a) and SEM image showing a denudation zone (b) in P-3: Power 156; Speed 1299.6; Hatch 0.072

Figure 20 represents P-4. The microstructure is coarser than most of the other microstructure except for P-2. Speed and power were high, hatch spacing was low. All dendritic grains are aligned in the same direction. MPBs are longer and less sharply curved than in other specimens. Small needlepoint features are present as in P-1 and P-2. As in P-1, nano-sized pores and nano-inclusions are present.

Figure 20: Optical micrograph showing coarse features(a) and SEM image showing nano-sized pores(b) in P-4: Power 234; Speed 1299.6; Hatch 0.072
P-5 (Figure 21) was built at low laser power, low travel speed, and high hatch spacing. The microstructure is relatively coarse. Even though the specimen had the same GED as P-3, no noticeable faceted voids are present. Columnar dendritic structures are not as long as in P-4 and some misalignment is again apparent. Figure 21b indicates how the layer-by-layer process and complicated metal-laser interaction affects the microstructure. The image was taken near the center of the specimen. With a few exceptions, dendrites tend to be oriented roughly perpendicular to the MPBs.

Figure 21: Optical micrograph showing relatively coarse microstructure (a) and SEM image showing highly varied grain growth directions (b) in P-5: Power 156; Speed 866.4; Hatch 0.108

P-6 was produced under high laser power, low travel speed, and high hatch spacing. Fewer cellular structures appear as demonstrated by Figure 22a. Columnar dendritic grains are shorter than in the specimens shown so far. Analysis on the SEM revealed isolated denudation zones and a faceted void (Figure 22b). No overall porosity was noted.
Figure 22: Optical micrograph showing more dendritic and fewer cellular regions (a) and SEM image showing a denudation zone (b) in P-6: Power 234; Speed 866.4; Hatch 0.108

P-7 was built at the lowest GED of 1.11; low power, high travel speed, and high hatch spacing were used. Several large faceted voids were clearly present in Figure 23. SEM imaging reveals unmelted particles within the voids.

Figure 23: Optical micrograph showing faceted voids (a) and SEM image showing unmelted powder particles (arrow) (b) in P-7: Power 156; Speed 1299.6; Hatch 0.108

P-8 was built using high power, high speed, and high hatch spacing. The GED was 1.67. It appears to have the finest microstructure of all the samples. A long, thin faceted void is visible
in Figure 24a. Porosity, columnar dendritic zones, and cellular zones intersect around a similar void in Figure 24b. The voids look like either solidification cracking or keyholes.

![Figure 24: Optical micrograph showing an extremely fine microstructure (a) and SEM image showing a keyhole (b) in P-8: Power 234; Speed 1299.6; Hatch 0.108](image)

Finally, P-9 was built with the original build parameters that was used for mechanical testing and built at various build angles as a control specimen. More short dendrites are present than in the other parameter sets. Some longer dendrites are visible, but there are more cellular structures than in most other parameters as shown in Figure 25a. Figure 25b reveals porosity and nano-inclusions, but discontinuities were not observed otherwise.

![Figure 25: Optical micrograph showing high cellular region density (a) and SEM image showing a lack of porosity (b) in P-9: Power 195; Speed 1083.0; Hatch 0.90](image)
3.2. Tensile test results

Tensile tests were performed to determine mechanical properties under static loading. The 0.2% offset yield strength (YS) is the stress where notable plastic deformation begins. The ultimate tensile strength (UTS) is the stress at the highest point on the stress-strain curve. Young’s modulus or the elastic modulus (E) is the relationship of stress to strain. It is the slope of the linear region of the stress-strain curve. Figure 26 contains the stress-strain curve for each orientation.

![Average Tensile Results](image)

**Figure 26:** Stress-strain curves for wrought specimens and 3D printed specimens built at varying orientations

Table VI summarizes values of each mechanical property of interest except for elongation, along with the standard deviation within each build. In all cases the standard deviation is low (less than one), suggesting that 3D printed components may be as reliable as traditional wrought components.
### Table VI: Summary of tensile properties

<table>
<thead>
<tr>
<th>Orientation</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>E (GPa)</th>
<th>Std. Dev (YS)</th>
<th>Std. Dev (UTS)</th>
<th>Std. Dev (E)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0°</td>
<td>527</td>
<td>629</td>
<td>185</td>
<td>0.14</td>
<td>0.18</td>
<td>0.403</td>
</tr>
<tr>
<td>30°</td>
<td>499</td>
<td>606</td>
<td>186</td>
<td>0.58</td>
<td>0.49</td>
<td>0.322</td>
</tr>
<tr>
<td>60°</td>
<td>518</td>
<td>617</td>
<td>188</td>
<td>0.29</td>
<td>0.49</td>
<td>0.131</td>
</tr>
<tr>
<td>90°</td>
<td>469</td>
<td>536</td>
<td>165</td>
<td>0.66</td>
<td>0.18</td>
<td>0.812</td>
</tr>
<tr>
<td>Wrought</td>
<td>371</td>
<td>646</td>
<td>187</td>
<td>0.53</td>
<td>0.10</td>
<td>0.274</td>
</tr>
</tbody>
</table>

Figure 27 is a bar graph comparing the YS, UTS, and E for specimens built at each build angle.

![Tensile Properties](image)

**Figure 27: Average tensile properties of four specimens**

All build conditions except for the 90° had a Young’s modulus from 185 GPa to 188 GPa, having no more than about 1% difference from the wrought. Young’s modulus for the 90° sample was 165 GPa. There was a 12% difference between the value for 90° and the wrought.

### 3.3. Fatigue

Figure 28 is the S-N curve summarizing the fatigue test data. It displays the fatigue life, or number of cycles until failure (N), and the stress that each sample was tested at (S). Too few
specimens were available to repeat trials, so only a preliminary study could be performed. Scatter is clearly visible in the 3D printed specimens but cannot be measured. Fatigue testing of the wrought yielded a smooth S-N curve. In the 3D printed S-N curves, some specimens survived more cycles at higher stress than at lower stress. Except for the 90° specimens, the printed had higher fatigue strengths than the wrought.

![Fatigue Life](image)

**Figure 28: Fatigue test results for wrought and printed specimens**

Four fatigue test specimens were available for each orientation. Fatigue strength is highly sensitive to surface discontinuities, therefore several samples are typically tested at each stress level. Four samples are not statistically significant for fatigue testing. Only a preliminary study could be performed. Some of the specimens that did not break were rerun at other stress values to increase the number of data points. Mechanical problems hampered testing of the 60° specimens. Due to various laboratory infrastructure events, testing of the 60 was hampered. Data for the 60° specimens are less reliable than for other specimens.

Too few data points were available and too much scatter was present to determine a concrete value for fatigue strength of the 3D printed specimens. Again, only a preliminary study
could be performed. Table VII contains a range of stress values that the fatigue strength may fall within. All build orientations except for the 90° were stronger than the wrought in fatigue, even at the low end of the given ranges.

Table VII: Estimated range of fatigue strengths

<table>
<thead>
<tr>
<th>Build Condition</th>
<th>Fatigue Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90°</td>
<td>270-280</td>
</tr>
<tr>
<td>60°</td>
<td>376-402</td>
</tr>
<tr>
<td>30°</td>
<td>330-350</td>
</tr>
<tr>
<td>0°</td>
<td>345-350</td>
</tr>
<tr>
<td>Wrought</td>
<td>305</td>
</tr>
</tbody>
</table>

3.4. Fractography

Fractography was performed on one tensile fracture surface and one fatigue fracture surface for each build angle.

3.4.1. Tensile

Fracture surfaces of the wrought, 0°, 30°, 60°, and 90° orientation specimens are presented. Figure 29 shows the fracture surface of a wrought tensile specimen. Porous honeycomb structures (Figure 29a) and cracking (Figure 29b) are visible.

Figure 29: Fracture surface of wrought tensile specimen.
Figure 30 shows a fractograph of a 0° tensile specimen. Dimples and fibrous structures are present in Figure 30a. A quasi-cleavage plane believed to be a melt pool boundary\textsuperscript{8,17} is among the fibrous structures. Large craters are scattered around the surface. Craters are so deep that the bottom cannot be seen as shown in Figure 30b.

Figure 30: Fracture surface of 0° tensile specimen.

Figure 31 is a fractograph of a 30° build specimen. An unmelted powder particle was found near the edge of the surface (Figure 31a). Fibrous structures are present within the center of the surface in Figure 31b. Some ductile ridges resemble MPBs more than grain boundaries. An incompletely fused particle is surrounded by a relatively smooth surface.
Figure 31: Fracture surface of 30° tensile specimen.

Figure 32 is a fractograph of a 60° specimen. A honeycomb structure surrounds a quasi-cleavage plane. The quasi-cleavage plane in Figure 32a is thought to be either a melt pool boundary or a delamination site. The edge of a crater is to the left of the quasi-cleavage plane. An unmelted particle about 1 µm in diameter is wedged within a void in Figure 32b.

Figure 32: Fracture surface of 60° tensile specimen.

Figure 33 is a 90° specimen fractograph. In Figure 33a a powder particle is at the bottom of a crater that did not fully develop. A highly ductile honeycomb structure is present. In Figure 33b secondary cracks follow MPBs.
3.4.2. Fatigue

Fatigue fracture surfaces are divided into three zones: the crack initiation zone, propagation zone, and final fracture zone. According to Dowling\textsuperscript{15}, several different crack initiation zones commonly form, but only one dominates failure. Plastic fracture occurs during crack propagation and striations commonly form\textsuperscript{27}. Once the crack has grown to a critical length, fatigue specimens fail catastrophically by brittle fracture. A rough fracture surface is observed due to the rapid failure and tearing of metal in the final fracture zone.

Two possible crack initiation zones were detected in the wrought specimen (Figure 34). One was a vertical crack at the edge of the specimen shown in Figure 34a. The tip of the vertical crack acted as a stress concentrator allowing a horizontal crack to initiate. Another possible initiation point was where the metal folded during rolling (Figure 34b). Faint striations travel away from the fold. Porosity and dimpling were present in the final fracture zone in Figure 34d.
SEM images of the different fracture zones of a 0° build specimen are presented in Figure 35. Linear details diverging from the edge indicate the dominate crack initiation zone in Figure 35a. A higher magnification of the edge (Figure 35b) indicates that a cluster of subsurface pores was the root cause of failure. Striations were visible in the crack propagation zone in Figure 35c. Figure 35d reveals faceted voids within the final fracture zone. Faceted voids were not found in the propagation zone.
Figure 35: Fractograph of 0° build fatigue specimen (a,b) crack initiation site, (c) propagation zone, and (d) final fracture zone.

Figure 36a is a possible crack initiation site in a 30° build orientation specimen. An unfilled space about the size of a particle is present (Figure 36b). Figure 36c is a high magnification view of the crack propagation zone. Striations radiate from a secondary crack. Extensive secondary cracking appears in the final fracture zone from Figure 36d.
Figure 36: Fractographs of $30^\circ$ fatigue specimen (a,b) crack initiation site, (c) propagation zone, and (d) final fracture zone.

Figure 37 is a fracture surface of the $60^\circ$ fatigue specimen. Two large voids in Figure 34a are believed to be crack initiation sites. In Figure 37b the cracks converge and travel away from a single void. Secondary cracking and striations are present in the main crack propagation zone in Figure 37c. A rough fracture surface reveals tearing in the final fracture zone (Figure 37d).
A void at the surface of the specimen is in Figure 38a. Faint and highly visible linear features run perpendicular to each other in the crack propagation zone (Figure 38b). The more highly visible features are likely slip bands which indicate high levels of plastic deformation\textsuperscript{36}. Figure 38c shows fibrous features in the final fracture zone. A higher magnification image of the final fracture in Figure 38d zone reveals many particulates. Secondary cracking was not observed on the 90° fracture surface.
Figure 38: Fractographs of 90° fatigue specimen (a) crack initiation site, (b) propagation zone, and (c, d) final fracture zone.
4. Discussion

4.1. Microstructure as a function of orientation

Orientation had a small effect on mean grain size and shape distribution. Discontinuity density was low at all orientations. Compactness followed a near-normal distribution, but the size distributions were non-normal. The 0° and 90° build angles had more outliers in grain length, while the 30° and 60° had grain lengths less concentrated around the mode. Denudation zones and islands were the most commonly observed discontinuities.

While grain size had little dependence on build angle, the 0° and 30° build angle specimens did have larger maximum grain sizes than either the 60° or 90° build angle specimens. Theoretically, the highest thermal gradient is from the top layer down to the build plate. The 0° and 30° x-y cross-sections are more in line with that expected thermal gradient than the 60° and 90° x-y cross-sections. Grains are longer because the cross-sections are more in line with the thermal gradient.

Leica Grain Expert measured larger grains in the 90° than in the 30° or 60°, but that was not observed in the micrograph. As suggested by Zhong et al\textsuperscript{31}, cellular structures were subgranular. The selected etchant may not have sufficiently revealed the actual grain boundaries sufficiently for the naked eye to see, but well enough for the computer to identify.

4.2. Microstructure as a function of GED

P-7 was the only specimen with a pore size of over 1 µm. Pores were low compactness faceted voids. Voids were irregularly spaced and concentrated near the center of the specimen, so the percentage porosity reported is not truly representative. A truly representative value requires use of Archimedes’ principle, but the presence of unmelted particles would also cause measurement errors\textsuperscript{10}.
Parameters 8, 5, and 2 had the largest gaseous pore size and the highest porosity. All three were built at high GED. P-2 had the highest GED of all samples, and was expected to have the highest level of gas porosity; this was not the case. P-5 had both the largest pore size and highest percent porosity. Maybe because of the high energy density available, enough heat was available for the metal to diffuse into and close some pores in P-2. In most specimens, pore size and percent porosity depended on whether the region was cellular, columnar, or near a melt pool boundary (MPB). Measurements could be affected if the micrographs were not representative of the periodic microstructure.

Complex correlations exist between build parameters and microstructure. Micrographs indicate that not just overall heat input but each parameter affecting heat input impacts the microstructure differently. The optical and SEM image of P-5 each show a complex microstructure with columnar grains aligned in various directions. Both images were taken near the center of the specimen. Recall that during 3D printing the build platform was rotated by 67° after each layer. The thermal gradient changed direction correspondingly, causing a change in direction of grain growth. Laser tracks intersect near the center. The varying alignment of dendrites is likely a result of rotating the stage. The images suggest that the laser affects the microstructure even several layers below the working layer.

Most dendritic regions were perpendicular to MPBs, but some ran parallel. Dendrites growing perpendicular to MPBs grew from top to bottom. Dendrites that grew parallel to MPBs may have grown in the direction of laser travel. As the laser traveled, more heat was present in the scanning direction. Some dendrites were thus encouraged to grow perpendicularly to others.

Under certain building parameters bright particles were found within gaseous pores. The particles were thought to be oxide nano-inclusions. Normally inclusions are detrimental to
mechanical properties, but under specific conditions they can become strengthening agents. To increase strength, particulates (including nano-inclusions) must be: nearly spherical, below a critical size, and uniformly distributed throughout the matrix.

Nano-inclusions were within the expected size limit. They looked fairly round at the available magnification. Gaseous pores containing nano-inclusions were located within specific regions of the microstructure, so the nano-inclusions were not uniformly distributed; however, the periodic microstructure suggests that clusters of nano-inclusions were also uniformly distributed. Nano-inclusions may still act as strengthening agents, thus they contribute to mechanical properties.

A potential oxygen source is a preexisting oxide layer on powder particles. Whatever the source, the oxygen is so low and the cooling rates are so high that many nano-inclusions nucleate but lack the energy to grow larger.

P-4 and P-8 were selected for further mechanical testing. P-8 was mainly chosen for the apparently large proportion of cellular grains and uniform cellular grain size. P-4 was chosen because it lacked long dendrites and again several grains within a narrow size range. Narrow size distributions theoretically make it simpler to model relationships between microstructure and mechanical properties.

Further observations on the SEM revealed that P-3 and P-6 may have been preferable because of the lack of observed porosity. Quantitative measurements of the microstructure show that all four had similar grain size and shape distributions, so all are worth considering for further analysis.
4.2.1. Laser power

According to Saad et al\textsuperscript{9}, denudation zones were caused by spatter where the laser impinges on the powder bed. Increasing laser power increases heat input and thermal gradient. Three effects are expected by increasing the laser power: coarser grains, more gas porosity, and more denudation zones.

With all other parameters held constant, specimens built at higher laser power have longer dendrites than those built at lower power. Fewer but larger dendritic regions tend to form at higher laser powers. Except for the pair one pair built at high hatch spacing, components built at higher laser powers tend to have higher porosity than specimens built at lower laser power. Also excepting the same pair, specimens built at higher laser power tended to have more compact grains. Interactions between laser power and hatch spacing must be responsible for the differences in observations.

Cellular structure size was more varied in specimens built at high power and low speed. The lower dwell time implied by high travel speed imparted less energy for grain growth. Interactions between high laser power and low travel speed result in the growth of larger cellular structures. Each grain grows at the expense of the surrounding grains, so more varied grain sizes are observed. Building at low power and low speed gives a less varied microstructure which theoretically makes it easier to derive a relationship between microstructure and mechanical properties.

4.2.2. Travel velocity

At constant hatch spacing, combinations of either high power and low speed or low power and high speed yielded more varied dendrite direction. Either condition gives a higher GED. Increasing the GED at constant hatch spacing raised the thermal gradient. Combined with
the rotation of the build platform, the high thermal gradient caused grains to grow in various directions.

Specimens built with higher travel velocity had longer grains and had larger dendritic regions, but were more compact on average. The longer grains suggest a higher thermal gradient in the laser travel direction. That the average compactness also increased indicates the presence of extremely fine cellular grains in sufficient number to counteract the effect of the longer grains.

Less porosity was usually present in specimens built at high travel speeds, except for specimens built at high hatch spacing. Having both high speed and high hatch spacing decreases the GED, which may cause increased porosity. Specimens built at high power, low speed, and high hatch spacing are favorable because they have lower porosity and less aligned dendrites. Lower porosity is always expected to increase strength. Dendrites add directional properties to metals, but having them aligned in different directions brings specimens closer to isotropic.

4.2.3. Hatch Spacing

Specimens built at low hatch spacing had larger maximum grain lengths than those built at high hatch spacing. In general specimens with lower hatch spacing had more varied dendrite direction. When travel speed is also high, however, higher hatch spacing gave more varied dendrite direction. High laser speed probably increased the thermal gradient in the laser travel direction, allowing dendrites to grow in different directions each time the build platform rotated.

Specimens with higher hatch spacing usually had higher porosity except for P-2, which also had the highest GED of all the specimens. Based on all of the observed results, low laser power, high travel speed, and low hatch spacing are the recommended parameters. Extreme GED values lead to much higher porosity or void space, so values should be between 1.50 and 3.0 J/mm².
4.3. Effect of orientation on elastic modulus

Most build conditions gave a consistent elastic modulus with less than or equal to 1% difference from the wrought. In the case of the 90° build angle specimens, the elastic modulus was much lower than that of the wrought. Elastic modulus is a physical property independent of microstructure and processing. It should not change significantly for any two specimens of the same alloy.

The lower elastic modulus may have been caused by discontinuities between layers. During deformation areas of incomplete bonding increase the actual stress. Discontinuities often concentrate at mold walls during casting\textsuperscript{22}. Similarly, discontinuities may concentrate between layers in 3D printing\textsuperscript{31}. Balling has been found to occur at the substrate due to oxidation of solidified metal and poor wetting\textsuperscript{21}. Higher strain would result and the incorrectly calculated strain would make the elastic modulus appear lower than it truly was.

Another possibility is that delamination occurred in more than one layer. Weak spots may have caused delamination to initiate in one layer. After the crack grew a certain length, delamination at a weak point in a separate layer became more energetically favorable. Sequential delamination of separate layers would continue until the stress was so high that on crack grew the entire length. Such a failure mode would cause much higher elongation and result in lowering the measured elastic modulus.

4.4. Effect of orientation on yield strength

Wrought specimens had lower YS values but higher UTS values than 3D printed specimens. It is not surprising that the wrought YS was lower than the 3D printed YS because of the fine microstructure. Grain boundaries increase the strength of metals with small grains and
fine microstructures. Grain boundaries are barriers to dislocation motion, causing the increase in strength\textsuperscript{27,28} of 3D printed specimens over wrought specimens.

The plane of stress on the 0° build angle specimen was orthogonal to the layers. Delamination was unlikely, which may have caused the higher strength compared to other 3D printed specimens. On the 30° and 60°, the planes of stress were oblique to the layers. Delamination became more likely, weakening the metal. Both had similar yield strengths, suggesting that the layered structure was the most significant factor in tensile strength. The 90° had the lowest YS of the 3D printed specimens. The plane of stress was parallel to the layers. Again discontinuities are believed to concentrate between layers. Additionally heat transfer is less efficient between layers than within the layers; stress profiles similar to bending develop\textsuperscript{12}. All factors lead to the lower strength of 90° components.

Similarities in grain shape provide further evidence that delamination contributed to failure more than microstructural properties. Regardless of orientation, compactness followed a nearly normal distribution centered at values near 0.5. Shape was fairly consistent for each orientation. Finally the 90° had the smallest mean grain size which supports that delamination was the dominant factor in strength.

The 30° and 60° build angle specimens had similar strengths. The 60° was slightly stronger. Both were oblique to the tensile axis. If delamination was the cause of failure, the 60° layers were closer to 90° than the 30° and thus more in line with the tensile axis than. The 60° was not expected to have been as strong. Similarities in grain size and the fact that the 60° had slightly larger grains than the 30° suggest that grain size did not cause the difference in yield strength.
Heat effects and differences in discontinuity population may be responsible for differences in YS at build angles between 0° and 90°. As the angle changes, so does the cross-sectional area of the build plane. Specimens with smaller cross-sections have shorter times between laser scan tracks and remelting. Less time was available for heat dissipation. Increased heat accumulation may have caused the formation of more discontinuities and thermal stress. Differences in YS with build angle then result from the differences in discontinuity volume and thermal stress. Qualitative analysis of discontinuity population is needed to evaluate the hypothesis.

4.5. Effect of orientation on tensile strength

The UTS followed the same trend as the YS with respect to orientation. Similar phenomena are therefore expected to be responsible for the differences in UTS as YS across build angles. The 0 specimens were strongest because delamination could not occur, while delamination was energetically preferred over yielding in the 90 specimens. Discontinuity volume or thermal stresses may have been responsible for differences in strength between the 30 and 60 specimens.

Unlike the YS, the UTS was lower for the 3D printed specimens than for the wrought specimens. Usually UTS and YS increase together\textsuperscript{27,28} and the 3D printed specimens should have a higher UTS than the wrought. One hypothesis is that the microstructure is so fine that it limits strain hardening. During strain hardening dislocations move through the crystal lattice as the metal plastically deforms. Dislocation motion generates new dislocations which begin to impede further dislocation motion. The strength of the metal increases\textsuperscript{27}. Fine grain microstructures have more grain boundaries than coarse microstructures. Grain boundaries increase YS by hindering dislocation motion\textsuperscript{27,28}. Too fine of a microstructure may impede dislocation motion to the point
that too few new dislocations form before a grain boundary cuts off motion. Strain hardening would be significantly impaired, and the UTS would be lower than for a coarser microstructure.

Alternatively the fine grains may promote grain boundary sliding. At high temperatures, grain boundaries act like pipelines that greatly increase the diffusion rate\textsuperscript{34}, creating a new pathway for plastic deformation. Grain boundary sliding is often the dominant creep mechanism in metals\textsuperscript{15,27}. Creep rate increases with smaller grains. Usually grain boundary sliding is only significant under high load and temperature\textsuperscript{15}. In such small grains the usual trend may not hold. Relationships established for more moderate grain sizes sometimes fail when grains are either extremely fine \textsuperscript{27}. Furthermore creep rate ($\dot{\varepsilon}$) temperature ($T$), and grain size ($q$) have the proportional relationship\textsuperscript{15}

\[ \dot{\varepsilon} \propto \frac{1}{d^q T} \quad (4) \]

where $q$ is a constant equal to 2 or 3 for grain boundary sliding. Rearranging the expression reveals that as the grain size decreases, the temperature required for the same strain rate rapidly decreases.

During low temperature static tensile tests, dislocation motion is the dominant mode of deformation\textsuperscript{27}. Dislocation motion in extremely fine grains may be hindered to the point that it no longer sufficiently disperses energy. Grain boundary sliding may become important even at room temperature after 3D printing because the microstructure is so much finer than observed after conventional processing. The UTS would not increase in that case because grain boundary sliding largely bypasses dislocation motion.
4.6. Effect of orientation on fatigue strength

Like tensile strength, fatigue strength depends on build orientation. The $60^\circ$ build orientation had the highest fatigue strength. Machine issues occurred during each of the trials that broke. Data for failed $60^\circ$ specimens may not be reliable, but the two survivors suggest significantly higher fatigue strength. It is unlikely that grain size was responsible for the differences in fatigue strength.

At all but one build angle 3D printed specimens had higher fatigue strengths than the wrought specimens. Other studies report that 3D printed specimens have a lower fatigue limit than their wrought counterparts\textsuperscript{12,17}.

As built 3D printed components have high surface roughness which greatly reduces fatigue life\textsuperscript{17}. Machining away the surface does increase fatigue strength, but internal porosity becomes external porosity and the strength of wrought specimens is not expected reached\textsuperscript{17}.

In the present study, the rough surface was machined away. It is not specified whether the specimens were as built or machined in Song \textit{et al}\textsuperscript{12}. In Mower and Long\textsuperscript{17}, machining did improve fatigue life but 3D printed specimens still performed worse than wrought specimens. Abstetar\textsuperscript{35} tested 3D printed aluminum after machining and still found the fatigue strength was much lower than for wrought. It may be that the porosity was sufficiently low that it did not negatively impact fatigue strength as much as in other studies. Alternatively, the specifications for the printer and the machinists may have coincidentally yielded an optimum surface roughness that was not reached in other studies.

Only the $90^\circ$ orientation had a lower fatigue limit than the wrought. Interfaces between layers are potential weak points in the metal. As mentioned earlier, discontinuities may concentrate at the substrate (previously melted layer)\textsuperscript{21,31}. The plane of stress was parallel to the
weakest part of the specimen. Poor interfacial bonding and the potential concentration of discontinuities both make layer interfaces potential weak points in the component.

### 4.7. Tensile Fracture

Each tensile specimen failed by ductile fracture. Quasi-cleavage planes on the 0° and 60° build angle fracture surface indicate weak binding sites. Such features are possible crack initiation sites. Brittle fracture occurred at the feature. The honeycomb structure then appeared as ductile fracture began. Both specimens failed by initial brittle fracture followed by transformation into ductile fracture.

Crack bridging is evident as the root cause of failure in the 90° build angle. Cracks are seen traveling between craters. Stress concentrates in material between nearby discontinuities. Cracks can easily initiate in such areas and grow between the discontinuities.

Each tensile fracture surface contained deep craters. Some regions of the microstructure contained clusters of long, highly aligned columnar dendrites. The craters may have been caused by such clusters pulling out of the surface. In that case, though, clusters of dendrites should be visible jutting out from other regions of the surface.

Alternatively the craters could be denudation zones. Spattering raises the edges of denudation zones, which may block powders from subsequent layers from filling the area. Deep pits may form because little powder entered the zone for several layers. However discontinuities were not found in the microstructural analysis in sufficient concentration to account for the number of craters.

Plastic deformation causes craters to elongate along with the bulk metal. Rather than having a large particle pull out, a small particle pull-out may have occurred. The appearance of
the craters would result from extensive plastic deformation. Unmelted powder particles, which can be extremely small and difficult to locate, may have provided nucleation sites for cratering.

### 4.8. Fatigue Fracture

Faceted voids were found within the final fracture zone. Faceted voids were not noticeable in the crack propagation zone. One possible explanation was that ductile fracture occurs in the propagation zone. Ductile flow may cause some metal to smear over and hide faceted voids. The final fracture zone fails from brittle overload. Faceted voids were not hidden in the final fracture zone because of the lack of plastic deformation during brittle fracture. Tearing in the final fracture zone may also have opened the faceted voids.

All 3D-printed fracture surfaces except the 90° build angle specimen showed secondary cracking. Most secondary cracking appeared to follow MPBs. In most orientations gravity pulls the melt pools parallel to the cross-section so that they overlap. In the 90°, gravity acted perpendicularly to the cross-section, so MPBs did not overlap. Weak bonding at MPB overlap sites may have created a path for secondary cracking which was not present in the discrete MPBs of the 90°.

#### 4.8.1. 0° orientation

A cluster of pores on the edge was determined to be the root cause of failure in the 0° build angle surface. Linear macroscopic features and fatigue striations both radiate from the cluster. Much more secondary cracking was present in the final fracture zone than for other build angles. Large voids with curved overhangs are present in the final fracture zone. Overhangs had curvature like MPBs. One possibility is that during final fracture some melt pools began to separate from the surface and pull out with the opposite surface.
4.8.2. 30° orientation

The main crack initiated at a faceted void. A rough surface in the void suggests an unmelted particle was present and was either pulled out or fell off the surface after fracture. Less heat is input near the edge than in the center because the powder not scanned acts as a heat sink. If faceted voids are going to form, they will be near the edge of the sample.

Secondary cracks in the final fracture zone had a slight curvature, similar to MPBs. Preexisting cracks had been observed near MPBs in some micrographs. Rather than delamination of layers, it is possible that preexisting cracks grew between MPBs. Once the crack actually reached an MPB the melt pools delaminated from the rest of the structure, causing the observed secondary cracking.

4.8.3. 60° orientation

Striations traveled in various directions. Several potential crack initiation zones were present in one small area of the fracture surface. A subsurface void seems to have been the primary crack initiation site. Following the striations shows that multiple cracks converged at a junction between two interior faceted voids. Beyond that point all striations and therefore the crack traveled in a single direction.

4.8.4. 90° orientation

Two sets of radial features traveling perpendicular to each other were present in the crack propagation zone. Fainter features filling the entire frame area are thought to be striations. The clearer features are thought to be slip bands\textsuperscript{36}. In ductile materials extensive plastic deformation precedes crack initiation and crack growth\textsuperscript{27}. Slip lines and ductile deformation are both consequences of dislocation motion. Slip lines grow perpendicularly to the crack\textsuperscript{36}. 
Unlike in the other orientations, secondary cracking was not present. Layers in the 90° build angles are parallel to the plane of stress. If secondary cracking was a consequence of delamination, then it would not appear in the 90° orientation.

Delamination of the MPBs would also be avoided because of the shape of MPBs. Melt pool boundaries are deformed hemispheres in shape. In the 0°, 30°, and 60° builds the melt pools are the rounded bottoms or sides of the MPBs as depicted in Figure 36. Stress concentrators are present at the vertices of each MPB giving a path for secondary crack growth. In the 90°, MPBs are nearly absent as the flat, elliptical surface is parallel to the plane of stress (Figure 39).

![Figure 39: Sketches of an MPB a) from the side and b) from the top](image)

Fatigue striations were most clearly seen in the 90° build angle specimen. Cyclic stress causes striations. In metals, ductile deformation occurs in the propagation zone. Striations show more clearly because of the permanent deformation\textsuperscript{15}. More brittle materials have less defined striations than ductile materials because fracture is abrupt without any permanent deformation. Striations were more prominent in the 90° specimen because it was the most ductile.

Studies on Inconel 718 suggest that striations only appear at rapid crack growth rates\textsuperscript{37}. Compared to the other specimens the 90° failed after relatively few cycles, indicating faster crack growth rates. Striations may have appeared in the 90° and not the other orientations because cracks in the 90° grew more rapidly. In the other orientations, striations may have been fainter because the crack grew much less rapidly.
5. Conclusions

- Build angle orientation had only a small effect on mean grain size, but did affect the grain size distribution. Specimens built at 0° and 90° had more and larger outliers, but the grain size was more concentrated around the mode. Specimens built at 30° and 60° had similar distributions with smaller outliers, but more grains were larger than the mode. Based on the data gathered, the layered structure was the most significant factor in the difference in strength between build orientations.

- Grain size did not appear to be responsible for the differences in strength between build orientations. Grain size changed little with orientation. The 60° build angle specimens had larger grains but higher strength than the 30° build angle specimens.

- Tensile test results had low standard deviations, indicating that 3D printed specimens are as reliable as wrought with higher yield strengths. The UTS is lower for printed specimens; strain hardening is insignificant. With the yield strength already higher than the wrought counterpart, and net shape forming being one of the goals, the low degree of strain hardening is not necessarily an issue.

- Excessive scatter was present in the fatigue data. Specific fatigue strengths could not be determined with the number of specimens available, but the 0°, 30°, and 60° orientations all gave higher fatigue limits than the wrought. Even at the lowest point in the curve these three performed better than the wrought. Specific values were not available and only a range of values could be drawn. More specimens are required for testing to provide a fully defined S/N curve. At all stresses the 90° failed at lower than the wrought.
• The 90° build angle had both the lowest YS and the lowest fatigue strength. It is most likely because the plane of stress is parallel to the layered structure. Extreme elongation in the 90° orientation may have been due to sequential delamination in multiple layers.

• Tensile failure was dominated by ductile fracture in all specimens. Cracks following melt pool boundaries, craters, balling, and unmelted particles were evident in many of the fracture surfaces. Planar or quasi-cleavage features are present. Any of the listed features may have acted as a crack initiation zone.

• Fatigue failure in specimens selected for fractography initiated at either pores or voids in every case. In two specimens voids looked as though they held unmelted particles. Some secondary cracks grew along melt pool boundaries. Other observed cracks may have been preexisting thermal cracks or solidification cracks.

• Changing parameters often led to conflicting results. Interactions between different parameters are suspected based on the inconsistencies observed. Porosity measurements and dendrite orientation in particular were inconsistent when considering individual parameters.
6. Recommendations

- A method for evaluating discontinuity volume may reveal the cause for differences in mechanical properties.

- More detailed grain size analysis is needed. A heterogeneous microstructure was present, so the average grain size of each type of structure is needed. Statistics software is currently being used to determine pdfs and statistics for various grain size measurements and shape factors.

- Further fatigue testing with sufficient numbers of specimens to complete a statistically viable S/N study is required. Such high scatter is present that methods typically reserved for ceramics and brittle materials may be needed. Statistical methods such as Weibull analysis are suggested.

- Mechanical testing is currently underway for specimens built at the two different GED values determined from microstructure.

- Results suggest laser power/travel speed and laser power/hatch spacing interactions occur. Further analysis to investigate the effect of interactions is recommended.
7. References Cited


[21] Investigation of crystal growth mechanism during selective laser melting and mechanical property characterization of 316L stainless steel parts.


doi:10.1016/j.jmatprotec.2014.06.001.


8. Appendix A: Histograms of Microstructure with Orientation

Figure 40: Length of a) 0 b) 30° c) 60° d) 90°

Figure 41: Histogram of Compactness a) 0 b) 30°
Figure 41: Compactness of a) 0° b) 30° c) 60° d) 90°
9. Appendix B: Variation of microstructure with GED

![Histogram of Compactness](image)

- **a)**
- **b)**
- **c)**
- **d)**
- **e)**
- **f)**
Figure 42: Compactness of a) P-1 b) P-2 c) P-3 d) P-4 e) P-5 f) P-6 g) P-8 h) P-9
Figure 43: Length of a) P-1 b) P-2 c) P-3 d) P-4 e) P-5 f) P-6 g) P-8 h) P-9
SIGNATURE PAGE

This is to certify that the thesis prepared by Penn Rawn entitled “3D PRINTING OF 316L STAINLESS STEEL AND ITS EFFECT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES” has been examined and approved for acceptance by the Department of Metallurgical and Materials Engineering, Montana Tech of The University of Montana, on this 18th day of October, 2017.

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