Effect of Laser Processing Parameters on Powder Bed Fusion Stainless Steel

By

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Abstract

Stainless steel has been used in the army for a wide variety of applications due to its high strength and durability. Additive manufacturing can be used to create parts at the point of need reducing the time and cost to repair critical equipment. However, due to field limitations post processing the as-built parts are difficult. This limits the ability to improve mechanical properties by traditional heat treating methods. During the additive manufacturing process material undergoes rapid solidification resulting in different material properties than that found using traditional manufacturing techniques. By having an open access system we are able to take advantage of this high cooling rate by manipulate the thermal history through manufacturing processing parameters. In this work the influence of laser scan strategy, part geometry, and laser energy density were examined experimentally. These processing parameters were measured for its effect on defect formation, microstructure, and mechanical properties. Based on the experimental results phase composition and grain size can be locally altered to a certain extent through manipulating the thermal history by creating regions with differing cooling rates.
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Chapter I: Introduction

1.1. Project Needs

For the army additive manufacturing provides ability to repair and replace existing parts in theater. Due to the remote field locations, long transportation time, and the sole source manufacturing of equipment it can be very costly and timely in order to replace critical equipment. The use of additive manufacturing can help overcome this challenge and produce parts “on-demand”.

It is important to note that the replacement parts do not need to meet original equipment manufacturers standards. Such as the use of a spare tire on a car, the additive manufactured components do need to have a predictable lifetime in order to complete the mission or remain operational until the appropriate replacement can arrive. This requires an understanding of the manufacturing process and the resulting properties. It is known that as built parts made from metal additive manufacturing techniques do not have the same mechanical properties as those made via traditional manufacturing. Unfortunately, the ability to post process in order to improve mechanical properties is difficult and time-consuming to do in theater. By having a better understanding of the influence of processing parameters on material properties, it is possible to customize critical parts with good enough properties for field use.

The high cooling rate experienced during the powder bed fusion (PBF) additive manufacturing process can create parts with nonuniform metastable microstructure and increased porosity compared to traditionally manufactured material. By understanding the process-property relation we will be able to better design and control the processing parameters in order to manipulate material properties such as porosity, strength and hardness. In this project, various process conditions including scan strategy, scan length of the laser, and powder bed energy density, have been studied to manipulate the thermal history within a part and to gauge their influence on microstructure and mechanical properties. The effect of flaw formation was also investigated and quantified.

1.2. Research Plan

The goal of this work is to evaluate the effect of various processing parameters on material and mechanical properties of powder bed fusion additive manufactured 17-4 stainless steel parts and propose how these parameters can be used in order to tailor properties for targeted applications.

The research plan is given in more detail in the following outline:

(1) Effect of Laser Scan Pattern on the Microstructure and Mechanical Properties (Published in Materials and Design 2017) and (2) Effect of Laser Scan Pattern and
Part Geometry on Mechanical Properties for Powder Bed Fusion of 17-4 Stainless Steel

PBF was used to fabricate 17-4 stainless steel tensile testing bars and compression samples based on ASTM standards. The thermal history was manipulated as a result of changing the laser scan path. Mechanical properties with tensile and compression testing and microhardness measurements. The effect of scan strategy on defect formation, material properties and mechanical properties were studied and discussed.

(3) Effect of Zone Creation to Locally Control Microstructure in Powder Bed Fusion 17-4 Stainless Steel

PBF was used to fabricate 17-4 stainless steel cylinders with regions containing different laser processing parameters. The effect of locally changing the thermal history was measured using micro hardness measures. The effect of local control on grain size and austenite retention was studied and discussed.

1.3. Thesis Organization

This thesis is organized into seven chapters. The first chapter is to familiarize the reader with the motivations and objectives driving this research. The second chapter is a literature review of relevant research relating to the PBF additive manufacturing process. Chapters III-V are papers that have been submitted or in the process of being submitted to journals as early outlined in the research plan. This thesis closes with conclusions (Chapter VI) and recommendations for future research (VII). Appendix A is a list of publications (accepted and in preparation) and conference presentations.
References

Chapter II: Literature Review

1. Introduction to Powder Bed Fusion

Powder Bed Fusion (PBF) is an additive manufacturing technique in which a specimen is created layer by layer using a computer controlled laser on a metal powder bed. A computer aided design (CAD) model of an object is sliced into layers that are sintered into a powder bed line by line to create a layer, Figure 1. The powder is recoated to create a new powder layer and the process repeats itself until the object is completed.

Figure 1: Schematic of experimental set up for selective laser melting.¹

Figure 2 schematically shows the generation of the melt pool in the powder bed caused from the interaction of the laser with the bed. The laser, with a Gaussian energy distribution, moves over the powder bed at a predetermined speed and power, creating arc shaped melt pools that resemble those found in welding. These welds have a heat-affected zone that melts the area around the beam causing the welds to be larger than the laser spot size. The depth of these welds needs to be deep enough to allow for remelting and bonding between layers. Energy from the interaction of the powder bed with the laser is absorbed into the powder while energy can be lost due to heat conducted into the powder bed and heat lost due to radiation and convection at the surface.
2. Laser Processing Parameters

2.1 Energy Density

The laser controlled parameters can be quantified into a powder bed energy density equation that relates the amount of energy stored in the powder bed. From Lakshminaryan’s dissertation on SLS of ceramic materials equation 2 shows the interactions between the laser power (P) and velocity (v) for the spot size (D) on the powder layer, called a fluence parameter (f).

\[
f = \frac{P}{D \times v} \quad (1)
\]

By taking into account that sintered lines would connect in order to form a fully bonded layer, Nelson et al. developed a relation, equation 3, for the overlap with spot size (D) and hatching spacing (HS). A schematic of this is show in Figure 3.

\[
O = \frac{D}{HS} \quad (2)
\]
Multiplying the overlapping equation and the fluence parameter the energy density \( (\Psi) \) equation is formed by relating the laser power, laser speed, and hatching distance.

\[
E = \frac{P}{v \times HS}
\]  
(3)

This equation is used by researchers as the main parameters varied in order to create processing maps to gauge the feasibility of materials for optimal surface quality. Other variations of this equation include the thickness of the powder layer.

2.2 Single Line Wetting Properties

A desired post melted surface for PBF is smooth, flat, and without pores, allowing for easy powder recoating and reducing delamination. The molten material for lines that exhibit these characteristics are considered wetted. Poor surface quality has a high roughness and the sintered lines create pores by the formation of liquid droplets, known as “balling” or “beading”, caused by the need to reduce surface energy. A comparison of which can be seen in Figure 4.\(^5\). The cause of irregularity in the sintered lines is that PBF is localized melting which causes a large temperature gradient in the melt pool, influencing surface tension gradients. Additives and impurities to the powder can also influence the surface tension allowing for balling to occur.\(^6\).
The difference between wetting and non-wetting systems is governed by the interfacial free energy equation.

\[ \gamma_{SV} - \gamma_{SL} = \gamma_{LV} \cos(\theta) \]  

(4)

The molten metal is considered wet when the \( \cos(\theta) \rightarrow 1 \) or if \( \gamma_{SV} - \gamma_{SL} > \gamma_{LV} \) which is favorable to spreading. In 1997, Schiaffino and Sonin tested the stability of molten wax on Plexiglas and found that molten material is stable when the contact angle is less than \( \frac{1}{2} \pi \) and unstable when larger than \( \frac{1}{2} \pi \). The instability of the material leads the molten material to ball, breaking up the sintered track. There are several forces that reduce the metal’s ability to wet such as Rayleigh instability and Marangoni convection. Balling prevents a good recoating of the next powder layer leading to increased porosity, poor interlayer bonding, and potential delamination of layers.

Rayleigh instability is when a liquid breaks up into droplets as a result of surface tension, as seen in Figure 5. As the droplets break up the liquid pinches and can create necking in order to minimize surface area. The larger the viscosity of the melt, the greater the space between droplets and potentially larger droplet size.
Figure 5 Artistic rendition of how Rayleigh instability occurs\textsuperscript{10}.

Marangoni convection is the thermocapillary flow of fluid from areas with low surface tension to areas with high surface tension\textsuperscript{11}. This gradient determines the direction of fluid flow, as shown in Figure 6. If the gradient is negative, like in most metal alloys, the flow is outward. Certain impurities, such as oxygen in iron, can make the gradient positive. This causes the flow to be inward, producing a larger surface tension and results in balling.\textsuperscript{9}.

Figure 6 Marangoni convection in melt due to surface tension gradient.\textsuperscript{9}.

2.3 Feasibility of Material

The energy density of the laser parameters affects the powder melt pool, influencing the surface tension and wetting properties. The energy density equation can be amplified by increasing laser power and decreasing scan speed and hatching spacing. Materials for use PBF in initially start with a processing maps to see how the material melts and provides a good surface for the layer wise process. This is done by varying the parameters used in the energy density equation in order to optimize for desired surface quality starting with speed and power.
Increasing the laser scanning speeds can induce thermocapillary irregularities such as satellite particles and necking. This is due to the heat received by the powder particles from the laser is decreased with an increase in scanning speed, Figure 7. With an increase in the laser scan speed decreases the energy density applied to the powder, increasing the amount of instability in the molten liquid. This can cause splashing in the metal increasing surface roughness in the continuous lines. These sintered tracks can be classified as continuous, irregular, and balling, Figure 8. At low scanning speeds and high laser power the molten pool created can overheat and the sintered tracks become unstable and exhibit irregularity.
Figure 8 Track shapes in continuous sintered lines in a CoCrMo powder material using a Nd:YAG laser. a. continuous; b. irregular; c. cracking and balling defect and a schematic of what is occurring as the in the molten sintering track as the laser scan speed was increased while the laser power remained constant.\textsuperscript{14,15}

Balling can also be induced by the laser power. If the power is not great enough the energy density will be too low to induce enough liquid phase sintering to allow for bonding between powders. Gu et al. investigated balling phenomena in stainless steel powders by varying the laser power and velocity, Figure 9.\textsuperscript{15} It was found that when the power was too low for the laser velocity this type of balling would occur. The partial melting created coarsened balls of agglomerated powder, but no continuous melt. When the power level was increased there would be enough liquid melt to overcome the surface tension and allow for spreading creating necking, bonding powders allowing for a continuous melt.
Figure 9 SEM image of laser sintered tracks using a CO2 laser at 316L stainless steel at a laser velocity of .04 m/s showing balling caused by lack of liquid formation: a. P-350W; b. P-400W.  

Powder layer thickness also plays a crucial role in the stability of sintered lines. The energy density must be great enough to penetrate to the next layer to bond. Yadroitsev examined the influence the powder layer thickness had on single track formation with optical results shown in Figure 10. When the thickness was too great there is not enough energy to produce a continuous melt and causes balling by too low of a laser scan speed. This was done to find the critical thickness boundaries in which continuous stable sintered lines could be formed without irregularity or balling.

Figure 10 Effect of 316L stainless steel powder thickness layer on balling caused by increasing scanning speeds. Where laser power is 50 W.
A feasibility test uses only the laser power and scanning speeds to make individual melt lines. This is done to find where the single scan lines show the most ideal wetting properties, as shown in Figure 11. The results of the varying processing parameters are categorized as no melting, low melt, partial melting, melting with balling, melting with breaking, and the desired continuous melt. An example of this processing map can be seen in Figure 12.

Figure 11 An example of how increased laser scan speeds can cause balling in metal powder.
2.4 Multi-Layer Laser Processing Parameters
2.4.1 Effect of Laser Processing Parameters

The laser power and laser scan speed control the width of the melt pool and consequently the width of the final sintered line. In order to make a complete surface layer for PBF the hatching distance, distance between sintered lines, must be controlled so that there is overlap and bonding between lines. If the hatching distance is too large pores will occur because the sintered lines do not bond. Figure 13 shows varying hatching distances of 316L stainless steel. When the hatching distance is below 0.3mm the sintered lines are close enough leaving a smooth surface that is fully dense. As the hatching distance increases pores form when the sintered lines solidify and the previous layer is visible revealing the alternating hatching pattern.
The irregularity caused by increasing the laser velocity also increases the porosity as more layers occur. Li et al. investigated the balling behavior of PBF on 316L stainless steel by varying the laser scanning speed and keeping the laser power and hatching distance constant, Figure 14. At 50 mm/s the sintered lines were continuous and overlapping is smooth allowing for easy powder re-layering, continuous melted lines. By increasing the scanning speed to 400 mm/s splashing caused satellite particles which increased surface roughness, irregular. As the speed changes to 600 mm/s and greater balling and disconnecting of sintered lines occurred. The laser speed affects the size, shape, and wetting of the melt pool. By reducing the speed pores can occur between lines. As the layers are added this porosity is compounded and can create open spaces in a vertical direction. This can lead to delamination between layers due to the lack of sintered material for the layer to bond, Figure 15. This inconsistency occurring in a three dimensional layered process can cause structural weakness and failure in the final product, Figure 16. The problem can be limited by having an alternating layer scan strategy which would reduce the ability for vertical pores to occur because the laser scan lines would not transpire in the same place each layer.
Figure 14 SEM image showing balling of PBF 316L stainless steel induced by increasing scanning speed: a 50 mm/s; b 400 mm/s; c 600 mm/s; d 800 mm/s at a laser power of 190W and hatching distance of 0.15 mm. 16
Figure 15 SEM and schematic of the effect of scan speed on porosity through layers during additive manufacturing. 316L stainless steel powder at 100W, hatching distance of 0.1mm and layer thickness of 0.06mm. A. 900mms\(^{-1}\); b. 120 mms\(^{-1}\); c. 150 mms\(^{-1}\); d. 180mms\(^{-1}\).\(^{18}\)

Figure 16 Schematic showing the potential defects or failures caused by non-continuous lines.
2.4.2 Effect of Scan Strategy and Orientation

Since the process of SLM is localized melting and quick cooling, residual stresses can occur due to high temperature gradients. This can be further increased with the layering used in SLM in which the top layer is being expanded due to laser interactions and then shrinking during cooling, as shown in Figure 17a. These residual stresses can cause delamination of layers and cracking which can cause part failure. In order to reduce part failure Kruth et al. investigated the effect of the laser scan length on internal stresses. It was found that by reducing the cooling time the temperature gradient would decrease allowing for better wetting conditions, Figure 17b.19

Figure 17 A. Effect of the temperature gradient and (B) scan length on PBF.19

Little research has been done on the effect of laser scan strategy, i.e. the specific path the laser traverses on the powder bed to build the part, on the mechanical properties and microstructure of AM parts. Most research focuses on other laser processing parameters due to the difficulty of adjusting scan strategy parameters with commercial PBF equipment.20,21 Previous studies on laser patterns focused on how these strategies effected the dimensional accuracy and density of the final part.20,21,22 Most scan strategies are like in Figure 18b in that the laser moves as a continuous line. Other scan strategies exist such as the alternate hatching pattern, Figure 18a. When used on iron powder the alternate hatching pattern was found to have a greater hardness than the line hatching pattern, but was overall found to be inefficient because it would increase build time immensely without increasing density.23

Figure 18 Schematic showing alternating and line hatching patterns.23
Line hatching patterns can also alternate in the x and y direction for each layer, Figure 19. This can prevent anisotropy in the melt layers which can lead to failure caused by directionality in the solidification. This type of hatching pattern can also increase the hatching density of the final specimen.24

Figure 19 Schematic of hatching patterns investigating the effect hatch length on density.24

For the metal additive manufacturing process columnar grains are common due to the strong thermal gradient between the build plate and the melt pool at the top most layer of the part. A deposition strategy created by a cross directional pattern that rotates 90 degrees every layer can reduce the formation of dendrites in the build direction.25 This effect was found to be reduced with an increase in energy density.26 For iron powder in PBF process, the cross directional pattern had an increased density over the singular direction strategy in the x and y direction. However, density decreased as the aspect ratio of the area increased.27

The effect of build direction on the mechanical properties has also been investigated. It was found that for specimens orientated flat on the plate versus orientated at 90 degrees had no change in mechanical properties, however the surface roughness increased for the top of the specimen with the change in orientation.28 However, studies have shown that 17-4 stainless steel specimens had lower elongation properties due to defects created by the layering process.29,30
References


Chapter III: Effect of Scan Pattern on the Microstructure and Mechanical Properties of Powder Bed Fusion Additive Manufactured 17-4 Stainless Steel

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**Highlights**

1. The length and direction of the raster pattern, at constant energy density was shown to have a strong influence on the cooling rate and resulting microstructure and defects within a final part.
2. Scan lines oriented along the loading direction resulted in brittle fracture orientated in the build direction due to the increased length in scan line and the laser scanning patterns being identical for each build layer.
3. A large variation in elongation from 3.3 to 17.6% was observed based on laser scan strategy.
Effect of Scan Pattern on the Microstructure and Mechanical Properties of Powder Bed Fusion Additive Manufactured 17-4 Stainless Steel

Abstract

Additive manufacturing (AM) of metallic parts is generating significant interest due to the ability to produce complex parts in a short period of time with minimal finishing required. However, the effect of laser scan strategy on the properties of finished parts is not well understood. In this paper the effects of laser scan line strategy on the microstructure and mechanical properties of stainless steel produced using metal Powder Bed Fusion (PBF) AM were characterized. Microstructure and phase identification were measured using x-ray diffraction and quantitative optical microscopy which found that all samples had a dual phase austenite-ferrite composition. Shorter scan lines perpendicular to the load direction resulted in 25% retained austenite, while elongated scan lines parallel to the load direction more than doubled the amount of austenite retained. A change of direction within the scan line path resulted in increased delamination porosity along the melt pool boundary and changes in volume fraction of retained austenite. Fractography, revealed cracks that propagated along melt pool boundaries. Understanding the effect of strategy on the microstructure and mechanical properties allows the producer of AM parts to implement materials by design strategies.

Introduction

Powder Bed Fusion (PBF), as defined by ASTM International, is a layer wise additive manufacturing technique for fabricating metal parts by which thermal energy selectively fuses regions of a powder bed [Standard, ASTM "F2792. 2012"]). This technique uses a three-dimensional computer aided design (CAD) that is sliced and melted layer-by-layer in order to create a final part.

Previous PBF work, such as those performed by Gu et al. [2009], Zhou et al. [2015] and Jia et al. [2014], focused on studying the effect of the laser energy density by adjusting the laser processing parameters. The equation for volumetric laser energy density \( E \) (J/mm\(^3\)) is shown in equation 1, where \( P \) (W) is the laser power, \( v \) (mm/s) is the laser scanning speed, \( h \) (mm) is the hatch spacing—the distance between laser scan lines, and \( t \) (mm) is the powder layer height [Williams et al. 1998].

\[
E = \frac{P}{v \cdot h \cdot t}
\]  

(1)
The variables from equation 1 were used as the processing parameters to adjust for improving final part density. Irrinki et al. [2016] used this equation to study the effect of gas versus water atomized 17-4 PH powder in order to investigate reducing the processing costs of PBF by substituting the feedstock source. It was found that gas atomized specimens overall had superior densification (87% to 97%), elongation (7% to 23%), and ultimate tensile strength (470 MPa to 850 MPa) at 64 J/mm³. Water atomized specimens properties were almost comparable to that gas atomized when the powder bed energy density was increased to 104 J/mm³ [Irrinki et al. 2016]. The energy density equation is an easy way to quantify the amount of energy being applied to the powder bed, but the laser processing parameters are not interchangeable. Keeping the energy density constant and varying the laser processing parameters does not yield the same results. Gu et al. [2013] found that as the power (190-70W) and scanning speed (800-287mm/s) decreased, the porosity increased while the energy density was maintained at 61 J/mm³ [Gu et al. 2013].

The effect of build direction on the mechanical properties has also been investigated. It was found that for specimens orientated flat on the plate versus orientated at 90 degrees had no change in mechanical properties, however the surface roughness increased for the top of the specimen with the change in orientation [Delgado et al. 2012]. However, studies have shown that 17-4 stainless steel specimens had lower elongation properties due to defects created by the layering process [Yadollahi et al. 2017 and Mower et al. 2016].

Little research has been done on the effect of laser scan strategy, i.e. the specific path the laser traverses on the powder bed to build the part, on the mechanical properties and microstructure of AM parts. Most research focuses on other laser processing parameters due to the difficulty of adjusting scan strategy parameters with commercial PBF equipment [Carter et. al 2014 and Kamath et al. 2014]. Previous studies on laser patterns focused on how these strategies effected the dimensional accuracy and density of the final part [Yadroitsev et al. 2010, Yadroitsev et al. 2007, and Kruth et al. 2010]. Samples produced using alternating pulsed scanning strategies were investigated as an alternate to continuous scan lines on powder and were found to have a greater hardness. However, the pulsed scanning strategies were found to be inefficient due to the strategy increasing the build time without increasing density [Rombouts 2004].

For the metal additive manufacturing process columnar grains are common due to the strong thermal gradient between the build plate and the melt pool at the top most layer of the part. A deposition strategy created by a cross directional pattern that rotates 90 degrees every layer can reduce the formation of dendrites in the build direction [Liu et al. 2011]. This effect was found to be reduced with an increase in energy density [Parimi et al. 2014]. For iron powder in PBF process, the cross directional pattern had an increased density over the singular direction strategy in the x and y direction. However, density decreased as the aspect ratio of the area increased [Simchi et al. 2003].

These previous studies show that laser processing conditions can be used to control melt pool properties and can be adjusted to better control final part properties. The laser scanning pattern can influence part porosity and microstructure due to changing the thermal history of each layer without having to adjust laser processing parameters. In this study the influence of laser scan strategy on AM 17-4 stainless steel is studied with regard to the influence of raster directionality
and length on tensile properties. The surface roughness, microstructure, hardness, mechanical properties and fracture mechanisms were examined for the effect of the scanning pattern.

**Materials and Methods**

17-4 is a martensitic precipitation hardened (PH) stainless steel and was chosen due to its resistance to corrosion up to 300\(^\circ\)C and tailorable strengthening by copper precipitates. The excellent weldability of 17-4 PH stainless steel and its ability to be heat treated to improve mechanical properties make it a good material choice for PBF.

Stainless steel 17-4 PH powder feedstock from 3D Systems (Rock Hill, SC, USA) was used in this study. The particle size distribution was measured via laser diffraction analysis using a Beckman-Coulter LS13 320. It was found that the average particle size was 14.5 \(\mu\)m, the \(D_{10}\) was 3.28 \(\mu\)m, and the \(D_{90}\) was 30.14 \(\mu\)m. The particle size distribution is shown in Figure 20A. In Figure 20B, the 17-4 PH powder has a roughly spherical morphology as imaged by scanning electron microscopy (SEM) in secondary electron (SE) mode. The composition of the 17-4 PH feedstock powder is provided in Table 1.

![Image of particle size distribution and SEM micrograph.](image)

**Table 1** Chemical composition of 17-4PH powder feedstock [Washko and Aggen 1990].

<table>
<thead>
<tr>
<th>Composition (wt. %)</th>
<th>Fe</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Si</th>
<th>Mn</th>
<th>Nb</th>
</tr>
</thead>
</table>
ASTM E-8 rectangular tensile specimens were additively manufactured on a 3D Systems ProX300 in a nitrogen atmosphere. The specimens were oriented parallel to the powder spreading direction and flat on the build plate (Figure 21). For this study, the laser energy density, Equation 1, was kept constant at 62.5 J/mm³. The laser power, scanning speed, hatch spacing, and layer thickness are given in Table 2. These parameters are the default processing parameters provided by the manufacturer for processing of 17-4PH and were used as a baseline. Six different laser scanning strategies were prescribed as shown schematically in Figure 22. Schematic representation of laser scan (x-y) strategies investigated in this paper. The following patterns were used, where the name in parentheses is the name which will be used to refer to that pattern throughout this paper:

![Schematic of laser scan strategies](image)

Figure 21 Orientation of tensile bars with respect to the build plate. Z-direction (normal to the page) corresponds to the build direction and the X-direction corresponds to the loading axis of the tensile specimens.
Table 2 Laser process parameters used in this study

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Laser Scanning Speed (mm/s)</th>
<th>Hatch Spacing (µm)</th>
<th>Powder Layer Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>1200</td>
<td>50</td>
<td>40</td>
</tr>
</tbody>
</table>

Figure 22 Schematic representation of laser scan (x-y) strategies investigated in this paper.

1. Hexagon pattern (“Hexagon”) which consists of 25,000 µm in diameter hexagonal islands with a 100 µm overlap of hexagons within the layer. The hexagons are fused at a 45° angle to the loading axis of the tensile specimens. From layer to layer, in the Z direction, the pattern angle alternates between 315° and 225°. The hexagon pattern is the default scan strategy for 3D Systems metal printers.

2. Concentric pattern (“Concentric”) which consists of successive outlines of the part. In this case the outer perimeter of the part is processed by the laser first, and the outlines move successively inward towards the center of the part.

3. 90 degree vertical hatch pattern one direction (90-BF-F). This pattern is uni-directional in the axis perpendicular to the loading axis of the tensile bar.
4. 90/270 degree vertical hatch pattern (90-BF-T). This pattern is bi-directional to the axis perpendicular to the loading axis of the tensile bar.

5. 0 degree horizontal hatch pattern (0-BF-F). This pattern is uni-directional to the axis parallel to the loading axis of the tensile bar.

6. 0/180 degree horizontal hatch pattern (0-BF-T). This pattern is bi-directional to the axis parallel to the loading axis of the tensile bar.

Although the energy density input to the powder bed is kept constant, the time to scan each layer varies from strategy to strategy as the total distance the laser travels varies due to the geometric constraints imposed by the specific scan strategy. The build time for a single specimen for each laser scan line strategy can be found in Table 3.

Table 3 Summary of time required to build a single part using each scan strategy

<table>
<thead>
<tr>
<th>Scanning Strategy</th>
<th>Scan Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexagon</td>
<td>40</td>
</tr>
<tr>
<td>Concentric</td>
<td>35</td>
</tr>
<tr>
<td>90-BF-F</td>
<td>48</td>
</tr>
<tr>
<td>90-BF-T</td>
<td>39</td>
</tr>
<tr>
<td>0-BF-F</td>
<td>47</td>
</tr>
<tr>
<td>0-BF-T</td>
<td>37</td>
</tr>
</tbody>
</table>

After the build was complete, the specimens were removed from the build plate via electrical discharge machining (EDM). Specimens from each scan line strategy were sectioned and mounted for metallographic preparation which included polishing. Specimens were etched with Murakami’s reagent at 80°C to color the ferrite phase [Voort and Manilova, 2005]. Vickers hardness was measured on a Tuckon 1202 using 1 mm x 1 mm grid mapping. Microstructure was examined with a Keyence VK X-200 confocal microscope. Density was measured using the Archimedes method. The size, shape and volume percent of pores were measured using optical microscopy and quantified with ImageJ software [Schneider et al. 2002]. Pores under 3 µm² were not considered for this work due to software limitations. Surface topography measurements of the as built specimens were measured at a 20x objective using laser profilometry with the Keyence confocal microscope. Tensile testing was conducted at room temperature on an Instron 1331 following ASTM E8M-09 standards using a constant displacement rate of 1.25 mm/min. Fractured tensile test specimens were examined using Hitachi S4700 Field Emission SEM for analysis of failure mechanisms. X-ray diffraction (XRD) was performed on both the powder and additive manufactured (AM) specimens using a Panalytical X’pert Pro MPD diffractometer using cobalt radiation. Quantitative XRD analysis was only possible on the as atomized 17-4 PH powder due to extensive texturing present on the AM parts. Rietveld whole pattern fitting using the Sonneveld and Visser background fitting was used to quantify the volume fraction of each phase observed in the powder [Sonneveld and Visser 1975]. This was then compared to the area fraction of each
phase observed in the AM parts which was determined using optical microscopy images and ImageJ.

Results and Discussion

Density, Porosity and Surface Roughness

Relative density was determined using the Archimedes method and compared to martensitic solution annealed wrought Condition A 17-4 stainless steel (7.80 g/cm³) to determine porosity. It is noted that the absolute density of the materials may vary due to variations in microstructure and not only porosity, e.g., ratio of austenite to martensite, and the choice of Condition A as reference density is chosen merely as a standard reference to facilitate relative comparisons. Results can be found in Table 4 [Washko and Aggen 1990]. There is not a strong change in the overall density observed by changing the scan strategy.

Representative defects that were found within the PBF AM parts are shown in Figure 23. These can be classified in general terms by their characteristic morphology and will be referred to in discussion as Type I, Type 2, and Type 3 porosity. Type I porosity is caused by entrapped gas, and are spherical due to the vapor pressure of the entrapped gas that can be influenced by powder packing density and the powder manufacturing process [Zhao et al. 2008]. Type II porosity is caused by various AM process related factors, examples of which include: cracking due to thermal stresses, incomplete melting of particles, and balling caused by the inability of the melt pool to overcome surface tension which creates incomplete coverage for the next powder layer [Gu et al. 2012]. This type of porosity is expected to have a more irregular shape than the entrapped gas pores and can occur from sub-micron to macroscopic in size [Sames et al. 2016]. Type III, lack of fusion porosity, is caused by the incomplete melting of the powder layer into the layer below leading to interlayer cracking causing elongated voids along the melt pool boundaries [King et al. 2014]. These voids are expected to be elongated along the melt pool boundary with a very low circularity. Voids within the AM part can have deleterious effect on the mechanical properties due to the ability to act as crack initiation sites [Yadollahi et al. 2017].
The area fraction of voids was determined by image analysis and is listed in Table 4. The scan strategy did not have a statistically significant effect on the overall void concentration, however different scan strategies had different distributions of void circularity and area. This result indicates that the scan strategy has a role in the type of defects produced but not the absolute concentration of voids. Figure 24A shows the normalized frequency of circularity (4π the ratio of the area to the squared perimeter of the void) of the voids for each of the scan strategies. The distribution is skewed towards voids having a circularity of 1 (perfectly circular) which indicates that the dominant defect is Type I porosity. Type II porosity is the second most prevalent and occurs over a large range (0.3 to 0.9) of circularities due to the irregularity of the shapes that can occur. Type III, with a circularity less than or equal to 0.3, is the least frequent type of porosity observed for all scan strategies. For the scan strategies orientated perpendicular to the loading direction (90-BF-F and 90-BF-T), there is an increased amount of voids that are larger in area (over 1000 µm²) compared to the other scan strategies. This is due to the orientation of the specimen being parallel to the scan strategy so an increased amount of Type III porosity along the layer boundaries is visible as opposed to viewing the surface perpendicular to the scan strategy. There is a decreased amount of voids due to entrapped gas in the bi-directional scan patterns.
Figure 24 (A) Histogram of the normalized frequency of the circularity of the porosity with one being perfectly circular; (B) Histogram of the normalized frequency of the void area.

Table 4 also shows the average surface roughness (Ra) and the average between the five highest peaks and lowest valleys (Rz) for each scan strategy. Representative surface roughness maps showing the specimen topology measured from the center of the gauge length can be seen in Figure 25. The bi-directional scan strategies did not appear to reduce the formation of balling and satellite particles caused by the surface temperature of the melt pool. The non-uniform surface roughness indicates that the scan strategy did not prevent splashing caused by the surface overheating [Simonelli et al. 2015]. Reducing this phenomenon would require a change to the linear energy density parameters (laser power and laser scanning speed) [Calignano et al. 2013]. Balling can be potentially detrimental to the recoating process causing poor powder coverage for the next layer and in severe cases cause failure of the build [Gong et al. 2014].
Figure 25 Surface topology of 17-4 PH tensile specimens: (A) Hexagon; (B) 90-BF-F; (C) 0-BF-F; (D) Concentric; (E) 90-BF-T; (F) 0-BF-T.

Table 4 Summary of results for Archimedes density, void concentration, surface roughness, and ten point average surface roughness.

<table>
<thead>
<tr>
<th>Scanning Strategy</th>
<th>Relative Density (%)</th>
<th>Void Concentration (%)</th>
<th>Ra (µm)</th>
<th>Rz (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexagon</td>
<td>98.9</td>
<td>0.3 ± 0.1</td>
<td>9.53</td>
<td>103.6</td>
</tr>
<tr>
<td>Concentric</td>
<td>98.2</td>
<td>0.3 ± 0.1</td>
<td>11.07</td>
<td>107.6</td>
</tr>
<tr>
<td>90-BF-F</td>
<td>98.5</td>
<td>0.2 ± 0.1</td>
<td>9.37</td>
<td>98.74</td>
</tr>
<tr>
<td>90-BF-T</td>
<td>98.8</td>
<td>0.4 ± 0.1</td>
<td>11.13</td>
<td>125.31</td>
</tr>
<tr>
<td>0-BF-F</td>
<td>98.7</td>
<td>0.3 ± 0.2</td>
<td>9.90</td>
<td>110.19</td>
</tr>
<tr>
<td>0-BF-T</td>
<td>98.7</td>
<td>0.4 ± 0.1</td>
<td>7.78</td>
<td>96.14</td>
</tr>
</tbody>
</table>

Microstructure

The concentric specimen has a valley through the center of the gauge length with roughly the depth of one powder layer caused by overlapping scan lines re-melting the center. Double melting creates a keyhole defect in the surface because the scan line area is not in plane with the rest of the surface due to the double melting of the specimen by decreasing the distance between melt pool boundaries [Yasa et al. 2011]. This double melting further consolidates the specimen, causing incomplete powder coverage within each layer due to the height discrepancy compared to the adjacent sintered lines [Li et al. 2012]. Since the laser scan strategy is the same for each layer, the self-closing mechanism that would correct this defect takes more than one layer, causing repeated porosity through the build height [Zhou et al. 2015].
Other valleys in the specimens follow the laser path direction indicating that valleys are influenced by the scan strategy. This can be caused by a less than fully dense powder coverage that allows for more consolidation of powder and an uneven surface [Gong et al. 2014].

Figure 26 shows the XRD spectrum of the 3D Systems 17-4 feedstock powder and of the different scan strategies in the build direction. The 17-4 powder has austenite (γ, FCC) and ferrite (α, BCC) microstructures. After AM, the austenite microstructure is retained, as seen in the γ, (111), peak, but the concentration of which is affected by the scan strategy choice. Strong texturing of both the austenite and ferrite phases was observed in the AM parts which prevented Rietveld phase analysis.

![XRD pattern](image)

Figure 26 XRD pattern of 17-4 PH samples with a cobalt source: (A) Hexagon; (B) 90-BF-F; (C) 0-BF-F; (D) Concentric; (E) 90-BF-T; (F) 0-BF-T.(G) 17-4 stainless steel powder feedstock.

AM samples were etched with Murakami’s reagent at 80°C which colors ferrite, but not austenite. Optical micrographs of etched AM parts for each scan strategy are presented in Figure 27. Micrographs are orientated perpendicular to the loading axis with the build direction indicated in Figure 27. The average austenite percent by volume was determined using image analysis, results can be found in Table 5. The distance in the build direction between the visible melt bands, examples of which are represented by the dotted lines in Figure 27, are not the same as that of the powder layer thickness, 40 µm, due to penetration [Lienert et al. 2011], i.e. the laser melting the
powder layer and into the layer below forming a new melt pool. Differences in distance between melt pools vary depending on homogeneity of the powder layer and the solidified layer underneath.

![Micrographs of etched AM parts with lines indicating melt pool boundaries.](image)

**Figure 27** Optical micrographs of etched AM parts with lines indicating melt pool boundaries. (A) Hexagon; (B) 90-BF-F; (C) 0-BF-F; (D) Concentric; (E) 90-BF-T; (F) 0-BF-T.

Table 5 Summary of results for percent of average austenite determined from image analysis for each scan strategy. (*) The volume fraction of austenite for the 17-4 PH powder was determined using quantitative XRD analysis.

<table>
<thead>
<tr>
<th>Scanning Strategy</th>
<th>Average Austenite (vol. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>17-4 PH powder</td>
<td>62.9*</td>
</tr>
<tr>
<td>Hexagon</td>
<td>58.3±2.1</td>
</tr>
<tr>
<td>Concentric Middle</td>
<td>82.4±5.3</td>
</tr>
<tr>
<td>Concentric Edges</td>
<td>50.3±7.6</td>
</tr>
<tr>
<td>90-BF-F</td>
<td>27.2±6.5</td>
</tr>
<tr>
<td>90-BF-T</td>
<td>25.5±2.6</td>
</tr>
<tr>
<td>0-BF-F</td>
<td>43.6±7.9</td>
</tr>
<tr>
<td>0-BF-T</td>
<td>69.9±9.7</td>
</tr>
</tbody>
</table>

The micrographs confirm that both austenite and ferrite are present. The lath-like martensitic phase was not visible; however, a third phase occasionally is found on the edge of the melt pool for all samples. This phase was too fine for compositional analysis via the SEM and will be the subject of future work. All samples exhibited texturing in the build direction which has been observed in previous published work of the same material type [Murr et al. 2012]. The high thermal conductivity in the build direction through the previous built layers allow for the continuation of grains orientated in the build direction by the partial re-melting of the layer below [Carter et al. 2014 and Thijs et al. 2010]. These elongated grains are positioned more towards the center of the
melt pool boundary and cut across multiple melt pool boundaries in the vertical direction. For the hexagon scan strategy these phases are broken up in places due to the cross sectional layer to layer scan pattern.

The 90-BF-F and 90-BF-T specimen micrographs show a single laser path line for each build layer. This orientation in comparison to the line direction reveals grain colonies on the order of one build layer in height that occur due to the overlap of scan lines. These colonies of grains are not as frequent with the uni-directional scan strategies, but may occur at the melt pool boundary.

For the concentric scan strategy, when the laser melt pool moves from parallel to perpendicular to the loading axis, the average austenite phase concentration increases from 50.3% to 82.4%. For this scan strategy the raster vector changes from parallel to perpendicular to the load direction causing a change in thermal gradient due to accumulated residual heat [Cheng et al. 2016]. This can cause delamination to occur along the melt bands when the laser path changes directions. The section of concentric raster pattern lines that are parallel to the loading axis has austenite concentrations similar to that found in the 0-BF-F and 0-BF-T samples. For scan strategies that are perpendicular to the load direction (90-BF-F and 90-BF-T), the austenite phase concentration is about a third of the concentric scan strategy in the same direction. For the 90-BF-F and 90-BF-T scan strategies, the melt pool moves from one side of the specimen to the other cooling as it goes. In contrast, the concentric scan strategy has a thermal gradient moving inwards for each layer. As a result, as the single line moves from parallel to perpendicular to the load direction, the melt pool line follows in the same direction and is now almost completely surrounded by the previously melted and now cooled solidified material. This solidified material acts as a heat sink for the melt pool allowing for the formation of more austenite than samples with similar scan strategies due to the bulk material having greater thermal conductivity then the powder bed [Montes et al. 2003].

**Mechanical Properties**

Hardness maps were generated on the cross section of the specimens to quantify the homogeneity of the properties. Vickers hardness measurements were taken using a 1 x 1 mm grid, and are presented in Figure 28. The average hardness of the entire cross section can be seen in Table 6. For 0-BF-F and 0-BF-T hardness increases as the laser moves from one end of the horizontal distance to the other for each layer. For the 90-BF-F and 90-BF-T the hardness maps shows a single scan line along the length of the horizontal distance repeated throughout the height of the build and is fairly uniform for each scan line, but decreases as the build height increases. The concentric hardness decreased to 305 HV towards the center of the specimen. This follows the change in scan strategy where the scan strategy turns from parallel to the loading axis to perpendicular to the loading axis. This decrease in hardness can be attributed to a change in phase composition and an increase in porosity caused by the mid line change in direction of the scan strategy.
Figure 28 Vickers hardness maps of cross section of the grip perpendicular to the load direction of the tensile specimens. (A) Hexagon; (B) 90-BF-F; (C) 0-BF-F; (D) Concentric; (E) 90-BF-T; (F) 0-BF-T. The build direction is parallel to the z direction.

Table 6 Mechanical properties for each scan strategy

<table>
<thead>
<tr>
<th>Scanning Strategy</th>
<th>Avg. (HV)</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Strength (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexagon</td>
<td>346.3 ± 13.8</td>
<td>798 ± 21</td>
<td>1101 ± 27</td>
<td>15.8 ± 3.5</td>
</tr>
<tr>
<td>Concentric</td>
<td>356.1 ± 12.8</td>
<td>824±30</td>
<td>916 ± 59</td>
<td>4.2 ± 2.4</td>
</tr>
<tr>
<td>90-BF-F</td>
<td>350.2±11.7</td>
<td>810 ± 15</td>
<td>948 ± 36</td>
<td>4.8 ± 1.1</td>
</tr>
<tr>
<td>90-BF-T</td>
<td>355.3 ± 19.8</td>
<td>773 ± 3</td>
<td>1043 ± 2</td>
<td>17.6 ± 2.6</td>
</tr>
<tr>
<td>0-BF-F</td>
<td>346.7 ± 22.4</td>
<td>873 ± 10</td>
<td>951 ± 17</td>
<td>5.3 ± 2.7</td>
</tr>
<tr>
<td>0-BF-T</td>
<td>350.3 ± 18.8</td>
<td>866 ± 10</td>
<td>935 ± 8</td>
<td>3.3 ± 0.6</td>
</tr>
</tbody>
</table>

An engineering stress-strain curve for each scan strategy is presented in Figure 29 and the average yield strength (YS), ultimate tensile strength (UTS), and elongation are presented in Table 6. All scan strategies exhibited similar elastic behavior. Uni-directionality does not appear to affect yield strength with 0-BF-F and 90BF-F exhibiting yield strengths similar to their respective bi-directional compliments. The laser scanning strategy does affect plastic deformation with the average ultimate strength ranging from 916-1043 MPa and elongation ranging from 3.3 - 17.6 %.
The hexagon and 90-BF-T scan strategy demonstrated defined yielding behavior, with the hexagon strategy exhibiting necking.

Figure 29 Tensile engineering stress-strain curves of 17-4 stainless steel AM samples for each scan strategy.

**Fracture Mechanisms**

The hexagon scan strategy was oriented 45° to the tensile load direction, had a lower yield strength, the highest UTS, and high elongation. Figure 30 A is a SE micrograph of the fracture surface of the hexagon specimen which exhibited a highly ductile failure. The 0-BF-F, concentric, and 0-BF-T scan strategies had laser scan lines parallel to the load direction, exhibited the highest YS, moderate UTS but poor ductility. The fracture surfaces for these were examined in SEM in SE mode and are provided in Figure 30 C, 11 D and 11 F respectively. Both the 90-BF-F and 90-BF-T scan strategy had weld lines perpendicular to the load direction. However, the 90-BF-T exhibited a much higher strength and elongation than the 90-BF-F specimen. The 90-BF-F fraction of cleavage failure is also much higher than the 90-BF-T (see Figure 30 B and 11 E, respectively). But, the microstructure of the two specimens (see Figure 27 B and 8 E), including the volume fraction of austenite, are very similar. The difference in thermal history due to differing scan
strategies may affect the ultrafine microstructure in the melt pool. Additional investigation into this phenomena will be deferred to a later study.

Figure 30 SEM secondary electron micrographs of the fracture surfaces: (A) Hexagon; (B) 90-BF-F; (C) 0-BF-F; (D) Concentric; (E) 90-BF-T; (F) 0-BF-T. The build direction is from the bottom to the top in all images.

The fracture surfaces were examined using SEM to investigate the effect of scan strategy on failure mechanisms. All specimens fractured at roughly 45° angles with the load direction and with a mixture of ductile and cleavage failure (quasi-cleavage failure). The quasi-cleavage failure is commonly found in complex steels which consist of a fine aggregates of multiple phases where one phase will fail by cleavage, such as the more brittle, BCC \( \alpha \)-Fe, and the other phase is ductile, such as the more ductile, FCC \( \gamma \)-Fe. A higher magnification SE micrograph illustrating the cleavage lines is provided in Figure 31. The specimens with greater than 10% elongation (Hexagon and 90-BF-T) had far less cleavage failure than their more brittle counterparts (90-BF-F, 0-BF-F, and 0-BF-T).
In the more brittle specimens, such as with the concentric scan strategy, a significant portion of the center of the fracture surface consists of flat cleavage-type failure that closely resembles the respective microstructure in Figure 30 D. The crack propagated in such a way that the weld lines became isolated during fracture. This indicates that the crack readily propagated through 1) the weld line overlap controlled by the hatch spacing, 2) the melt pool boundary, and 3) by cleavage of the ferrite. This failure can be reduced by having a cross directional non-repeatable scan strategy. This failure mechanism is illustrated schematically in Figure 32.
Figure 32 schematic of the crack propagation along the weld line and through the melt pool boundary. The hatch spacing, h, between the laser scan lines is 50 µm, and t is the theoretical powder layer thickness, 40 µm. The dotted lines represent the center of the laser on the melt pool of each scan line. The areas filled with a diagonal green lined pattern represents regions with ferrite phase present and the red line represents the crack propagation path. (A) Representation of scan line strategies that are parallel to the loading axis; (B) Representation of scan line strategies that are perpendicular to the loading axis.

The fracture surfaces in Figure 33 show some of the different types of defects, such as a lack of fusion between layers, entrapped gas, and scan strategy aligned porosity that can influence fracture mechanics. Figure 33 A shows lack of fusion between layers along the scan line for 0 BF-T where the scan line is visible. Inside the void is a mixture of partially melted powder and balling of various sizes attached to the scan line and the layer above it indicating poor melting in this area. Specimens manufactured using the 0-BF-T scan strategy had the longest raster length through the gauge length out of all the scan strategies used in this study. The increased length of the melt pool could induce Rayleigh instabilities and Marangoni forces which can cause improper wetting of the melt pool due to the inability of the melt to overcome surface tension [Attar et al. 2014 and Rombouts et al. 2006]. This can create scan strategy-induced porosity aligned in the loading axis of the specimen [Qiu et al. 2015]. The concentric and 0-BF-F scan strategy also have long continuous raster lines through the gauge and are expected to exhibit similar defects. Figure 33 B shows porosity caused by layer delamination and there is an unmelted particle as well as balling occurring within the void. Figure 33 C shows a nearly spherical pore caused by entrapped gas. Porosity in the part can act as easy propagation sites for brittle fracture. Some regions of
microplasticity, as seen in Figure 33 D, were observed in the parts with low elongation which indicate limited ductility.

Figure 33 characteristic fracture surfaces showing various manufacturing related defects and fracture surface characteristics: (A) delamination around a melt pool for 0-BF-T scan strategy; (B) porosity due to lack of fusion acting as a crack initiation site for 0-BF-F scan strategy; (C) ductile and brittle fracture surface with gaseous pore acting as brittle fracture initiation site for 90-BF-F (indicated by the white arrow); (D) regions of microplasticity on the fracture surface indicate regions of limited ductility for the 90-BF-T scan strategy. The build direction is from bottom to the top in all images.

Due to the nature of the concentric scan strategy, there are repeating voids corresponding with the layer thickness in the build direction, as shown in Figure 30 D. As discussed previously, this scan strategy has an almost complete overlap through the center of the sample; this double melting encourages porosity in this area throughout the build height. These voids occur at the periodicity of roughly one layer thickness, ~40 µm.

The alignment of melt pool boundary delamination porosity through the gauge section in the specimens with the concentric, 0-BF-F, and 0-BF-T scan strategy would be in the load direction. In contrast, the 90-BF-F and 90-BF-T melt pool boundary delamination porosity would be perpendicular to the load direction. These delaminations can act as stress concentration sites and potential crack initiation sites. Pores that are aligned perpendicular, especially Type III that are more elongated in the laser scan direction, are more prone to crack initiation than those that are parallel direction to the tensile load [Yadollahi et al. 2017]. Other porosity created during
manufacturing could also act as crack initiation sites decreasing ductility. The hexagon island cross hatching strategy reduced thermal stress and heat distortion due to reduced scan line length. The hexagon scan strategy can reduce the size of the pores due to the pattern changing layer to layer allowing for the self-closing mechanism to occur in fewer layers than the other scan strategies [Zhou et al. 2015]. 0-BF-F, and 0-BF-T scan strategies have long raster lines that go the length of the specimen causing a high layer thermal gradient in the load direction. For the 0-BF-F this long raster line increases the overall build time due to the laser having to move back before rastering another line, further increasing the residual stress for each layer. The 90-BF-T scan strategy has a shorter overall build time, compared to its uni-directional counterpart, from the lack of laser jump time due to the bi-directional movement. The reduction in laser path length, in comparison to the 0-BF-F and 0-BF-T strategies, decreases the thermal gradient within each layer, reducing residual thermal stresses and heat distortion [Kruth et al. 2004]. This is also exhibited with the hexagon scan strategy. This allows for reduced residual stresses throughout the samples, improving mechanical properties.

Conclusions

In this work the effect on microstructure and properties of additively manufactured 17-4 stainless steel as a function of laser scan path at constant energy density were evaluated. The length and direction of the raster pattern was shown to have a strong influence on the resulting microstructure and defects within a final part. The overall void concentration was roughly the same for all scan strategies but the concentration of each type of void present varies depending on specific scan strategy employed. Three types of defects were observed in each part: Type I which is entrapped gas, Type II or defects caused by the AM manufacturing process, and Type III which are voids caused by interlayer de-bonding. Type I was the most common type of void in general but was slightly less frequently observed in the bi-directional scan patterns. Scan strategies oriented perpendicular to the loading direction contained more Type III voids. Double-melting caused keyhole defects in the concentric scan from overlapping scan lines re-melting the center throughout the build height.

The uni or bi directionality of the scan strategy did not prevent the formation of balling or satellite particles. All samples exhibited these defects that can be detrimental to the build and potentially cause the formation of defects.

Austenite and ferrite were the primary phases observed in both XRD and optical microscopy. A third phase was observed occasionally at the edge of melt pool boundaries. Identifying this third phase will be the subject of future studies. The grains were textured along the build direction for all scan strategies except the hexagon scan strategy due to the repeating nature of the scan line strategy. These effects can be mitigated by a cross directional layer to layer scan strategy. Small grain colonies (~1 build layer thickness) formed due to scan layer overlap and were less frequently observed with the uni-direction scan strategies. The scan strategies oriented parallel to the loading axis had a higher concentration of austenite (43.6% 0-BF-F and 69.9% 0-BF-T) than their perpendicular counterparts (~25%). The concentric scan strategy had the highest concentration (82.4%) in areas with the laser scan direction perpendicular to the load direction and a moderate
concentration (50.3%) of austenite in areas with laser scan direction parallel load direction. The hexagon scan strategy also had a high concentration, 58.3%, of austenite.

The uni-or bi-directionality did not have a strong effect on the homogeneity of the micro hardness across the sample. With the hexagon scan strategy having the most uniform distribution of hardness across the sample. The 0-BF-F and 0-BF-T scan strategies had increasing hardness as each layer developed. The concentric scan strategy had a reduced hardness in the center of the specimen due to the increased amount of retained austenite as well as the deleterious effect of the delamination porosity caused by the change from parallel to perpendicular to the load.

In general, the uni- or bi-directionality of the scan strategy did not affect the yield strength of the part however, it may be a critical factor in determining the ultimate strength and elongation of the part. For example, both of the scan strategies oriented parallel to the loading direction had similar ultimate strengths and elongation but the 90-BF-T and 90-BF-F had very different mechanical properties. Considering that 90-BF-T and 90-BF-F have similar microstructures, failure is likely to be dominated by the types of voids observed in each. The concentric scan strategy parallel to the load direction had a higher yield, lower elongation and a moderate ultimate tensile strength. The part produced with the hexagon strategy exhibited necking, a lower yield, the highest ultimate tensile strength and a high elongation

All samples failed through a quasi-cleavage failure. Failure occurred through: 1) weld line overlap, 2) the melt pool boundary, and 3) cleavage of ferrite. Defects such as pores serve as crack initiation sites. Pores that are aligned perpendicular to the tensile load are more prone to crack initiation. Crack propagation along the melt pool boundaries and overlaps, controlled by the laser scan strategy and hatch spacing, and decreased the overall mechanical properties.

Most scan strategies move from one end of the specimen to the other. The concentric scan strategy is a good study of the changing scan strategy within a part and how this effects the resulting microstructure compared to those with similar raster pattern direction and length. The changes in raster length mid pattern in the concentric patterns may lead to changes in thermal gradients causing formation of defects along the melt pool boundary. This effect should be investigated further, including experimental quantification of the complex thermal gradients resulting from each scan strategy. This work serves as the basis to enable design of manufacturing strategies which manipulate process parameters to facilitate local control of microstructures and properties within complex AM parts. Additionally, the current state of the art in metal AM uses open loop control systems in which the process parameters are fixed during a build. The understanding of the relationships between process parameters and laser strategies and their role in defect formation is necessary for development of feedback or feed forward control algorithms which seek to optimize process parameters on the fly to mitigate the formation of defects, and optimize performance of complex AM parts.

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References


Chapter IV: Effect of Laser Scan Pattern and Part Geometry on Mechanical Properties for Powder Bed Fusion of 17-4 Stainless Steel.

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**Keywords:** Additive manufacturing; powder bed fusion; stainless steel; processing-microstructure-property relations; failure analysis

**Highlights**

- Hexagon scan strategy reduced texturing in the build direction due to cross directional layer to layer scan strategy
- Having an inward moving thermal history with a decreasing laser scan length and reducing the time between laser passes resulted in refined grains and decreased hardness.
Abstract:

Additive manufacturing has the ability to produce specimens with geometry that would not be able to be manufactured by traditional means. The pattern that the laser moves to fuse the powder layer has an effect on the mechanical properties and microstructure of the resulting part. The goal of this study is to determine the effect that scan strategy and specimen geometry has on compression properties, hardness and microstructure. Defect formation was examined using micro computed tomography and found that pores were aligned with the laser scan direction. Grain size was measured using electron beam scatter diffraction which found that by reducing the laser scan length and decreasing time between laser passes resulted in refined grains and decreased hardness.

Introduction:

Powder Bed Fusion (PBF) is an additive manufacturing (AM) technique in which a specimen is created layer by layer using a computer controlled laser to selectively fuse regions of a powder bed. A computer aided design (CAD) model of an object is sliced into layers that are sintered into a powder bed line by line to create a layer. The powder is recoated to create a new powder layer and the process repeats itself until the object is completed. This technique can be used to create near net shape parts that are not able to be created using traditional manufacturing processes. Previous studies on PBF processing parameters have studied the effect of laser processing parameters such as laser power and speed on defect formation and mechanical properties. Other variables that can be used to change material properties in PBF include inerting gas, powder properties, and laser scan path. For 17-4 stainless steel the shielding gas is important to final phase concentrations, Nitrogen greatly increases the retention of austenite compared to Argon due its influence on the martensite transition temperature. This is also true of the gas used for powder atomization with nitrogen gas atomized powders having a greater concentration of austenite in the final part than argon gas atomized powders.

Utilizing a smaller scan length resulted in smaller distortions in due to a decrease in local thermal gradient compared to a long scan strategy. However a decreased scan length can increase in shrinkage potentially increasing part porosity. As well as decrease the ratio of length to width of the melt pool. Previous studies to this research paper investigated the effect of scan pattern on tensile properties found that by manipulating the cooling rate by reducing the scan length with constant laser energy parameters influenced the defect formation and microstructure composition in 17-4 stainless steel.

17-4 stainless steel is an industrial material that is useful due to its resistance to corrosion up to 300°C. The excellent weldability of 17-4 makes it a good choice material candidate for the PBF process because both manufacturing techniques experience high cooling rates. For traditional manufacturing processes and heat treatment 17-4 has a precipitate hardened martensitic microstructure, however for most PBF processes the resulting microstructure is a duplex stainless
Heat treatment has been investigated for PBF 17-4. Yadollahi et al. found that strength increased with heat treatment compared to as built, but defects still occurred and had a deleterious effect on mechanical properties. Lebrun et al. found heat treated PBF samples had reduced strength compared to wrought material due to an increased amount of retained austenite. With heat treatment alone as built parts do not meet wrought manufactured 17-4 mechanical properties.

The aim of this study is to investigate the microstructure, defects and mechanical properties of 17-4 stainless steel fabricated with PBF with the change in laser scan pattern while keeping all other processing parameters constant. This study explores how the control of thermal history within a part can be used in order to tailor material and mechanical properties in order to design for manufacturability in order to limit the need for post processing.

**Methodology**

Stainless Steel 17-4 PH powder feedstock from 3D Systems (Rock Hill, SC, USA) was used in this study. The particle size distribution (measured via laser diffraction analysis using a Beckman-Coulter LS13 320) is shown in Figure 34 a. The SEM image in Figure 34 b demonstrates that the 17-4 PH powder has a morphology that is roughly spherical. The composition of the feedstock powder is found in Table 7. X-ray diffraction (XRD) was performed on the powder and AM specimens using a Panalytical X'pert Pro MPD diffractometer using cobalt radiation. Cobalt was chosen to avoid fluorescence between copper radiation and the steel samples. The XRD spectra of the powder is biphasic FCC and BCC stainless steel and is shown in Figure 34 c.
Figure 34 (A) particle size distribution of 17-4 stainless steel by laser diffraction; (B) SEM micrograph of the 17-4 stainless steel gas atomized powder morphology, (C) XRD micrograph showing dual phase FCC and BCC microstructure of the 17-4 stainless steel.

Table 7 Chemical composition of 17-4PH powder feedstock\textsuperscript{17}

<table>
<thead>
<tr>
<th>Composition (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
</tr>
<tr>
<td>Balance</td>
</tr>
</tbody>
</table>

17-4 stainless steel cylindrical and cuboid compression samples of each scan strategy were manufactured on a Powder Bed Fusion (PBF) machine (3D Systems ProX300 DMP) in a nitrogen environment. Laser processing parameters used in this study are shown in Table 8. Samples were removed from the build plate via EDM.

Table 8 Laser processing parameters used in the bulk of the samples for this study

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Laser Scanning Speed (mm/s)</th>
<th>Hatch Spacing (µm)</th>
<th>Powder Layer Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>1200</td>
<td>50</td>
<td>40</td>
</tr>
</tbody>
</table>
Compression samples were 16 mm in height and 10 mm in diameter for the cylindrical samples and 10 mm in width for the cuboid samples, Figure 35.

![Figure 35 Schematic of the compression cylinder and cuboid.](image)

The following scan strategies were used, where the name in parentheses is the name which will be used to refer to that pattern through this section:

1. Hexagon pattern (“Hexagon”) which consists of 25,000 µm in diameter hexagonal islands with a 100 µm overlap of hexagons within the layer. The hexagons are sintered at a 45° angle to the x-direction of the compression samples. From layer to layer, in the Z direction, the pattern angle alternates between 315° and 225°. The hexagon pattern is the default scan strategy for 3D Systems metal printers.

2. Concentric pattern (“Concentric”) which consists of successive outlines of the part. In this case the outer perimeter of the part is processed by the laser first, and the outlines move successively inward towards the center of the part.

3. 0 degree horizontal hatch pattern (“Zero”). This pattern is unidirectional to the axis parallel to the x-direction of the compression sample.

A schematic of the scan strategies can be seen in Figure 36.
Figure 36 Schematic of the scan strategies for the compression cylinders and cuboids.

Two dimensional size, shape and volume percent of pores were measured using optical microscopy and quantified with ImageJ software. Pores under 1 µm² were not considered for this work due to software limitations. Density was measured using the Archimedes method. To characterize the three dimensional porosity samples were Micro Computed Tomography (Micro CT) CTed using a Zeiss Xradia 520 Versa. Due to size limitations only a quarter of the cuboidal samples were measured by Micro CT. Visual representations of porosity was created using CTvox. Pores under 1000 µm³ were not considered for this study. Porosity size, shape, and orientation were examined using CTan software.

Samples were compressed using a 55kip MTS Hydraulic Universal Test Frame using constant displacement control set at 0.05 inch/minute. Vickers hardness was measured on a Tuckon 1202 using 1 mm x 1 mm grid mapping. X-ray diffraction (XRD) was performed on both the powder and additive manufactured (AM) specimens using a Panalytical X’pert Pro MPD diffractometer using cobalt radiation. Specimens were electro-etched with 10 % Oxalic acid for 45 seconds. Microstructures was examined with a Keyence VK X-200 confocal microscope. Electron backscatter diffraction (EBSD) was performed using the EDAX software TEAM in an FEI NovaLabTM 600 SEM. EBSD scans were run using a beam voltage of 20kV and a beam current of 2.4 nA.

Experimental Data and Discussion
Effect of Scan Strategy on Porosity

Density and Porosity results are shown in Table 9. Archimedes densities show that there is very little variation in density except for the cuboid with the concentric scan strategy which has a reduced average Archimedes density of 7.29 g/cm³. Further investigation into the porosity was completed using micro CT (to show three dimensional defects greater than 1000 µm³ in volume) and optical microscopy (to show defects that would be outside the limitations of micro CT down to 1 µm² in area).

Table 9 Archimedes density and Micro CT porosity.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Archimedes Density (g/cm³)</th>
<th>Micro CT Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexagon Cylinder</td>
<td>7.72 ± 0.02</td>
<td>0.022</td>
</tr>
<tr>
<td>Hexagon Cuboid</td>
<td>7.73 ± 0.02</td>
<td>0.022</td>
</tr>
<tr>
<td>Concentric Cylinder</td>
<td>7.69 ± 0.01</td>
<td>0.207</td>
</tr>
<tr>
<td>Concentric Cuboid</td>
<td>7.29 ± 0.05</td>
<td>3.400</td>
</tr>
<tr>
<td>Zero Degree Cylinder</td>
<td>7.70 ± 0.01</td>
<td>0.031</td>
</tr>
<tr>
<td>Zero Degree Cuboid</td>
<td>7.69 ± 0.02</td>
<td>0.069</td>
</tr>
</tbody>
</table>

Representations of the normalized frequency of the circularity of defects determined using optical microscopy of cross sections in the build direction are shown in Figure 38 A and B. Circularity is the measure of roundness of an object and is represented by equation 1.

\[ \text{Circularity} = \frac{4\pi A}{P^2} \] (1)

Where A is the area of the defect and P is the perimeter, the circularity of a perfect circle would be one. Optical micrographs show that the majority of pores found are a result of entrapped gas (examples represented in Figure 37). All samples except for the concentric cuboid had over 50% of the defects having a circularity of greater than 0.9 represented as gas pores. The concentric cuboid scan strategy resulted in a delamination defects in the build direction on the radially outer part of the sample that was reduced as the pattern moved inward (Figure 37 B) increasing the frequency of less circular pores. Figure 38 C show the cumulative frequency of the defect area as determined through optical microscopy. For all samples over 90% of the determined defects were under 100 square microns.
Figure 37 Binarized optical micrographs for the hexagon cuboid sample (A) and the concentric cuboid sample (B) of the plane parallel to the build direction.
Figure 38 Frequency diagrams for the circularity of the pores determined from image analysis for the (A) Cylinders and (B) Cuboids for each scan strategy. (C) Cumulative frequency of the area of the defects found using optical microscopy.

Visualizations of defects are shown in Figure 39 parallel and perpendicular to the build direction. The zero degree cuboid sample also shows cracking, most likely caused by thermal or residual stresses from the manufacturing process. Concentric specimens are shown to have a decrease in porosity concentration moving towards the center of the specimen. There is however a line of pores corresponding to the start and stop of the contour. There is also a line of porosity occurring at the center of the sample due to the incomplete melting in that area. Table 9 shows the porosity percent for each of scan strategy and geometry specimens found using Cтан software (Bruker micro CT, Kontich, Belgium) image analysis software. The hexagon and zero degree samples had similar porosity concentrations with the zero cuboid having a slightly higher porosity due to cracking found within the sample. Concentric samples had the most porosity with 3.4% for the concentric cuboid and 0.2% for the concentric cylinder. On the outer areas of the concentric samples there
was incomplete bonding in between scan lines and balling can be seen occurring in-between scan paths (Figure 37 B).

Figure 39 Micro CT two Dimensional projections of defects from parallel for the cylinder (A) and cuboid (C). Two dimensional projects of defects perpendicular to the build direction for the cylinder (B) and cuboid (D) for each scan strategy.

The alignment of pores relative to the plane perpendicular to the build direction are shown in Figure 40. For the Zero Degree samples defects are most frequently orientated in the direction of the scan strategy ex. 0, 180 and 360 degrees. The Hexagon scan strategy has most frequent defects oriented in variations of the 45 degree scan strategy ex. 0, 135, 315 degrees. Indicating that most defects were caused by the scan strategy due to layer or melt pool boundary delamination in the orientation of the x-y plane. For the Zero Degree and Hexagon samples there was little variation
in orientation due to the geometry of the samples. The orientation of the concentric cuboid and cylinder defects are shown in Figure 40B. The orientation of the pores in the concentric cylinder were randomly distributed while the concentric cuboid sample showed an orientation preference for every 90 degree direction which corresponds to the scan strategy direction the laser moves in. Most defects are orientated in the build direction Figure 40C due to residual stress being larger perpendicular to the laser scan direction.21

Figure 40 Frequency of the angle of orientation (φ) of pores: (A) zero degree, concentric, and hexagon cylinder samples and (B) concentric cylinder and cuboid for the x-y-plane. (C) Frequency of the defect angle of orientation (θ) where 90 degrees is the build direction and zero degrees is the x-y plane.

Sphericity of the defects compares how closely the elongation of a defect is to a perfect sphere represented by equation 2.

\[
Sph = \frac{3\sqrt{3}/(6\nu)^{2/3}}{s} \quad (2)
\]
Where V is the sphere volume and S is the sphere surface area. A perfect sphere would have a sphericity of one. The distribution of volume defects and its sphericity can be found in Figure 41 A and B. Most defects have a sphericity between 0.6 and 0.9 suggesting that most porosity determined using Micro ct is a result of lack of fusion and delamination. The concentric cuboid had the most elongated defects with 10% of defects having a sphericity of less than 0.6 while the cylindrical compliment had less than 5%. The increased frequency in elongated defects can be attributed to the contour strategy. The concentric specimens also have the largest defect volumes and have 10% of pores greater than 27000 cubic microns.

![Figure 41 Frequency of defect sphericity (A) and cumulative frequency of the volume (B) of defects.](image)

**Effect of Scan Strategy on Mechanical Properties**

**Elastic Modulus**

The effect of scan strategy on elastic modulus for each scan strategy and geometry is shown in Figure 42. The zero degree samples had the highest elastic modulus. Elastic modulus for the concentric cuboid was the lowest of all samples due to the highly angled porosity (60 % orientated away from the build direction) which can be deleterious to the elastic modulus.
Figure 42 Compression elastic modulus of 17-4 stainless steel samples for each scan strategy and geometry

Hardness

Average hardness of the transverse direction of the samples are shown in Figure 43. Vickers hardness maps shows the average hardness of the x and build direction of the samples are in Figure 44. The zero degree samples had the least variation and highest average hardness. Zero degree scan strategy had the least variation in hardness and shows the highest hardness in the cross sectional. This follows the other mechanical property results where the zero degree samples also had the highest elastic modulus. This could be due to a lack of a cross directional layer to layer scan strategy since the samples were manufactured using the same laser processing properties and exhibited similar defects the only difference would be the change in thermal history resulting from the different scan strategies.
Figure 43 Average Vickers hardness for the plane parallel to the build direction

Figure 44 Vickers hardness maps of the x and build direction of the samples: (A) Hexagon Cylinder; (B) Concentric Cylinder; (C) Zero Degree Cylinder; (D) Hexagon Cuboid; (E) Concentric Cylinder; (F) Zero Degree Cuboid.

Cuboid concentric hardness maps have a reduced hardness in the outer area due to the porosity caused by the 90 degree change in direction (Figure 44E). Once the sample became less porous towards the middle the hardness increases and becomes comparable to the zero degree and hexagon samples (this area is also shown in the concentric cylinder in Figure 44 B). The hardness in the
middle of the sample decreases by 100 HV. The concentric cylinder has a similar decrease in hardness towards the middle of the sample with an increase at the center.

**Effect of Scan Strategy on Microstructure**

Figure 45 shows the XRD spectrums for the cuboids as measured across the vertical reference plane (PBF layers are orientated normal to the surface studied). All samples demonstrate a duplex stainless steel exhibiting BCC and FCC microstructure. There is a double peak for the FCC microstructure at the 2θ of 50 degrees for all samples that was not present in XRD spectrum of the pre-manufactured powder (as shown in Figure 34 C).

![XRD spectra for cuboid samples using a Co source.](image)

Optical micrographs of the vertical reference plane are shown in Figure 46. The microstructure generally followed the scan strategy with the melt pool boundaries that are easily recognized. There was no change in microstructure for geometries for the zero degree and hexagon scan strategies. Zero degree specimens exhibited a strongly BCC microstructure with the melt pool boundaries being stacked on top of each other. Hexagon also had a mostly BCC microstructure, but has an increased concentration of smaller refined grains that exist on the grain boundaries due to the cross directional layer to layer scan strategy.
The concentric scan strategy has a non-homogenous microstructure with three distinct zones (Figure 47 A and B). Zone I corresponds to the majority of the area created by the concentric scan strategy. This area is also shown via Micro ct to contain the greatest amount of pores greater than 1000 cubic microns, especially in the cuboid sample. Zone I microstructure is similar to that found in the zero degree and hexagon specimens and this is reflected in having comparable hardness measurements in the concentric cylinder sample (Figure 44 B and E). The concentric cuboid sample has a reduction in hardness due to the high concentration of porosity on the outer areas of the sample. In Zone II refined grain areas are exhibited and the melt pool boundaries that the laser creates are still visible. For the concentric samples as the layer develops the unmelted powder acts as an insulator preventing the transfer of heat away from the center (Figure 50 b). As the laser moves to the middle of the sample the center the length of the scan line and the time between laser passes decreases (Figure 47 C). This reduces the amount of time between laser passes allowing for this area to maintain heat more efficiently than the rest of the surface. This area was represented in the in the hardness maps as an area with reduced hardness (~250 HV). Zone III is the area closest to the center and encompasses the last two scan lines for each layer. This zone has a mixture of extra refined and large BCC grains as well as scan strategy induced porosity caused by lack of laser interaction with the center of the specimen. This area correlates to the increased hardness in the center of the specimen as represented by area C in the hardness profile in Figure 48.
Figure 47 Optical micrograph of the cylinder (A) and cuboid (B) showing the area perpendicular to the build direction. Zone I: shows the steady state microstructure with mostly BCC grains with smaller grains on the melt pool boundaries. Zone II Refined mostly BCC grains. Zone III Center of the concentric part: Refined grains with an increase in austenite with large BCC grains found in the heat affected zone with Scan strategy induced porosity on the order of the diameter of the diameter of the final circle of 80µm. (C) Estimated time between laser passes (in seconds) as
moving inward toward the center of the concentric samples with the location of the corresponding zones noted.

Figure 48 Optical micrographs of the concentric sample parallel to the build direction. With A and E corresponding to Zone I, B and D corresponding to Zone II, and C corresponding to Zone III. With F representing the micro hardness profile across the sample parallel to the build direction in the x-direction with the corresponding micrographs noted.

Figure 49 A and B show the IPF Map produced from the EBSD data from the build direction of the zero degree and hexagon scan strategies to show the effect of cross hatching has on grain orientation and size. The cross directional layer to layer pattern as a result of the hexagon scan strategy is visible within the orientation of the grains and the breaking up of the elongated grains. The IPF map also shows that smaller grains occur along the melt pool boundaries that are shown to have a random orientation with no specific texture. Small amounts of austenite likely to occur in regions with refined grains. Figure 49 C shows the effect of scan strategy has on the distribution of grain size for the hexagon and zero degree scan strategies. The hexagon sample has an increased concentration of smaller grains under 10 µm in diameter compared to the zero degree sample due to not having a repeating layer to layer pattern. Figure 50 shows the difference...
in grain size distribution of the zones of the cross section of the concentric cylinder specimen. Moving towards the center of the part there is a significant decrease in grain size between Zone I and Zone II (average grain size decreases from 14.83 to 2.65 microns). Zone III has the smallest grain size with an average of 1.91 microns as well as porosity at the center of specimen caused by the incomplete melting in that region that can cause keyholing porosity.²⁶

Figure 49 IPF EBSD maps showing the effect Zero Degree (A) and Hexagon (B) scan pattern for grain orientation and diameter. (C) Distribution of grains taken from measurements in the build direction for cylindrical samples.
Figure 50 IPF EBSD maps of Zone I (A), Zone II (B), Zone III (C), Hexagon (D), and Zero Degree (E) for the grain orientation. (F) Distribution of grain diameters taken from measurements transverse to the build direction for the concentric cylinder specimen.

Table 10 Grain size characteristics for the cylinder samples perpendicular to the build direction

<table>
<thead>
<tr>
<th></th>
<th>Zone I</th>
<th>Zone II</th>
<th>Zone III</th>
<th>Hexagon</th>
<th>Zero Degree</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Grain Diameter (micron)</td>
<td>14.83</td>
<td>2.65</td>
<td>1.91</td>
<td>16.29</td>
<td>21.57</td>
</tr>
<tr>
<td>Largest Grain Size (micron)</td>
<td>45.15</td>
<td>11.09</td>
<td>8.20</td>
<td>45.50</td>
<td>62.68</td>
</tr>
</tbody>
</table>

Unlike the zero degree and hexagon samples that have the thermal history moving in one direction for each layer the concentric samples the heat moves inward. This allows for higher temperatures for areas closest to the center where as the outer areas have four times the amount of time to cool (Figure 47 C). Due to the reduced time between scan lines the area closest to the center (Zone II) act as a heat affected zone (HAZ) to fusion zone created by the local laser welding occurring adjacent. This causes a reduction in grain size and hardness in this HAZ.27-28 Zone II also
experiences a lower cooling rate than that of Zone I, thereby potentially increasing the amount of austenite retained.\textsuperscript{29}

**Conclusions**

Directional porosity negatively affect the elastic modulus such as those found on the concentric cuboid samples.

For geometries with no change in shape in the vertical direction there is no significant change in microstructure from cuboid to cylinder.

Hexagon scan strategy reduced texturing in the build direction due to cross directional layer to layer scan strategy

Having an inward moving thermal history with a decreasing laser scan length and reducing the time between laser passes resulted in refined grains and decreased hardness.
References


Chapter V: Effect of Zone Creation to Locally Control Microstructure in Powder Bed Fusion 17-4 Stainless Steel

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Keywords: Additive manufacturing, powder bed fusion, stainless steel, processing-microstructure-property relations

Highlights

1. Reduction in scan length decreases grain size and reduces porosity.
2. Formation of zones creates defects due to shrinkage.
3. By creating areas with different laser processing parameters were able to decrease the average grain size by 75%.
Abstract

Powder Bed Fusion is an additive manufacturing technique that utilizes localized melting in order to manufacturing metallic parts. The effect of adjusting laser processing parameters in different regions in order to locally tailor mechanical properties within a part has not been explored. In this work the effect of zone creation through selectively changing the laser power and speed and double melting different regions were characterized. Defect formation was examined using micro computed tomography and found that pores were aligned along zone boundaries due to shrinkage. Microstructure and phase identification were measured using optical microscopy and electron beam scatter diffraction which found that by reducing the laser scan distance the grain size decreases and the austenite concentration increases compared to bulk. Mechanical properties were measured using microhardness measurements showing that zones were softer in hardness compared to the bulk of the sample. Understanding the effect of scan length and how this can be used to change material properties allows for the tailoring of properties within a part.

Introduction

Powder bed fusion (PBF) is an additive manufacturing technique used to produce metallic components utilizing selective laser melting of a powder layer.1 This manufacturing technique allows for the formation of parts with unusual geometries that could be difficult to make using traditional manufacturing methods.2-3 The rapid solidification of the additive manufacturing process can be exploited to potentially make tailored mechanical properties.

Previous studies investigated the laser scan speed and power to measure its effect on single weld line quality.4-6 This was done to obtain laser speed and power combinations that displayed good wetting properties by exhibiting smooth continuous lines with no balling or beading.6-7 It was found that increasing scan speed can induce thermocapillary irregularities due to the reduction of heat being applied to the powder creating incomplete melt pools.8 On the other hand, too slow scan speed can cause overheating of metal powder and poor material properties due to splashing.9-10 If the power is not great enough the energy applied to the powder bed will be too low to induce enough liquid phase sintering to allow for bonding between powders.11 Gu et al. investigated balling phenomena in stainless steel powders by varying the laser power and velocity.6 It was found that when the power was too low for the laser velocity there was not enough energy to overcome the surface tension causing balling to occur. This partial melting can create coarsened balls of agglomerated powder and a noncontiguous melt. As the laser power was increased there was an increase in liquid formation overcoming the surface tension and allow for spreading creating necking and bonding of powders allowing for a continuous melt. When the laser power was too high overheating can occur causing an irregular scan lines due to splashing.
In order to measure the influence of the laser powder and speed for multilayer laser processing of PBF, the volumetric energy density equation was proposed to quantify the amount of energy applied to the powder bed. The equation for volumetric laser energy density \( E (J/mm^3) \) is shown in equation 1, where \( P (W) \) is the laser power, \( v (mm/s) \) is the laser scanning speed, \( h (mm) \) is the hatch spacing—the distance between laser scan lines, and \( t (mm) \) is the powder layer height.\(^{12}\)

\[
E = \frac{P}{v \cdot h \cdot t} \quad (1)
\]

For multilayer PBF, the hatching distance needs to be optimized so that adjacent scan lines are bonded. If the hatch distance is too large, it can result in a reduction of overlap between scan lines, increasing porosity.\(^{11}\)

By having a constant energy density, but with varying laser power and scanning speed does not result in the same properties. Gu et al. [2013] found that as the power (190-70W) and scanning speed (800-287mm/s) decreased, the porosity increased while the energy density was maintained at 61 J/mm\(^3\).\(^{13}\) The volume fraction of martensite was fairly consistent for all samples.

Previous research has shown that by only changing the laser scan strategy and maintaining all other laser processing parameters can result in drastically different material microstructures and mechanical properties.\(^{14}\) By having a better understanding of the influence of additive manufacturing processing parameters on mechanical, material, and defect properties can reduce the need for heat treatments and other post processes. Previous studies have only investigated the bulk effect of laser processing parameters. By quantifying the effect of locally changing the laser processing parameters within a part, we can better understand how to customize material properties. Manipulating the thermal history of the PBF manufacturing process and investigating its influence on microstructure including phase concentration, orientation, and grain size as well as defect formation can broaden our design capability and allow us to tailor mechanical properties.

**Experimental Methodology**

Four 17-4 stainless steel cylindrical samples were manufactured on a Powder Bed Fusion (PBF) machine (3D Systems ProX300 DMP) in a nitrogen environment. Bulk laser processing parameters used in this study are shown in Table 11. The scanning strategy of the bulk of the cylinders was done with the manufacturers default “hexagon” island based laser scan strategy with either double melting or an inset of the zone.

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Laser Scanning Speed (mm/s)</th>
<th>Hatching Spacing (µm)</th>
<th>Powder Layer Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>1200</td>
<td>50</td>
<td>40</td>
</tr>
</tbody>
</table>

79
The zones used for this study are the letters “ARL” with the width of each line of text being 0.25 to 0.3 mm in width. Schematics of the scan strategy for each cylinder are shown in Figure 51. This text was used to show examples of the effect of zones with curved as well as straight edges.

![Scan Strategy Schematic](image)

**Figure 51** Scan strategy schematic for the samples

The following scan strategies were used, where the name in parentheses is the name which will be used to refer to that pattern through this section:

1. (“Reference”) The bulk has a hexagon pattern which consists of 25,000 µm in diameter hexagonal islands with a 100 µm overlap of hexagons within the layer. The hexagons are sintered at a 45° angle to the x-axis of the cylinder samples. From layer to layer, in the Z direction, the pattern angle alternates between 315° and 225°. The hexagon pattern is the default scan strategy for 3D Systems metal printers.

2. (“Double Melting”) The bulk scan strategy has the same hexagon pattern as in the reference. After the cylinder is printed each layer, the zone area is “remelted” with the same laser processing parameters and scan strategy as the bulk.

3. (“Equivalent Energy Density”) The bulk scan strategy has the same hexagon pattern as in the reference. The bulk is printed with the laser melting an outline of the “ARL” zone. After the bulk is complete the laser than processes the inset with a hexagon scan strategy with a reduction of the laser scanning speed from 1200 to 600 mm/s and the laser power from 150 W to 75 W. The powder bed laser energy density is maintained at 62.5 J/mm³.

4. (“Reduced Speed”) The bulk scan strategy has the same hexagon pattern as in the reference. The bulk is printed with the laser melting an outline of the “ARL” zone. After the bulk is
complete the laser then processes the inset with a hexagon scan strategy with a reduction to the laser scanning speed from 1200 to 800 mm/s. An increase in the powder bed energy density from 62.5 J/mm³ to 93.75 J/mm³.

The processing parameters of the specimens including the laser speed and power are summarized in Table 12.

Table 12 Cylinder processing parameter for bulk and zone regions.

<table>
<thead>
<tr>
<th>Cylinder Name</th>
<th>Type</th>
<th>Scan Strategy</th>
<th>Bulk Power (W)</th>
<th>Bulk Speed (mm/s)</th>
<th>Bulk Energy Density (J/mm³)</th>
<th>Zone Power (W)</th>
<th>Zone Speed (mm/s)</th>
<th>Zone Energy Density (J/mm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
</tr>
<tr>
<td>Double Melting</td>
<td>Double Melting</td>
<td>Hexagon</td>
<td>150</td>
<td>1200</td>
<td>62.5</td>
<td>150</td>
<td>1200</td>
<td>62.5</td>
</tr>
<tr>
<td>Equivalent Energy Density</td>
<td>Inset</td>
<td></td>
<td>75</td>
<td>600</td>
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<td>Reduced Speed</td>
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<td>150</td>
<td>800</td>
<td>93.75</td>
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Samples were removed from the build plate with electrical discharge machining. Densities were measured by Archimedes. To characterize porosity Microcomputed Tomography (Micro CT) was done using a Zeiss Xradia 520 Versa with a pixel size of 10 µm. Three dimensional representations of defects within specimens were made using CT Vox. Defect size, shape, and orientation were characterized using Ctan software. Specimen’s microstructure was revealed using 10% oxalic acid electro etchant. Optical micrographs were created using a Keyence VK X-200 confocal microscope. Vickers hardness measurements were completed using a Tuckon 1202 using a 0.25 mm x 0.25 mm grid mapping. Electron backscatter diffraction was performed using the EDAX software TEAM in an FEI NovaLabTM 600 SEM. The scans were run using a beam voltage of 20 kV and a beam current of 2.4 nA.

**Results and Data Analysis**
Effect of Zone Formation on Defects

Relative densities are shown in Table 13. Specimens exhibited no significant change in density due to the addition of zones. Micro CT was completed to measure the effect of zones and double melting on the defect formation and porosity concentration. The results of the percent porosity and defect characteristics are summarized in Table 13. The addition of zones did increase the porosity concentration within the part, especially for the Equivalent Energy Density (EED) sample. It should be noted that the bulk areas of the zone samples have a decreased porosity comparable to the reference samples for the EED and Reduced Speed (RS) specimens. This porosity is comparable to the found in the Reference and Double Melting (DM) samples. This was not done for the DM sample due to lack of change within the part due to the use of double melting.

Table 13 Archimedes density, Micro CT porosity and defect characteristics of the whole part and bulk sections
Sphericity of the defects relates how elongated a defect is compared to a perfect sphere and can be represented by the equation (2).

\[ Sph = \frac{3\sqrt{\frac{3V}{2\pi}}}{s} \] (2)

Where \( V \) is the defect volume and \( S \) is the defect surface area. A sphericity of one would correspond to a perfect sphere. The distribution of sphericity for the defects for samples within this study can be seen in Figure 52 and the average sphericity can be found in Table 13 Error! Reference source not found.. The majority of pores have a sphericity between 0.6 and 0.9 which is likely a result from the manufacturing process such as incomplete bonding, incomplete melting, keyholing, and delamination between layers.\(^{15}\) Pores caused by entrapped gas have a very high sphericity. Our previous using optical microscopy showed that these pores may be too small to be visible via Micro CT (Sub 200 µm\(^2\)).\(^{14}\) The average sphericity did not significantly change from the zone area compared to the bulk section, however the distribution of sphericity for the bulk versus the zone regions show that the more elongated pores were created as a result of the zone formation.

Figure 52 Defect sphericity frequency distribution for (A) the Zone regions of specimens and (B) the bulk area (represented by the areas of the circle not in black).

Figure 53 shows the distribution of defect volume found via Micro CT for the zone region (Figure 53 A) and the bulk region (Figure 53 B). The defect volume distribution for the EED sample resulted in a significant amount of large pores that resulted from the use of zones within the manufacturing process. In the bulk section the distribution of pores was comparable the rest of the samples for the EED and RD samples Error! Reference source not found..
Figure 53 Cumulative frequency of the defect volumes for the zone region (A) and the bulk region (B) (represented by the area of the circle not covered in black).

Visual representation of the porosity can be found in views perpendicular (Figure 54) and parallel (Figure 55) to the build direction. The EED and RS samples have an increased porosity corresponding to the interface between the zone and bulk of the sample, this results in the clearly visible outline of “ARL”. This is likely caused by the change in processing parameters as well as shrinkage of the bulk area in the zone as a result of the reduced scan length.\textsuperscript{16-17} The view perpendicular to the build direction show that porosity is non uniform throughout the build height. These defect patterns are not visible in the double melted sample. However, there is a clear line of porosity in the Figure 54 B that corresponds to the island overlap as a result of the hexagon scan strategy. It should be noted there was no detectable porosity inside the “A” and the “R” zones of the EED and RS samples.
Figure 54 Visual representation of defects with build direction being normal to the build direction plane for the Reference (A), Double Melting (B), Equivalent Energy Density (C), and Reduced Speed (D) specimens. With arrows for the Equivalent Energy Density and the Reduced Speed representing the porosity that aligns with the interface with the zone and the bulk part. For the Double Melting sample the arrow indicates the island based overlap caused by the hexagon scan strategy.
Effect of Zone Formation on Hardness

Hardness maps were generated on the cross section parallel to the build direction of the specimens to quantify the homogeneity and the effect of zone creation. The average hardness for the full, zone, and bulk regions are presented in Figure 56. For the Reference and the DM samples there is little variation in the average hardness between the bulk and zone regions of the sample. For the zoned samples the bulk areas had comparable hardness measurements to the specimens without zones. The area corresponding to where the zones occurred had a reduced average hardness compared to the bulk areas. Zones had an especially negative effect on the hardness properties for the EED specimen likely contributed from the high porosity concentration as shown in Figure 57. Double melting had little effect on the surrounding hardness in the zone area.
Figure 56 Vickers hardness results for samples for the full cross section, zone regions (area within box), and bulk area (area outside of box).
Effect of Zone Formation on Microstructure

The microstructure for all samples was revealed using an oxalic acid electro etchant. Optical micrographs of the bulk area (Figure 58) show that all specimens have comparable microstructure with not much variation. IPF EBSD maps of this area show that the austenite concentration varies due to colony concentration and the average grain size is between 13 and 18 microns in diameter, Figure 59. The concentration of austenite is low (less than 10%) and forms within colonies of smaller grains. Previous research has revealed that these colonies can occur along melt pool boundaries in the build direction.14
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Figure 59 IPF EBSD Maps for the Reference (A), Double Melting (B), Equivalent Energy Density (C), and Reduced Speed (D) specimens for the bulk region of a cross section normal to the build direction. With the average grain size percent and concentration of Austenite (E) for each sample.

The zone areas for all samples resulted in a change in microstructure. Optical micrographs for all samples within the R area of the zone (representative of zones in each sample) are shown in Figure 58. For the EED specimen there are large grains present, but there is also visible porosity corresponding to incomplete bonding between the zone and bulk of the sample with an increase of smaller grains at the interface (Figure 58 C). For the RS specimen the grains are more refined within the zone, but larger BCC grains still occur although infrequently. There is a decrease of average grain size (a quarter of the size than those found in the bulk region) as well as a 75% increase in austenite concentration. For the DM sample there is an increase in smaller grains, however, it is not significant and does not appear to have an effect on the microstructure in the surrounding areas.

Optical micrographs show that there is delamination along the melt pool boundaries for the EED. Delamination also appears to occur along the boundaries of the Double Melt zone, but not in the RS areas. Micro CT images demonstrate that there is porosity that does exist along these boundaries for the RS sample (Figure 54). For the RS sample the scan speed is reduced, but the power is kept constant increasing the amount of heat applied to the powder (compared to reference). A reduction of speed increases the width and depth of the melt pool which could have improved the wettability decreasing the porosity at the interface in comparison to the EED sample. However, this interaction is also power dependent which is why the EED exhibited higher porosity at the interface at a much lower laser scan speed (800 to 600 mm/s).
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Optical micrographs for the center of the R are shown in Figure 62. It was observed that in zone regions represented by “A” and “R” there is a reduction of defect formation which may be as a result of reduction of scan length. There are inclusions in these areas, but their high circularity suggests that they are formed due to entrapped gas, not a result of the manufacturing processing parameters. For the DM and the Reference samples the resulting microstructure is similar to that found in the bulk regions of the sample.

EBSD maps show there is a finer microstructure being exhibited in the areas within the A and R for the RS and EED samples (Figure 63). There is a decrease in grain size (10-15 microns in the bulk to under 5 microns in diameter). This could be due to the heating up of this area as a result of the shorter scan length and the reduced length of time between scans. This can refine grains having a tempered like effect like that seen previous studies. Softening and a reduced grain size can occur as a result of the heat affected zone created by the reduced length scan strategy. This process can also increase austenite retention which can be seen in Figure 63 E. For the EED and RD samples the Austenite concentration in the reduced length areas was over 10%. This indicates that there is a scan length that could be used with the hexagon scan strategy that would allow for similar microstructure to occur.
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Conclusions

A detailed study has been conducted to investigate the effect of using zones and double melting on defect formation of 17-4 stainless steel, and the following conclusions can be drawn:

In the PBF of 17-4 stainless steel the Austenite formed by the process occurs only in small grains (sub 5 micron in diameter).

Locally double melting areas did not result in defect formation and did not have a strong effect on the surrounding microstructure.

Local formation of zones did not have an effect on defects and microstructure in the bulk of the part.

Maintaining energy density, but reducing power and laser scan speed resulted in similar microstructure, but had decreased wettability properties increasing porosity.

By reducing the laser scan length the formation of small grains with higher concentrations of austenite was observed. Suggesting that there is a critical scan length with which used in an island strategy could result in a complete part composed of sub 5 micron grains.
References


Chapter VI: Research Conclusions

This thesis presented a series of experimental investigations into the effect of additive manufacturing processing parameters on 17-4 stainless steel powder bed fusion. The following conclusions can be drawn.

The influence of scan strategy on the material and mechanical properties of PBF 17-4 has been studied. (Chapter III and IV)

- Keeping the laser processing parameters constant and varying the scan strategy can change the thermal history and result in different material and mechanical properties.
- The as-made PBF 17-4 parts were composed of BCC/Martensite and Austenite
- The length and direction of the raster pattern, at constant energy density was shown to have a strong influence on the cooling rate and resulting microstructure and defects within a final part.
- A large variation in tensile elongation properties from 3.3 to 17.6% was observed based on laser scan strategy.
- Reducing the laser scan length and decreasing time between laser passes resulted in refined grains and decreased hardness.
- Hexagon scan strategy reduced texturing in the build direction due to cross directional layer to layer scan strategy

The influence of local control of material properties through zone formation was studied. (Chapter V)

- In the PBF of 17-4 stainless steel the Austenite formed by the process occurred only in small grains (sub 5 micron in diameter).
- Locally double melting areas showed decreased grain size, but did not result in local defect formation and did not have a strong effect on the surrounding microstructure.
- Local formation of zones did not have an effect on defect formation and microstructure on the bulk of the part.
- By reducing the laser scan length the formation of small grains with higher concentrations of austenite was observed. Suggesting that there might be a critical scan length that used in an island scan strategy could result in a complete part composed of sub 5 micron grains.
Chapter VII: Recommendations for Future Work

Metal additive manufacturing (AM) is a manufacturing technique that has many potential benefits for the army if implemented in theater. Due to it being a relatively recent technology the understanding of its capabilities and its ability to be certified for use in the field is limited. The goal of my research is to develop the fundamental knowledge of the additive manufacturing process in order to improve mechanical properties and increase repeatability in order to qualify parts for use in the field.

With the open access capabilities of the powder bed fusion AM machines, the ability to manipulate the thermal history locally could potentially allow for the tailoring of microstructure. Previous research has shown that by changing only the laser scan strategy and maintaining all other processing parameters can result in drastically different material and mechanical properties. By having a greater understanding of the additive manufacturing process and its effect on mechanical, material, and defect properties, the need for heat treatments and other post processing can be reduced. Manipulating the thermal history of the manufacturing process and investigating how these changes affect microstructure (phases, orientation, and grain size) and defect formation can broaden our capability to tailor mechanical properties within a part to design for additive manufacturability. A few future directions may include:

**Defect Formation Characterization**

One benefit of additive manufacturing is that it allows for more complex geometries than traditional manufacturing. However, the effect of changing the geometry through a part such as increasing and decreasing areas layer to layer has not been thoroughly investigated. It is important to better understand how the change of thermal history within a part due to part geometry affects microstructure and defect formation. The use of non-destructive micro Computed Tomography (micro CT) can visualize and characterize manufacturing defects and part accuracy. Micro CT provides the ability to quantify the sphericity, orientation and size of pores within a part. This will be used to relate defects to manufacturing processing parameters for as built, post processed, and mechanically tested parts in a non-destructive fashion.

**High Strength Materials**

For Army applications, AM has the benefit of being able to fabricate parts from materials that are not commonly wieldable. Materials such as tungsten with high strength are of particular interest due to its use in munitions and projectiles. However, there is very little prior AM investigations into tungsten due to it being susceptible to brittle failure under stress induced thermal shock. A future research topic could be to increase wettability and decrease cracking of PBF tungsten through decreasing the particle size. This could potentially reduce thermal cracking compared to increasing the thermal gradient by adjusting laser processing parameters alone. The effect of particle size distribution on part density, material, and mechanical properties will be quantified for pure tungsten. This also requires not only an understanding of the manufacturing process but the feedstock powder as well. The powder flowability, conductivity, particle size distribution, and
sphericity (relating to powder packing density) will also be quantified to see how tungsten AM is effected by particle size.

**Powder Characteristics**

The feedstock powder used for the AM process has an effect on the final part material and mechanical properties. It is important to characterize and understand how powder properties influence the AM process in order to manufacture reliable parts.

The ability to reuse powder especially in the field is extremely beneficial especially compared to traditional top down manufacturing. It is important to quantify powder feedstock characteristics such as flowability, sphericity, and size distribution, with an emphasis on how reusing this powder affects these characteristics and how this translates to final part defect and mechanical properties.

Investigation of the effect of inert gas used in powder atomization and inert gas on feedstock, mechanical properties, defect formation, and microstructure of 17-4 stainless steel should be carried out. Optimization of which can improve mechanical properties and streamline manufacturing process to reduce time and cost and increase reliability of the manufacturing process.
Appendix A: Publications and Presentations

Accepted Publications:

Journal Publications in Preparation
Andelle Kudzal, Tomoko Sano, Scott Coguill, Joshua Taggart-Scarff, Jianyu Liang, Brandon Mcwilliams, *Effect of Laser Scan Pattern and Part Geometry on Mechanical Properties for Powder Bed Fusion Stainless Steel*

Andelle Kudzal, William Hornbuckle, Clara Hofmeister, Joshua Taggart-Scarff, Jianyu Liang, Brandon Mcwilliams *Effect of Zone Creation to Locally Control Microstructure in Powder Bed Fusion Stainless Steel*

Conferences:
Solid Freeform Fabrication
Andelle Kudzal, Clara Hofmeister, Daniel Galles, Chad Hornbuckle, Brandon McWilliams, Jianyu Liang, “Effect of Zone Creation to Locally Control Microstructure in Powder Bed Fusion 17-4 Stainless Steel”

Clara Hofmeister, Andelle Kudzal, Joshua Taggart-Scarff, Ryan Rogers, Jennifer Sietins, Brandon McWilliams, “Effect of a mid-build halt on the microstructure and porosity in powder bed fusion stainless steel parts”

Andrew Gaynor, Terrence Johnson, Andelle Kudzal, Brandon McWilliams, “Projection-based topology optimization algorithms for manufacturing design constraints in powder bed fusion and vat-based additive manufacturing.”


MS&T
Krista Limmer, Andelle Kudzal, Efrain Hernandez, Mark Tschopp, Brandon McWilliams, “Design of Experiments Approach for DMLS”

TMS
Andelle Kudzal, Clara Hofmeister, Joshua Taggart-Scarff, Brandon McWilliams, Jianyu Liang, “Effect of geometry on the porosity and microstructure of additively manufactured titanium” Phoenix, Arizona 2018

Brandon McWilliams, Andelle Kudzal, Jian Yu, “Effect of Laser Scan Strategy on Microstructure-property Relations in Additively Manufactured Stainless Steel”

Brandon McWilliams, Brahmananda Pramanik, Andelle Kudzal, Bruce Madigan, “Effect of Laser Scan Strategy and Post Processing on High Strain Rate Deformation Response of Additively Manufactured Stainless Steel”
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