



# **NAVAL POSTGRADUATE SCHOOL**

**MONTEREY, CALIFORNIA**

## **THESIS**

**MECHANICAL AND MICROSTRUCTURAL PROPERTIES  
OF MARAGING STEEL SAMPLES PRODUCED BY  
ADDITIVE MANUFACTURING (3D PRINTING)**

by

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June 2020

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**MECHANICAL AND MICROSTRUCTURAL PROPERTIES  
OF MARAGING STEEL SAMPLES PRODUCED BY  
ADDITIVE MANUFACTURING (3D PRINTING)**

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## **ABSTRACT**

In recent years, additive manufacturing (AM) techniques have been compared to those of traditional manufacturing (TM) in order to compare and contrast part quality. Selective Laser Melting (SLM) is an AM technique which uses a standard triangle language (STL) file to 3D print a part layer by layer. A high-power laser is used to melt metal particles to obtain fully dense parts. This work studied the mechanical and microstructural properties of SLM parts by performing tensile and microhardness tests, along with optical and scanning electron microscopy (SEM) microscopy. Parts produced via SLM were found to be comparable to those produced by TM and to contain adequate mechanical properties. Feature quality of SLM parts was studied and mechanical and microstructural properties observed. Microhardness tests and optical microscopy revealed that feature quality increased with feature size. Furthermore, print parameters require optimization to produce adequate features and parts containing minimum porosity.

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## LIST OF ACRONYMS AND ABBREVIATIONS

|      |                                   |
|------|-----------------------------------|
| AM   | additive manufacturing            |
| AP   | as printed                        |
| CNC  | computer numerical control        |
| EDS  | energy dispersive spectroscopy    |
| EHT  | electron high tension             |
| HT   | heat treated                      |
| HV   | hardness according to Vickers     |
| MS   | maraging steel                    |
| PD   | powder depleted                   |
| QA   | quality assurance                 |
| QC   | quality control                   |
| RPM  | rotations per minute              |
| SB   | sandblasted                       |
| SEM  | scanning electron microscope      |
| SLM  | selective laser melting           |
| STL  | standard triangle language        |
| TM   | traditional manufacturing         |
| TRIP | transformation-induced plasticity |
| XRD  | x-ray diffraction                 |
| YAG  | yttrium aluminum garnet           |
| YB   | femtosecond ytterbium             |

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# **I. INTRODUCTION**

## **A. OVERVIEW**

The idea of turning bits into atoms began as early as the 1860s with the first attempt to replicate solid objects using photographs and tracing [1]. Francois Willeme, a Frenchman from Paris, began by simultaneously photographing a solid figure surrounded by twenty-four cameras, then using their silhouettes to create a 3-dimensional (3D) representation of that same object. From the photographs, he obtained layer data for his model. Each image was then projected onto a screen where it was translated to the physical movements required to re-create each layer of the model. A pantograph was used to trace and cut out each layer. All layers were then compiled to create a photo sculpture of the object [1].

Over the next few centuries, several technological advances would lead to what is now known as 3D printing, otherwise termed additive manufacturing (AM). From this, many methods were developed to create objects from several materials including plastics, glass, waxes, and metals. This research will focus on the AM of metals and maraging steel 300 in particular.

## **B. MARAGING STEEL**

In recent years, a variety of high strength low weight steels were developed to meet specific requirements in nuclear, aerospace, and defense applications, maraging steel was among these steels. The term “maraging” is derived from its martensitic microstructure obtained through age-hardening processes [2]. The characteristics of maraging steel make it well suited for safety-critical aircraft design which require high strength and damage tolerance. Maraging steel is a form of high strength, high toughness steel with a relatively high concentration of nickel. The presence of nickel in concentrations from 10 to 30 wt% causes the formation of a microstructure consisting of a cubic martensitic matrix with a relatively high density of nanometer-sized Ni-based precipitates. These precipitates are introduced by an aging treatment [3]. The strength of maraging steel is derived from the precipitation of these Ni-based intermetallic compounds, vice carbon content manipulation

like most steels. Additionally, high carbon content steels are commonly found to have high levels of corrosion and quench cracking. These defects are sometimes not identified until a part is in service, causing that part to fail prematurely. Reduced carbon found in maraging steel reduces susceptibility to corrosion [3]. Finally, the lack of carbon enables maraging steel to have good weldability, making it ideal for additive manufacturing.

Unlike polymeric materials which can be directly printed, however, maraging steel parts need to be post-processed in order to develop the desired properties. Recall precipitates, which strengthens the material, is formed during an appropriate post printing heat-treatment. Without the proper treatments, the 3D printed steel will not perform as expected and will pose a risk to systems which it is integrated into. What will be seen in later chapters are the effects of the printing process on the printed part. The unique AM technique used to print maraging steel imparts a cellular structure which was observed to improve the mechanical properties (increased strength without losing ductility) over TM maraging steel.

### **C. ADDITIVE MANUFACTURING**

As the manufacturing industry has adopted 3D printing methods, so has the U.S. Navy. AM techniques are predicted to reduce cost, as well as part lead time, resulting in greater equipment availability for both the Navy and Marine Corps alike [4]. Huntington Ingalls Industries' Newport News Shipbuilding division, the sole producer of U.S. Navy aircraft and submarines, is at the leading edge of the Navy's integration with advanced technologies. Since 2017, Newport News Shipyard has used digital shipbuilding to increase efficiency, safety, and affordability [5]. Digital shipbuilding, also known as 3D modeling, allows for consolidated drawings, laser scanning of spaces, augmented reality, modeling and simulation. The state-of-the-art technique of 3D modeling has enabled shipbuilders to model, customize, and optimize parts to reduce material consumption and increase functional space while maintaining issues that may cause premature failure [6].

Building on their successes with digital shipbuilding, in 2018, Newport News Shipyard partnered with 3D Systems, the originator of 3D printing [5]. Through this partnership the Navy plans to migrate from traditional to more AM methods. This includes

developing ways to design, build, and certify 3D printed parts quickly and efficiently. The ProX DMP 320 metal printer is the first 3D printer installed at a U.S. Navy Shipyard. The printer is used to build marine-based alloy replacement parts for castings, valves, housings, and brackets [4]. Following the addition of the ProX DMP 320 printer (Figure 1), the Navy approved its first additive manufactured part to be installed aboard a warship. The aircraft carrier USS *Harry S. Truman* (CVN 75) installed a prototype metal drain strainer orifice for its steam system. These milestones mark huge turning points in the Navy's effort to "design for affordability," while building at higher rates with reduced cost per unit [6]. Ultimately, the use of AM will optimize the Navy's processes for design, maintenance, and lifestyle management of its assets.



Figure 1. ProX DMP 320 metal printer. Source: [7].

#### **D. ADDITIVE VERSUS TRADITIONAL MANUFACTURING**

Since AM emerged within its industry, many manufacturers have performed research as to whether AM or traditional manufacturing (TM) is more beneficial in terms of cost, part reliability, and production volume. In this section, we will explore the many factors that contribute to both manufacturing methods, and the pros and cons of each.

AM is the process of creating a model from an electronic or computer-aided design (CAD) model then constructing that model layer by layer. TM is a subtractive manufacturing method whereby material is either removed via machining, drilling, grinding, or cast in a mold [8]. Depending on a manufacturer's needs of a part, either method is acceptable. Historically, TM has been used to produce parts at high rates and in high volumes. AM offers the ability to produce complex parts at low rates and in low volumes. This is commonly seen during the process of prototyping a model. During prototyping, a model is built, tested, and optimized before it is mass produced. In TM a machine may need to be re-tooled in order to make necessary changes to the final part. This process can take weeks, and sometimes months, to complete. On the other hand, AM does not require re-tooling. Changes can be made electronically to a CAD file. Simulations may be run prior to printing in order to optimize a part. When considering prototype production, AM saves on time, material, and labor costs [8].

Production waste includes any material left unused in the process to construct a final model [8]. Waste material can be found in both TM and AM. In TM, large amounts of scrap material are used to produce parts. Although most of this material is recycled, it still requires large amounts of time and labor to do so. In AM, only what is extruded from the printer itself is used to build the model. In some cases, such as in raw powders, this material can be used several times before needing to be discarded for good. With regard to production waste, AM is a more efficient method [8].

When it comes to large production volume, many argue that AM is simply "too slow" to keep up with TM methods that have led the industry for decades. Since AM prints a model layer by layer, it can take several hours to construct a model that is only a few millimeters in length, width, and height.

For AM methods to produce near the same capacity as TM, the actual volume of the part must be relatively small. Several factors must be considered when deciding on a manufacturing method. This includes “cost, part complexity, material usage, material property requirements, time, energy consumption, sustainability” and many others [8]. There is not one method that works for every industry. A study from Hopkinson and Dickens et al. modeled the cost of AM versus a TM technique (particle injection molding). The study revealed that AM costs less than particle injection molding for small production volumes (<10,000). At commercial scale production levels, TM dominates manufacturing, however, AM is better suited to fabricating tooling and fixtures with complex geometry at smaller volumes [8].

The scale of parts and complexity must be considered when comparing AM to TM. The scale of parts in AM are restricted to the size of the printer. Entry level printers can range from several inches in area, while production level printers can range from several feet in area. The type of printer used will determine how large a part can be. TM is less restricted by part size. This has no restriction on AM as things as small as gameboard pieces to as large as houses have been built using AM techniques.

The complexity of a part is a significant benefit of AM. As shown in Figure 2, AM offers much more design proficiency as well as complex geometries. Figure 2 shows the original component (made up of several parts assembled/ welded together) and the two resulting 3D printed parts that are purpose-built and designed exclusively for the application without regard to TM concessions. AM allows the capability to print with multiple integrated parts, gradients of colors, and a wider range of materials other than resin and metals [9]. There are some parts that are difficult, if not impossible to make using TM methods. Manufacturers mostly use AM for rapid prototyping, as changes can be made quickly before a part is mass produced.



Figure 2. Geometrical complexity of AM versus TM part. Source: [9].

The downside of AM is that little is known about the mechanical properties and quality control of printed parts. “The complex geometry, internal lattice structure, surface finish, layer orientation, and topology optimization all contribute to the mechanical aptitude of an AM part” [8]. Loose powders commonly found in AM parts pose a health and safety risk and can negatively impact the mechanical and structural integrity of a part. Similarly, the size, orientation, sharpness, location, and type of defects also found in TM can have an undesirable effect on part quality [9]. Because TM methods have been around for centuries, quality control (QC) and quality assurance (QA) measures have been put in place and improved over time to produce the highest quality parts. Batch testing is the most widely used QA/QC measure to test mechanical properties in TM. During these tests, one part is reflective of all parts within that same batch. This method is more difficult to use in AM as more dependent variables must be considered. Best part imperfections, standard mechanical testing cannot detect “volume mismatch, layer removal and undesirable internal finish” [8]. However, several studies exist highlighting many consistencies between AM parts using the same print parameters.

Although it is difficult to produce the volume of parts with AM compared to TM, AM is capable of producing parts with enhanced mechanical properties through the unique technique in which they are processed. AM parts contain microstructures that cannot be achieved through TM processes alone. The intent of this paper is to present a body of work that helps bridge the gap between legacy methods of TM and state-of-the-art methods used

in AM to help researches develop more sustainable techniques that yield the desired outcomes for specific part application. In doing so, the mechanical and microstructural properties of AM versus TM maraging steel are studied through experimentation using diverse feature dimensions and subsequent test of properties, as well as observation of microstructural details.

#### **E. CASE STUDY: SELECTIVE LASER MELTING**

Selective Laser Melting (SLM) is an AM technique widely used in 3D metal printing. SLM has revolutionized 3D metal printing due to its ability to produce near dense parts with mechanical properties that meet, and in some cases, exceed those of TM. Though, one of the most important factors in achieving great properties is ensuring print parameters are set accordingly for the specific metal powder used during printing to control the amount of porosity present in a sample. This case study aims to explain the importance of using high quality powder in conjunction with the proper print settings to achieve parts that contain high density and minimum porosity.

In this process, a high power-density laser is used to melt and fuse metallic powders, as seen in Figure 3 [10]. Powder is melted by the laser beam, adhering to the previously laid layer. Finally, the build platform lowers by the preset layer thickness and a new layer of powder is applied. This process repeats until a 3D part is fully printed. Many factors affect the quality of a part. In general, print parameters and powder morphology have the biggest influence on the ultimate material properties. More specifically, laser power, scan rate, and layer thickness are major parameters that affect product properties and the number of trapped pores. Print parameters specific to this research will be discussed in more depth in follow on chapters.

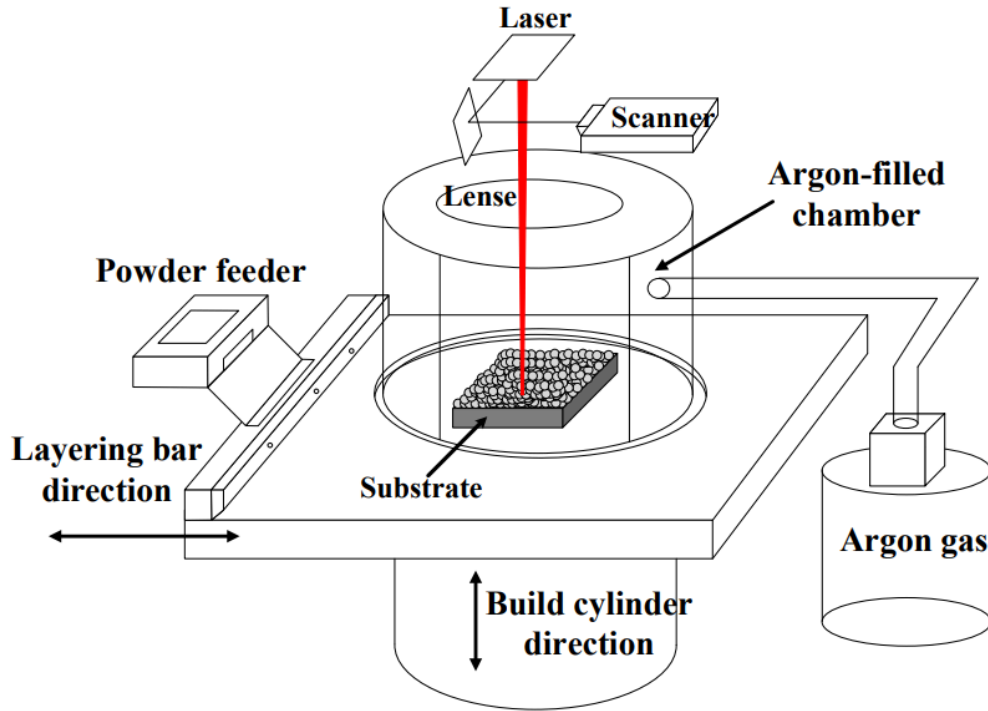


Figure 3. Schematic of SLM system. Source: [10].

A laser beam is used in the SLM process. A laser strikes the surface of the powder bed and energy is dissipated. Some of the particles absorb the laser energy, while the remaining energy is scattered away [10]. Higher absorptivity leads to more reflections of laser radiation between powder particles. Higher packing densities have increased absorptivity as seen in Figure 4. Going from (a) to (d), the powders change from a fine powder with uniform particle size in (a) to a powder with a distinct bimodal distribution of particles in (d) (i.e., the powders are coarsening). How fine or coarse the powders are will affect the packing density. The finer the powders, the higher the packing density and absorptivity results (i.e., more energy absorbed). Therefore, SLM conducted on fine powders leads to dense printed parts while the same conducted on coarse powders leads to a porous part [10].

Irregular pores are formed due to a lack of fusion or melting of powder particles [10]. Insufficient laser power leads to inadequate heat input which results in a lack of fusion between particles. The size of pores depends on powder morphology. In a study performed

by Jeon et al., porosity increased with the size of non-spherical powder mixed with spherical powder. “The difference in pore density was caused by variations in powder packing density. When large non-spherical powders are mixed with spherical powders, various pores are generated as a result of reduced powder packing density” [10].

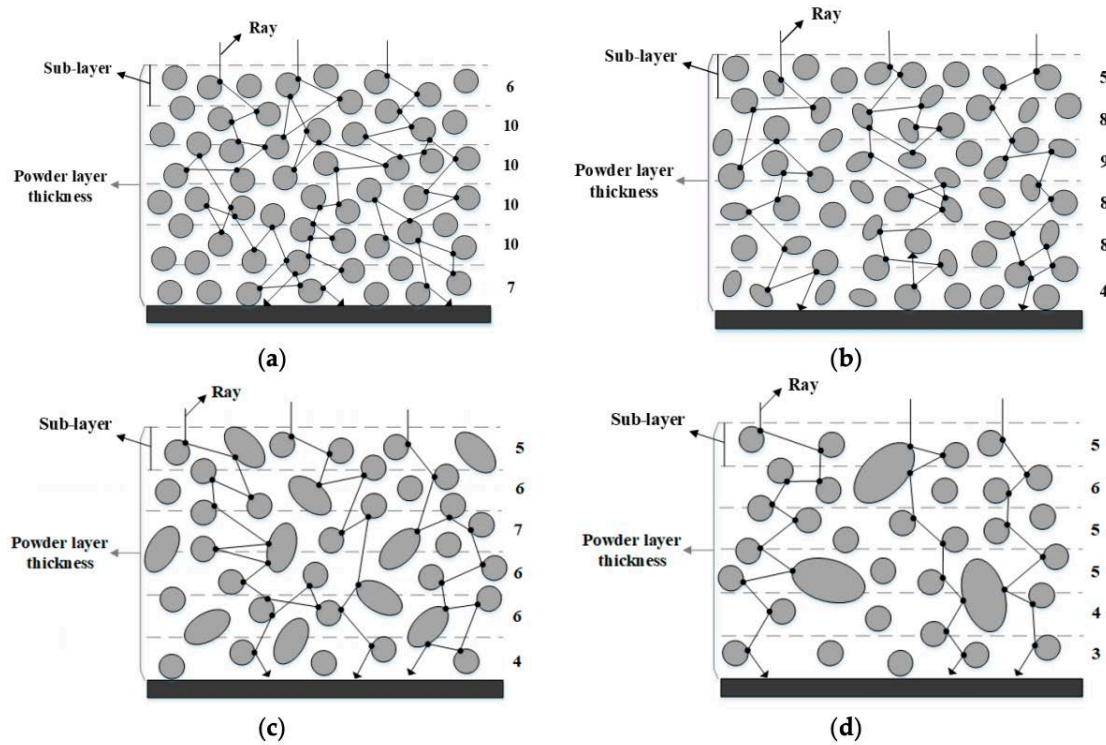


Figure 4. Model of four powders with different packing densities. Source: [10].

Another cause of porosity is the presence of zones depleted of particles (PD) during the printing process. A PD zone occurs when “material from the melt pool formed from laser beam interaction with the bulk material detaches from the neighboring powder. This forms an agglomeration separated from the bulk which increases the overall porosity of the build,” as seen in Figure 5 [10]. The shape and size of these agglomerations affects the shape and size of PD zones ultimately changing porosity in a build.

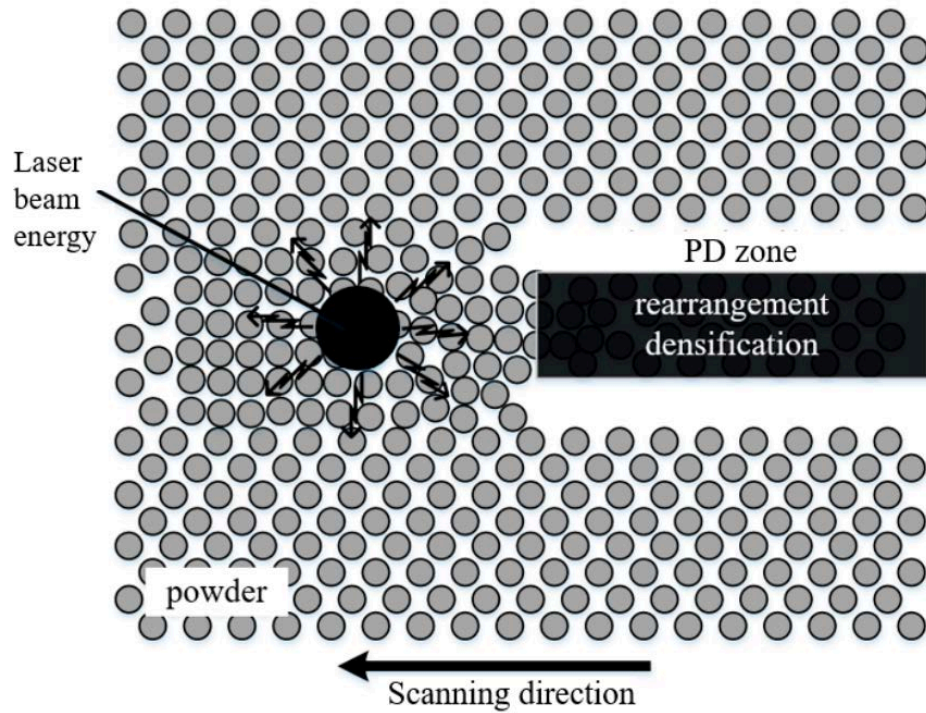


Figure 5. Schematic of rearrangement consolidation kinetics. Source: [10].

An increase in layer thickness and scan rate also causes an increase in porosity. High heat concentration is required in order to obtain a fully dense layer. Increased layer thickness and scan rate reduces heat concentration, therefore increasing the likelihood of porosity [10]. Dense layers are formed when a portion of the previous layer is re-melted. High heat concentration is required for this to take place [11].

Since its induction in 1981, the SLM technique has improved drastically in an effort to reduce porosity and create more fully dense parts [12]. SLM remains an attractive method as it allows for high resolution parts containing less weight and higher strength. There is not a part that exists that cannot be built via SLM methods [13]. Additionally, SLM is ideal for parts that need to be produced within hours at reduced cost. This is likely the case for small scale productions. AM via SLM requires little human interaction and yields lower amounts of material waste compared to TM. This is a key benefit for manufacturers looking to save money in processing costs. As will be seen in later chapters,

SLM is capable of producing parts with mechanical strengths comparable to those produced from TM methods.

The correct selection of fabrication parameters will enable the generation of parts free of defects (such as pores) and the transition to AM technologies that could not only provide high quality components but to also shorten the time required to resupply the fleet. The many variables involved in the AM process have several impacts on the mechanical and microstructural properties of a part. These variables must be optimized to ensure the desired properties are achieved during printing.

## **F. OBJECTIVES**

This research aims to compare the mechanical and microstructural properties of TM and AM (specifically SLM) maraging steel produced parts by conducting tensile tests and analyzing the results. Similarly, SEM analysis will be performed on raw powder material, tensile fracture surfaces, and cross sections. Analysis of data will be used to provide further insights on the quality and properties of parts generated by AM of maraging steel.

Additionally, the effects of feature size in the properties of AM parts printed here at NPS has not been widely explored. The size and quality of features will be examined in order to make correlations between feature size, dimension of features, as well as mechanical and microstructural properties as it pertains to feature sizes. Feature sizes range from 3 mm to 0.1 mm for both as-printed (AP) and heat-treated (HT) samples. The hardness, grain structure, and porosity of both samples will be analyzed to determine adequate print parameters.

Experimental methods for tensile tests, as well as results and discussion of parts printed at Albany Marine Logistics Base and analyzed by NPS are enclosed in Chapter II. Experimental methods and mechanical and microstructural properties as they relate to feature sizes of specimens printed at NPS laboratories are discussed in Chapter III. The conclusions and recommendations are presented in Chapter IV.

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## II. MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF MARAGING STEEL TENSILE SPECIMENS

This chapter presents all the information gathered in regard to tensile specimens printed at Marine Corps Logistics Base Albany analyzed at NPS. It also addresses the experimental methods employed to fabricate samples, prepare them for analysis, and explain the instruments and techniques employed to characterize and test them. Additionally, this chapter presents the data gathered, and its analysis and discussion of those results. The chapter ends by summarizing the main milestones and learnings achieved.

### A. EXPERIMENTAL METHODS

#### 1. Background

An EOS M400 SLM printer located at the Marine Corps Logistics Base Albany, GA was used to print maraging steel tensile samples. Maximum laser output power was 1000 watts. Laser power varied depending on the current location of the laser relative to the part. A scanner was used to direct laser emissions. Powder layer thickness was controlled by the height of the build platform. Argon gas was used as a shield to create an inert environment during printing and prevent oxidation. Raw powders used were EOS MS1, maraging steel 300 (18Ni-300) atomized powder designed specifically for printing maraging steel parts in EOS printers. The chemical composition of the powder is listed in Table 1.

Table 1. EOS MS1 raw powder composition. Source: [14] .

| Element | Fe      | Ni   | Co  | Mo   | Ti   | Al   | Cr   | C    | Si   |
|---------|---------|------|-----|------|------|------|------|------|------|
| wt%     | balance | 17.3 | 9.1 | 2.85 | 1.25 | 0.05 | 0.73 | 0.26 | 0.05 |

Initially, seven tensile specimens as well as, MS1 raw powder were provided for mechanical and microstructural analysis. The parts were printed as 16 mm diameter and 130 mm long bars with circular dog bone shapes prescribed by ASTM standard E8/E8M-

13 [15]. Four specimens were AP, as seen in Figure 6, and three specimens were HT, as seen in Figure 7. Printing conditions are listed in Table 2. AP specimens were received directly from the print chamber. HT specimens were printed, heated to 490°C for 6 hours, then furnace cooled.



Figure 6. AP tensile test specimens.



Figure 7. HT tensile test specimens.

Table 2. EOS M400 printer settings

| Volume Rate              | Layer Height | Powder Dosing | Pressure | Scan Rate  |
|--------------------------|--------------|---------------|----------|------------|
| 5.5 mm <sup>2</sup> /sec | 5.5 um       | 120 – 130 %   | 280 Pa   | 260 mm/sec |

## 2. Tensile Test Preparation

Tensile properties were observed using the Instron 5982 Tensile Tester IAW ASTM E8 (Figure 8). Testing rate was 2 mm/min. Custom stainless-steel sample holders were designed and manufactured in house to support the circular shape of tensile specimens. The INSTRON 5982 was originally equipped with clamps to hold flat rectangular shaped specimens.



Figure 8. Instron 5982 used to measure tensile properties of parts.

### 3. Metallographic Sample Preparation

After tensile testing, specimens (seen in Figure 9) were cut using the Secotom-10 cutting saw to expose cross-sections. Samples were then prepped for microstructural analysis and microhardness testing by mounting in an epoxy resin consisting of 15 parts resin to 2 parts hardener by volume. Samples were polished to remove surface scratches. Polishing consisted of 5 minutes with 500 grit, 10 minutes with 800 grit, 30 minutes with 1200 grit, 60 minutes with 2500 grit, and 2 hours with felt padding adding 1um alumina polishing compound every 5 minutes. Polishing speed was 260 RPM using 5 lbs. force. The fracture surfaces of all samples were cut.

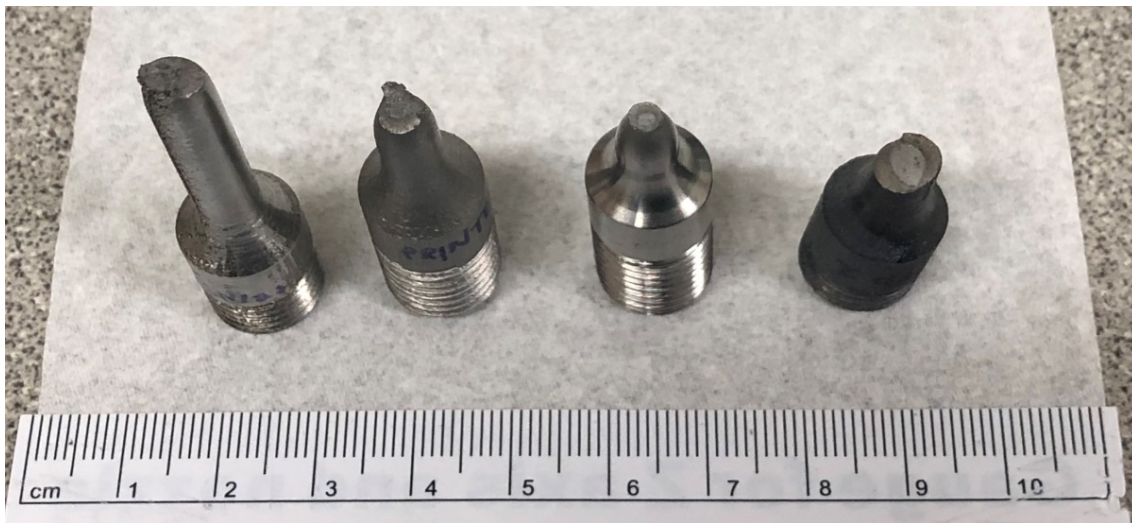


Figure 9. Fracture surfaces of tensile specimens

### 4. Microhardness Test Preparation

Hardness testing was conducted using a Durascan Microhardness Tester. Grid size was set to 0.1 mm x 0.1 mm. The test method used was hardness according to Vickers-HV1. The test method measures the depth of penetration of an indentation under a specified load [3]. In this case a test load of one-kilogram force (kg-f) was used. Series measurements were taken using horizontal and vertical diamond-shaped indentations. Twenty-six horizontal and eighteen vertical test points were taken. Hardness measurements were

conducted on the sample cross-section from the bulk material to the fracture area. The Vickers scale was used for all hardness measurements.

## **5. Microscopy Preparation**

Cross-section samples underwent visual inspection using an Epiphot 200 reflective optical microscope. Cross-section, fracture surfaces, and MS1 raw powder samples were mounted using double-sided adhesive in preparation for microstructural and compositional analysis using a Neon 40 dual-beam scanning electron microscope (SEM) with attached energy-dispersive spectroscopy (EDS) detector. Samples were cleaned using ethanol prior to being placed inside the SEM chamber. EHT was set to 20 electron volts (eV), with a working distance of 5 mm.

## **B. RESULTS AND DISCUSSION**

This section discusses the results of tests performed on tensile specimens. Characterization of raw powders was performed, and its effects on surface roughness are analyzed. The mechanical and microstructural properties of all tensile specimens are examined. This includes yield strength, ultimate tensile strength, microhardness, optical microscopy, and SEM analysis. Porosity is examined through SEM analysis. Austenite reversion is defined, and evidence of this process presented in HT samples. Print parameters and their effect on energy density is investigated. The energy density of samples studied in this experiment are compared to those found in literature. Print setting recommendations specific to this research are suggested.

### **1. Powder Precursors**

Analysis of raw gas atomized powder using the SEM in Figure 10 revealed that powders consisted mostly of spherical  $\mu\text{m}$  size particulates. Few irregularities were present. Mean particle size was 13.871  $\mu\text{m}$  with a maximum particle size of 83.655  $\mu\text{m}$ , and a standard deviation 10.903  $\mu\text{m}$ . There was evidence of sintered particulates which were commonly found in powders produced by atomization routes.

Gas and water atomization are the two most common powder metallurgy techniques used in the processing of high-speed steel powders. Gas atomization generally caused

spherical particle formation, while water atomization produced more irregular shaped or angular particles [16]. SLM of gas atomized particles resulted in a homogenous sintered structure with high surface density unlike SLM of water atomized particles which resulted in poor surface density with large inter-agglomerate pores. Parts built from gas atomized powders typically lead to higher packing densities, thus lower porosity as compared to water atomized powders [17]. To produce near dense parts, powders mostly spherical in shape were used during this experiment.

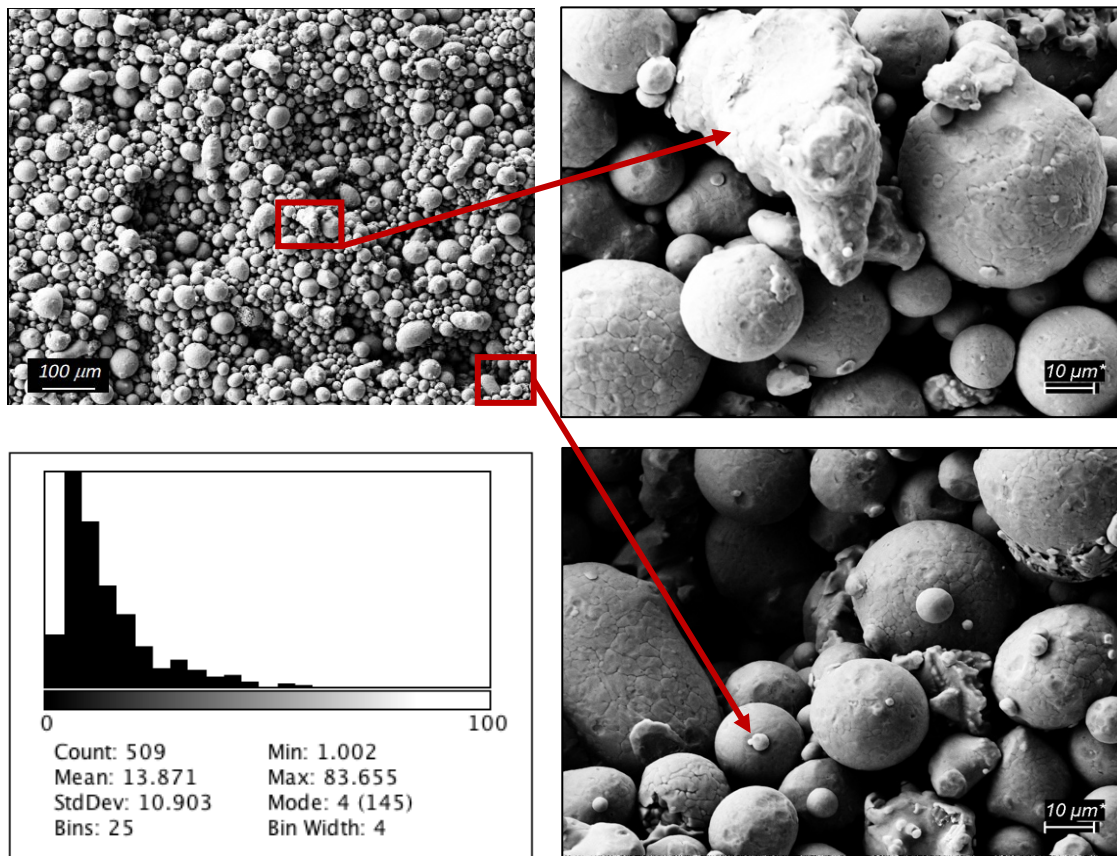


Figure 10. SEM images of raw MS1 powders.

## 2. Surface Roughness

A laser power of 148 watts, scan speed of 400 mm/s, and a spot diameter of 90 μm was used to print the parts in Figure 11. Visual observation revealed a high amount of

surface roughness commonly found in AM parts. In the case of these tensile specimens, the rougher surfaces were generated as part of the support structure during printing, not a result of the printing conditions. A study by Demir et al. showed that both pulse duration and remelting directly influenced the average surface roughness of SLM parts. Pulse duration has a direct impact on pulse energy, which is defined as “the laser power per unit area” [18]. From pulse duration, the energy density of a part can be calculated. Energy density (E) is defined as the laser power (P), divided by the laser scan rate (V), times the spot diameter (d), as seen in Equation 1.

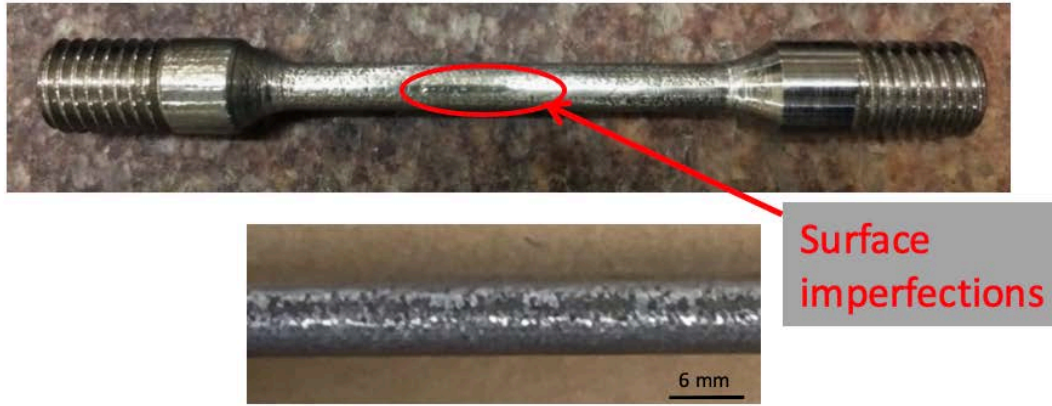


Figure 11. Surface roughness seen on an AP sample.

$$E = \frac{P}{V \cdot d}$$

Equation 1: Equation for Energy Density. Source: [19].

The calculated energy density for this experiment was 4.11 J/mm<sup>2</sup>. Energy density is important because it reflects the heat absorbed by the powder during SLM. The more heat absorbed, the more fully melted the powders become, ultimately resulting in denser parts. In a study done by Bai et al. on AM maraging steel 300, energy density increased linearly with increased laser power when scan speed and scan line spacing remained

constant [20]. One may assume that higher laser power results in denser parts. This could not be further from the truth, as when the laser reaches a high enough power, vaporization and spattering occur within the metal molten pool. These effects cause insufficient amounts of molten metal to fill molten tracks, leading to voids and decreased relative density.

As scan speed and scan line spacing vary, so does energy density. At high scan speeds, a decrease in relative density is seen. Similarly, when scan speed is too low, relative density also decreases. This is due to insufficient cooling of the metal in both cases, correlating to input power to the molten pool being either too high or too low [20]. Scan line spacing works in the very same way, in as if it is increased or decreased too much will lead to voids and unmelted powders, respectively, in the overlapping zone, as seen in Figure 12.

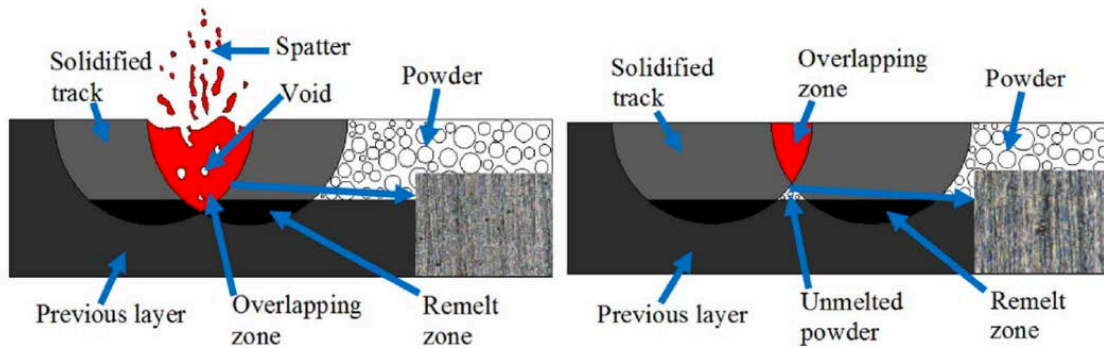


Figure 12. Effect of scan spacing on overlapping zone. Source: [20].

The image on the left shows the effects of excessive input power as the molten pool begins to spatter, causing voids. The image on the right shows the effects of insufficient input power as the molten pool is too low, resulting in unmelted powder [20]. Figure 13 displays the optimal parameters of laser power, scan speed, and scan line spacing producing relative densities of greater than 99% in maraging steel 300 [20]. Through interpolation it was calculated that given the laser power of 148 Watts and scan speed of 400 mm/s used in this experiment, a scan line spacing of 0.09 mm would be required to produce parts of greater than 99% density.

| Number | Power/W | Scanning speed/mm s <sup>-1</sup> | Scanning space/mm |
|--------|---------|-----------------------------------|-------------------|
| 1      | 100     | 300                               | 0.05              |
| 2      | 100     | 400                               | 0.07              |
| 3      | 100     | 500                               | 0.09              |
| 4      | 100     | 600                               | 0.11              |
| 5      | 130     | 300                               | 0.09              |
| 6      | 130     | 400                               | 0.11              |
| 7      | 130     | 500                               | 0.05              |
| 8      | 130     | 600                               | 0.07              |
| 9      | 160     | 300                               | 0.11              |
| 10     | 160     | 400                               | 0.09              |
| 11     | 160     | 500                               | 0.07              |
| 12     | 160     | 600                               | 0.05              |
| 13     | 190     | 300                               | 0.07              |
| 14     | 190     | 400                               | 0.05              |
| 15     | 190     | 500                               | 0.11              |
| 16     | 190     | 600                               | 0.09              |

Figure 13. Orthogonal experimental factor design (three factors and four levels). Source: [20].

To reduce surface roughness and stress concentration points, one AP part was sand blasted for 2 minutes as seen in Figure 14 on the right. Sandblasting is a mechanical abrasive treatment that uses sand particles and compressed air to smooth out a rough surface in this case. Through the technique of sandblasting, impurities and weak boundary layers are eliminated, and or reduced [21]. After sandblasting, parts were cleaned using compressed air and wiped down with laboratory dry clothes. No solvent cleaning was performed, therefore chemical composition remained the same between AP and sandblasted samples.



Figure 14. AP versus sandblasted surface.

### 3. Mechanical Properties

Figure 15 illustrates that the sandblast surface treatment had a small effect on the tensile properties of AP maraging steel. Elongation also changed with values between 8.6–10.3%. These values were similar to EOS mechanical properties for material, which are rated at an elongation of 11–13%. In a study by Kempen et al. on the microstructural and mechanical properties of maraging steel 300 after SLM, elongation values ranged from 11.4–15.2% [3]. Our elongation percentage is slightly lower than EOS and Kempen et al., however, printing parameters vary, affecting mechanical properties.

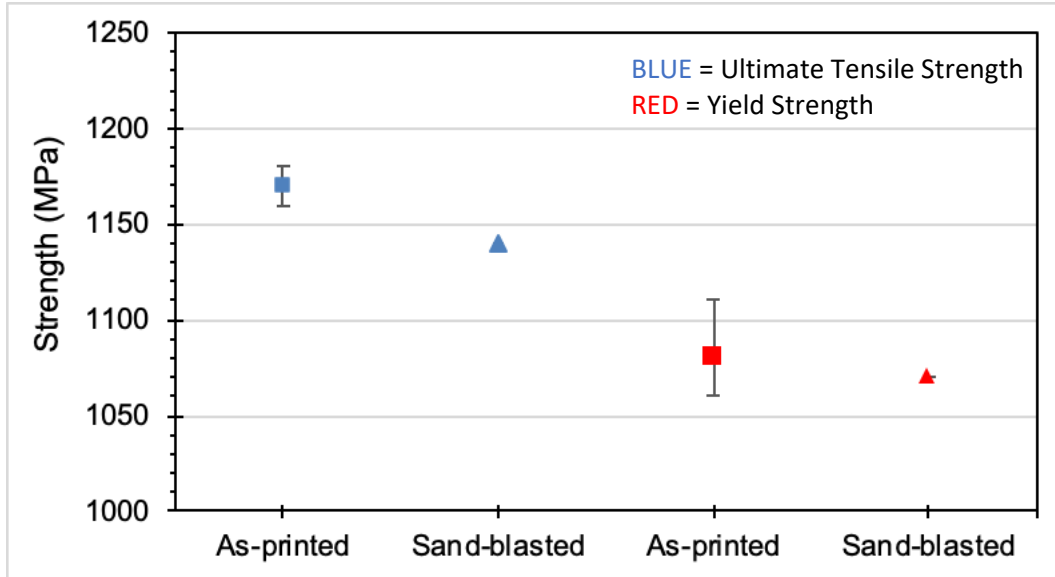


Figure 15. Strength of AP samples compared to sandblasted samples.

Several locations resembling lack of fusion were seen in AP samples, as shown in Figure 16. Crack initiation may have been caused by a lack of fusion. Spalling due to radial expansion of stress may also have led to crack initiation. Crack initiation appears from the center of the fracture surface on the sandblasted sample seen in Figure 17. Crack initiation from the center, resulting in a “cup and cone” shape is preferred. This feature signifies uniform stress during necking as a result of tensile stresses. The presence of stress concentration points and/or voids is exploited when the fracture surface results in any form other than a “cup and cone.” Although sandblast treatment changed failure mode, the location of crack ignition had a small effect on the mechanical properties of AP samples.

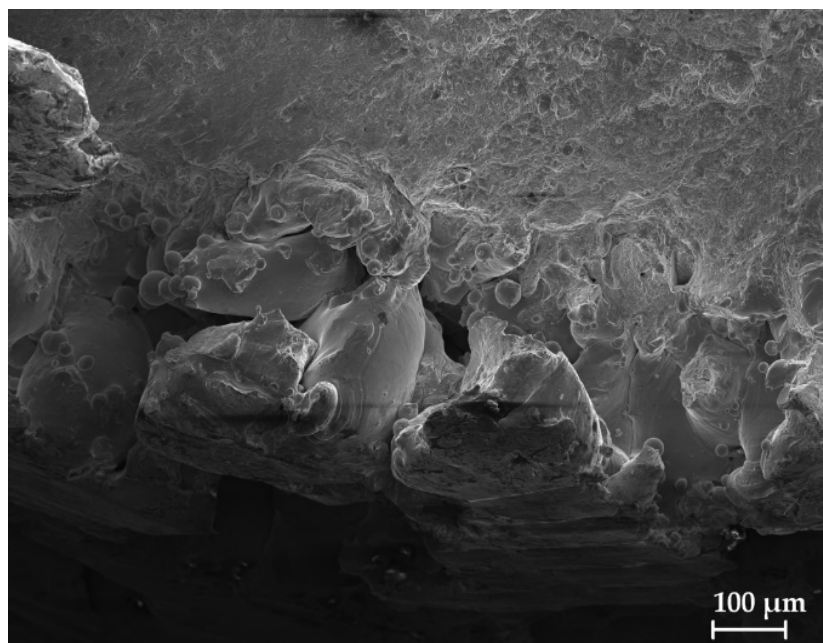


Figure 16. SEM images of fracture surface of AP sample showing lack of fusion.

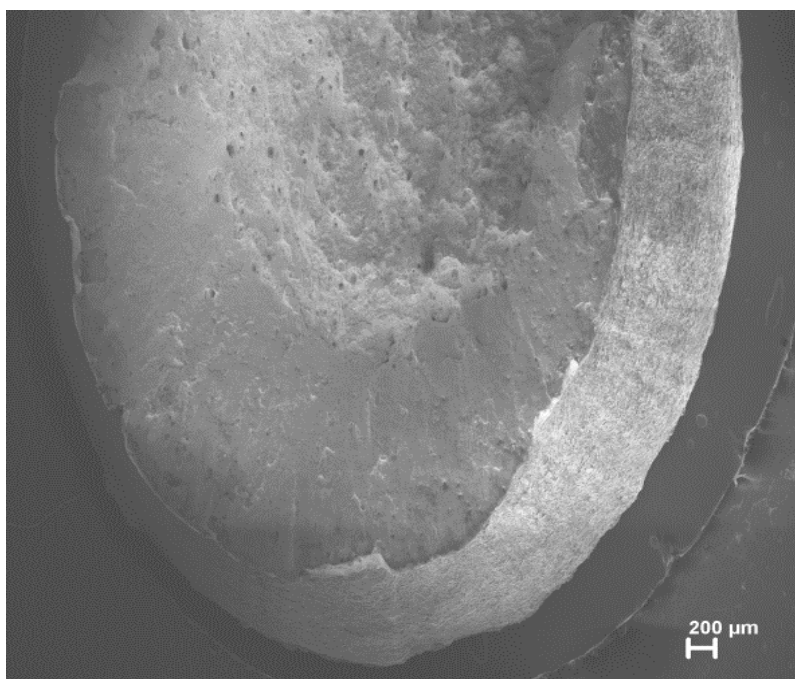


Figure 17. SEM image of fracture surface of sand-blasted tensile sample.

A closer look through the SEM seen in Figure 18 shows a small inclusion of an AP cross-section on the left, and an area containing lack of fusion on an AP fracture surface on the right. Both features are a result of unmelted powder particles. Lack of fusion and unmelted powder seen both on the surface and within the cross section of AP samples show that print quality was consistent throughout. It is likely that these voids led to stress concentration points resulting in potentially premature material failure. While these features may cause a decrease in strength, they were seldomly seen across all AP samples and had little effect on mechanical properties, as illustrated in Figure 19.

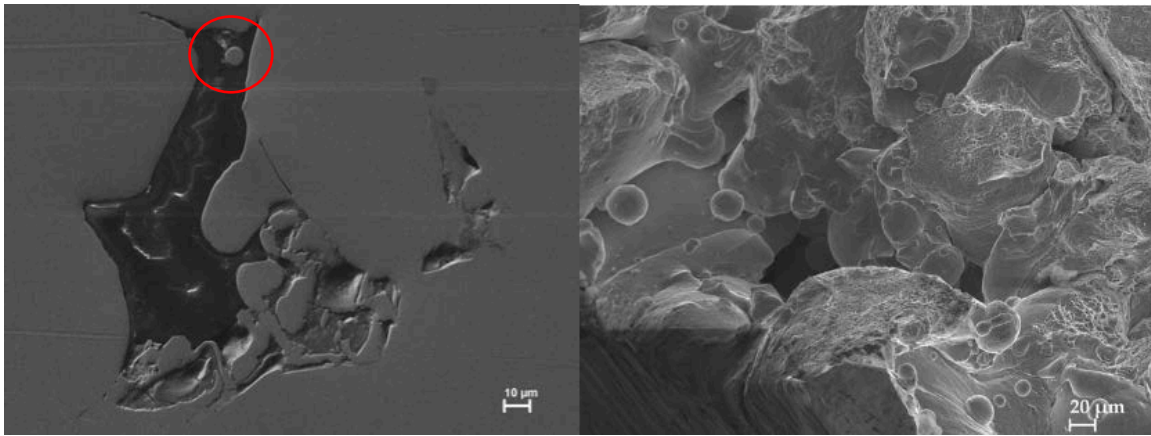


Figure 18. AP cross-section versus fracture surface sample.

Mechanical properties of AP samples were also compared to computer numerical control (CNC) and HT samples as seen in Figure 19. The AP samples outperformed CNC samples in total strength by roughly 200 MPa. The HT sample had twice the amount of yield and tensile strength when compared to AP samples. “The ultimate tensile strength of AP samples (1200 MPa) was similar to values rated by EOS for printing this material ( $1100 \pm 100$  MPa). Similarly, an ultimate tensile strength of 2000 MPa for the HT sample met EOS standards of  $1950 \pm 100$  MPa after age hardening” [3]. Higher yield and ultimate tensile strengths of AP samples are due to the natural aging process of SLM. The initial melting, then subsequent re-melting of layers contributes to the unique microstructure of SLM parts. SLM parts are known to have a very fine microstructure of elongated grains

because of cellular solidification. These grains have a diameter of less than 1  $\mu\text{m}$ . As a result, high strength and hardness values are achieved through SLM [3].

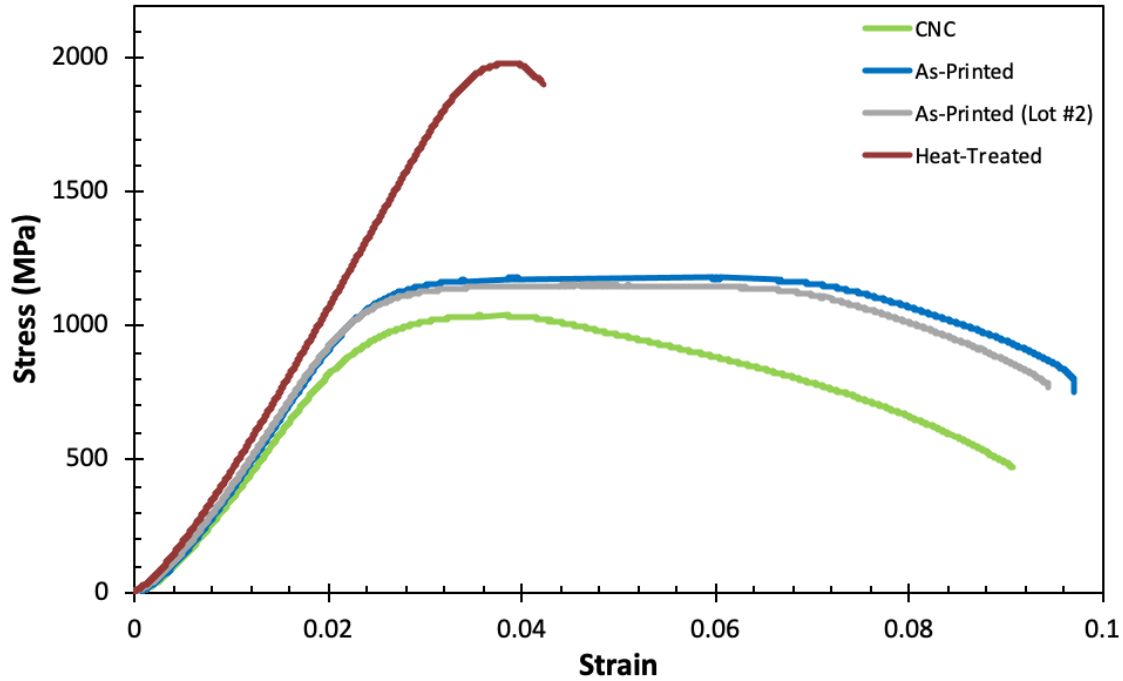


Figure 19. Stress-strain curve comparing mechanical properties of CNC, AP, and HT samples.

#### 4. Microhardness

Microhardness tests were performed on the cross sections of AP and HT samples. Results of these tests are displayed in Figures 20 and 21 for AP and HT samples, respectively. AP samples had an average hardness of 377 HV1 with a maximum of 385 HV1 and a minimum of 370 HV1. Standard deviation was 4.2 HV1. Our samples were printed with a layer thickness of 5.5  $\mu\text{m}$  and scan speed of 260 mm/s. According to a journal published by Yasa E. et al., hardness values for the same maraging steel printed using the SLM technique had slightly higher values. Their printed samples had an average value of 400 HV0.5 with a maximum of 412 HV0.5 and a minimum of 390 HV0.5 [22]. Parts were printed with a layer thickness of 30  $\mu\text{m}$  and scan speed of 200 mm/s. To produce higher

microhardness values in our experiment, cooling rates must be increased, thus layer thickness and scan rate adjusted appropriately. It is worth noting that although cooling rates were the primary factor that influenced microstructural properties, mechanical properties could have been enhanced with an increase in scan speed [22]. A higher scan speed may have resulted in a reduction in hardness due to increased porosity.

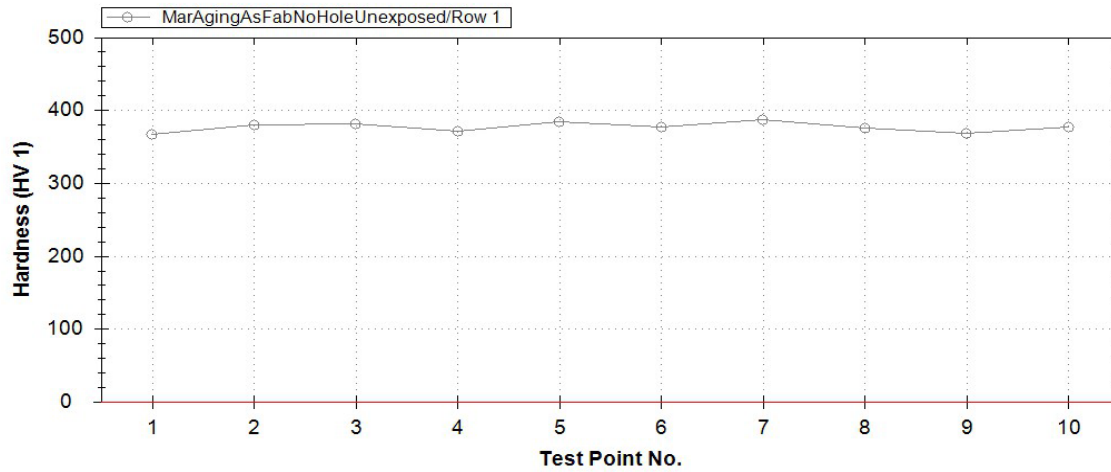


Figure 20. Microhardness test results for AP samples

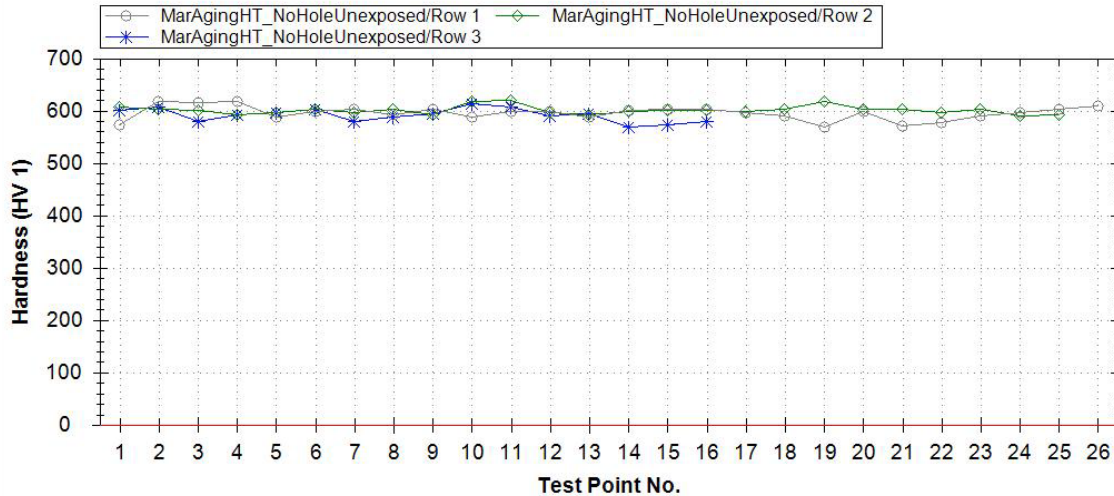


Figure 21. Microhardness test results for HT samples.

Heat treatment of maraging steel is essential in achieving excellent strength, toughness, and hardness properties. During treatment a uniform distribution of nickel-rich intermetallic precipitates is formed, giving the metal increased strength through the aging of martensite [22]. In comparison to AP samples, HT samples had significantly higher hardness values as expected. As mentioned in the previous chapter, our samples were heat treated for 6 hours at 490°C. HT samples had an average hardness of 596 HV1 with a maximum of 620 HV1 and a minimum of 568 HV1. Standard deviation was 11.9 HV1. When compared to research conducted by Yasa et al., the standard treatment applied to maraging steel after SLM was 3 hours at 480°C which increased hardness above 634 HV. The study stated that the optimal heat treatment was 5 hours at 480°C which produced higher microhardness values closer to 650 HV [22]. Exposure to increased temperatures for an extended amount of time can lead to over aging of the metal. This is likely the cause of lower hardness values in this experiment compared to Yasa et. al., as our samples were exposed for a longer period of time at a higher temperature.

Over aging may be suspected when hardness values decrease with prolonged heat treatment. During over aging the reversion of metastable martensite and coarsening of intermetallic precipitates takes place. These two phenomena together decrease the hardness as the part is kept at elevated temperatures for a prolonged time, [22]

## **5. Microstructural Properties**

As discussed in the previous section, there is a high possibility that austenite reversion occurred during heat treatment. Austenite reversion is inevitable at prolonged heat treatments (or higher temperature heat treatments) because the martensite is metastable and transforms to the stable austenite [22]. During austenite reversion, the redissolution of Ni, Ti, and Mo precipitates are released back into the Fe matrix. Due to the high Ni content in maraging steel, Ni-rich areas formed through segregation and cellular-dendritic growth are more likely to make this transition first [3]. It is important to note that all AP samples produced through SLM retain some form of austenite. Austenite reversion is measured through the change in austenite and martensite percentages similar to the values seen in Table 3. An increase in percent austenite after heat treatment is

evidence that austenite reversion has taken place. Further analysis is required to determine the percent of austenite and martensite conversion that took place during this experiment.

Table 3. Quantification of present phases through Rietveld refinement analysis. Source: [3].

|                        | Austenite $\gamma$ | Martensite $\alpha$ |
|------------------------|--------------------|---------------------|
| As produced            | 5.8%               | 94.2%               |
| Age hardened 480°C, 5h | 9.4%               | 90.6%               |

SEM analysis revealed further evidence that austenite reversion took place during heat treatment. Figure 22 shows an AP sample on the left which displays an intercellular structure formed through laser induced rapid solidification, a common feature seen in SLM of maraging steel. This microstructure features dendrites with secondary arms, as well as several cellular and columnar regions. This unique structure contributes to the excellent strength properties of maraging steel.

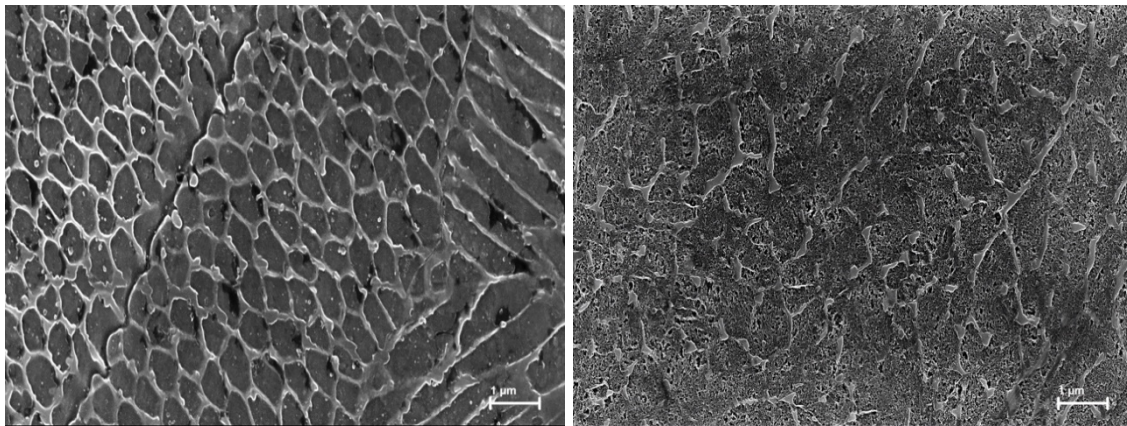


Figure 22. AP (left) versus HT (right) microstructure. Scale bar: 1  $\mu\text{m}$ .

In comparison, the figure on the right displays the microstructure of a HT sample. This sample contains a broken cellular structure. As austenite forms within cells and breaks through cell boundaries, the HT microstructure shown in Figure 22 is formed. As cellular structure becomes broken, strength decreases. Through SLM processes, martensite is

formed as layers are subsequently re-melted and cooling rate is relatively high. Martensite is a metastable phase; however, excessive heat treatment causes the transformation of martensite to austenite. When the heat treatment temperature is set above the austenite start temperature, austenite reversion occurs [23]. For maraging steel 300, this temperature is around 250°C. At temperatures between 455°C and 510°C, austenite reversion is slow enough to allow Ni-based precipitates to come out of solution [24]. “At temperatures higher than 510°C, the transformation rate of martensite to austenite is increased and precipitates do not form or dissolve back into solution” [23].

The interplay between the austenite and martensite phases makes maraging steel a unique iron alloy [25]. On one hand, too much austenite will soften the material, yet ductility will increase via transformation induced plasticity (TRIP). Corrosion resistance may also increase though this may be due more to phase homogenization rather than due specifically to austenite over martensite. With appropriate heat treatment, a mixture of the two phases can be achieved and precipitation of Ni rich nanometric precipitates together can lead to a high strength steel with an adequate level of ductility (i.e., high toughness material) [20].

Optical micrographs were taken on CNC and AP samples in Figure 23 and a HT sample in Figure 24 for comparative analysis. All images were taken on the middle cross-sections of samples. Through etching, laser track marks were made visible. Laser track marks were in the same direction of laser scanning during printing. Both pores and inclusions are seen on the cross-sections of AP and HT samples. The CNC sample contained a low density of pores at relatively smaller sizes. A higher density of pores at a relatively larger size were seen in the AP and HT samples. The largest pore size was approximately 40  $\mu\text{m}$  in the AP sample. New phases within the solidification cells and at cell boundaries are seen in both AP and HT samples. This is a major feature of the SLM process. The HT sample contained a smaller density of pores compared to the AP sample; however, pore size was larger than the AP sample. The largest pore size in the HT sample was approximately 90  $\mu\text{m}$ . These pores lessen the mechanical properties of the metal, especially in the HT sample where the maraging steel is more brittle than the AP sample. Despite this porosity, mechanical properties of the printed samples compared favorably

with TM (CNC) parts, and in all cases exceeded the tensile strength of TM parts. This fact is attributed directly to the printing process to which laser remelting likely plays a role. An open question is whether the cellular structure is directly responsible. An open question is whether the cellular structure is directly responsible or not.

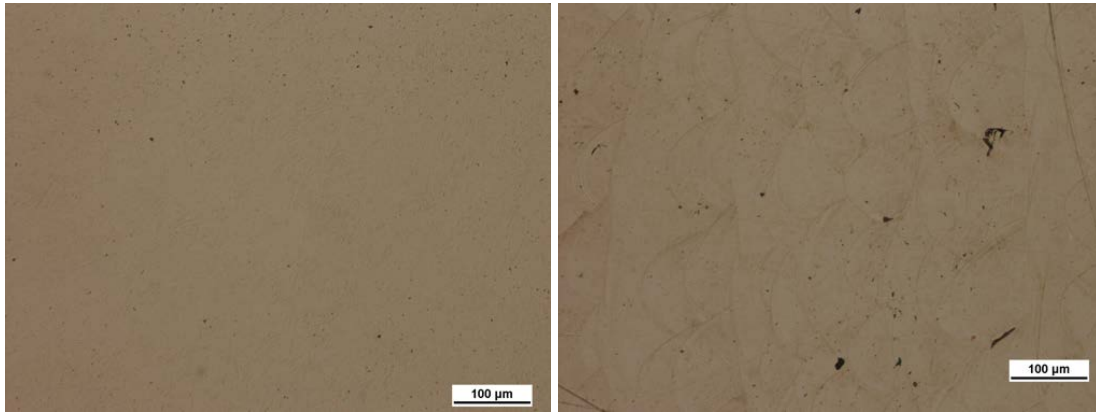


Figure 23. Optical images of the microstructure of a CNC sample (left) and an AP sample (right).

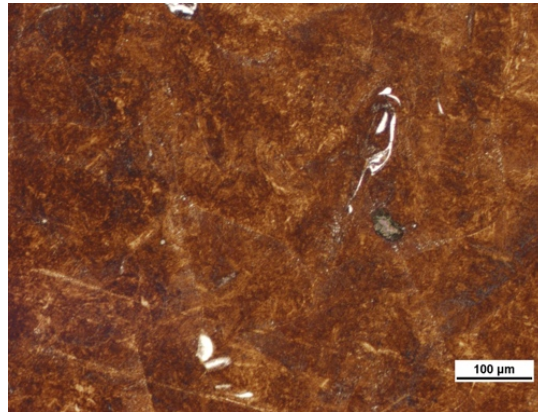


Figure 24. Optical image of HT microstructure.

## 6. Overall Results

In this chapter, we presented and analyzed the results of tensile tests and microscopy of specimens provided by Marine Corps Logistics Base Albany. Atomized powders used in SLM processes produced parts that exhibited higher yield and ultimate

tensile strength values than CNC parts. Samples contained surface roughness due to support structure removal. Sandblasting was used to mitigate the effects of surface roughness on mechanical properties. Tensile tests of AP and sandblasted samples revealed that surface treatment had a small effect on the mechanical properties of tensile specimens. Microhardness values were lower than those found in literature. The maximum AP hardness value in this research was 27 HV lower than that found by Yasa et al. Similarly, the maximum HT hardness value was 54 HV lower than values found in the Yasa et al. study.

The energy density of samples in this experiment was  $4.11 \text{ J/mm}^2$ , 176% higher than the average maximum energy density for maraging steel produced via SLM. A higher energy density may have led to spatter and voids within the overlapping zone, resulting in increased porosity and lower hardness values as observed.

Limited austenite reversion was seen in the microstructure of HT samples. HT samples were exposed at a higher temperature for a longer period of time when compared to optimal heat treatment of maraging steel found in literature. Heat treating samples at the optimal conditions of  $480^\circ\text{C}$  for 5 hours may have resulted in higher hardness values. SLM of maraging steel remains an excellent method for producing maraging steel parts. Post-printing treatment parameters, however, can be optimized to improve properties.

### **III. CORRELATION OF FEATURE SIZE AND MECHANICAL PROPERTIES ON MARAGING STEEL**

#### **A. EXPERIMENTAL METHODS**

This chapter discusses the part design and print parameters for maraging steel samples used to study feature size and quality. The EOS M100 printer is rated to print a minimum feature size of 0.1 mm [26]. Microhardness tests and optical microscopy were performed on feature sizes ranging from 0.1 mm to 3 mm for comparative analysis. Additionally, overall part quality to include porosity was analyzed and compared to similar samples found in literature.

##### **1. Part Design**

Samples were created using Solidworks 2018. A 25 mm in length, 5 mm in width, and 10 mm in height part was created with (2) 0.1 mm, (2) 0.5 mm, and (2) 0.8 mm non-cubic features on each sample. Another part with the same base dimensions but with different feature sizes was created. The second sample included (2) 1 mm, (2) 2 mm, and (2) 3 mm non-cubic feature sizes. All features were separated by 1 mm spacing. Each part was printed four times for a total of eight parts. Parts were saved in the standard triangle language (STL) format then transported to Magics software for AM configuration.

##### **2. Print Parameters**

The support structure for all parts was created using Magics software. In order to maximize strength a support structure was created. Support structures are a vital part of the AM process because they help offset the effects of residual tensile stresses created during printing. In the SLM process powders are heated in compression and cooled in tension which creates residual tensile stresses. These tensile stresses may lead to cracking, delamination, and distortion in a part once printed. The design of the support structure must be strong enough to support the weight of the part, but not too strong to prevent clean removal. Support structure design can range from multiple weaker joints to a few strong joints, or a combination of both. In this experiment we chose to use a combination of both

due to the geometry of the part and the strength of maraging steel. A grid support was used in combination with eight cone structure supports for each part.

The grid support was 3 mm in height printed using the thinnest layer possible. The grid support allows for the first layer of the part to be built evenly. The cone structures were used to anchor the grid supports so the weight of the part did not break the support structure. Cones were built up through the grid along the z-axis. They were placed in pairs along the length of the part, as seen in Figure 25. This experiment marks the first time MS1 powders were used on an EOS M100 printer at NPS, therefore, little is known about the thermal effects of printing maraging steel. In previous prints of Ti-6Al-4V, parts were known to warp during cooling. The same theory was applied during the design of the support structure for maraging steel parts. Cones were inserted to prevent warping of parts.



Figure 25. Live imaging of the support structure during printing of maraging steel parts.

All parts were built in the vertical direction along the z-axis with the least significant plane facing down. A stainless-steel build plate was used, as well as stainless steel settings. Default 316L stainless-steel settings were used to print all parts. Print parameters are shown in Table 4. The top layer specifies the top two layers of each build while the bottom layers are the four bottom layers.

Table 4. Process parameters for printing maraging steel on an EOS M100 printer using stainless steel settings.

|               | <b>Laser Power</b> | <b>Scan Speed</b> | <b>Scan Spacing</b> | <b>Layer Thickness</b> |
|---------------|--------------------|-------------------|---------------------|------------------------|
| Top Layers    | 54.6 Watts         | 600 mm/s          | 70 $\mu\text{m}$    | 40 $\mu\text{m}$       |
| Bulk Material | 77.1 Watts         | 827 mm/s          | 70 $\mu\text{m}$    | 30 $\mu\text{m}$       |
| Bottom Layers | 35.0 Watts         | 1594.3 mm/s       | 60 $\mu\text{m}$    | 80 $\mu\text{m}$       |

An image of newly printed parts is seen in Figure 26. Parts were removed from the support structure using traditional machining methods. Minor damage was done to the bottom plane during support structure removal. No cracks or warping were seen on any part. This would suggest that the grid support structure height of 3 mm with added cones was adequate to support the weight of the part, as well as assist in clean removal of supports. Note for all builds, the side faces are square while depth of each feature is equal to the depth of the bulk. As will be seen in later sections, these dimensions will play a role in the mechanical properties of the builds.



Figure 26. Maraging steel feature size samples printed from EOSM100.

### 3. Heat Treatment and Metallurgical Characterization

After parts were printed, two of each sample were heat treated. A Thermo Scientific Blue M furnace was used. Tube volume was 230 mL which required a 20-minute Argon flush prior to placing parts inside the tube. The argon flush was used to prevent the alloy from oxidizing from contact with environmental gases. Parts were heat treated to 490°C for 5 hours while Argon continued to flow. After 5 hours, parts were quenched by removing the tube, with the builds inside, from the furnace and placing the tube on a cooling rack in air at ambient temperature. Samples remained inside the tube and were left to air cool. Once heat treatment was complete, parts were ready to be mounted and polished.

All parts were mounted using a Struers CitoPress Mounting Press. Mounting material was ConduFast, an acrylic hot mounting resin with iron filler. Samples were auto polished with 500 grit paper until scratches appeared in the same direction. Polishing speed was 260 RPM using 5 lbf. Scratches appeared uniform after 5–10 minutes of polishing. Next, 800 and 1200 grit papers were used following the same methodology. Afterwards, samples were hand polished using 4000 grit paper until scratches were uniform. Polishing

speed was 120 RPM. Lastly, micro cloth paper was used with 1  $\mu\text{m}$  alumina for 5 minutes, then 0.5  $\mu\text{m}$  alumina for another 5 minutes or until the surface appeared shiny with minimal scratches.

Microhardness testing was conducted using a Durascan Microhardness Tester. The test method used was hardness according to Vickers- HV1. Point measurements were taken on all feature sizes, while series measurements were taken on the bulk material. Small (0.1 mm, 0.5 mm, and 0.8 mm features) samples consisted of 8 total point measurements. Large (1 mm, 2 mm, 3 mm features) samples consisted of 58 total point measurements. Both small and large samples underwent series measurements on the top, middle, and bottom portions of the bulk material.

Microstructural properties were observed using an Epiphot 200 reflective optical microscope. Test Method was HV1 and objective lens was 10x. Bright and dark field imaging was used to observe contrast differences between samples. Results of microhardness tests and optical microscopy are discussed in the next section.

## **B. RESULTS AND DISCUSSION**

### **1. Microhardness**

To see the effects of cooling rates on feature sizes, cooling rates were controlled while feature size remained variable. Figure 27 displays the microhardness data for all AP samples. The average hardness values for AP features, indicated on the graph with an “x” was 326.7 HV1 for 0.1 mm, 383 HV1 for 0.5 mm, 374.1 HV1 for 0.8 mm, 365.8 for 1 mm, 363.6 HV1 for 2 mm, and 360.2 HV1 for 3 mm features. Insufficient data was gathered on the 0.1 mm feature. This was mostly due to the indenter touching both the sample and mounting resin during testing. To correct for this, a smaller load could have been used, perhaps 0.5 HV. The smaller the feature size, the less data points were able to be gathered due to the small size of the feature relative to the indenter. Two data points in total were collected on the 1 mm feature for small samples compared to 32 data points total on the 3 mm features for large samples. Because of this, more data points, and in turn, more accurate data was gathered as feature size increased. Except for the 0.1 mm feature, smaller features had higher hardness values than larger features, thus allowing for the formation of

metastable phases. Additionally, because smaller features have faster cooling rates than larger features, grains have less time to grow. Smaller grain sizes due to rapid cooling leads to increased hardness. On the other hand, larger features have slower cooling rates, allowing more time for grains to grow and an equilibrium state to be reached, resulting in decreased hardness values due to larger grains.

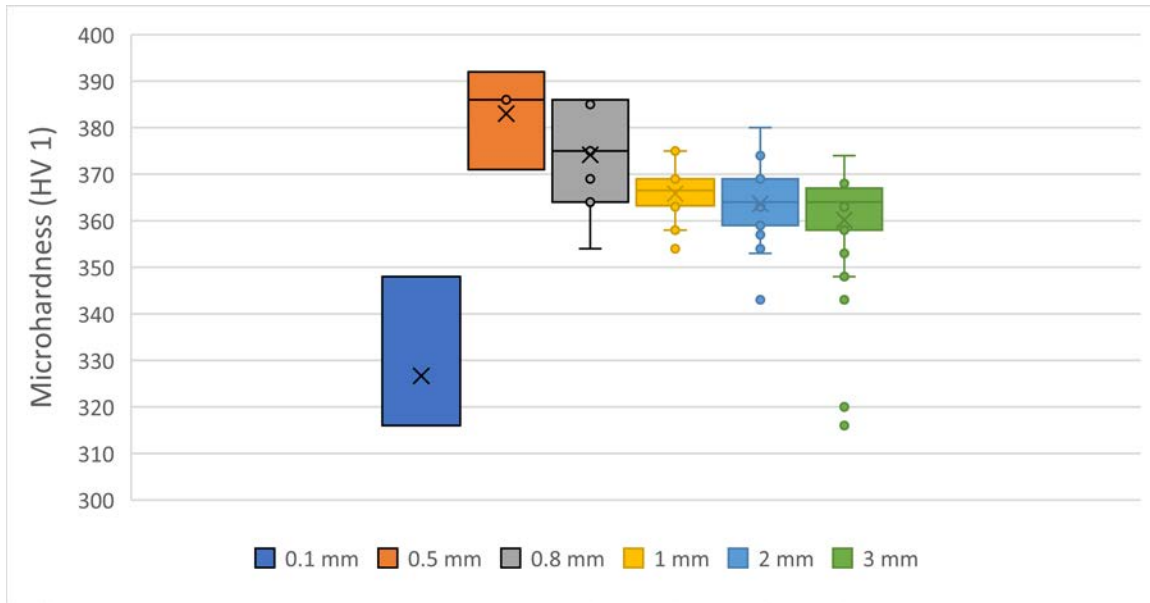


Figure 27. AP microhardness data for feature size samples

AP bulk hardness values remained consistent throughout top, middle, and bottom series measurements, as seen in Figure 28. Small bulk hardness values varied by 8.7 HV1, while large bulk hardness values varied by 1.4 HV1. The average bulk hardness value was 363.8 HV1 for small samples and 359.3 HV1 for large samples. Both small and large samples had the same bulk material dimensions. Little variation was seen between bulk hardness values of small and large samples. As expected, bulk hardness values were less than their respective feature sizes in both samples. It was concluded that mechanical properties within the bulk material were consistent, as shown by slight variation in bulk hardness values.

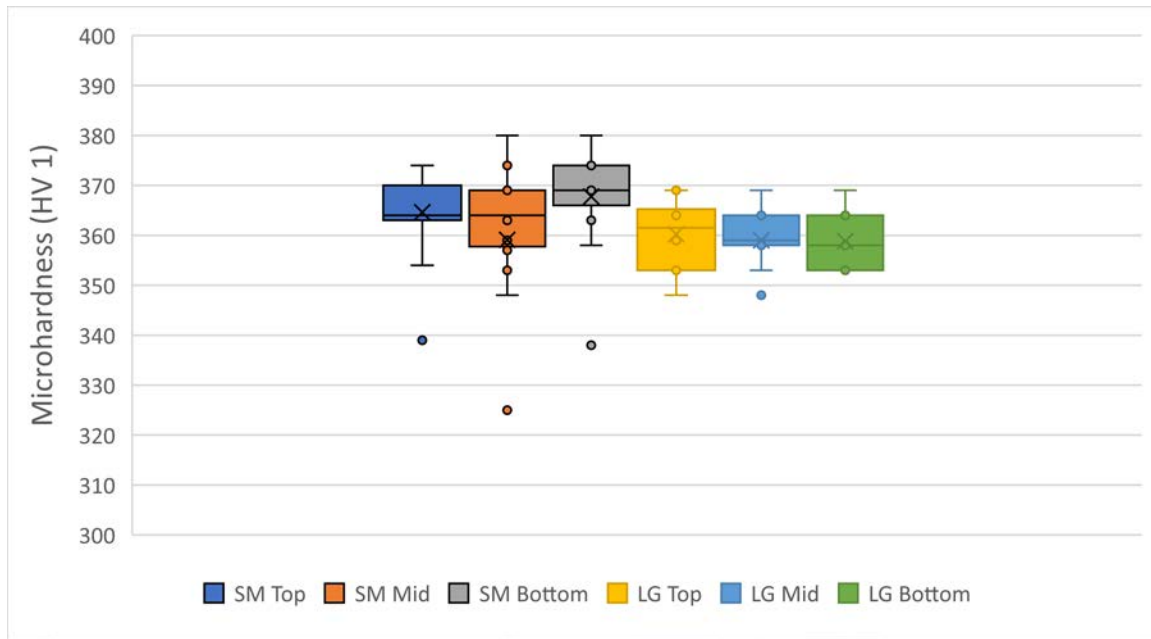


Figure 28. AP microhardness data for bulk material

The same amount of data was collected for each feature size between HT and AP samples. Figure 29 displays the microhardness data for all HT samples. The average hardness values for HT features was 600.3 HV1 for 0.1 mm, 582.8 for 0.5 mm, 589.4 HV1 for 0.8 mm, 592.5 HV1 for 1 mm, 587.9 HV1 for 2 mm, and 588.5 HV1 for 3 mm features. Similar to the AP samples, the 0.1 mm feature for HT samples had a large disparity. Despite this disparity, the HT 0.1 mm feature still exhibited higher hardness values than larger features. The other HT samples did not follow the expected trend of higher hardness values with decreasing feature size. The 0.8 mm feature had a higher average hardness value than the 0.5 mm feature and the 1.0 mm feature had a higher average hardness than the 0.8 mm feature. In addition, the 3 mm feature had a higher average hardness value than the 2 mm feature. The smallest feature exhibited the highest hardness values likely due to a faster cooling rate. Cooling rates of HT samples was likely slower than that of AP samples due to a smaller grain size.

Data point measurements were taken as linear as possible for consistency, however, avoiding areas of large pore size made taking linear measurements very difficult. In a study by Yasa et al. hardness values for heat treated maraging steel under the same conditions

was  $648.4 \pm 9.8$  HV [22]. The smallest HT feature size with the highest expected hardness was lower than values found in the experiment by Yasa et al. by 48.1 HV. As previously mentioned, dissimilarities in cooling rates, as well as the size and density of pores present in the metal are the biggest contributing factors for this disparity.

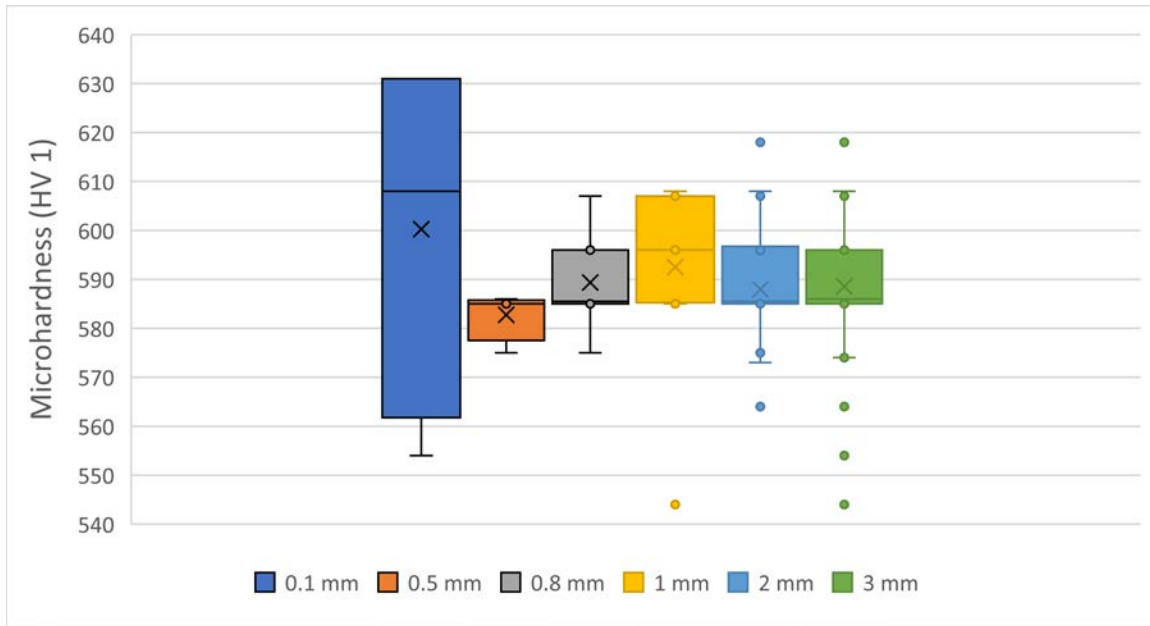


Figure 29. HT microhardness data for feature sizes samples

Bulk hardness values for HT samples decreased with top, middle, and bottom measurements, respectively, as seen in Figure 30. Small hardness values varied by 11.9 HV1, while large hardness values varied by 12.4 HV1. Both small and large samples experienced similar variations in bulk hardness values. The average value for small samples was 572.3 HV1, and 590.2 HV1 for large samples. Bulk hardness values were less in both samples compared to their respective feature sizes. The HT samples experienced more outlying data points in bulk measurements than compared to AP samples. Bulk hardness measurements made up the majority of variation in HT samples.

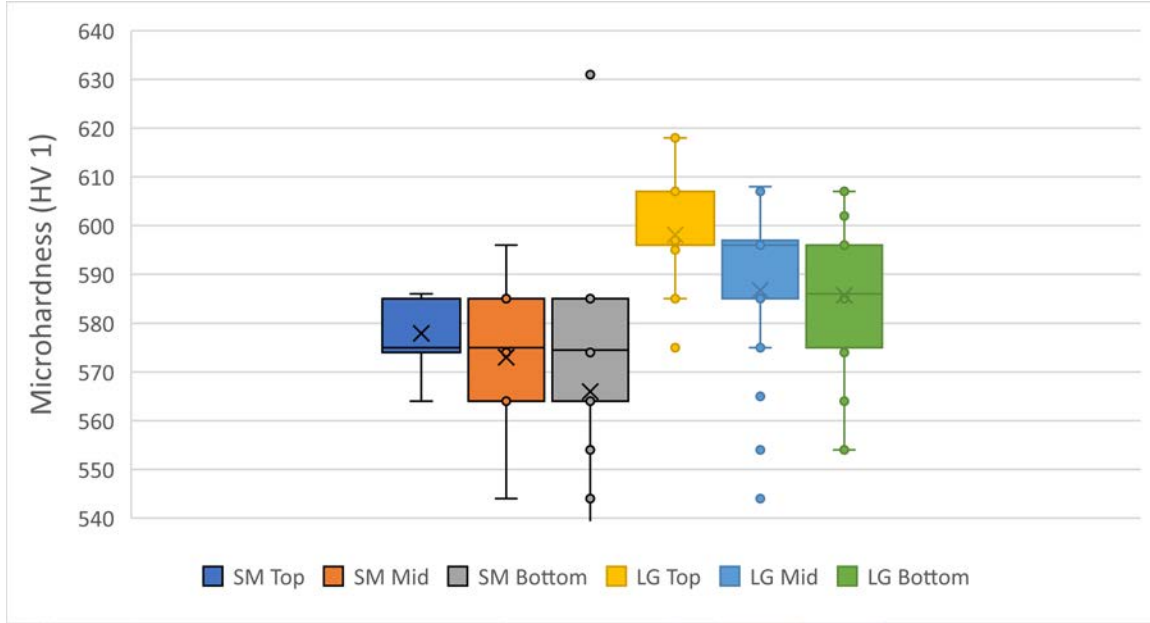


Figure 30. HT microhardness data for bulk material.

## 2. Microstructural Properties

Figure 31 shows the AP samples with fingers of varying sizes. If feature quality is defined as how closely the printed feature matches the shape and volume of the 3D model, then what was observed was a decrease in feature quality with a decrease in feature size in both AP and HT samples. As seen in Figure 31, dimensions varied greatly in printed samples. The 0.1 mm feature is seen to have very rounded edges, while the 0.8 mm and 2 mm features have very jagged top layers, which however, matches the digital model more closely than the 0.1 mm feature. A study by Frazier et al. observed that surfaces of a build sitting at right angles to the energy source is several times rougher than surfaces parallel to the same energy source. “It is also noted that SLM printers produce surfaces about twice as smooth as the electron beam manufacturing (EBM)” [27]. One of many reasons, SLM is the preferred method for producing high quality parts with small feature sizes. Print parameters have a major impact on the quality of parts. Adjustments in parameters may lead to increased part quality and mechanical properties in the future.

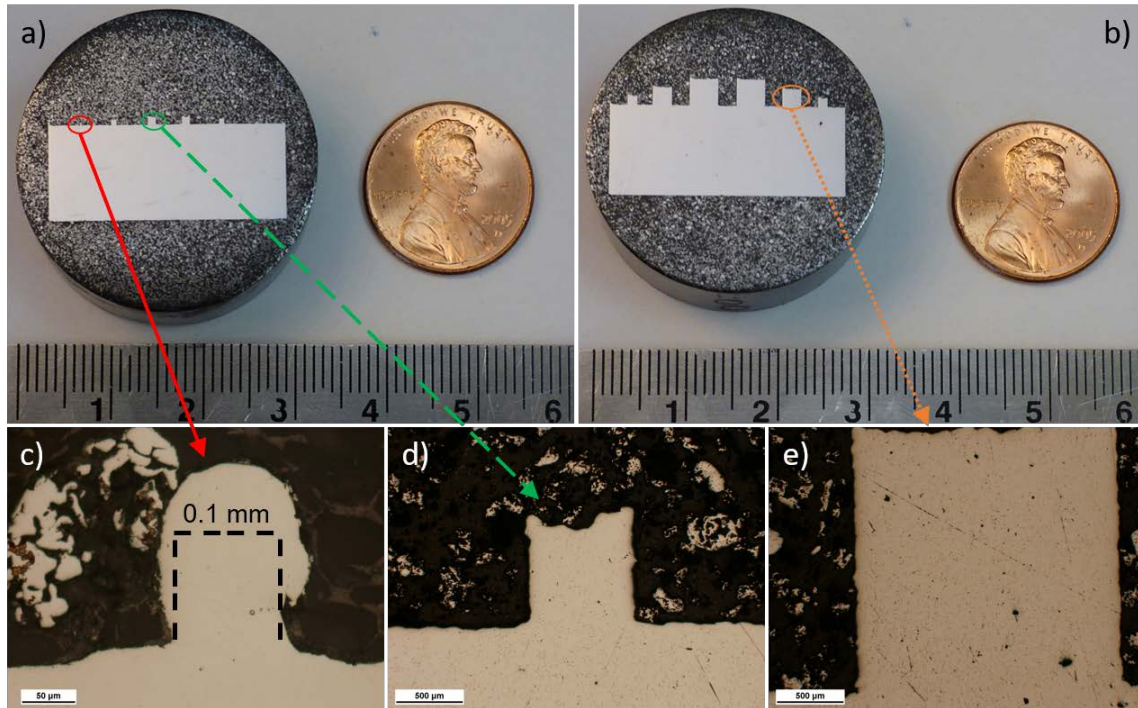


Figure 31. Small and large AP samples showing feature size and quality

Figure 32 illustrates the relationship between laser power, build rate (scan speed), and feature definition. The graph shows that feature quality and resolution decrease as build rate increases. In a study by Jeon et al. optimum bead shapes for spherical and non-spherical powder types were 110 mm/s and 73 mm/s, respectively [10]. Yasa et al. reported the optimum scan speed for SLM maraging steel 300 on an EOS M270 machine to be 750 mm/s [22]. This experiment used a scan speed of 827 mm/s on an EOS M100 machine which may have been too high to produce quality parts greater than 99% density at a constant scan speed, scan line spacing, and layer thickness. Yasa et al. also showed that as scan speed increased, the hardness values significantly decreased due to increased porosity. At higher scan speeds, a smaller layer thickness should be used to obtain higher density parts.

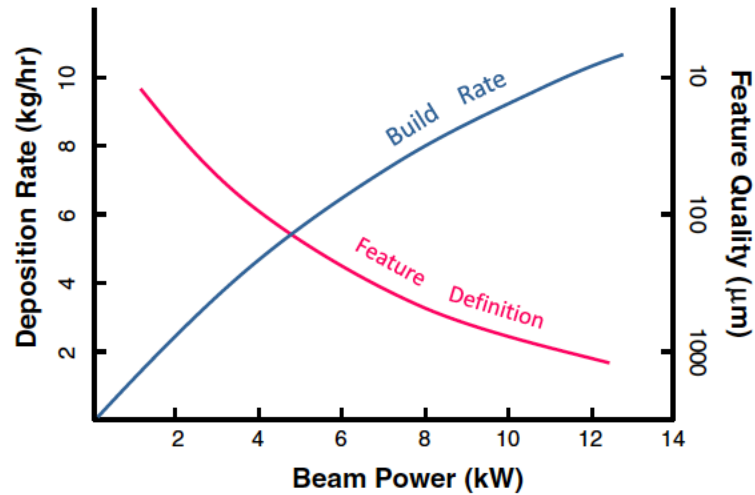


Figure 32. Relationship between power, build rate, and feature definition.  
Source: [27].

AM is an art, as there is not a “one size fits all” approach to produce high-quality fully dense parts. Depending on part application and desired mechanical and microstructural properties, different print parameters and heat treatment techniques must be applied. A study by Gorse et al. showed different laser powers and scan speeds for a given layer thickness during SLM. Several laser power and scan speed parameters are characterized into 4 different melting zones for a given layer thickness as illustrated in Figure 33.

Zone I is the fully dense zone [28]. In this zone, parts have little to no porosity. Zone II is characterized as the over melting zone. Zone II contains a significant amount of porosity due to the use of excess energy. Zone III is the incomplete melting zone. This zone also contains a significant amount of porosity; however, this is due to excessive energy input. Lastly, Zone IV is the over heating zone characterized by low scan speeds at high laser powers. Parts made in this zone contain several defects due to excess energy input. At a laser power of 77.1 Watts and a scan speed of 827 mm/s, the samples produced in this experiment border Zones I and III. This is logical as optical imaging revealed samples with relatively high amounts of porosity [28].

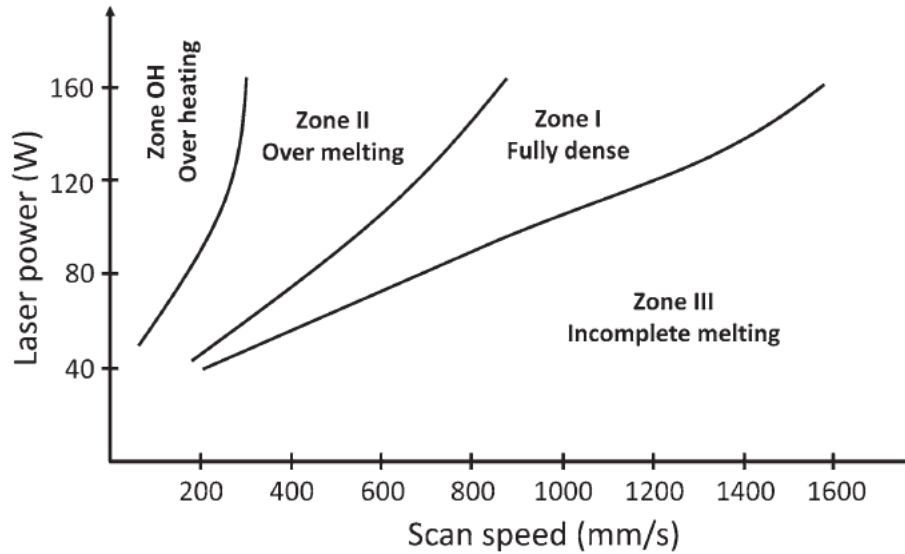


Figure 33. Process window for SLM-produced samples. Source: [28].

Porosity can form due to insufficient energy input or due to excessive energy input [28]. Figure 34a is a dark field image showing the laser track marks created during SLM. Figure 34b is a bright field image of Figure 34a showing the number of pores present within the sample. Figure 34c is a higher resolution image of the bulk material of an AP sample. Within this image laser track marks containing SLM-specific microstructure were observed. Porous regions were seen across all samples. Triple points, areas where 3 or more pores conjoined were seen across AP and HT samples alike. Due to the size of triple points, these features significantly degrade the mechanical properties of maraging steel. Using Equation 1, the energy density for this experiment was calculated. Despite the amount of triple points and inclusions present in samples, relative density and percent porosity were similar to values found in literature. Samples in this experiment had an energy density of  $2.33 \text{ J/mm}^2$ . In a study by Casalino et al. on the optimization of SLM of maraging steel, the average maximum energy density was  $2.78 \text{ J/mm}^2$  [19]. Although lower than the maximum average energy density, samples produced in this experiment still classified as fully dense.

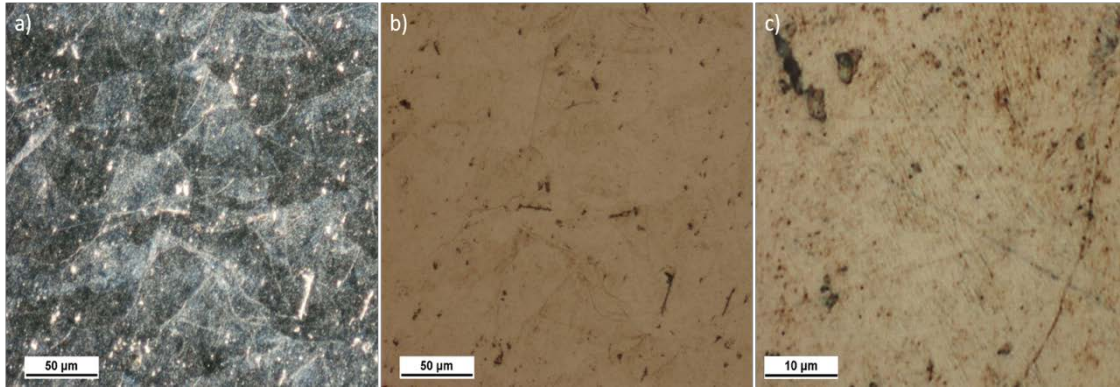


Figure 34. Optical imaging of an AP sample showing laser track marks, bulk material, and microstructure.

Percent porosity was determined by comparing samples in this experiment to those studied in Casalino's research. Figure 35a contained 94.5% density, had a laser power of 57 Watts, scan speed of 180 mm/s, and energy density of  $1.58 \text{ J/mm}^2$  [19]. As previously stated, Figure 35b had an energy density of  $2.33 \text{ J/mm}^2$ . Figure 35c contained greater than 99% density, had a laser power of 100 Watts, scan speed of 200 mm/s, and energy density of  $2.50 \text{ J/mm}^2$  [19]. An accurate porosity measurement could not be obtained using ImageJ on the sample shown in Figure 35b. However, by comparison it is estimated to have a density between 94.5% and 99%. That results in 1–5.5% porosity. The true density of maraging steel is  $8.01 \text{ g/cm}^3$ . Parts between 90.9 and 99.9% are considered fully dense [19]. The number of pores observed in Figure 35b were relatively similar to those found in Figures 34a and 34c. However, pore size was smaller than Figure 34a and larger than 34c. From this, it can be concluded that pore size has a direct effect on part density.

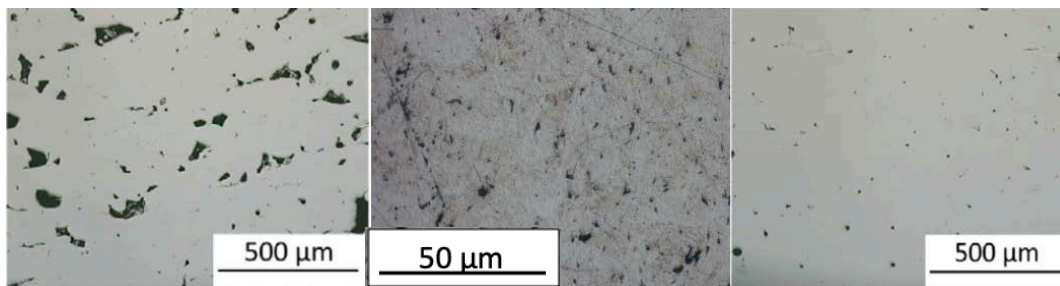


Figure 35. Feature size density compared to literature. Source: [19].

The high nickel concentration in maraging steel allows for the formation of nickel precipitates during heat treatment. The metal is strengthened as these precipitates prevent the movement of dislocations. Pores within the HT samples appeared smaller and contained fewer triple points. Percent porosity was calculated using ImageJ on the sample seen in Figure 36a. Figure 36b shows the same image as 35a following binarization processing using ImageJ. This sample was measured to contain 3.1% porosity. This equates to a density of 96.9%.

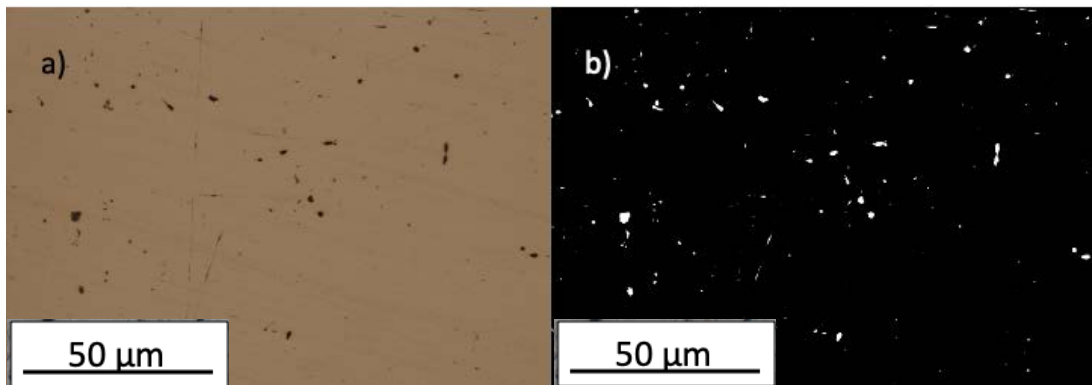


Figure 36. Bulk material of a HT sample at 10x magnification.

Different ranges in process parameters apply to different SLM printers. Scan speed, as well as the type of laser used during printing seem to be the most varied parameters found in literature regarding SLM of maraging steel. It must be noted that scan speeds in Casalino's experiment ranged between 180–220 mm/s. In addition, Casalino used a Nd:YAG laser source [19]. Yasa et al. compared the relative densities obtained using a Concept Laser M3 printer equipped with an Nd: YAG laser, to an EOS M270 printer equipped with an Yb-fiber laser. The Concept Laser M3 printer produced parts with 99% density at an optimum scan speed of 200 mm/s. On the other hand, the EOS M270 produced parts with 99% density at an optimum scan speed of 750 mm/s. Low scan speeds had a significant impact on the density of parts in the EOS M270 machine, an effect not seen on the Concept Laser machine [22]. From this, it is deduced that fully dense maraging steel parts are produced using a variety of SLM technologies. However, process parameters

amongst SLM printers must be optimized to produce desired part quality. In regard to this experiment, printing could have been optimized by decreasing scan speed, and increasing laser power. While 316L stainless steel settings produced fully dense parts, optimizing laser power and scan speed parameters would likely produce parts greater than 99% density.

### **3. Overall Results**

In summary, this chapter addressed the results of feature size quality using maraging steel powders and 316L stainless-steel print settings on an EOS M100 3D metal printer. Print quality of 0.1 mm features was inadequate; however, feature quality significantly improved with an increase in feature size. Perhaps, test methods on 0.1 mm features should have been decreased from HV1 to HV0.5 to obtain accurate data. In general, higher hardness values were observed in smaller features when compared to larger features. This was mostly due to rapid cooling rates of smaller features compared to larger features. Smaller features have faster cooling rates, thus leading to smaller grains and higher hardness values. The opposite is true regarding larger features that contained higher cooling rates. The hardness values amongst AP and HT bulk material were consistent throughout.

In order to gain further insight on the effect of feature size on maraging steel samples, it may be beneficial to print features with heights greater than those used for builds in this research. If material is spaced further away from the bulk, than faster cooling rates will be experienced, and hardness values and microstructure will see more dramatic changes than what was seen in this work. Though print quality of features were adequate, it is highly recommended that either EOS designated maraging steel settings are used or print settings are manually optimized for maraging steel printing in the EOS M100 machine to achieve parts with lower porosity.

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## **IV. CONCLUSION**

### **A. MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF MARAGING STEEL TENSILE SPECIMENS**

Mechanical and Microstructural properties of tensile specimens provided by Marine Corps Logistics Base Albany were successfully analyzed. Properties of CNC, AP, and HT samples were observed through tensile tests, microhardness tests, optical microscopy, and SEM analysis. Samples that contained surface roughness due to support structure removal underwent sandblasting to improve surface quality. Sandblasting showed to have a small effect on the mechanical properties of printed specimens. Percent elongation of both AP and sandblasted samples were within tolerance of EOS standards. The AP samples outperformed CNC samples in total strength by roughly 200 MPa. The HT sample had twice the amount of yield and tensile strength when compared to AP samples. Excellent mechanical properties were achieved, implying that print settings were adequate. Through mechanical testing of tensile specimens, it is proven that SLM is a viable option for the manufacturing of maraging steel parts.

Optimal heat treatment of maraging steel is 490°C for 5 hours. Samples that were heated at 490°C for 6 hours experienced some additional austenite reversion, resulting in lower microhardness values. SEM analysis revealed austenite reversion within the microstructure of HT samples. Although HT samples in this experiment exhibited lower hardness values than those provided in literature by 30 HV, they still presented excellent mechanical properties.

The microstructure of AP samples exemplified that associated with the SLM technique. Grain structure enclosed by laser tracks were observed on the cross sections of printed samples. New phases within the solidification cells at cell boundaries were also observed. The energy density for all samples was 4.11 J/mm<sup>2</sup>, 176% higher than the average maximum energy density for maraging steel produced via SLM. While a higher than normal energy density may have contributed to increased porosity, this finding had no effect on the mechanical properties of samples.

## **B. CORRELATION OF FEATURE SIZE AND MECHANICAL PROPERTIES ON MARAGING STEEL**

Feature size samples were successfully printed on an EOS M100 machine at NPS using maraging steel powder, and mechanical properties analyzed. Print quality for this experiment was adequate. Print quality increased significantly with an increase in feature size. Print settings should be optimized to obtain better results in print quality for features smaller than 0.8 mm. Overall, smaller features portrayed higher microhardness values than larger features. Smaller features had higher cooling rates which contributed to restricted grain growth and smaller grains. The abundance of smaller grains acted as a barrier to the movement of dislocations causing increased hardness. On the contrary, larger features had slower cooling rates which contributed to more grain growth and larger grains. Less boundaries were available for the agglomeration of dislocations which resulted in softer material and decreased hardness values.

Density of printed samples was adequate, however, adjusting print settings would have resulted in increased density closer to 99%. Samples in this experiment had an energy density of  $2.50 \text{ J/mm}^2$ , 16% lower than the average maximum energy density found in literature. It is likely that decreasing scan speed while increasing laser power would have resulted in a more optimum energy density yielding more fully dense parts.

## **C. FUTURE WORK**

The stated objectives for this experiment were met. It has been proven that AM via SLM produces parts comparable to those created by TM methods. SLM provides a unique microstructure that contributes to excellent mechanical properties in parts.

Going forward, more maraging steel samples should be printed with proper settings in order to further research how mechanical and microstructural properties are influenced by varying print parameters. Additionally, samples with various support structures should be observed to determine how support structure design can be optimized in order to decrease surface roughness. Acquiring proper licensing for maraging steel settings would allow future students to establish a baseline for part quality. Lastly, XRD (X-ray diffraction) analysis should be used in order to gain further insight on the phenomena of

austenite reversion. This technique may provide more detailed data on the volumetric percentage of austenite and martensite present before and after aging, leading to improvements of heat treatment techniques.

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