

**Repeatability of the Mechanical Properties of  
Additively Manufactured  
Precipitation Hardened 17% Chromium – 4% Nickel Stainless Steel**

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**Master's Thesis**

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"And He said unto me, My grace is sufficient for thee: for my strength is made perfect in weakness. Most gladly, therefore, will I rather glory in my infirmities, that the power of Christ may rest upon me."  
2 Corinthians 12:9

# Abstract

Additive manufacturing is a state-of-the-art technology quickly becoming invaluable to the engineering field because of its versatility and cost-effective part creation methods. However, while these methods are exciting, their ability to create reliable and consistent components with mechanical properties comparable to those of wrought parts of the same material is under review.

This work studies the repeatability of the mechanical properties and material behavior of powder bed fusion additively manufactured 17% Chromium -4% Nickel precipitation hardened stainless steel alloyed structures. Specifically, the repeatability of the material hardness, yield strengths, and ultimate strengths was examined. Therefore, tensile tests of meso sample scale specimens were run, and Vickers microhardness measurements were obtained from zig-zag-shaped structures. The results show that the mechanical properties and material behavior were repeatable and that geometry and sample orientation consistently impact the sample properties.

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# List of Commonly Used Acronyms

|        |                                                  |
|--------|--------------------------------------------------|
| 3D     | Three Dimensional                                |
| 3DP    | 3D Printing                                      |
| AM     | Additive Manufacturing                           |
| CAD    | Computer Aided Design                            |
| DED    | Direct Energy Deposition                         |
| DLP    | Digital Light Processing                         |
| DMD    | Direct Metal Deposition                          |
| DMLS   | Digital Metal Laser Sintering                    |
| EBM    | Electron Beam Melting                            |
| EDM    | Electric Discharge Machining                     |
| FDM    | Fused Deposition Modeling                        |
| FEUP   | Faculdade de Engenharia da Universidade do Porto |
| FFF    | Fused Filament Fabrication                       |
| LENS   | Laser Engineered Net Shaping                     |
| LMD    | Laser Metal Deposition                           |
| LOM    | Laminated Object Manufacture                     |
| MJM    | Multi-Jet Molding                                |
| PBF    | Powder Bed Fusion                                |
| PBF-LB | Laser Beam Powder Bed Fusion                     |
| PBF-EB | Electron Beam Powder Bed Fusion                  |
| SCP    | Smooth Curvatures Printing                       |
| SDL    | Selective Deposition Lamination                  |
| SL     | Stereolithography                                |
| SLA    | Stereolithography Apparatus                      |
| SLS    | Selective Laser Sintering                        |
| SLM    | Selective Laser Melting                          |
| STL    | Standard Tessellation Language                   |
| SEM    | Scanning Electron Microscopy                     |
| UAM    | Ultrasonic Additive Manufacturing                |
| UMBC   | University of Maryland Baltimore County          |

# **1 Introduction**

This thesis resulted from a partnership between the University of Maryland Baltimore County (UMBC), industrial and professional scientists and engineers, and the Faculdade de Engenharia da Universidade do Porto (FEUP). This thesis is the final component of the master's in mechanical engineering degree at FEUP.

UMBC hopes to characterize additively manufactured (AM) precipitation hardened 17% Chromium-4% Nickel stainless steel (17-4 PH SS). Furthermore, they hope to understand the repeatable nature of the mechanical properties of AM 17-4 PH SS components.

## **1.1 Background**

Additive Manufacturing (AM) is a viable method to form Precipitation Hardened (PH) Stainless Steel (SS) alloys, which are desirable for next-generation vehicles due to high strength and corrosion resistance. Layer-by-layer construction of AM components causes a series of thermal transients. These heating cycles encompass temperatures that exceed the material melting point, provide stress relief, and combine time-temperature profiles for solution treatment and age hardening. In addition, complex thermal gradients achieved in Powder Bed Fusion (PBF) AM processes permit the tailoring of as-built microstructures by matching thermal heat flow to the build's structural geometry, potentially reducing the need for heat treatment[1].

The complex thermal history created by AM processes impacts the microstructure of the printed material. The microstructure of any material determines that material's mechanical properties. Therefore, it is necessary to determine if the PBF AM process created repeatable mechanical properties in 17-4 PH SS.

## **1.2 Goals**

This work aims to assess the repeatability of mechanical properties of 17-4 PH SS part builds printed on three EOS M290 3D printers in three separate locations. Therefore, parts from each build underwent microhardness and tensile testing to obtain Vickers Hardness, Young's Modulus, yield strength, strain to failure, and ultimate tensile strength. Subsequently, statistical analysis and observation of sample fracture surfaces and gauge sections detailed any variation of properties across the three builds.

## **2 State of the Art**

### **2.1 Overview of Additive Manufacturing**

Additive Manufacturing (AM) is a state-of-the-art technology that creates parts by consecutive stacking of material, representing a field of significant development and research.

#### ***2.1.1 Additive Manufacturing vs. Traditional Manufacturing***

An understanding of the differences between additive manufacturing and traditional manufacturing will make transparent additive manufacturing's value. The first difference is in the method of part creation. Additive Manufacturing (AM) is a part creation method that consecutively stacks layers of the desired material on each other in a predefined arrangement based on a computer-aided design (CAD) file [2]. According to the American Society for Testing and Materials (ASTM), AM is a "process of joining materials to make objects from three-dimensional (3D) model data, usually layer by layer, as opposed to subtractive manufacturing methodology" [3]. Conversely, subtractive manufacturing (SM) or traditional manufacturing transforms an existing material block into the desired design by removing material from the material block [4]. Examples of SM include milling and sawing.

Another difference between AM and SM is the ease of complex part creation. AM readily and inexpensively forms intricate parts that would be expensive, difficult, and often impossible to manufacture by SM. In addition to effortlessly creating complex parts, AM can also print entire operational assemblies as a whole, combining manufacturing and assembly into one step. In contrast, assemblies created by SM require the separate creation of each part in the assembly. Formed parts later join to form the desired assembly[4].

AM and SM also differ in the required technology for their manufacturing processes. For example, SM requires multiple steps that each need a different machine. Comparatively, AM uses a single device to create any component. A single machine simplifies the manufacturing process and reduces the number of necessary operators[4].

Most importantly, AM decentralizes the manufacturing process, making it possible to manufacture almost anywhere, bringing manufacturing closer to the end-user. SM is limited to factories with large assembly lines[4]. AM's decentralization of manufacturing is especially useful for remote areas such as oil rigs and naval warcraft where spare components need to be made quickly and wait time impacts critical vehicle functions.

### ***2.1.2 History of Additive Manufacturing***

The first form of AM, known as rapid prototyping, was developed in the 1980s. As the name suggests, rapid prototyping quickly helped engineers create prototypes of design concepts. Stereolithography (SL) was the first and most popular rapid prototyping process. SL was so popular that rapid prototyping and SL became interchangeable terms. Rapid prototyping by SL uses a liquid polymer resin that cures in the presence of ultraviolet lasers. Like every AM process, SL operates based on information provided by a Standard Tessellation Language (STL) file. STL files are formed by discretizing or slicing CAD file geometry into layers of triangles, an accompanying normal vector, x, y, z coordinates, and file header. Next, a build plate is placed in the liquid polymer resin, and an ultraviolet laser cures the resin according to the STL file. This process is repeated as the build plate incrementally decreases its vertical position. Finally, any remaining resin is removed and saved for reuse[2]. Figure 2-1 shows the components of stereolithography.

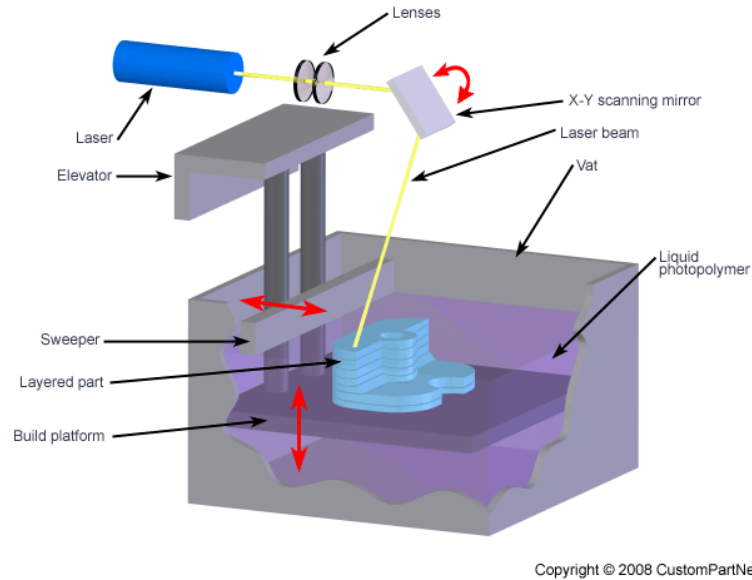


Figure 2-1:Stereolithography (SL) setup [4]

### 2.1.3 Classification of Additive Manufacturing Methods

AM has become increasingly popular because of its relative simplicity compared to traditional or wrought manufacturing, which requires extensive manual labor, resources, and multiple steps like forging, molding, and machining [5]. Since the advent of rapid prototyping and SL, the field of AM has advanced significantly. ASTM divides the AM field based on process methodologies, creating seven categories: direct energy deposition (DED), powder bed fusion (PBF), binder jetting, material extrusion, material jetting, sheet lamination, and vat photopolymerization. Figure 2-2 shows the seven categories of AM and provides additional details for each.

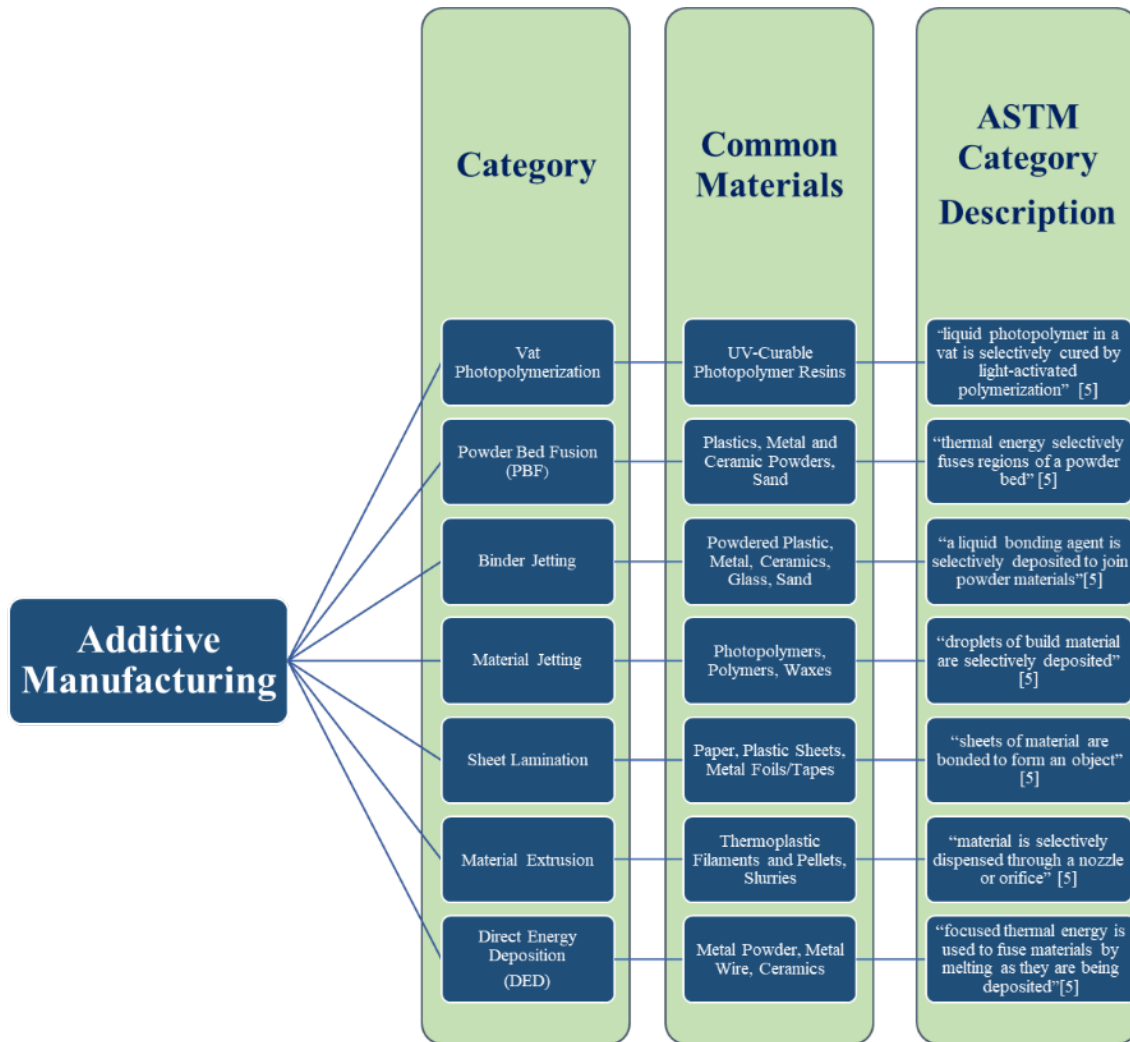


Figure 2-2: Seven Categories of Additive Manufacturing as Described by ASTM  
(Adapted from [6])

Vat photopolymerization, also known as stereolithography apparatus (SLA) and digital light processing (DLP), creates parts using selective curing. Selective curing is the selective exposure of liquid photopolymer resin to a light source such as a laser or projector. This exposure begins the process of polymerization and changes the liquid resin into a solid. AM parts created by this method can have high design complexity, accuracy, and a smooth surface finish. Additionally, vat photopolymerization allows for large build volumes[6].

Powder bed fusion (PBF) melts plastics, metals, ceramics, and sand selectively using a laser or electron beam to form components. The unmelted powder remains in the powder bed to support any component features that may extend from the primary geometry. Because of the supporting powder, PBF is well-suited for components with intricate designs. Commonly used materials in PBF are powdered plastics, ceramics, and metals, and sand. Note that other terms for PBF are selective laser sintering (SLS), electron beam melting (EBM), and direct metal laser sintering (DMLS)[6].

Binder jetting applies liquid bonding agents selectively onto layers of powdered material, forming parts in consecutive layers. Following printing, ceramic and metal parts enter a furnace. Note that binder jetting allows for full-color printing and is known for its high productivity rates. Commonly used materials for binder jetting are powdered plastics, metal, ceramics, glass, and sand. Alternate names for binder jetting include 3D printing (3DP), ExOne, and Voxeljet[6].

Material jetting, also known as Polyjet, smooth curvatures printing (SCP), multi-jet modeling (MJM), and Projet, creates parts by depositing material droplets one layer at a time. Material jetting variants include depositing photocurable resin that cures under ultraviolet (UV) light and depositing molten materials that later solidify at ambient temperatures. Material jetting produces parts with high accuracy and allows for full-color and multi-material components. Photopolymers, polymers, and waxes form parts made by material jetting[6].

Sheet lamination forms parts by staking and laminating material sheets together. The lamination method varies by part material. Adhesives or chemicals laminate paper and plastic parts while ultrasonic welding or brazing laminate metal parts. Material excess is cut layer by layer during the forming process and removed after the process finishes. Sheet lamination is a process with high volumetric build rates and low costs. In addition to printing parts of either

paper, plastic, or metal, the process prints combinations of the same materials. Other names for sheet lamination include laminated object manufacture (LOM), selective deposition lamination (SDL), and ultrasonic additive manufacturing (UAM)[6].

Material extrusion extrudes material, usually thermoplastic filaments and pellets, liquids, and slurries with a nozzle or orifice in beads or tracks, and then combines the material into layered parts. This method is a low-cost process that can print parts with sound structural integrity in multiple colors. Material extrusion is also called fused filament fabrication (FFF) and fused deposition modeling (FDM)[6].

Essentially an automated method of welding, direct energy deposition (DED) feeds metal powder or wire into a melt pool that continually forms layers of the part. A laser or electron beam is the energy source that melts the material into the melt pool. DED can create parts regardless of direction or axis and can repair and add features to existing parts. Additionally, this method can create multi-material parts. DED has the highest single-point deposition rates of the AM methods. DED is also called laser metal deposition (LMD), laser engineered net shaping (LENS), and direct metal deposition (DMD)[6].

Of these seven AM processes, DED and PBF are the most useful for industry, especially in the defense sector[5].

#### ***2.1.4 Additive Manufacturing Applications***

Additive manufacturing provides many advantages over traditional manufacturing, making it a coveted manufacturing method for many industries. In addition to rapid part creation, AM allows for the easy creation of parts with complicated designs. This section presents advances made in four popular sectors of industry.

##### ***2.1.4.1 Aerospace Industry***

The ability to form complex parts with ease makes AM a top choice in the aerospace industry. For example, in traditional manufacturing, it is difficult, if not nearly impossible, to create hollow cross-sections for vehicle structures that these two industries desire for fuel-efficiency, cost-effectiveness, and weight efficiency[2]. Specifically, in the aerospace industry, engineers design vehicles for fuel efficiency. This need for fuel efficiency means that components of the vehicle must have large strength-to-weight ratios, requiring minimum material use. Structures with high strength-to-weight ratios often have complicated designs. AM is convenient for such scenarios as complex and diverse shapes are more easily formed additively than subtractive manufacturing methods. In addition to metallic structures, these industries also use AM to create nonstructural parts from nonmetallic materials. Beginning in the 1990s, leaders in aerospace such as Boeing and Bell Helicopter created nonstructural polymer components using AM [7].

Another reason that AM is attractive to the aerospace industry is the potential for part consolidation. To reduce the number of small parts in an aircraft, engineers design the complicated shapes mentioned previously with as few fastening components as possible. This reduction means that intricate designs often transform from numerous small parts to a single piece. The advantages of having fewer components include decreased production and inspection

costs, shorter assembly lines, and fewer tools required for assembly. For example, another industry leader, GE Aviation, reduced one design from 855 parts to about 12. This part reduction caused significant weight loss, increasing fuel efficiency by 20%. Equally impressive is Airbus's simplification of a 126 component hydraulic housing tank to one AM component [7].

#### *2.1.4.2 Automotive Industry*

The automotive industry values AM because it shortens the design development process and lowers manufacturing and production costs. More specifically, in the industry's motorsports segment, lightweight, intricate structures are necessary for success. These structures are printed in small numbers, making traditional manufacturing unattractive because of unnecessary expense. AM is desirable in this situation as these intricate structures can be printed immediately from design at a lower cost, and no excess production footprint is required [8].

AM's innovation was evident when CRP Technology, based in Italy, used AM to design and create titanium F1 gearboxes. These gearboxes were 20% to 25% lighter than traditionally manufactured gearboxes and had about 20% of traditional gearbox volume. Furthermore, these new gearboxes had a torsional stiffness two times greater than conventional gearboxes, lower gear wear, and lower power use [8].

Prometal provided another example of the effectiveness of AM in automotive production. Using AM, the company produced engine parts such as engine blocks, cylinder heads, and intake manifolds for engines of both passenger and motorsports vehicles. The time needed for engine component creation was significantly less than the necessary time using traditional methods. For example, one complex engine block, including oil recirculation lines and cooling passages, was formed in just one week [8].

#### *2.1.4.3 Biomedical Industry*

AM has many biomedical industry applications, including implants, medical devices, biological chips, and artificial organs. Specifically, in the orthopedic and dental fields, AM is used to create complicated designs, roughened surfaces to support bones' integration, and make personalized implants for every patient. For example, Arcam used AM to create knee, shoulder, and hip implants, several of which are market certified. Additionally, several biomedical companies have made crown and bridge copings for dental work [8].

Furthermore, biomedical research is turning to AM to produce better biomedical implants. For example, EBM has created titanium alloy implants that simulate bone stiffness to reduce stress. Additionally, cellular titanium alloy bone implants with connecting pores showcase tissue ingrowth and vascularization feasibility [8].

In addition to the advances mentioned above, the biomedical industry uses AM for bio fabrication of living cells. These living cells are the basis of biological substances, processes, and devices, including drug delivery, tissue engineering, organ printing, and in-cell printing. For example, researchers at Drexel University created an AM system used to print 3D, biopolymer-based, liver cell-embedded tissue constructs[9].

#### *2.1.4.4 Energy Industry*

The energy industry benefits significantly from advances in AM. Specifically, AM's fast and flexible nature allows for prototype creation at low cost and short production times. As such, AM lends to unique and innovative designs that improve energy efficiency and power density. One example is the use of AM for the fabrication of a graphite composite bipolar plate with a novel "leaf" flow field design for use in polymer electrolyte membrane fuel cells. As a result of

AM fabrication, the fuel cell had an increased power density of 20% over traditionally produced designs [10].

## **2.2 Metal Additive Manufacturing**

As seen in Section 2.1.4, multiple industries seek to make AM beneficial and profitable, taking it from concept to practical reality. For this reason, "functional printing" is emphasized as essential for the future of AM. "Functional printing" means that AM should create components capable of advanced functionality. Some examples of advanced functionality include printing multi-materials or composites and metallic materials [10]. Of particular interest is the printing of metallic materials.

### **2.2.1 Metal Printing**

There are three typical methods of metal printing based on the deposited material phase. The first and most conventional metal printing method is atomic deposition. An atomic deposition aims to create layers of metallic material either using thermal or plasma evaporation in a vacuum or electrolytic reduction processes to reduce an ionic metal in a solution. Both processes are nonlocal deposition processes, requiring intricate pretreatment and posttreatment steps to create a 2D pattern. Note that because metal atoms react according to their environment, environmental conditions must be selected and monitored carefully during these processes [10].

The second and most typically used method of metal deposition for AM utilizes multiple nano or microparticle phases. Because these particles are less likely to react with their processing environment, less care and monitoring of the environment is necessary. Nano and microparticles allow for less stringent processing environments and allow for greater flexibility in their deposition method. The use of these particles for metallization involves two sequential processing steps. The first step is the deposition or printing of the particles, and the second step

is sintering. Sintering is a thermochemical process that binds the deposited particles, creating solid metal. The method of deposition and sintering are particle-dependent. AM methods vary by printing and sintering processes [10].

The third method of metal printing transfers molten metal droplets from a bulk phase. In this method, thermally resistant material composes the dispenser. This material is chosen based on the melting temperature of the transferred metal. Transfer of molten metal droplets is best for metals with low melting points like gallium alloys, aluminum, and solder. In addition to the metal melting point, droplet size limits molten metal droplet transfer. The droplet size limitation is due to the high viscosity inherent to molten metal and dispenser engineering [10].

Note that metal AM causes intricate thermal cycles within the metals regardless of the metal deposition method. These thermal cycles necessitate an improved understanding of the relationship between metal AM microstructure, processing, and properties [11].

### ***2.2.2 Metal Additive Manufacturing Process Categories***

Metal AM has two main process categories- directed energy deposition (DED) and powder bed fusion (PBF). Each of these methods operates on a different AM principle and method of feedstock material delivery. Additionally, these methods differ in feedstock material and sources and states of fusion. However, PBF and DED share a common characteristic. Figure 2-3 outlines the two approaches, detailing the AM principle, material delivery method, feedstock materials, and sources and states of fusion for DED and PBF. The following sections will describe in detail each of the methods.

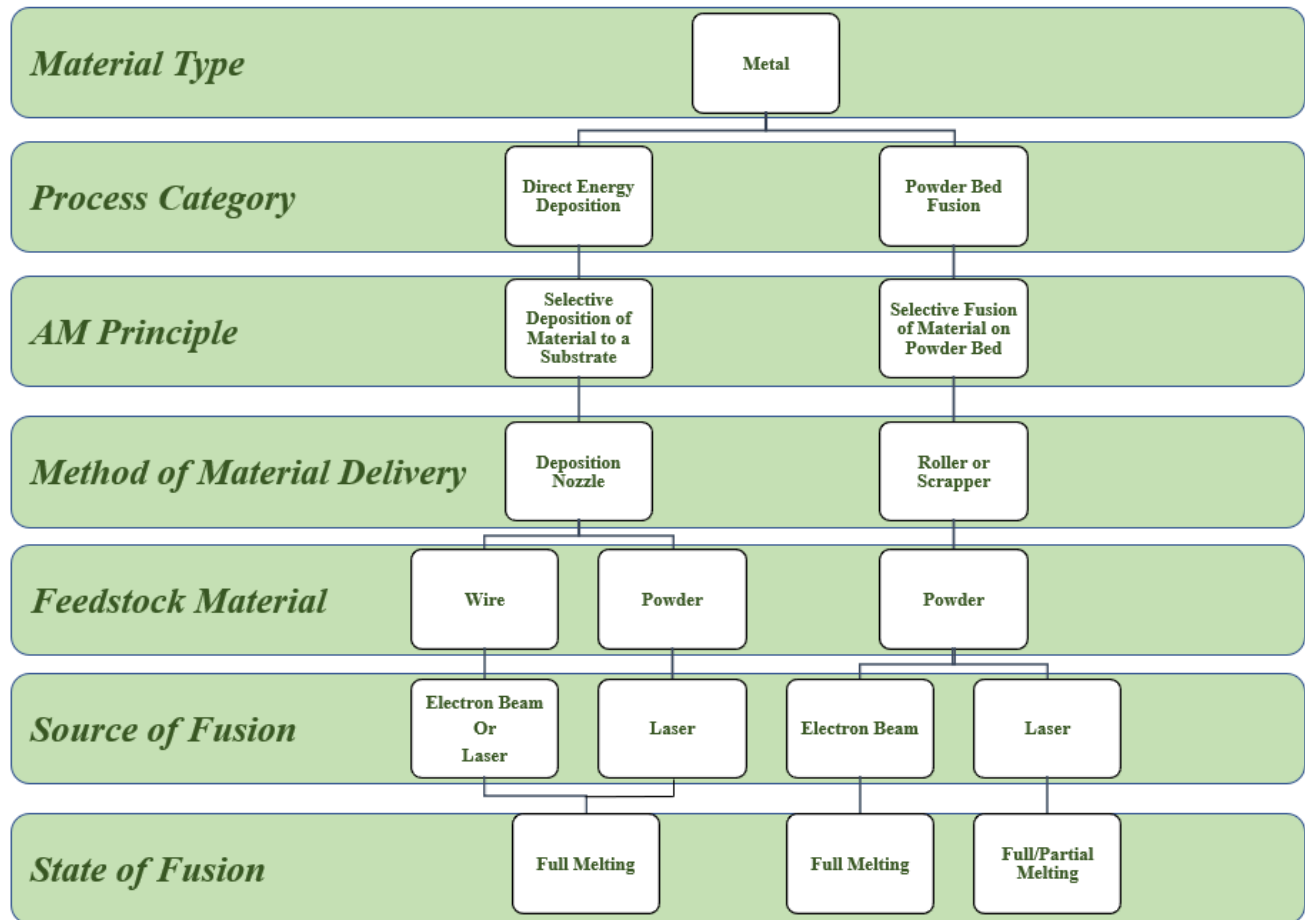


Figure 2-3: Classification of Metal AM (Adapted from [3])

#### 2.2.2.1 Direct Energy Deposition (DED)

Direct energy deposition operates by selective deposition of feedstock material onto a substrate. A deposition nozzle deposits the material onto a substrate. Note that the feedstock material for DED can either be in wire or powder form.

While many DED process category variants exist, their formation methods are identical. DED forms components by the creation of a melt pool on the print bed. Additional material injected into the melt pool increases its mass and volume. This process repeats for each layer of the component design file until the desired component forms. Melt pools are essential to the

DED process, especially when the material feedstock is powder, as they provide feedstock particles with the bond strength needed to bind to the substrate [3].

Regardless of the feedstock material used, every DED system comprises three subsystems: a directed energy source (laser or electron beam), feedstock injection unit (nozzle or deposition head), and substrate (also known as a print bed) controlled either by a CNC table or robotic arm. When the feedstock material is powder, the energy source for fusion is a laser, the state of fusion is total melting, and the AM machine is a powder feed system. When the feedstock material is wire, the energy source for fusion is either an electron beam or laser, the state of fusion is total melting, and the AM machine is a wire feed system [3]. Note that in DED, feedstock material and energy source deposit simultaneously to form the component design.

#### *2.2.2.1.1 DED Powder Feed Systems*

Powder feed systems use a deposition head or nozzle to provide powder transported by a carrier gas to the build plate. A laser, guided by a beam guidance system and focused by a lens, melts the deposited powder into the intended component format. These two steps iterate until the component is complete [2]. Figure 2-4 shows a general setup for a powder feed system.

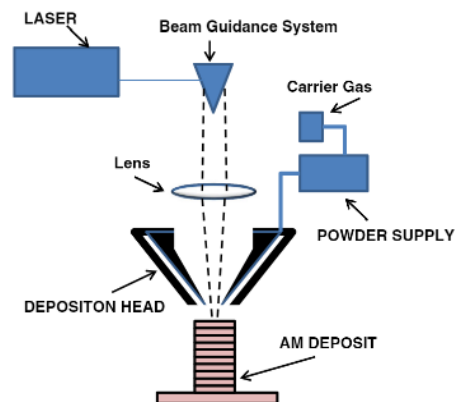


Figure 2-4: Design of a Standard AM Powder Feed System [2]

There are currently two main kinds of powder feed systems available. In the first kind of system, the component is stationary during its formation while the deposition head or nozzle moves by design to produce the desired final part. However, in the second kind of system, the deposition head remains stationary, leaving the build plate to move to create the intended design. Systems with fixed deposition heads can produce large build volumes and can also repair fatigued or broken components.

In general, powder feed systems typically form larger build volumes than those built by powder bed systems. For example, the Optomec LENS 850-R, a powder feed system, can print components with build volumes larger than 1.2 cubic meters [2].

#### *2.2.2.1.2 DED Wire Feed Systems*

Wire feed systems create components more rapidly than powder feed systems because of their high deposition rates. Additionally, these systems are more cost-effective than powder feed systems because of their high catchment efficiency. Catchment efficiency is the ratio of deposited material weight to supplied material weight [3].

Note that while wire feed systems can perform high deposition rate processes and efficiently form large build volume components, components formed by wire feed systems often need more post-process machining than those formed by powder feed systems. In a wire feed system, the material begins as a single bead and continually grows by subsequent material depositions to form the desired 3D component [2]. Figure 2-5 shows the general setup of a wire feed system.

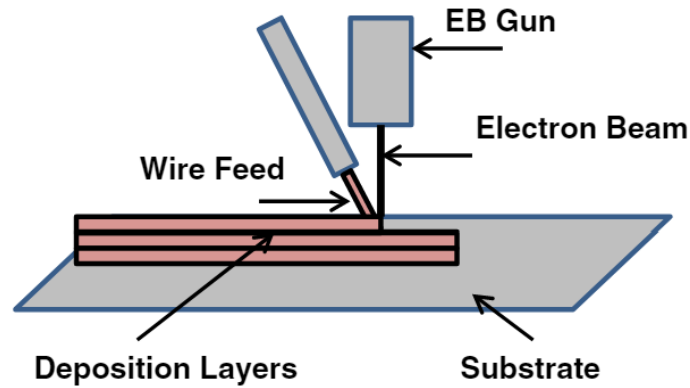


Figure 2-5: Design of a Standard AM Wire Feed System with Electron Beam Energy Source [2]

#### 2.2.2.2 Powder Bed Fusion (PBF)

Powder bed fusion (PBF) is the process carried out via powder bed printing. Each layer of the STL file for a component has a calculated heat source path which determines the boundary contour and filling sequence. In most cases, the filling sequence is a raster pattern that guides the heat source, typically a laser beam or electron beam. Every layer of the STL file bonds successively with the others. A roller or blade evenly distributes the powdered metal over the system build plate to prepare a new layer. Note that preferred layer thickness depends on operating conditions and the powdered metal used. Typical layer thicknesses range from 25 to 100 micrometers. Because the PBF process spreads powdered metal over the previously formed layer, manufacturing via PBF is not continuous but discrete [12].

PBF has multiple variants, each classified by the heat source and formed material. The two most common variants of PBF are laser beam PBF (PBF-LB) and electron beam PBF (PBF-EB). LPBF requires a ytterbium fiber laser to narrow into a small spot with enough energy intensity to melt a powdered metal layer. Moving galvanometer mirrors direct the spot over the build plate, creating a solid metal path connecting to surrounding paths and previous layers. The

shielding gas removes any emissions created during this process and prevents the metal from oxidizing. Weld path width depends on laser spot diameter, with each path two to three times greater than the laser spot diameter. Weld paths connect and overlap to form a single solid layer. Note that weld paths must have enough depth to remelt the previous layer slightly[13].

The two methods for powder melting are continuous scanning and modulated scanning. Continuous scanning continually provides laser energy and melts the metal powder by moving the laser beam over the build plate to solidify the desired powder regions. In this method, weld paths overlap and remelt the previous weld path to create a solid layer of welded metal. Alternatively, modulated scanning turns the laser on and off in short bursts. The powdered metal melted by each burst slightly covers the previously melted metal to create weld paths via two methods which are shown in Figure 2-6 [13].

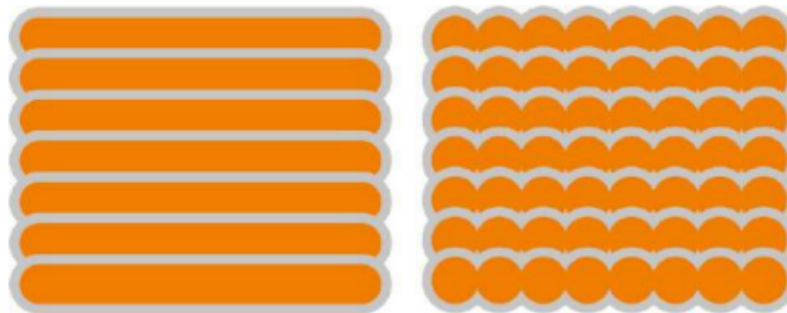


Figure 2-6: Weld paths created by continuous scanning (left) and modulated scanning (right) [13]

PBF-LB has several forms, including selective laser sintering (SLS) and selective laser melting (SLM). SLS is a trademarked expression describing a process that sinters powdered polymers using a laser. Like SLS, SLM is also a trademarked expression describing a process that uses lasers. However, unlike SLS, SLM completely melts materials instead of sintering them. This process usually melts metal powders, including aluminum and titanium alloys and

stainless steel. In addition, SLM uses inert atmospheric gas such as argon within the build chamber to prevent material oxidation during the forming process [12].

The most common form of PBF-EB is electron beam melting (EBM). EBM is an identical process to SLM with two exceptions. The first exception is that EBM uses an electron beam instead of melting powdered metal with a laser like SLM. The second exception is the use of a vacuum environment instead of inert gas inside the operating chamber. Note that a little bit of helium gas is often inserted into the vacuum environment to control the EBM process parameters[12].

Regardless of the form of PBF, postprocessing often improves the component properties. Heat treatments are applied to decrease component residual stress and improve mechanical properties. Surface treatment, often in grit blasting and chemical or laser polishing, enhances component surface finish [12].

#### *2.2.2.2.1 Powder Bed Systems*

Powder bed systems traditionally form components with build volumes of less than 0.03 cubic meters. Figure 2-7 shows the layout of a standard powder bed system. The basis of all powder bed systems is the powder bed. The powder bed takes form as the roller or rake spreads powder across the build area. The energy source of a powder bed system is either a laser beam or an electron beam. This energy source follows programming, melting, or sintering specific portions of the powder bed to create the designed component. As the process proceeds, the powder bed is incrementally adjusted vertically and refilled with powder. The energy source selectively melts or sinters the powder, building the component layer by layer [11].

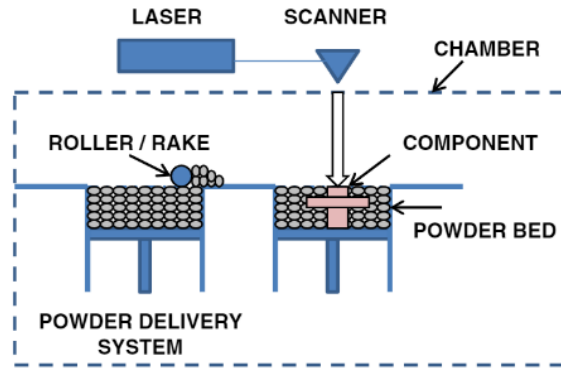


Figure 2-7: Diagram of Standard AM Powder Bed System [11]

Powder bed systems provide the ability for the creation of high-resolution features and internal passages in components. Additionally, powder bed systems allow for consistent dimensional control [11].

### 2.2.3 Process of Metal Additive Manufacturing

Regardless of the metal AM processing category, every metal AM has five phases, design, pre-processing, printing, quality assurance, and postprocessing[14]. Figure 2-8 displays the flowchart of these five parts.

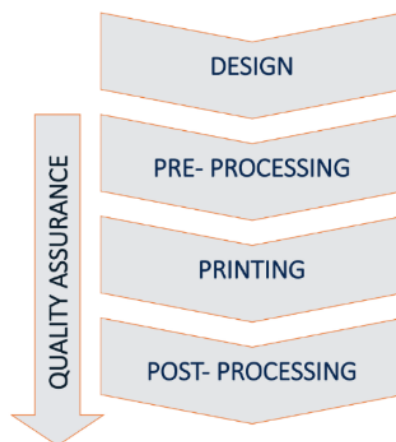


Figure 2-8:The five phases of the metal AM process [14]

The initial phase of any metal AM process is design. The goal of the design phase is to create a component that can perform the desired function and be efficiently manufactured. Note that the intended metal AM process category must be kept in mind during design as each one has parameters that may dictate component build volume and geometry [14].

The pre-processing phase follows the design phase in preparation for the printing phase. Two steps are involved in pre-processing. The first is converting the 3D CAD design file into a Standard Tessellation Language (STL) file[14]. CAD files must be translated to allow the printer to understand the design. STL files are formed by discretizing or slicing CAD file geometry into triangles and an accompanying normal vector, x, y, z coordinates, and file header. Note that triangle size dictates STL file accuracy. Smaller triangles lead to a higher accuracy file[2].

In addition to creating slices of the CAD file, the STL file forms a toolpath for the metal AM machine to follow. This toolpath includes information on position and printing parameters, such as printing power, for each layer. Determining the previously mentioned printing parameters is complex and often iterative as the metal component's quality and accuracy are dependent on printing parameters. The next step in pre-processing is machine preparation, including alignment of the build plate, preparation of the printing atmosphere with shielding gas, and loading the desired printer feedstock[14].

Following pre-processing, printing begins. Printing requires minimal supervision or attention. In many cases, printing takes place without any human involvement or observation. Depending on build volume and the AM machine used, printing time ranges from minutes to days. In many cases, the build plate must heat before printing, adding significant time to the overall build time. Another source of additional build time is heat treatment during the printing

cycle for processes that require stress relief. However, except for heat treatment, most of the printing phase is hands-free[14].

The postprocessing phase is vital to the quality of metal AM components. The steps of the postprocessing phase depend on the metal AM process, equipment, and application. These steps often involve iteration and testing to meet component specifications. Postprocessing includes five steps, build removal, part separation, machining, surface finishing, and heat treatment [14].

Build removal is the process of removing leftover material from the build and printer, removing the build geometry from the AM printer, and inspecting the build geometry for quality and accuracy. Part separation separates parts from the build plate, other parts, and any component supports. Electrical discharge machining (EDM), bandsaws, or clippers perform this process[14].

Machining removes any supports remaining on the components after build removal, smooths component surfaces, and adjusts dimensions to achieve design tolerance. This step also includes the creation of any custom fixtures for additional manufacturing phases[14].

Surface finishing involves two processes. The first process is the polishing of surfaces that do not meet the required component surface roughness. The second process is tumbling or shot peening which smooths and work-hardens surfaces and removes excess powder on unfinished components [14].

Heat treatment of the component takes place at various steps throughout the metal AM printing process. At each of these steps, heat treatment performs a different role. Heat treatment after build geometry removal reduces residual stress. Hot Isostatic Pressing (HIP), a heat

treatment used in powder feedstock processes, after part separation lessens component porosity. Heat treatment tempers the material before and after machining to meet component hardness and microstructural requirements[14].

Quality assurance (QA) is the inspection, measurement, analysis, and documentation performed to ensure that the component consistently meets design specifications. QA is present throughout the entire metal AM process because metal AM's repeatability depends on difficult-to-control process parameters[14].

#### ***2.2.4 Metal Additive Manufacturing Process Parameters***

Desired material properties drive metal AM process parameters selection. Chosen parameters must satisfy material strength and integrity requirements. In addition, process parameters are crucial for AM build repeatability as they ensure quality and consistency [5]. This section outlines the process parameters for the two most common metal AM processes, PBF and DED.

All AM metal printing methods' production goals and benefits are the same: high-resolution components, simple process, low production cost, and short production and processing timeframe [1]. Additionally, the defects found from AM metal printing methods share a root cause. This cause is the incorrect control of process parameters. Process parameters maintain product quality and repeatability. Process parameters also influence the melt pool size and shape, determining the component quality and consistency [2].

#### 2.2.4.1 LPBF Process Parameters

LPBF process parameters must produce the required laser energy and laser energy speed for a given material. There are six critical process parameters for LPBF- laser power, spot size, scanning velocity, hatch distance, and layer thickness. *Laser power* is the total energy emitted by the laser per unit of time. *Spot size* is the diameter of the laser beam after it is focused. The laser spot size is either constant or variable, depending on the LPBF machine. *Scanning velocity* is the speed with which the laser spot follows a scan vector across the build plate. *Hatch distance* is the amount of space between scan vectors. This space depends on the amount of remelting needed to form a complete metal layer. Finally, the *layer thickness* is the thickness of each powder layer melted. Note that process parameter selection is a multi-variable problem because the listed parameters can be adjusted individually[13].

Regardless of material or metal AM process, process parameters must result in a continuous, non-porous component. This criterion is essential as it allows the component to meet its strength requirements. The task of deciding the correct group of process parameters seems daunting. However, a simplified understanding of the relationship between the parameters makes the task straightforward. For any component geometry, several things remain constant, reducing the number of variables. The first two constants are powder composition and particle size distribution, as the same material composes the entire component. Layer thickness is also constant to accomplish the desired component resolution and surface finish. Laser spot size is also constant since most powder AM machines do not allow for its variance[13].

Now only three variables remain- laser power, scanning velocity, and hatch distance. When plotted against each other, laser power and scanning velocity give information about

component density. Figure 2-9 shows the plotted parameters and potential defects when the parameters reach extreme values[13].

High values of either parameter lead to defect formation within the printed component. Suppose scanning velocity is high and laser power is low. The part then has areas that do not completely melt, creating defects called lack of fusion defects and porosity. Conversely, high laser power and low scanning velocity overheat the melt pool leading to deep energy penetration and the formation of keyhole defects which contribute to porosity. In the middle of the two extreme cases is an ideal operating window. In this region, laser power completely melts the powder in the current layer and remelts the correct metal beneath the powder layer. Figure 2-9 shows that laser power and scanning velocity can be increased simultaneously for faster build speeds. While this is true, it is also true that melt pool stability limits build speed. The melt pool becomes unstable if the build speed is too great, creating material beading or "balling up." High laser power also increases material spatter[13].

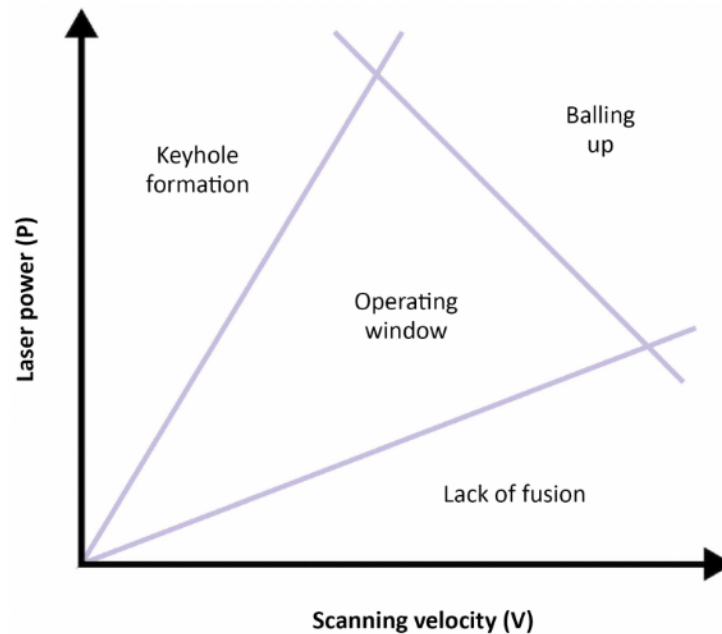


Figure 2-9:Laser power vs. scanning velocity plotted with possible defects and an ideal operating window [13]

When laser power and scanning velocity are within the ideal operating window, laser recoil pressure forms a shallow cavity in the melt pool. The laser moves the cavity by heating its front face and creating a metal vapor column that ejects perpendicular to the cavity surface and travels behind the cavity. Because the cavity does not permit internal reflections, no additional melting takes place. Instead, the heat energy flows turbulently into the melt pool, causing some weld splatter. Note that the traveling metal vapor column creates an agitated environment that pulls some powder towards the laser beam and then pushes it out. After passing through the laser, some powder heats while the remainder blows in the shielding gas flow around the laser[13].

#### 2.2.4.2 Defects in LPBF

Describing possible defects in materials formed by the LPBF process gives insight into the relationships between process parameters.

##### 2.2.4.2.1 Lack of Fusion Defects

Lack-of-fusion defects occur because of insufficient laser power at a given scanning speed. This combination of parameters forms a smaller than typical melt pool, which solidifies quickly and has low turbulence and little spatter. The metal vapor column or plume from these parameters is less forceful than typical, pulling less powder into the laser. The low laser energy causes shallow laser penetration, causing the incomplete melting of both the powder and the previous solid metal layers. Unmelted powder rests beneath the melt pool and causes increased porosity and higher delamination risk [13]. Figure 2-10 shows the formation process resulting in lack-of-fusion defects.

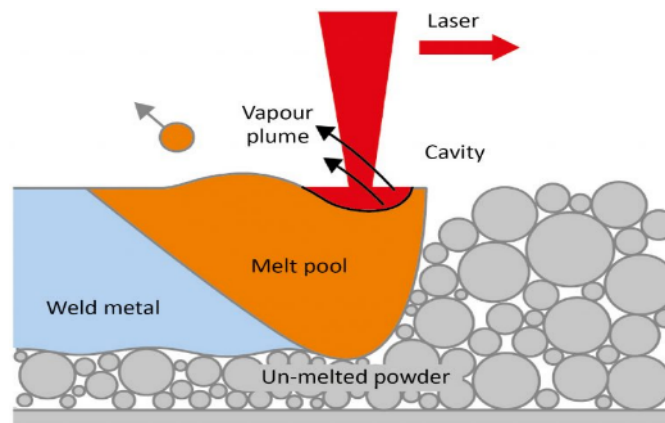


Figure 2-10: Formation process of lack of fusion defects in PBF (modified from[13])

##### 2.2.4.2.2 Keyhole Defects

Keyhole defects are the result of excessive laser power for a given scanning speed. The laser passes through the metal powder layer and into the previously formed metal, creating a

keyhole. The high laser power creates a deep melt pool and causes the metal vapor column to eject more vertically than is typical. Additionally, as the laser energy reflects in the cavity, heat becomes trapped deep within the material, creating a melt pool that is deeper and longer-lasting than usual.

The laser energy also creates increased turbulence in the melt pool, additional material spatter, and more powder accumulation within the laser path. If keyhole deformations become unstable, the melt pool collapses into the cavity and creates an inert gas pocket that causes porosity below the formed metal's surface. It is important to note that this porosity impacts the material microstructure [13].

#### *2.2.4.2.3 Impact of Laser Power, Scanning Speed, and Melt Pool Depth on Defects*

The National Institute of Standards and Technology's (NIST) research shows that melt pool size depends on scanning speed and laser power. If laser power is held constant, variations in scanning speed do not impact the melt pool length but affect the melt pool width. Low scanning speeds lead to broad melting pools and consequently form melt pools with large areas. Note that melt pool width and depth directly depend on each other. A wider melt pool has greater depth than a narrower one [8]. Recall that melt pool depth determines defect formation. Deep melt pools cause keyhole defects, while excessively shallow melt pools create lack of fusion defects. High scanning speeds cause melt pool instability and surface tension gradients which cause melt pool separation. The separated melt pool cools and solidifies into beads, leading to the "balling up effect" [5].

While melting during the PBF process impacts the component density, the primary determinant of component mechanical properties is material solidification. This solidification determines the material microstructure, which determines the material's mechanical properties. Recall that material microstructure, especially metal alloys', is complicated and often composed of multiple phases [5].

These phases depend on both alloy composition and temperature. This variety means that solidification does not occur instantaneously or uniformly across a metal layer. During cooling, most heat escapes from the melt pool and flows into the solidified material. The external edges of the melt pool reach a temperature below the liquidus temperature of the material, causing solidification of at least one phase of the alloy. Cellular dendritic crystals grow from the external edges of the melt pool into the center. The other liquid phases remain between the dendrites and only solidify when they reach their melting points, lower than those of the dendritic alloy phases. Grain boundaries form from opposing cellular dendritic growth fronts. As the material cools, strain accumulates on the grain boundaries, leading to porosity caused by solidification cracking. Solidification cracking is the most intense when there are vast phase solidification temperature differences [5].

The thermal response of any material depends on the melt pool size, duration, and cooling rate. A large melt pool lasts for an extended period and cools slowly, resulting in a coarse microstructure with large grains and thick dendrites. Conversely, if a melt pool is small, it cools quickly, forming a fine microstructure with small grains and thin dendrites. Deep melt pools lead to remelting of solidified material which impacts the material microstructure. Recall that deep melt pools result from high laser power. High laser power forms long columnar vertical grains that cross several material layers. The deeper the melt pool, the greater the contact with

previously solidified material, and the more heat conducted downwards, increasing vertical grain alignment. Vertical grain alignment causes material mechanical properties to differ with the component build direction [5].

#### *2.2.4.2.4 Energy Density*

An optimum operating window combines scanning speed and laser power to form a melt pool with perfect depth, width, and duration. This combination is called energy density which is the amount of energy applied to the material per unit volume. For a given material and layer thickness, an optimized energy density processes the material efficiently and provides the desired microstructure, fulfilling both material property and productivity requirements[13].

If the energy density is constant, laser power and scanning velocity relate inversely to each other. Thus, the plot of laser power and scanning speed in Figure 2-9 can expand to include energy density contours that radiate from the plot origin and couple the density to the contour gradient. Any chosen material and layer thickness has an ideal energy density that allows for efficient component processing and desired microstructure. Selected parameters should lie as far along the contour as possible without meeting the "balling up" area[13].

Knowledge of energy density is incomplete without the inclusion of hatch distance. Figure 2-9 assumes that hatch distance remains constant and determines energy density only from laser power and scan velocity. However, hatch distance can vary independently from laser power and scan velocity. Therefore, it is possible to adjust scan velocity, hatch distance, and laser power to reach the desired energy density contour. Figure 2-11 describes the relationship between energy density, laser power, scan velocity, hatch distance, and layer thickness[13].

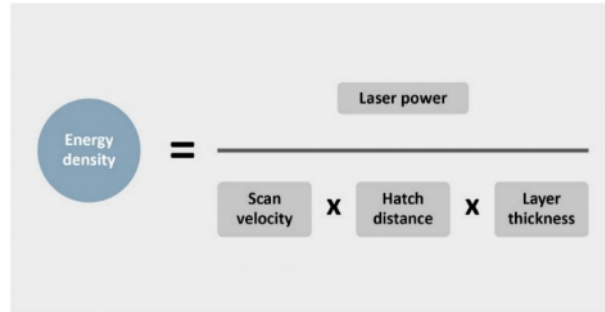


Figure 2-11: Relationship between energy density and four process parameters: laser power, scan velocity, hatch distance, and layer thickness [13].

Figure 2-12 shows three orange contours of equal energy density. Choosing a high ratio between laser power and scanning velocity requires increased hatch spacing to maintain the desired energy density. Deep penetrating laser beams create vast, deep melt pools that allow a lot of space between each other yet still fuse. Choosing a low ratio between laser power and scanning velocity requires decreased hatch spacing to maintain the desired energy density. Shallow penetrating laser beams create small, shallow melt pools that must be close to each other to fuse. Thus, choosing a hatch distance is best to allow a laser power-scanning velocity ratio far away from keyhole deformation or lack of fusion deformation processing regions [13]. Figure 2-12 denotes optimal hatch distance with a red X.

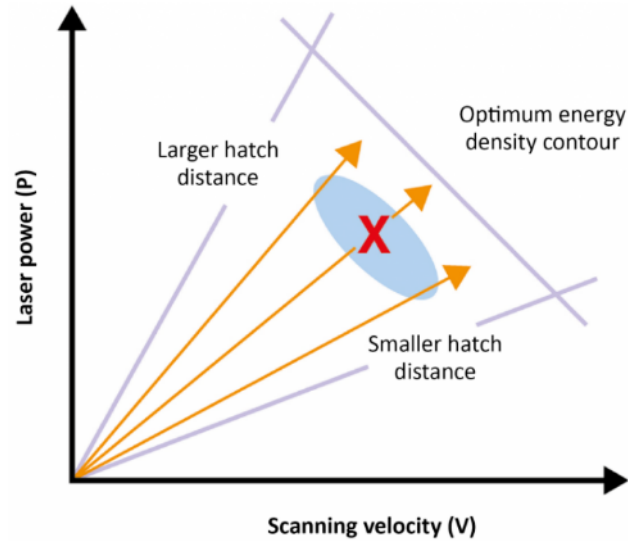


Figure 2-12: Impact of hatch distance on energy density; optimal hatch distance is indicated with a red X [13].

#### 2.2.4.2.5 Layer Thickness

Altering layer thickness impacts build rate and component surface finish. Thicker layers allow fast build rates but create rough surface finishes. There is a limit to increasing build rates via layer thickness. As layer thickness increases, laser penetration must also increase to fuse the layers completely. Layer thickness and energy input per layer increase simultaneously to fully melt the material and optimize volumetric energy. Thus, the slope of the energy density contour increases[13].

Increasing layer thickness also increases the lack of fusion defect region. It narrows the ideal operating window until the layers are so thick that the laser cannot penetrate them and simultaneously maintain a stable melt pool and adequate layer to layer fusion. Ideal layer thickness depends on material but is typically between 30 and 90 microns for laser spot diameters of 70 to 100 microns and laser powers of at most 500 W[13].

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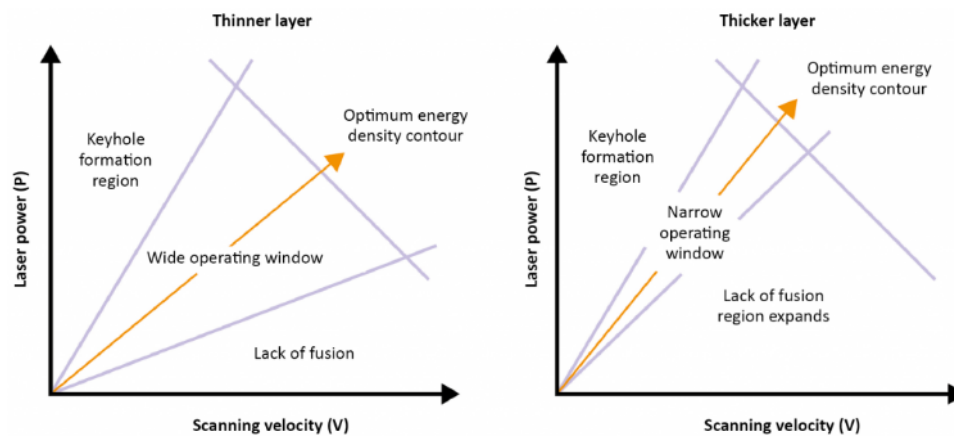


Figure 2-13: Narrowing of the ideal operating window as layer thickness increases[13].

Ideal layer thickness depends on material but is typically between 30 and 90 microns for laser spot diameters of 70 to 100 microns and laser powers of at most 500 W [13].

The best process parameters lie in the middle of a large operating window because they allow for a factor of safety. This safety factor is essential because thermal conditions vary in a component since each additional material layer causes heat conduction into the previous layers. Dispersion of the conducted heat depends on both component geometry and material properties.

Heat disperses most efficiently when the component has an excellent thermal connection to the build plate. However, if the component geometry has thin walls or inconsistent geometry, heat flows inefficiently and retains heat at the narrowest region[13]. Figure 2-14 shows the heat conduction for two different component geometries.

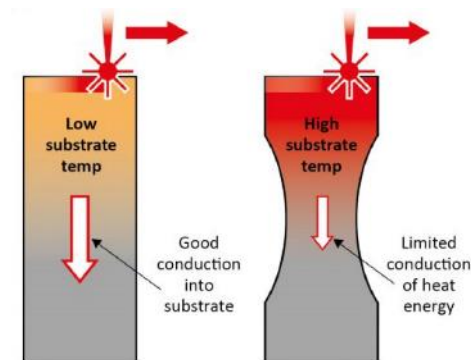


Figure 2-14:Heat conduction based on component geometry [13].

Note that these trends are most evident in materials with low thermal conductivity, like titanium aluminum alloys. From an understanding of heat conduction in various component geometries, it is clear that forming thin-walled components with thick material layers is complex and inefficient[13].

#### 2.2.4.3 DED Process Parameters

Like LPBF, DED succeeds only with the correct process parameters. Given the correct process parameters, a quality component forms from connected hatches and well-bonded material layers. Important DED process parameters include scanning speed, laser power, and powder and gas flow rates[15].

Note that all the primary DED processing parameters interact closely, making the system sensitive to small changes in any parameters[16]. Researchers determine the interactions of the parameters by performing single-track bead-on-plate material depositions. Specifically, these depositions help estimate hatch and layer spacing and their dependent laser, powder, and translation parameters. While process parameters are material-dependent, researchers have formed general conclusions about their interaction and impact on mechanical properties and component porosity[15].

DED is, by nature, a non-equilibrium manufacturing process with rapid cooling rates, transient temperature flow, transient heat flow, and complicated transport phenomena. Fast cooling and subsequent remelting repeat cyclically, leading to a complex thermal history throughout the formed component. These cycles impact the formed microstructure, defects, surface roughness, and mechanical properties. Figure 2-15 shows thermal cycling for three consecutive DED-formed layers. Note that the peak temperature increases with each layer. This increase is due to the accumulation of heat in the component [17].

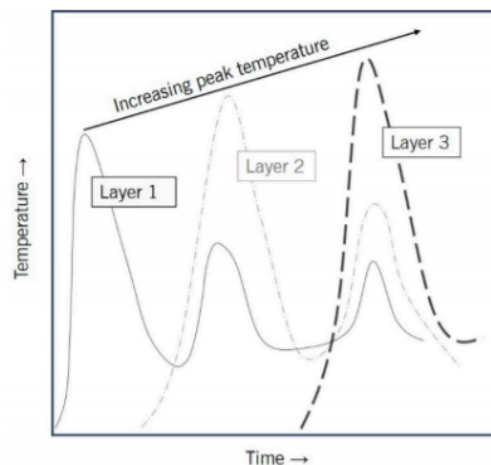


Figure 2-15: Depiction of three consecutive DED layers, indicating increasing peak temperature due to heat accumulation within the layers [17].

## **2.3 Impact of Process Parameters on Microstructure and Material Properties**

Traditionally manufactured components and AM components have differing metallurgical compositions because the AM process leads to inconsistent mechanical properties across geometry (anisotropy), defects, and residual stress[18]. AM results are directly related to the process parameters used during manufacturing. Inappropriate process parameters cause defects that directly impact the material surface finish and mechanical properties. The laser or electron beam movement causes a cycle of fast heating, melting, and subsequent alloy solidification during the AM process. The layer-by-layer nature of AM formation means that the cycle repeats multiple times in sections of the formed component, creating inconsistent material microstructure and properties[18]. This section will discuss potential defects in metal AM components, residual stress, and process parameter maintenance.

### **2.3.1 Defects**

#### *2.3.1.1 Loss of Alloying Elements*

AM of metal alloys creates melt pools with high temperatures. These high temperatures can vaporize alloying elements within the metal alloys, altering overall alloy composition [19]. This alteration changes the printed alloy's final microstructure, quality, and mechanical properties[18].

#### *2.3.1.2 Porosity and Lack of Fusion*

Two commonly occurring defects in additively manufactured metal alloys are porosity and lack-of-fusion voids. Both of these defects negatively impact an alloy's mechanical properties and must be avoided [18]. Three factors typically produce porosity and lack of fusion defects.

The first of these factors is power density. When power density is high, the process becomes a keyhole mode. Unless carefully monitored and controlled, keyhole mode melting creates unstable keyholes, which continually form and collapse, leading to voids [20]. These voids trap vapor and make spherical porosities whose size depends on the keyhole size and shape, as seen in Figure 2-16a [21].

The second factor capable of creating porosity and lack of fusion defects is powder atomization. During this process, gas may become trapped inside the powder particles, resulting in the formation of microscopic spherical pores[22]. Additionally, shielding gas and alloy vapors may become trapped inside the melt pool and form gas pores[18].

Finally, incomplete melt pool penetration into the substrate or deposited layer may create lack of fusion defects. These lack of fusion defects are at least 10 mm in diameter and are significantly larger than pores caused by trapped gas[19, 23]. Additionally, these lack-of-fusion defects have sharp edges, which create stress concentrations during external loading[18]. Figure 2-16b shows both lack-of-fusion pores and gas-induced porosity.

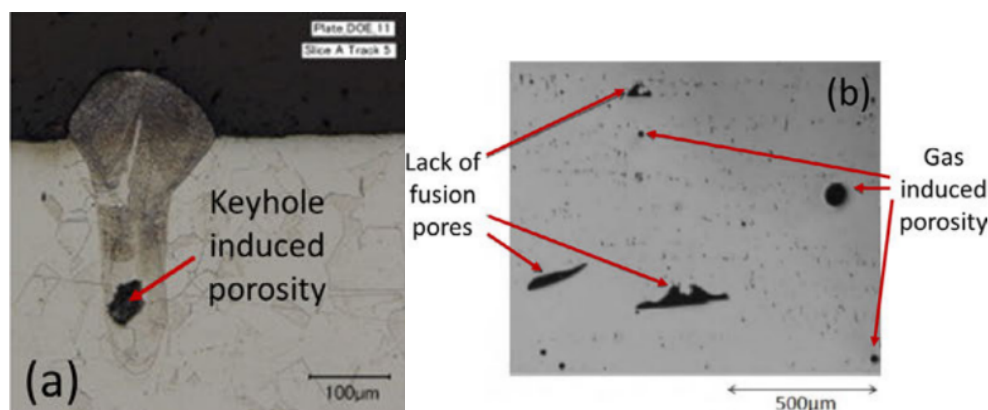


Figure 2-16: a) A keyhole porosity in AM 316L stainless steel[20] and b) lack-of-fusion pores and gas-induced porosity [24]

The size of lack-of-fusion defects is estimated using the "lack-of-fusion index" (LF) in Equation 2-1.

$$LF = \frac{\text{Melt Pool Depth}}{\text{Layer Thickness}} \quad (2-1)$$

The ideal melt pool is large with adequate penetration into the last layer deposited so that the layers bond correctly and contain minimal lack-of-fusion voids[19]. Thus, a sizeable lack-of-fusion index correlates to low numbers of lack-of-fusion voids. Melt pool depth is directly related to the ratio between laser power and laser scanning speed, known as the heat input per unit length. High heat input per unit length means deeper melt pools and fewer lack-of-fusion voids. Conversely, if the heat input per unit length is low, scanning speed is too high, reducing melt pool size and increasing the number of lack-of-fusion voids[25]. However, an increase in laser power will increase melt pool size and melt pool penetration, thus decreasing the porosity of the final material[26].

#### 2.3.1.3 *Cracking and Delamination*

AM components exhibit two common cracking mechanisms[27]. The first is solidification cracking, which occurs along the grain boundaries of the formed material [28]. Solidification shrinkage and thermal contraction cause the solidifying metal alloy to contract. Since the substrate and previously deposited layers have lower temperatures than the depositing layer, the depositing layer contracts more severely than either the substrate or the previously deposited layers, restricting the contraction of the solidifying layer. Thus, tensile stress forms at the solidifying layer. If the tensile stress is higher than the solidifying layer's strength, it causes the solidifying layer to crack along its grain boundaries[29]. Note that while an additively

manufactured metal's void fraction decreases with increased energy density, there is no direct relationship between energy density and cracking density [18].

The second cracking mechanism is liquation cracking, which occurs in either the formed material's mushy or partially melted zones[30]. Note that the mushy zone is the difference between the liquidus and solidus temperatures of an alloy.

The partially melted area experiences quick heating below an alloy's liquidus temperature, which causes several grain boundary precipitate phases to liquefy, including carbides with low melting points. As the material cools, a tensile force forms in the partially melted zone because of solidification shrinkage and deposit thermal contraction. This force causes the liquid around grain boundary phases and carbides to act as cracking sites. Alloys most susceptible to liquation cracking have broad mushy zones, high shrinkage due to solidification, and high coefficients of thermal expansion. Aluminum and titanium alloys tend to have many of these characteristics[28].

Cracks in additively manufactured parts can span multiple layers or remain in a single layer[31]. However, delamination causes two consecutively deposited layers to separate. This separation occurs when the residual stress at the layer interfaces is greater than the alloy's yield strength[32].

### **2.3.2 Residual Stress**

While defects are avoidable, residual stress is inherent to the AM process and can only be understood and mitigated. The root cause of residual stress is the deposition of hot liquid metal alloy on material layers or a substrate of a lower temperature. This temperature difference leads to thermal strain, a steep temperature gradient, and residual stress [33, 34]. The consequences of

excessive residual stress include part distortion, reduced geometric tolerance, layer delamination during the build process, and reduced fatigue life and fracture toughness of the final part[33, 34].

Residual stress may also negatively impact the AM printing process. For example, part distortion due to residual stress may be so significant that it prevents the roller of powder bed AM systems from distributing powder over the component surface. Optimally placed support structures, based on an understanding of thermal stress, help prevent such part distortion. Note, however, that residual stresses due to AM are both spatially diverse and build time-dependent [18]. Residual stresses in additive manufacturing originate from the spatial temperature gradient formed from thermal cycling due to the laser or electron beam, thermal expansion and contraction due to the thermal cycling, and variable distribution of inelastic strain (also known as strain compatibility)[18].

To reduce residual stresses and distortion during the AM process, researchers recommend preheating the substrate, decreasing the deposition length, increasing scanning speed, and decreasing layer thickness. Substrate preheating reduces residual stress by reducing the temperature difference between the manufactured part and the substrate, which reduces the thermal strain. Preheating the substrate is highly effective as it reduces the final residual stresses in a fabricated component and reduces the stresses during the fabrication process[35].

### ***2.3.3 Maintenance of Process Parameters***

AM process parameters must be consistent and accurate to produce high-quality AM parts. Therefore, for industrial AM applications, monitoring and in-situ measurement of process parameters is common. Currently, melt pool dimensions and temperature are the essential parameters to monitor[36]. This section outlines measurement methods for temperature and melt pool dimensions, including the advantages and disadvantages of each technique.

### *2.3.3.1 Temperature Measurement*

The most common process parameter measured is temperature[36]. A pyrometer, any instrument used to measure high temperatures, is sufficient for measuring temperature during the AM process. The most common pyrometers used for the AM process are digital cameras and photodiodes. Each measures the temperature by converting light to an electrical signal- in each case, light intensity and temperature are proportionate. Pyrometers are advantageous because they allow no-contact temperature measurement and sit near the heat source.

A disadvantage of pyrometers is that they only perform surface temperature measurements, making complete thermal knowledge of the melt pool impossible. Furthermore, since they convert emitted surface radiation to temperature, pyrometers depend on accurate physical properties. Unfortunately, these properties are not always easily obtained, especially when the manufactured alloys are rare[36-44].

Another typical instrument for temperature measurement is the thermocouple[45-48]. Thermocouples are less expensive than pyrometers. Additionally, they are spatially accurate and can measure AM build internal temperatures. To measure internal temperature, operators typically install thermocouples on the substrate before the deposition. Unfortunately, this placement is a disadvantage because the thermocouple will only contact the initially deposited layer, making temperature measurement of subsequent higher layers impossible[18].

### *2.3.3.2 Melt Pool Geometry*

For desirable final AM component quality, melt pool geometry must be consistent during the entire build time. Unfortunately, melt pool geometry typically varies throughout the build process because of inconsistent thermal cycles and accumulated heat. Therefore, high-speed digital cameras, which collect a large number of images in a short period, are typically used to

monitor changes in melt pool geometry. Processing all of the photos in real-time requires much data and processing power. Furthermore, digital cameras are limited to collecting images of the melt pool surface geometry, ignoring the impact of melt pool depth on material quality[18].

## **2.4 Material Information**

The most used feedstock materials for additive manufacturing using laser or electron beams are alloy powders. Alloy powders are favorable because they flow easily through the AM system, and it is easy to control their melting. Additionally, operators can mix several powdered alloys to create components with composite or gradient properties. Such parts are challenging to make using traditional manufacturing. While alloy powders are convenient, they can be difficult to manufacture at high quality because of their large surface area and tendency to oxidize. The manufacturing method of alloy powders is directly related to their final properties[18].

Four powder alloy manufacturing methods exist gas atomization, rotary atomization, plasma rotating electrode process, and water atomization. Gas atomization (GA) is a process that reduces a melted alloy into tiny particles using high-pressure argon and nitrogen gas[49]. The rotary atomization (RA) process reduces the alloy into particles by spinning poured melted alloy on a rotating disk. Particles of the alloy fly from the disk and solidify as a powder[50]. In the plasma rotating electrode process (PREP), powdered metal solidifies from metal ejected from a rotating bar[51, 52]. Finally, in the water atomization process (WA), a high-pressure water jet breaks the molten metal into particles, creating a powder[53].

Powders produced by the four manufacturing methods each have a different shape. PREP powders are perfect spheres with smooth surfaces. RA powders are also polished but have

another profile. GA powders are spherical with dimpled surfaces. Finally, WA particles have an irregular shape and rough surfaces, hindering their flow through an AM system. Because of their lack of flow, WA particles tend to form thinner layers than GA powders[54, 55]. WA particles also form components with high surface roughness[54]. Of note, PREP-created powders have the most uniform size distributions, while WA-processed powders have the least uniform size distributions[24, 54]. Size distribution uniformity is essential for homogeneous melting, structure, mechanical properties, sufficient bonding between part layers, and surface finish[18, 56]. Finally, GA-formed powders typically have trapped gas bubbles which cause part porosity[57].

When alloy powders have smooth surfaces and uniform size distribution, they flow smoothly through powder nozzles and create small melt pool sizes. Thus, DED and PBF AM processes provide the best results when using such alloy powders. Unfortunately, alloy powders of high quality tend to be expensive because the PREP process is costly with low powder yield. Powders. To help reduce the production cost, operators of PBF systems often reuse unmelted powder. Note, however, that reused powders have irregular shapes and create parts with rough surface finishes[56]. Thus, powdered alloy feedstock decisions must be well-informed, accounting for quality, cost, and AM process.

#### ***2.4.1 Material Properties of Precipitation Hardened Stainless Steels***

Most AM processes mimic standard welding processes, including arcs, lasers, and electron beams. Therefore, many of the structures produced by AM are similar to those produced by multipass welds. Since AM processes are nearly identical to welding processes, welding process-structure-property relationships and AM process-structure-property relationships should also be similar[28, 58, 59]. However, AM structures differ from traditionally welded structures

because the scanning speed of the heat source is greater in AM than in welding. This higher scanning speed allows for control of each material layer in the formed part[18]. This section outlines the variables that impact the final solidification, grain, and micro-structure of AM parts.

#### *2.4.1.1 Solidification of AM Metals*

Both the crystal texture and grain structure of an AM printed part are dependent on the melting and solidification of the melt pool. As heat from the melt pool flows from the melt pool into the substrate, it creates a curve in the melt pool shape. Therefore, process parameters directly impact melt pool form. For example, energy intensity and scanning speed determine the melt pool's surface and cross-section geometry [28, 58, 60, 61].

A melt pool's geometric features determine the material's solidification rate and the shape of future material grains in the fusion zone. Keyhole operation, commonly used in welding, uses a deeply penetrating beam. This beam penetrates the metal substrate using the minimum amount of heat input[61]. At high speeds, keyhole operation enters conduction mode[20, 62]. Conduction mode is ideal for AM processes because it is stable, reducing porosity in AM parts. Note that the shape of melt pools during AM is essential to create components with preferred grain structures[63, 64]. The following sections discuss the solidification of AM material, detailing nucleation and growth. Furthermore, the sections discuss the impact of essential parameters on AM material solidification microstructure, such as temperature gradient ( $G$ ), solidification rate ( $R$ ), and undercooling( $\Delta T$ )[18].

##### *2.4.1.1.1 Nucleation*

In a typical AM process, each layer of metal has an identical chemical structure as its predecessor. This identical chemical make-up means that nucleation of the next phase is unnecessary for the metal to regenerate epitaxially from the substrate since the layers all share

crystal composition[28, 58]. Note that the use of identical metals in AM fabrication does not need nucleation. Instead, they need only epitaxial growth from the print substrate. The lack of nucleation occurs randomly when the temperature is below liquidus temperature, and the activation energy barrier is non-existent. However, sometimes metals used in AM fabrication have different chemical compositions to create functionally graded materials, requiring nucleation at the melt pool boundary[65, 66]. In such cases, new phases require nucleation before any solid-phase growth happens in the melt pool. When nucleation is necessary at the liquid-solid interface, the solid phase must overcome an energy barrier. This energy barrier impacts the microstructure and mechanical properties of the printed part[67, 68].

#### *2.4.1.1.2 Growth*

In AM solidification, growth takes place in the material layers. This growth shapes the crystal structure of the printed part since the thermal cycling in the AM process partially or entirely melts previous layers[69]. Therefore, high heat intensity is vital to create quality AM parts since higher heat intensity penetrates multiple previously deposited layers. Additionally, the higher heat intensity degrades oxide films, eliminates impurities at the material surface, and atomically cleans the solid-liquid interface [70]. As an AM alloy solidifies, its microstructural composition varies with location. Closest to the melt pool boundary, the microstructural composition is primarily the base metal[25, 28, 71].

In contrast, the primary composition away from the melt pool boundary is competitive growth[28, 72]. When dendrites in polycrystalline materials have multiple crystal orientations, competitive growth occurs[28, 73]. The competitive growth phenomenon occurs in many alloys, including nickel, titanium, and tantalum[67, 68, 74-76]. The dendrites that achieve the most growth during solidification have easy-growth directions situated near the path of the highest

heat flow at the solid-liquid interface. Table 2-1 lists crystal structures and their respective easy growth directions.

Table 2-1: Direction of Easy Growth for Material Crystal Structures [28]

| Crystal structure              | Easy-growth direction        | Examples                                     |
|--------------------------------|------------------------------|----------------------------------------------|
| Face-centered-cubic (fcc)      | $\langle 100 \rangle$        | Aluminum alloys, austenitic stainless steels |
| Body-centered-cubic (bcc)      | $\langle 100 \rangle$        | Carbon steels, ferritic stainless steels     |
| Hexagonal-close-packed (hcp)   | $\langle 10\bar{1}0 \rangle$ | Titanium, magnesium                          |
| Body-centered-tetragonal (bct) | $\langle 110 \rangle$        | Tin                                          |

Note that materials with face-centered and body-centered cubic crystals share an easy growth direction of  $\langle 100 \rangle$ . As such, during solidification, polycrystalline body-centered and face-centered materials experience preferred growth when the base metal grains align with the  $\langle 100 \rangle$  direction. In addition, the shape of the melt pool boundary determines the direction of heat flow at the liquid-solid interface. As dendrites aligned with the temperature gradient grow more quickly than those not aligned, columnar grain structures form[63, 64, 77, 78]. Therefore, both growth direction and solidification structure depend on crystal orientation and direction of heat flow. Finally, note that competitive dendrite growth helps determine the final solidification structure of AM materials [18].

#### 2.4.1.1.3 Determination of Solidification Structure

AM solidification structure depends on processing parameters and scanning strategy. Processing parameters include scanning speed, laser power, laser spot size, powder layer thickness, and powder grain size distribution, while the scanning strategy determines the quality of the finished AM material [79]. For each combination of processing parameters, the temperature gradient ( $G$ ), solidification rate ( $R$ ), and undercooling ( $\Delta T$ ) determine the solidification microstructure of the printed AM material.

A solidification map displays the relationship between plots  $G$  and  $R$  and their relationships  $GR$  and  $G/R$ . Note that the  $G$  to  $R$  ( $G/R$ ) ratio directly impacts solidification mode, and  $GR$ , the product of  $G$  and  $R$ , determines the size of the solidification microstructure[80].

Figure 2-17 is an example of a solidification map. Figure 2-17 demonstrates that an AM material's solidification microstructure is planar, cellular, columnar dendritic, or equiaxed dendritic depending on the ratio between  $G$  and  $R$ . A high  $G/R$  value means planar microstructure, and a low  $G/R$  value means an equiaxed microstructure. The size of the solidification microstructure depends on the cooling rate. A high cooling rate creates a polished microstructure, while a low cooling rate forms a coarse microstructure. Note that solidification maps outline an alloy's final microstructure after solidification. Therefore, solidification maps are essential for an accurate study of alloy solidification, particularly for nickel-based, single crystal alloys[49, 81-100].

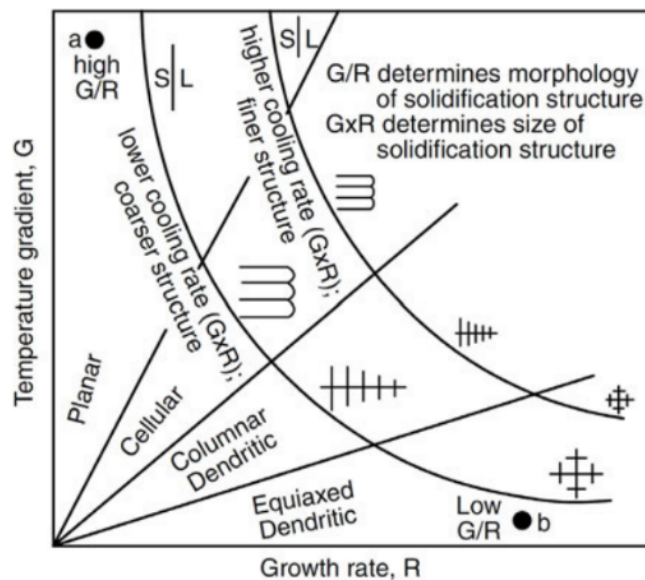


Figure 2-17: A solidification map demonstrating how the relationship between  $G$  and  $R$  impacts the solidification microstructure[28]

Once solidified, an ideal single-crystal AM microstructure has aligned columnar dendrites which grow from the substrate. Additionally, the perfect microstructure has no equiaxed dendrites. Solidification growth rate and temperature gradient characterize individual crystals placed by the additive manufacturing process. Based on the growth rate and temperature gradient, AM crystal structure will either shift from columnar to equiaxed, transition from oriented to misoriented or, create single grains[69, 70, 86, 92, 94, 98-102].

#### *2.4.1.1.4 Undercooling*

Undercooling stimulates dendrite growth. Total undercooling is the difference between the material's equilibrium liquidus temperature and local dendrite tip temperature. Total undercooling comprises four parts, solute diffusion, thermal diffusion, attachment kinetics, and solid-liquid interface curvature[28, 78]. When typical metal alloys solidify under normal conditions, solute diffusion dominates the undercooling composition. Note that solute diffusion undercooling is also called constitutional supercooling. An increase of constitutional supercooling leads to microstructural variation within the solidified AM metal [18].

#### *2.4.1.2 Grain formation of AM Metals*

The structure of grains created during solidification impacts a material's ability to resist solidification cracking and influences its mechanical properties. This section discusses grain size, grain morphology, and the direction and speed of grain growth. Additionally, this section shows grain structure evolution under different conditions[18, 103].

#### *2.4.1.2.1 Experimental Observations on Grain Growth Direction*

Powder bed AM machines with rapid scanning speed result in materials whose grain growth direction is in line with the  $\langle 001 \rangle$  direction [103-107]. However, grain growth direction often differs from the build direction in a DED process with a low scanning speed. Unlike columnar grains, which align with the build direction, curved columnar grains remain in a single material layer instead of going through multiple layers. Note that melt pool shape and size impact the orientation of the grain structure [68, 73, 75, 108].

#### *2.4.1.2.2 Mechanism of Grain Growth in Metal AM*

As mentioned in the above section, DED and PBF processes create differently shaped different grain orientations. This difference in grain shapes is because the melt pools in the two methods have different forms. Usually, columnar grains grow away from the grain boundary to the melt pool center. Grains of polycrystalline materials grow along the direction of maximum heat flow, which is perpendicular to the surface of the solidifying melt pool [28, 63]. The PBF process creates shallow, elongated melt pools. The shape of PBF melt pools shows that a material solidifies due to heat flowing in the opposite direction of the build direction. Conversely, a melt pool created during the DED process is deep and short, with a curved edge at its back end. Cooling in such a melt pool is dependent on the melt pool boundary position, which impacts the direction of heat flow.

When the scanning speed of the DED process is fast, the melt pool bottom is horizontal. As a result, the grains grow perpendicular to the melt pool from the layer beneath the melt pool, along the build direction. Note that the melt pool shifts upward as it cools, and the columnar grains continue to grow upwards. Such grain growth is location-independent.

#### *2.4.1.3 Grain Growth Rate*

Grain growth rate ( $R$ ) depends on melt pool motion. Melt pool motion depends on scanning speed and the melt pool boundary location. At the edge of the melt pool, the grain growth rate is zero and increases as grains approach the melt pool center. Additionally, as scanning speed increases, the grain growth rate increases [63].

##### *2.4.1.3.1 Grain Size*

Grain size in AM parts depends on cooling rate and scanning pattern. The grain structure is small for a high cooling rate, while the grain size is larger at low cooling rates. When the cooling rate is high, the growth rate is low, resulting in small grains. Recall that the cooling rate decreases with an increasing number of layers [109, 110]. Therefore, layers furthest from the build plate will be more coarse in an AM part than those in lower layers because of their lower cooling rate [108]. If scanning strategy leads to competitive growth, the resulting solidified structure will have few, large grains[68, 111].

#### *2.4.2 Phase Transformation of Metal alloys*

As mentioned in previous sections, processing parameters impact the microstructure of a solidified AM structure. However, after the AM metal has cooled beneath its solidus temperature and solidified, the material continues to cool until it reaches room temperature. As it continues to cool, the metal will experience more phase transformations. Furthermore, when the AM layers experience reheating during the printing process, phase transformations may occur, the residual stress state of the part may change, and the formed part may experience distortion. This section outlines the typical solid-state phase transformations that heat-treatable alloys experience[18].

#### *2.4.2.1 Heat Treatable Alloys*

Heat-treatable alloys experience phase transformations during heating and cooling. These phase transformations change the properties of the alloys. Usually, traditionally formed alloys undergo heat treatment to increase their strength and make other positive changes to their mechanical and physical properties. Common heat-treatable alloys are aluminum alloys, high strength steels, titanium alloys, and nickel-based alloys.

Note that heat-treatable alloys are hard to manufacture using AM methods because they have high strength, reducing their ductility. This reduced flexibility can cause cracking during the AM process [112]. Furthermore, to create parts with consistent, high-strength properties, AM parts typically undergo post-heat treatment [112, 113]. Phase transformations often occur during post-heat treatment.

#### *2.4.3 Microstructure of AM Manufactured Stainless Steel Alloys*

The macro and microstructure of an AM material determine the mechanical properties of that material. Recall that AM processing parameters, which determine solidification rate ( $R$ ) and temperature gradient ( $G$ ), also shape the microstructure of the melt pool as it solidifies [80]. Therefore, these particular processing parameters,  $G$  and  $R$ , also shape the melt pool. The melt pool shape impacts the growth direction of the grains. Recall that the preferred growth direction of the grains in the fusion zone (FZ) is along the temperature gradient. This growth direction causes the grain structure within the FZ to be columnar [63, 64, 80]. Because these columnar grains are in the same direction as the temperature gradient, they grow perpendicular to the melt pool surface [114].

Most additively manufactured austenitic stainless steels have microstructures composed of cells and columnar dendrites. Such steels rarely have equiaxed structures because the temperature gradient of AM processes is too high [18]. In addition to austenite, austenitic stainless steels also contain some ferrite [108]. This structure forms because of solute rejection at the solidification interface. Note that the concentration of ferrite in the microstructure decreases as the cooling rate increases [115]. Microstructure, grain structure, and solidification structure depend on alloy chemical composition and manufacturing process [18].

## **2.5 AM Precipitation Hardening Steel Alloy Properties**

17-4PH SS is one of the most common precipitation hardening (PH) stainless steels manufactured with AM. Heat-treated, traditionally manufactured 17-4PH SS components have a higher yield strength than as-built AM components (not heat-treated) of the same material. This difference is because the AM components have soft austenite. Heat treatment of the AM components leads to some improvement of both their yield and ultimate strengths. However, even with the heat treatment, the yield and ultimate strengths of AM 17-4 SS are less than those of traditionally manufactured 17-4 SS [18].

## **2.6 Additive Manufacturing of 17-4 Precipitation Hardened Stainless Steel**

### **2.6.1 Background on 17-4 PH SS**

17-4 PH SS is a precipitation hardening steel that is desirable because it has high corrosion resistance, strength, and toughness[116]. This steel must often be heat treated after the AM process to be sufficiently strong and have good corrosion resistance [112, 116]. To achieve the desired state, AM 17-4 PH SS must experience aging heat treatment at temperatures between 480-620 °C because, at these temperatures, nanometric copper-rich precipitates precipitate uniformly [113, 117]. Aging treatment time is temperature-dependent and can take minutes or hours[18].

17-4 PH SS is valuable to many industries, including the chemical industry, marine industry, and power industry. One of the most used applications is creating molds for injection molding because 17-4 PH SS has high strength and wear resistance[18, 112, 113, 116-118]. Note that precipitation hardened stainless steels are stronger than both ferritic and austenitic stainless steels[119].

### **2.6.2 Microstructure of PBF 17-4 PH SS**

When produced via laser powder bed fusion (L-PBF) in an argon environment, the microstructure of 17-4PH SS was found to be mostly martensitic and contained some retained austenite [120]. PH stainless steels have three microstructural phases,  $\delta$ -ferrite, austenite, and martensite. The traditional order of solid-state phase transformation of these steels is first  $\delta$ -ferrite, then austenite, and finally martensite. Unfortunately, if undercooling rates are high, the typical phase diagram for 17-4 PH SS is no longer valid. Additionally, the cyclic heating and cooling during the L-PBF process may create anisotropy in the material properties and inconsistent microstructures in the final part [121]. This inconsistency means that the mechanical

properties in the build direction are different from those perpendicular to the build direction[122].

### **2.6.3 Conclusion**

As discussed throughout Section 2.2.4 and Section 2.3, process parameters determine an AM material's formation and resulting microstructure. Microstructure determines the resulting mechanical properties of the metal. Therefore, to determine the dependability and usefulness of 17-4 PH SS in industry, it is essential to understand how the printed metal responds to the chosen AM printing process.

### 3 Material Preparation

This section provides information about the tested material, including build parameters and the experiment design. The material used in this study is precipitation hardenable 17% Chromium- 4%Nickel Stainless Steel (PH 17-4SS). The manufacturing method for all tested samples was AM powder bed fusion. Note that the powder was argon atomized, and the printing environment was also argon. The powder bed fusion machine used for manufacturing was an EOS M 290, shown in Table 3-1. The machine operated at the standard specifications listed in Table 3-1. All samples used for this study underwent a post-process, three-stage heat treatment. The three stages of heat treatment performed were stress relief performed at 650 °C, solution treatment at 1050 °C, and aging at 550 °C.

Table 3-1: EOS M 290 PBF Printer Standard Specifications [123]

|                                              |                                                                   |
|----------------------------------------------|-------------------------------------------------------------------|
| Building Volume                              | 250×250×325 mm (9.85×9.85×12.8 in)                                |
| Note that height includes build plate height |                                                                   |
| Laser Type                                   | Yb fibre laser; 400 W                                             |
| Precision Optics                             | F-theta lens; high-speed scanner                                  |
| Scanning Speed                               | Up to 7.0 m/s (23 ft./sec)                                        |
| Focus Diameter                               | 100µm (0.004 in.)                                                 |
| Power Supply                                 | 32 A /400V                                                        |
| Power Consumption                            | max. 8.5 kW/average 2.4 kW/with platform heating up to 3.2 kW     |
| Inert Gas Supply                             | 7,000 hPa; 20 m <sup>3</sup> /h (102 psi; 706 ft <sup>3</sup> /h) |

Under the guidance of external scientists and engineers, engineers from the University of Maryland Baltimore County (UMBC) designed various structures to evaluate the AM of PH 17-4 SS. Each structure shows how geometry and build height impact the PBF AM build process and thermal history[124].

Figure 3-1 shows the designed build plate. Note that the build plate has four quadrants, as shown in Figure 3-2.

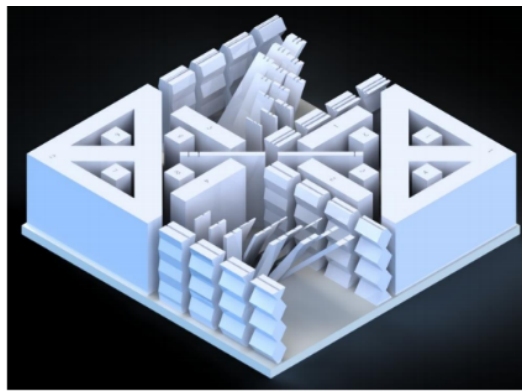


Figure 3-1: Build plate designed for this study[124]

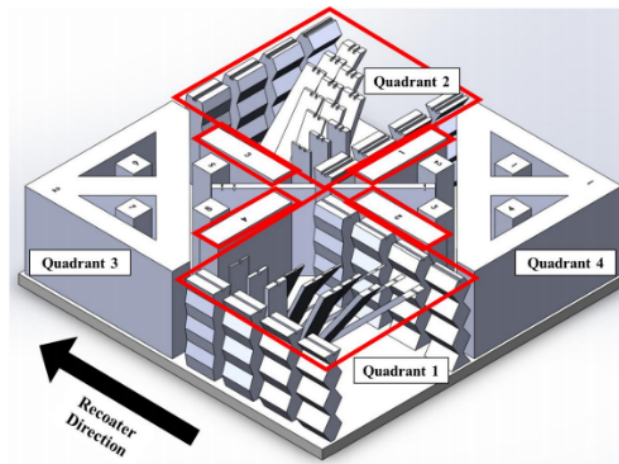


Figure 3-2:3D model of build plate with quadrants labeled[124]

Note from Figure 3-3 that Quadrant 1 and Quadrant 2 contain zig-zag structures, while Quadrant 3 and Quadrant 4 have rectangular structures.

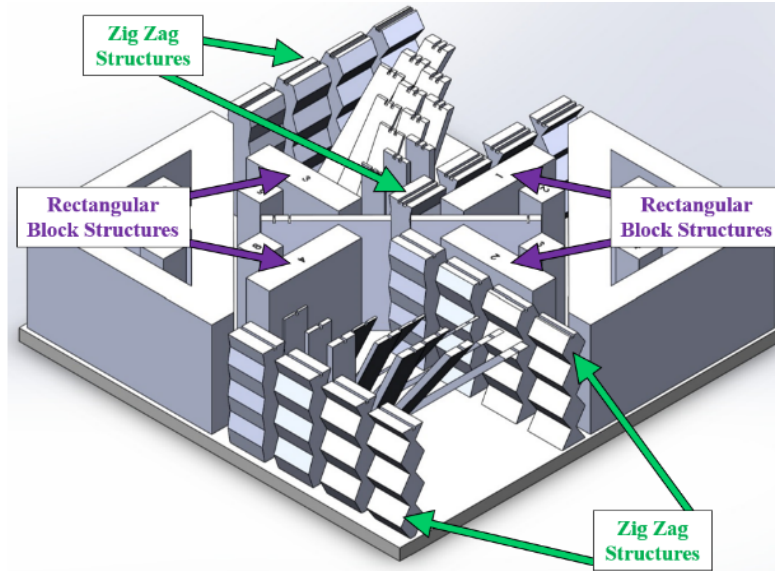


Figure 3-3: 3D model of the study build plate, indicating the zig-zag and rectangular structures[124]

Each build contained four zigzag structures with wide bases and four with narrow bases. Each set of four zigzag structures was printed in varying aspect ratios, with 20% the broadest and 80% the narrowest. The purpose of this variation in aspect ratio and base width was to show the impact of geometry on material hardness. Figure 3-4 shows the wide and narrow bases, while Figure 3-5 shows the variance in aspect ratio. Note that wide base structures are on the left of the build plate and narrow base structures are on the right side of the build plate [1].

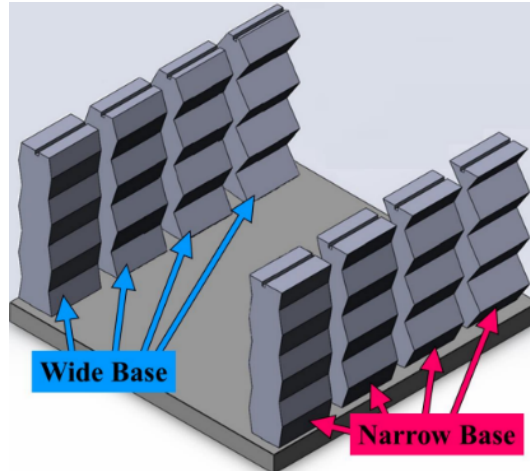


Figure 3-4: Wide and narrow base zig-zag structures on a 3D model of build plate[124]

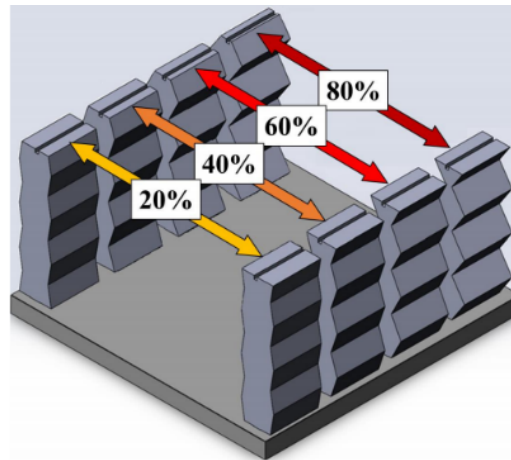


Figure 3-5: Zig-zag structures with reduction percentages labeled [124]

The rectangular pieces, Coupon 2 and Coupon 3, were in Quadrant 3 and Quadrant 4. As seen in Figure 3-6, each coupon was divided into five thinner slices. Meso tensile samples were cut from each piece, having midpoints 21.59 mm from the top of the slice.

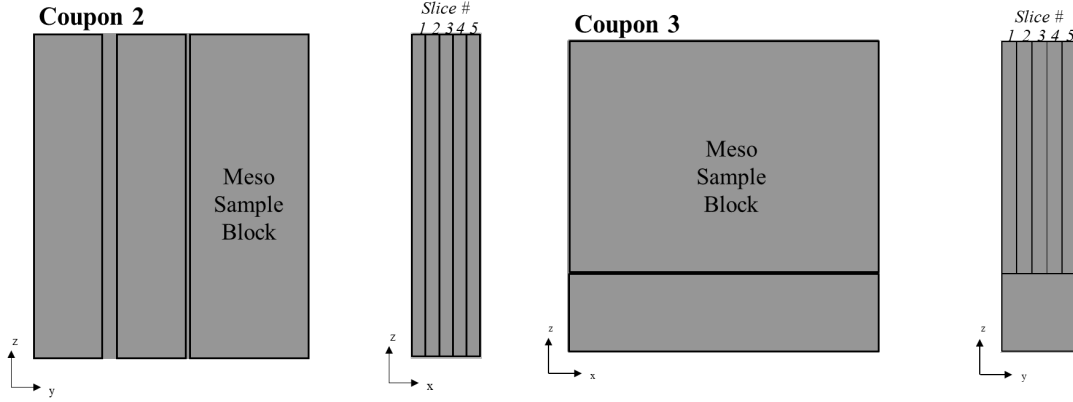


Figure 3-6: Rectangular bars from each build sliced into five sections

As seen in Figure 3-7 and Figure 3-8, meso samples cut from Coupon 2 were oriented differently than those cut from Coupon 3. Figure 3-7 shows that Coupon 2 meso samples have gauge sections parallel with the positive z-axis, while Figure 3-8 shows that Coupon 3 meso samples have gauge sections perpendicular to the same axis. These different orientations create two relations to the AM printing direction.

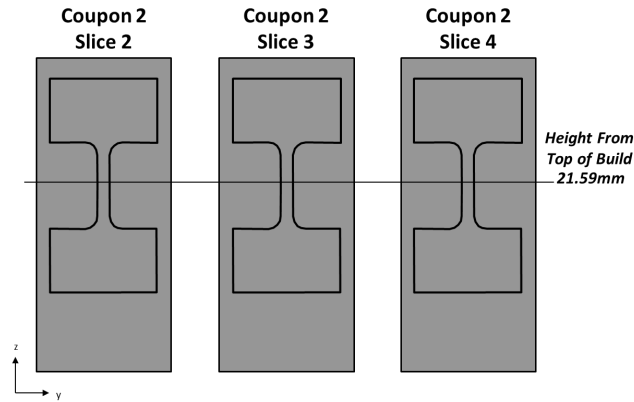


Figure 3-7: Mesosamples cut from Coupon 2 with gauge length parallel to the positive z-axis

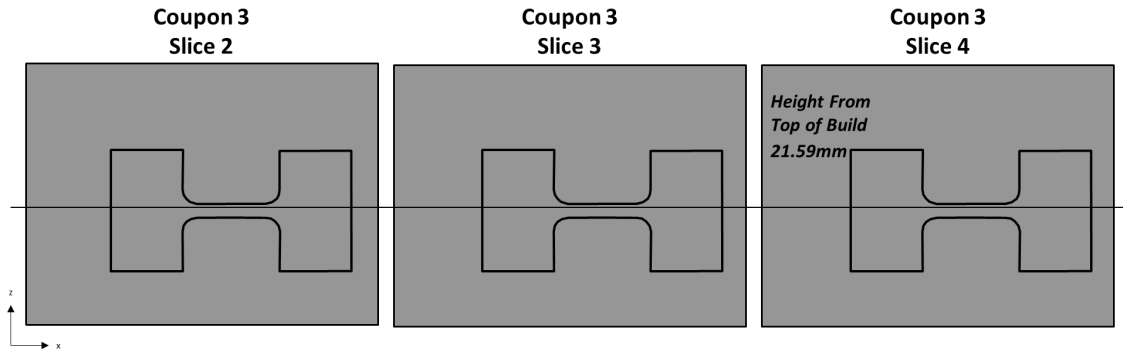


Figure 3-8: Mesosamples cut from Coupon 3 with gauge section perpendicular to the positive z-axis

Samples cut from Coupon 2 have gauge sections oriented along the printing direction, while Coupon 3 gauge sections are perpendicular to the printing direction. Figure 3-9 clearly shows this difference. For this study, only meso samples obtained from slices 2-4 underwent tension testing.

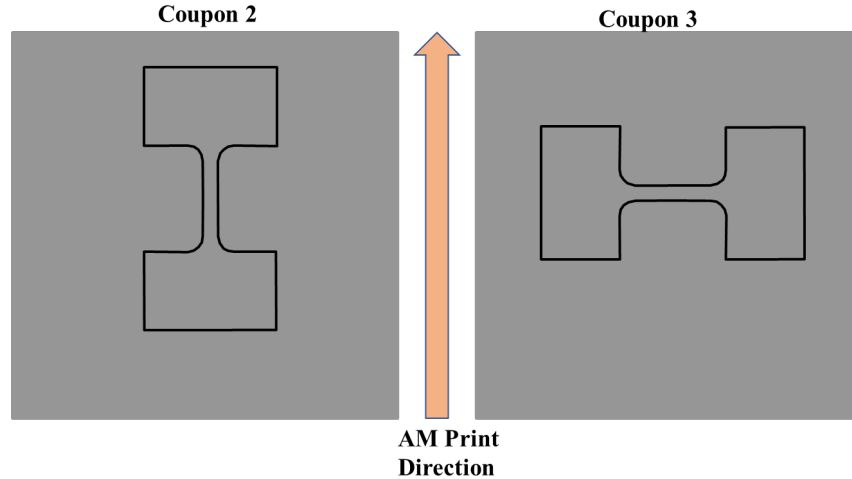


Figure 3-9: AM print direction and gauge section orientation for each coupon

In this study, three builds were printed on three EOS M290 machines housed at three separate locations. Table 3-2 shows details the number of builds formed on each printer.

Table 3-2: Number of AM builds manufactured at each printing location

| <b>Printer/ Location</b> | <b>Number of Builds</b> |
|--------------------------|-------------------------|
| <b>A</b>                 | 6                       |
| <b>B</b>                 | 1                       |
| <b>C</b>                 | 6                       |

Note that the build designation is in the order of printing. Meso sample naming is based on forming printer, build number, coupon number, and slice number. For example, a meso sample from the fifth build formed on printer C and cut from Slice 2 of Coupon 3 is designated sample C5-3-2. Zig-zag structure data in this study is averaged by base geometry, either wide or narrow. Thus, the narrow base zig-zag structures from the fifth printer C build are designated C5-N, while wide base structures from the same build are C5-W.

## 4 Hardness Testing

As noted in Section 3, zigzag structures were used to characterize the hardness of the material. Material hardness describes a material's resistance to indentation and gives qualitative information about material strength. An indent is made in the material to determine the material's hardness. Therefore, a load is applied to an indenter of a specific shape, creating a profile in the tested material. Two categories exist for hardness testing- macrohardness and microhardness testing. Macro hardness tests apply loads of more than 2 N, while microhardness tests use loads less than 2 N [125].

The method of hardness testing for this study was Vickers microhardness testing. This method depends on optical measurement and the use of a diamond-shaped indenter. First, the diamond-shaped indenter is placed under load and pressed into the material. Then, the size of the diamond-shaped indent created by the indenter is measured and subsequently converted to a Vickers Hardness value (HV). Note that this method requires smooth, well-polished materials for accurate results [126]. Figure 4-1 is an example of a Vickers indent with associated optical measurement markers.

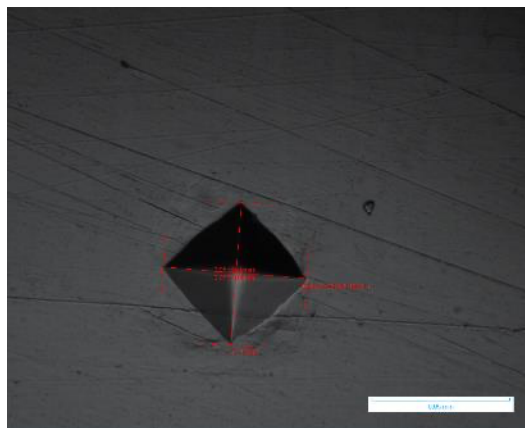


Figure 4-1: Vickers hardness indent from this study with optical measurement markers

## 4.1 Sample Preparation

Slices of 2.54 mm thickness were taken from each zigzag structure, as seen in Figure 4-2.

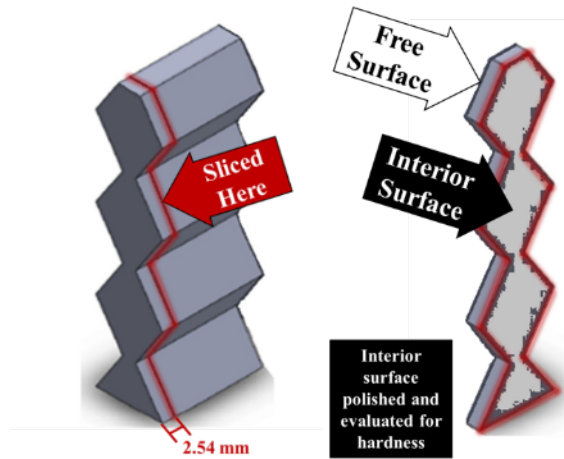


Figure 4-2: Illustration of zig-zag slicing [124]

Each slice's interior surface was polished using a typical polishing procedure to prepare the slice for hardness testing. First, each slice was polished on standard silicon carbide polishing sheets, starting with sheets of 180 grit and then continuing with 320, 600, 800, and 1200. At each grit, water was the polishing liquid. Note that polishing on the 320, 600, and 800 grits took place on the automatic polisher shown in Figure 4-3, while polishing on the 1200 grit was done by hand. Finally, each sample underwent final polish on a velvet fiber cloth, shown in Figure 4-3, with a polishing liquid of 0.05 $\mu$ m colloidal silica suspension [124].

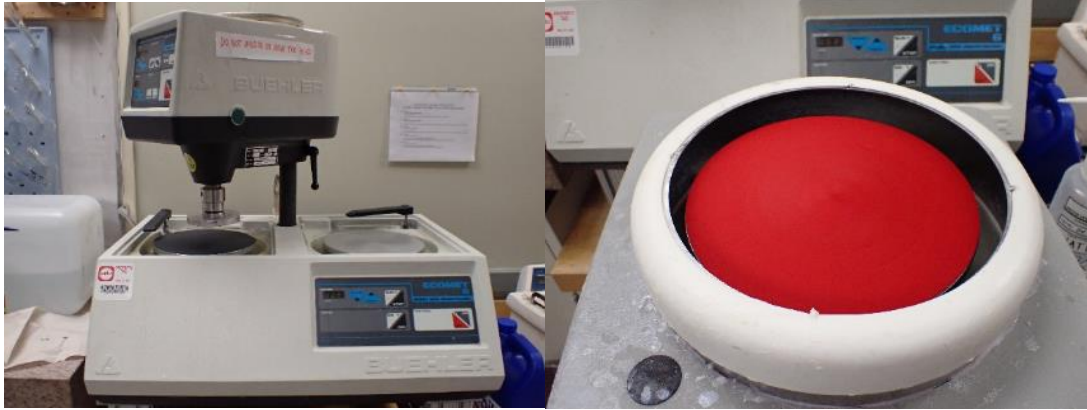


Figure 4-3: Automatic polisher used to perform initial polishing and the velvet fiber cloth used to perform the final polishing of samples

Next, hardness indents were made in 0.9 mm increments along the width of each sample slice. Note that for effective comparison, all hardness measurements were obtained from 20 mm above the build plate. From Figure 4-4, note that 20 mm is in a narrow portion of the component for the narrow base samples, while for the wide base samples, the 20 mm is in a wide portion of the sample. These heights were explicitly chosen to compare behavior at the different geometric configurations[1].

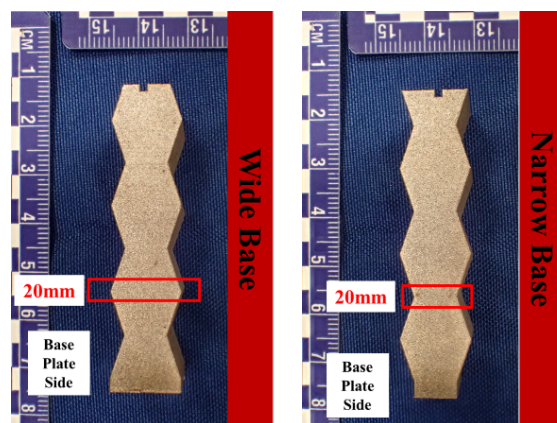


Figure 4-4: Wide and narrow base zigzag structures with hardness indentation locations marked [124]

## 4.2 Hardness Results

Figure 4-5 shows the relationship between build number and Vickers Hardness for wide base zig-zag structures printed at each print location.

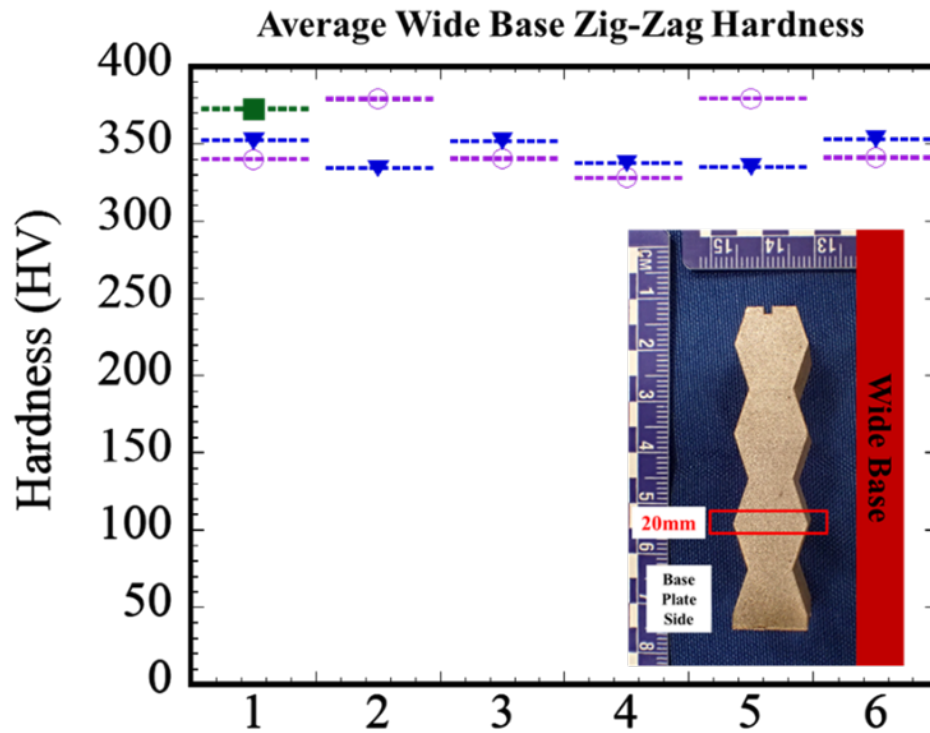


Figure 4-5: Average hardness of wide base zig-zag structures  
(Listed in printing order from left to right)

Figure 4-6 provides the same information for narrow base zig-zag structures. Note that the circular series of each plot is for print location A, the square series of each plot is for print location B, and finally, the triangular series is for print location C.

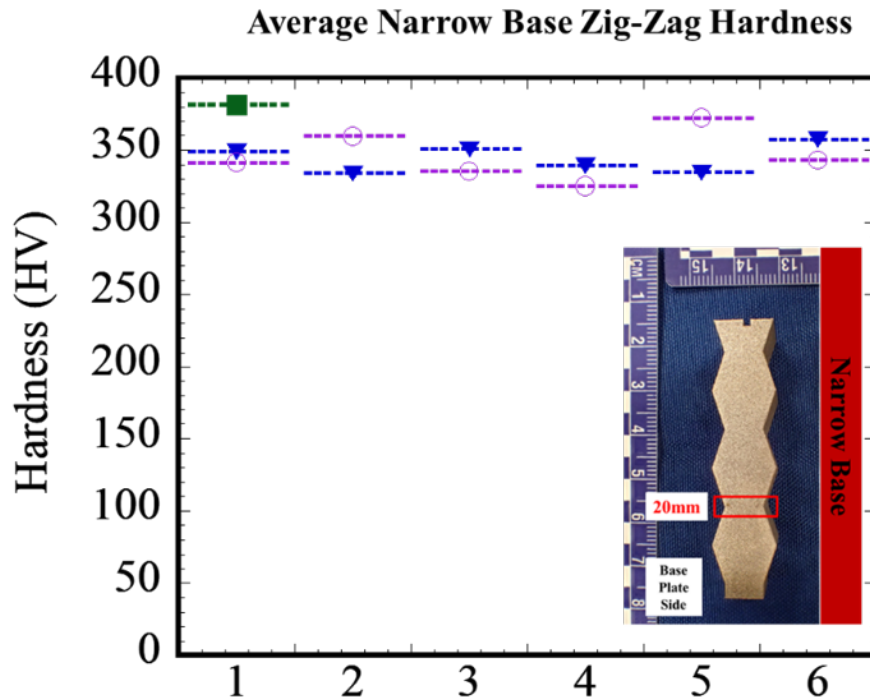


Figure 4-6: Average hardness of narrow base zig-zag structures  
(Listed in printing order from left to right)

In both plots, print location B has the highest average hardness of all the builds at any location. However, as print location B only created one build of samples, it is difficult to draw strong conclusions from these results. Table 4-1 and Table 4-2 list the average hardness values plotted in Figure 4-5 and Figure 4-6 respectively. These tables highlight that hardness values for builds from print locations A and C are quite similar and provide the coefficient of variation and standard deviation for the total hardness average at each location. It is worth noting that the hardness values of A-N and C-N are closer than those of A-W and C-W. Additionally, note that C-N has a higher average hardness than C-W.

Table 4-1: Average hardness values (in HV) of wide base zig-zag structures per build; average, standard deviation, and coefficient of variation are listed for each print location

| <b>Printer</b>                  |               |               |              |
|---------------------------------|---------------|---------------|--------------|
| <b>Build Number</b>             | <b>A-W</b>    | <b>B-W</b>    | <b>C-W</b>   |
| <b>1</b>                        | 339.86        | 372.62        | 352.10       |
| <b>2</b>                        | 378.92        | --            | 334.08       |
| <b>3</b>                        | 340.25        | --            | 351.67       |
| <b>4</b>                        | 327.84        | --            | 337.26       |
| <b>5</b>                        | 379.28        | --            | 335.04       |
| <b>6</b>                        | 341.12        | --            | 352.90       |
| <b>Average</b>                  | <b>351.21</b> | <b>372.62</b> | <b>343.8</b> |
| <b>Standard Deviation</b>       | <b>20.22</b>  | <b>0</b>      | <b>8.4</b>   |
| <b>Coefficient of Variation</b> | <b>0.058</b>  | <b>0</b>      | <b>0.024</b> |

Table 4-2: Average hardness values (in HV) of narrow base zig-zag structures per build; average, standard deviation, and coefficient of variation are listed for each print location

| Build Number                    | <i>Printer</i> |               |               |
|---------------------------------|----------------|---------------|---------------|
|                                 | A-N            | B-N           | C-N           |
| <b>1</b>                        | 341.32         | 381.25        | 349.02        |
| <b>2</b>                        | 359.84         | --            | 333.75        |
| <b>3</b>                        | 335.4          | --            | 350.59        |
| <b>4</b>                        | 324.79         | --            | 339.11        |
| <b>5</b>                        | 372.29         | --            | 334.65        |
| <b>6</b>                        | 343.03         | --            | 357.49        |
| <b>Average</b>                  | <b>346.11</b>  | <b>381.25</b> | <b>344.10</b> |
| <b>Standard Deviation</b>       | <b>15.68</b>   | <b>0</b>      | <b>8.82</b>   |
| <b>Coefficient of Variation</b> | <b>0.045</b>   | <b>0</b>      | <b>0.025</b>  |

It is worth mentioning that metal powder from previous builds was recycled, likely accounting for some variation within builds from the same location. Additionally, there is no direct conclusion on the impact of base geometry on the structure hardness.

## **5 Tension Testing**

Tension tests provided additional information about the properties and behavior of AM PH17-4 SS. This section outlines the procurement and preparation of tensile samples. Additionally, it outlines the knowledge gained from tensile testing.

### **5.1 Overview of Tension Testing**

Mechanical properties provide information about a material's response to load from force or movement. Specifically, when a component experiences a compressive or tensile force, its mechanical properties decide if it will survive the loading environment. Thus, tension testing provides the information needed to form a stress-strain curve-the applied load and the corresponding strain caused by the load. Stress is the ratio between applied load and the initial material cross-sectional area, and strain is the displacement or change in length of the material due to the stress [127].

Thus, stress-strain curves are the simplest way to understand how stress and strain are related to one another for any material. Figure 5-1 shows an example of a stress-strain diagram with the essential locations noted. This section describes the purpose of each of the sites and what material properties emerge from the stress-strain curve.

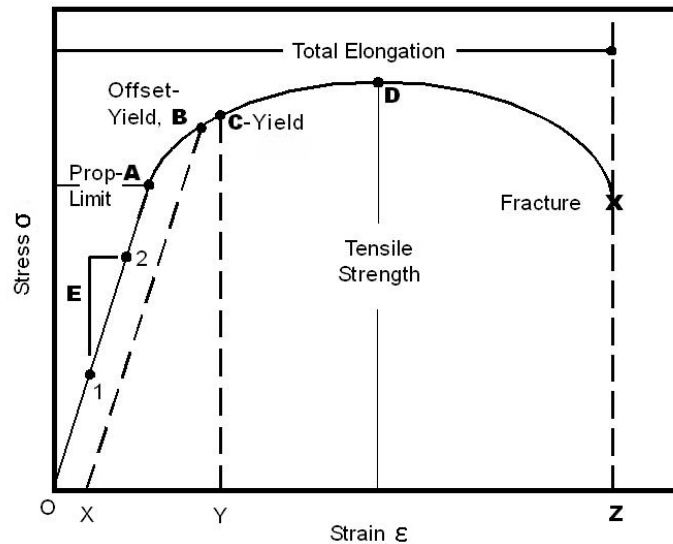


Figure 5-1: A typical stress-strain curve for a ductile metal with essential material properties denoted[127]

As seen in the figure, point A is the proportional limit of the material. Before the proportional limit, the stress and strain of the material relate proportionally, meaning that the ratio between the two quantities remains constant. In other words, if stress increases, the strain also increases. Therefore, the values used to calculate a material's Young's Modulus come from the proportional region. Essentially, Young's modulus is the slope of the stress-strain curve in the curve's proportional area. Equation 5-1 shows the calculation of Young's Modulus.

$$E = \sigma/\epsilon \quad (5-1)$$

This relationship is also Hooke's Law. Note that Young's Modulus describes a material's stiffness and allows for the calculation of a material's temporary deformation under applied load [127].

The proportional area of the stress-strain curve is also called the elastic region of a material. In this region, a material will deform during loading and return to its initial

configuration upon removal of the load. However, after the proportional limit, the material is no longer elastic. Instead, it behaves plastically, remaining deformed upon removal of the load. This area directly after the proportional limit is known as the elastic limit. In most materials, such as the one shown in Figure 5-1, the elastic and proportional limits are identical [127].

Point **C** of Figure 5-1 is the yield point of the material. At the yield point, a material becomes permanently deformed. Additionally, at this point, the material strain increases faster than the material stress, a phenomenon called strain hardening. Unfortunately, unlike the one shown in this figure, many materials lack a definite yield point, making the exact yield challenging to determine. As a result, the determination of the yield strength occurs at an offset. This offset yield strength is found by drawing a line parallel to the slope of the elastic region of the stress-strain curve and placing it at 0.002 on the strain axis. Then, the intersection point of the parallel line and the stress-strain curve is the offset yield strength, denoted as point **B** in the figure [127].

Point **D** in the figure is the tensile strength or ultimate tensile strength. At this point, the material experiences the most tensile stress that it can withstand without failure[127]. Following this point, the strain increases while the stress decreases until the material fractures. The strain at the fracture point is called the strain to failure.

## **5.2 Meso Sample Testing**

As mentioned in Section 5.1, tension testing demonstrates a material's response to loading, which allows the characterization of material behavior and mechanical properties. Mostly, these tests are performed on macro-sized samples. While macro samples can often provide the required

characterization behavior, they are often insufficient to provide more detailed information. For example, macro samples cannot determine biphasic material characteristics, fatigue life, or qualify an in-service component that cannot sustain a significant material loss [128].

Furthermore, macro samples are insufficient to characterize sheet metals because sheet metal specimens have a small thickness to length ratio, making them susceptible to buckling[129]. For the above reasons, the specimen dimension scale is reduced, creating smaller samples. This creation of smaller samples is called miniaturization[130]. Figure 5-2Figure 5-2 shows the standardized scales of miniaturization according to the National Institute of Standards and Technology (NIST).

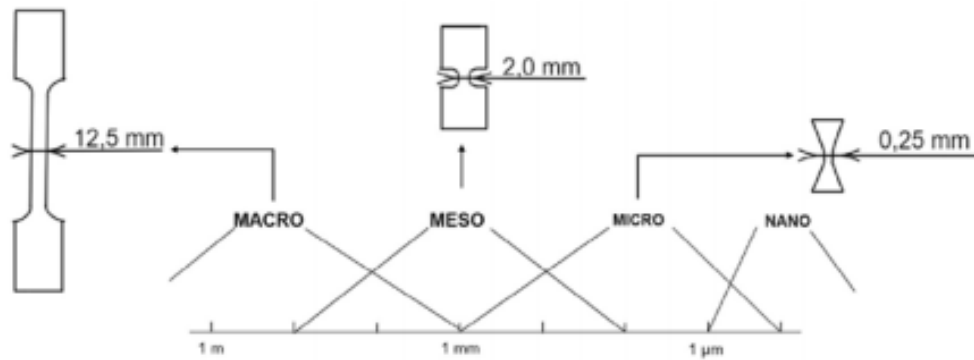


Figure 5-2:NIST Standardized Miniaturization Scales[130]

Figure 5-3 shows the dimensions of the mesoscale sample dimensions used for this study. The most relevant dimensions are the gauge section width and length. The gauge section width is 2.1 mm, placing the sample in the mesoscale. The gauge length of 9.8 mm is essential to the tensile testing process, as it is used to determine the sample strain.

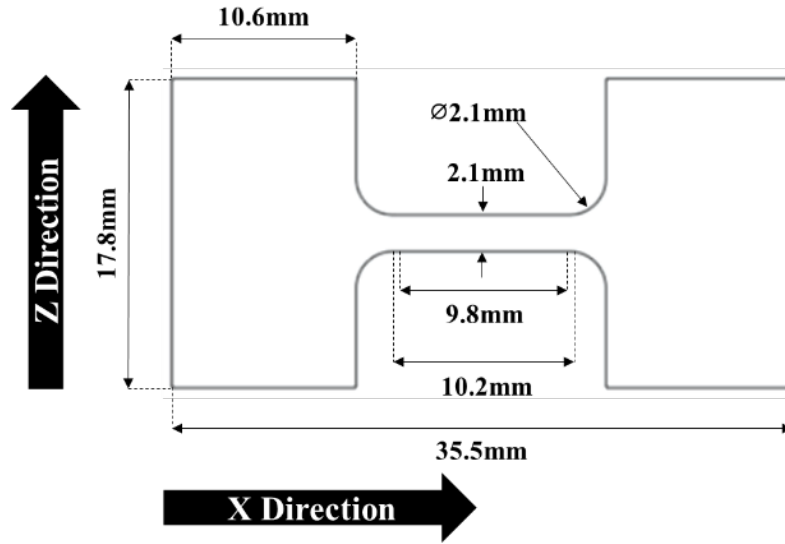


Figure 5-3: Study mesoscale sample dimensions [124]

### 5.3 Meso Sample Preparation from Prepared Material

Meso samples were cut via wire electrical discharge machining (EDM) from Coupon 2 and Coupon 3 of each build. Figure 5-4 shows a set of meso samples following the wire EDM process.



Figure 5-4: Meso samples directly after the wire EDM process

After the wire EDM process was complete, meso samples were separated by hand, labeled, and bagged. Once all samples for a particular build were separated, the polishing process began. The tension samples went through an identical polishing process as the zig-zag samples in Section 4.1.

Figure 5-5 shows a set of meso samples mounted and ready for the polishing process.

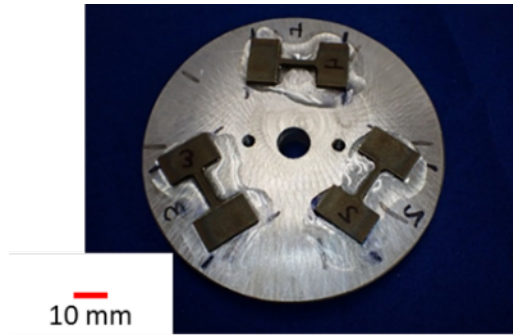


Figure 5-5: Meso samples mounted using crystal bond

#### 5.4 Tensile Testing Setup and Procedure

Before undergoing tension testing, each meso sample gauge length was measured using calipers. Then, a reflective tape was applied to the gauge section to indicate its height boundaries. The purpose of the reflective tape was to ensure that the external laser system used during tension testing would measure the gauge length accurately. Next, the sample was inserted into the Instron 3369 Dual Column Tabletop Testing System. Additionally, an external, non-contact laser extensometer was aligned with the system to measure the sample gauge length. Figure 5-6 shows the laser extensometer and tabletop testing system.



Figure 5-6: Non-contact laser extensometer and Instron 3369 Dual Column Tabletop Testing System

Figure 5-7 shows a taped meso sample mounted in the Instron 3369 Dual Column Tabletop Testing System.

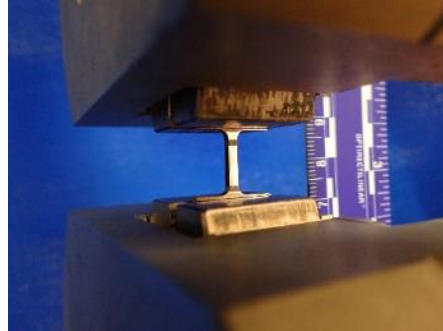


Figure 5-7: Meso sample mounted in the tabletop testing system

## 5.5 Tensile Results

Recall from Section 3 that for each print location, mesosamples were cut from Coupon 2 and Coupon 3. The difference between the mesosamples from each coupon is the orientation of the gauge section to the AM print direction. Figure 5-8, placed here for reference, is the exact figure shown in Section 3. The figure shows that Coupon 2 meso samples have gauge sections along the print direction, while Coupon 3 meso samples have gauge sections perpendicular to the print direction.

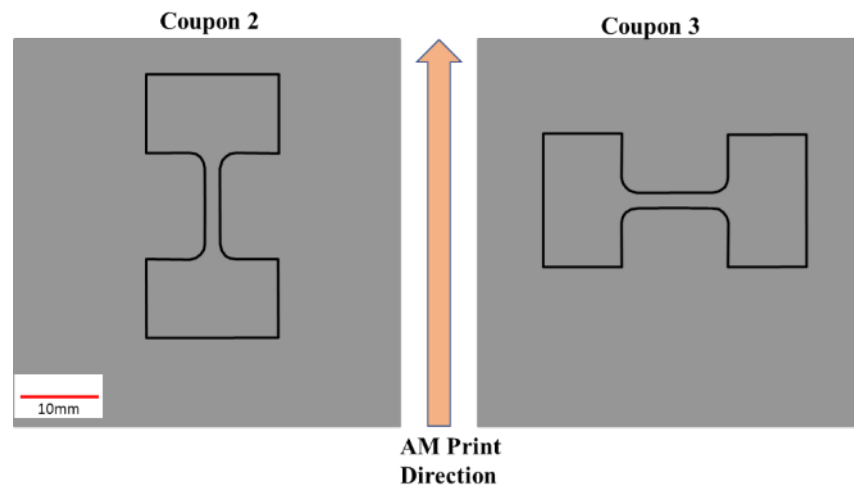


Figure 5-8: Coupon 2 and Coupon 3 orientation to AM printing direction

To understand if this difference in orientation impacts the mechanical properties of the printed 17-4 PH SS, Figure 5-9, Figure 5-10, and Figure 5-11 plot the stress-strain curves for both coupons on the same plot for Printer A, B, and C respectively.

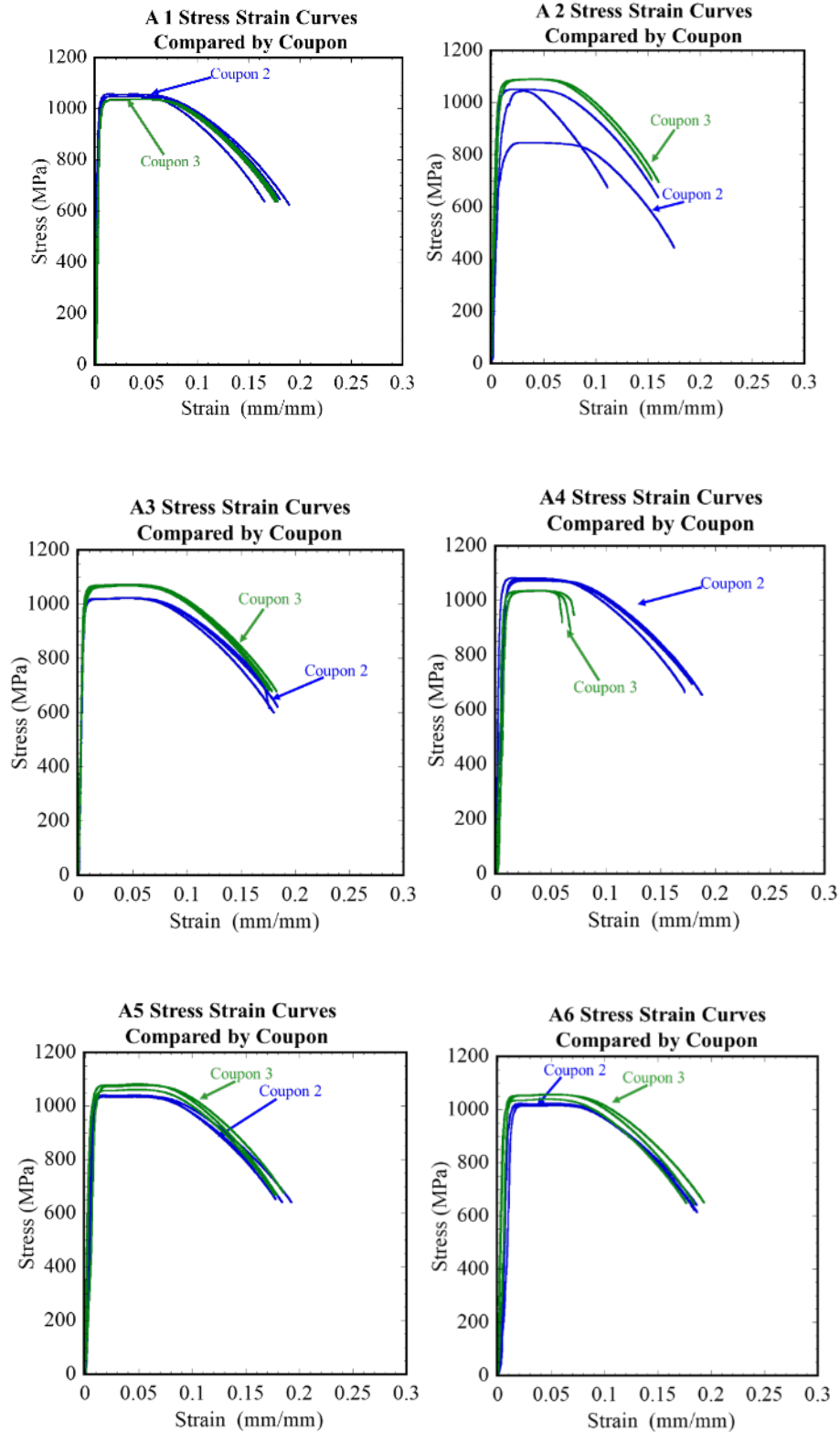


Figure 5-9: Comparison of Printer A stress-strain curves by coupon

From Figure 5-9, it is evident that for all Printer A builds except for A1 and A4, the Coupon 3 meso samples have higher yield and ultimate stresses than the Coupon 2 meso samples.

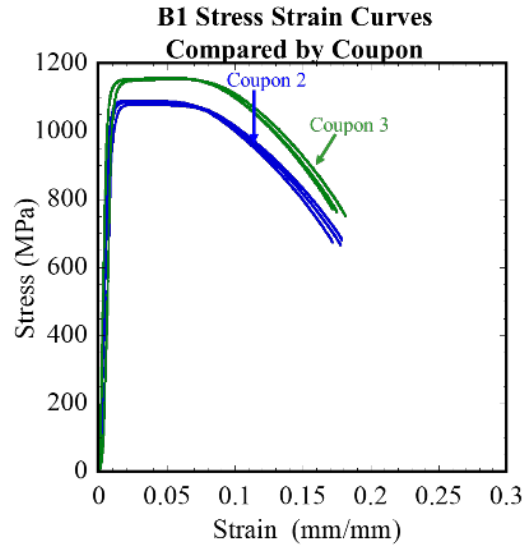


Figure 5-10: Comparison of Printer B stress-strain curves by coupon

Figure 5-10 shows that Printer B produced the same result, with Coupon 3 yield and ultimate stresses significantly higher than Coupon 2. Finally, Figure 5-11 demonstrates similar results from Printer C with exceptions in C1, C5, and C6. The potential reasons for these exceptions are discussed in Section 6.2.3 of this paper.

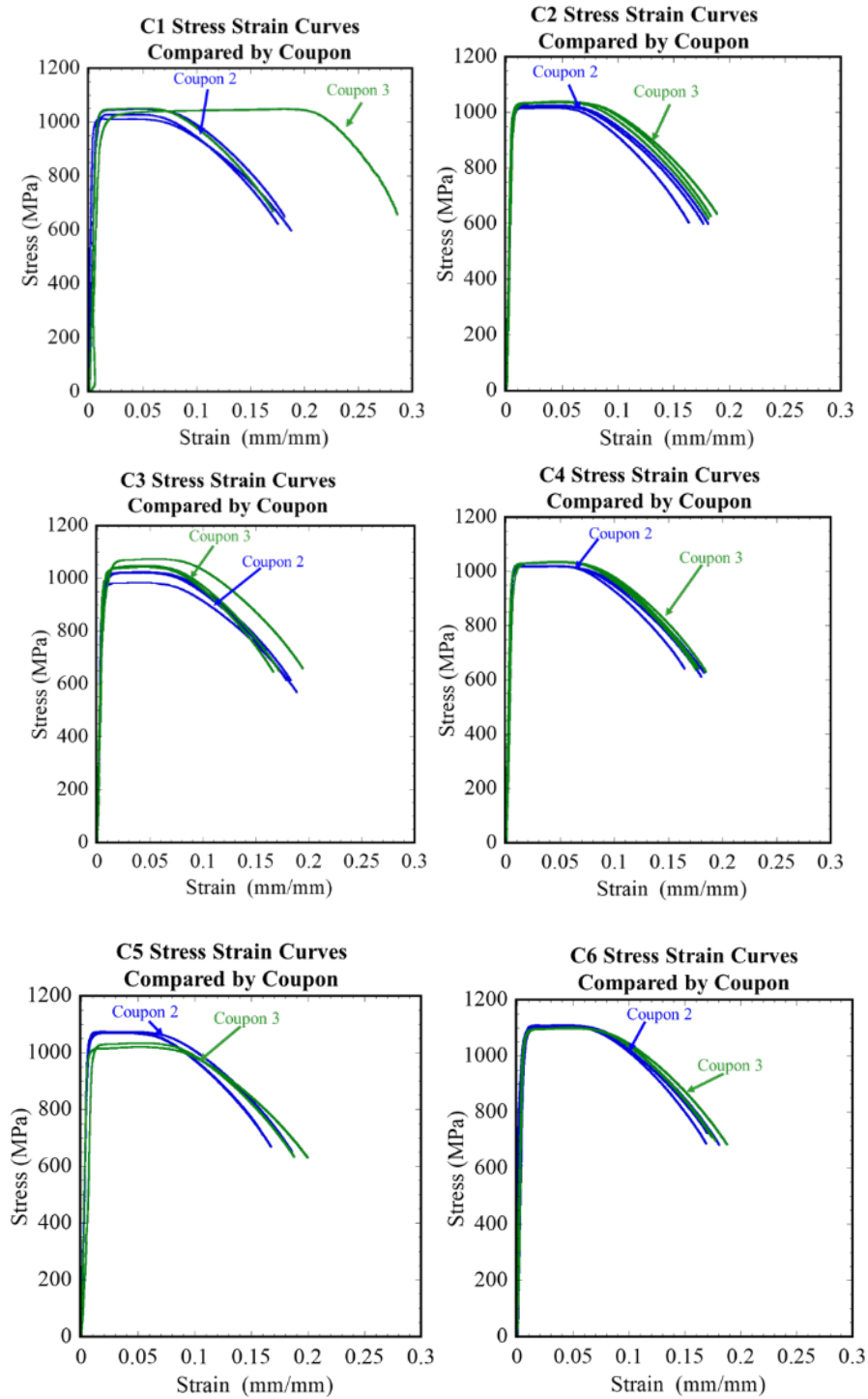


Figure 5-11: Comparison of Printer C stress-strain curves by coupon

The average mechanical properties listed for each printer in Table 5-1 to Table 5-6 further confirm these results as Coupon 3 average yield and ultimate stresses prove higher than the same properties in Coupon 2 samples.

Table 5-1: Printer A Coupon 2 average mechanical properties

| <b>Printer A<br/>Coupon 2</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1007.6                                    | 1034.6                                           | 0.176                                                | 202.4                                            |
| <b>Standard<br/>Deviation</b>       | 66.4                                      | 49.1                                             | 0.02                                                 | 56.3                                             |
| <b>Coefficient<br/>of Variation</b> | 0.066                                     | 0.047                                            | 0.102                                                | 0.278                                            |

Table 5-2: Printer A Coupon 3 average mechanical properties

| <b>Printer A<br/>Coupon 3</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1037.6                                    | 1057.8                                           | 0.157                                                | 190.9                                            |
| <b>Standard<br/>Deviation</b>       | 24.2                                      | 19.6                                             | 0.04                                                 | 57.0                                             |
| <b>Coefficient<br/>of Variation</b> | 0.023                                     | 0.019                                            | 0.270                                                | 0.299                                            |

Table 5-3:Printer B Coupon 2 average mechanical properties

| <b>Printer B<br/>Coupon 2</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1064.8                                    | 1084.3                                           | 0.176                                                | 163.9                                            |
| <b>Standard<br/>Deviation</b>       | 10.5                                      | 5.1                                              | 0.002                                                | 14.6                                             |
| <b>Coefficient<br/>of Variation</b> | 0.010                                     | 0.005                                            | 0.013                                                | 0.089                                            |

Table 5-4:Printer B Coupon 3 average mechanical properties

| <b>Printer B<br/>Coupon 3</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1103.7                                    | 1156.3                                           | 0.174                                                | 189.2                                            |
| <b>Standard<br/>Deviation</b>       | 41.9                                      | 2.1                                              | 0.01                                                 | 16.1                                             |
| <b>Coefficient<br/>of Variation</b> | 0.038                                     | 0.002                                            | 0.040                                                | 0.085                                            |

Table 5-5:Printer C Coupon 2 average mechanical properties

| <b>Printer C<br/>Coupon 2</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1027.6                                    | 1043.8                                           | 0.178                                                | 198.6                                            |
| <b>Standard<br/>Deviation</b>       | 42.7                                      | 36.8                                             | 0.01                                                 | 29.7                                             |
| <b>Coefficient<br/>of Variation</b> | 0.042                                     | 0.035                                            | 0.049                                                | 0.150                                            |

Table 5-6: Printer C Coupon 3 average mechanical properties

| <b>Printer C<br/>Coupon 3</b>       | <b>Average<br/>Yield Stress<br/>(Mpa)</b> | <b>Average<br/>Ultimate<br/>Stress<br/>(Mpa)</b> | <b>Average<br/>Strain to<br/>Failure<br/>(mm/mm)</b> | <b>Average<br/>Young's<br/>Modulus<br/>(Gpa)</b> |
|-------------------------------------|-------------------------------------------|--------------------------------------------------|------------------------------------------------------|--------------------------------------------------|
|                                     | 1024.94                                   | 1045.23                                          | 197.83                                               | 0.19                                             |
| <b>Standard<br/>Deviation</b>       | 14.80                                     | 10.68                                            | 40.04                                                | 0.03                                             |
| <b>Coefficient<br/>of Variation</b> | 0.01                                      | 0.01                                             | 0.20                                                 | 0.17                                             |

The impact of gauge section-print direction orientation on material yield and ultimate stresses is likely because of the different impacts of loading on the inter-layer strength of the printed component. Essentially, Coupon 2 samples are peeled apart layer by layer when placed under loading. In contrast, Coupon 3 samples do not experience this peeling phenomenon since the layers are parallel to the loading direction. Isostress and isostrain explain this behavior.

#### **5.5.1 Isostress and Isostrain**

When a sample is in an isostress state, the stress of the total sample is independent of the volume fraction of layers with a particular stress value. Instead, the stress of the entire sample is the same as the stress in each layer. Therefore, if one layer of an AM sample has low stress, the entire sample will have low stress. More importantly, the strain in each layer is not the same. This differing strain allows for areas of high strain, creating a “weak or soft link” situation. This increased strain leads to sample failure. Figure 5-12 provides an illustration of a high strain location that will lead to failure. Since the thermal cycling inherent to AM impacts the properties of a material, it is logical that Coupon 2 samples will have varying stresses.

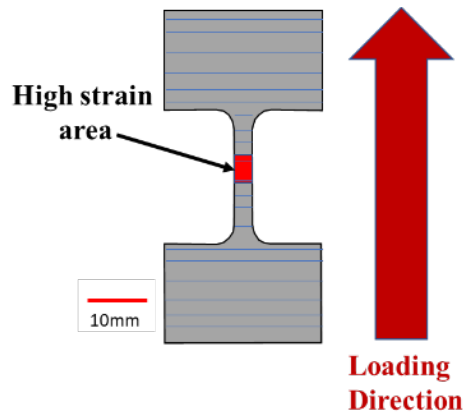


Figure 5-12: Example of a high strain location in an isostress sample

In contrast, Coupon 3 meso samples are in an isostrain state. In this state, total stress is determined by the volume fraction of each layer with a specific stress. Therefore, a single layer or a few low-stress layers have little impact on the total sample stress. To create low sample stress, most layers within a sample would have to have the same low stress. Thus, Coupon 3 samples have more consistent average stresses as they are less impacted by small variations in sample properties. Figure 5-13 illustrates the two different loading situations side beside each other for contrast.

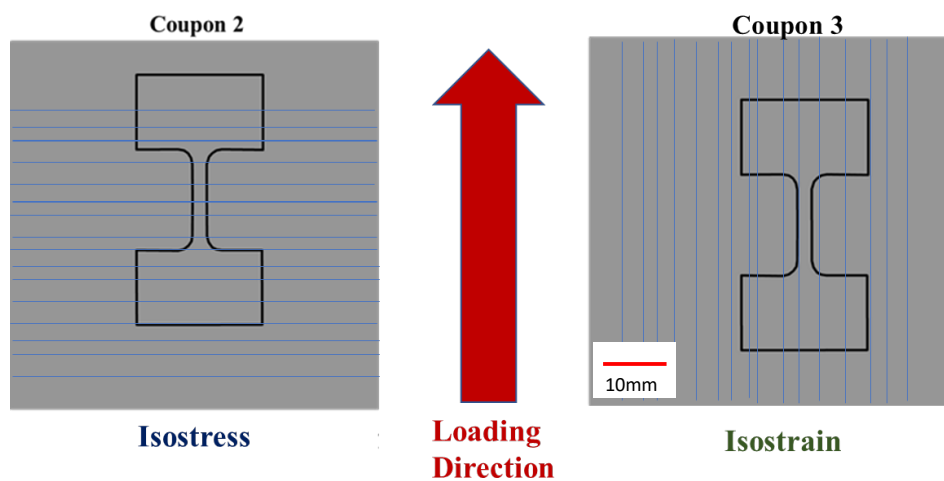


Figure 5-13: Illustration of AM layers and tensile loading direction with Coupon 2 in isostress and Coupon 3 in isostrain

### 5.5.2 Initial Tensile Test Conclusions

The average material properties of all builds, regardless of the printer, are displayed in Table 5-7 and Table 5-8. Table 5-7 displays these properties for all Coupon 2 samples, and Table 5-8 shows the properties for all Coupon 3 samples. These tables make the difference in yield and ultimate stress between Coupon 2 and Coupon 3 more apparent. Additionally, an inspection of the coefficient of variation for each coupon's yield and ultimate stress shows that Coupon 2 has a higher coefficient of variation than Coupon 3. This difference in coefficient of variation indicates that Coupon 2 samples have less consistent yield and ultimate stresses than Coupon 3 samples. Essentially, Coupon 2 results have a higher spread than those of Coupon 3. Stress-strain curves for Coupon 2 located in Section 6.2.3 make this higher spread apparent.

Furthermore, these results lead to an overall conclusion on print repeatability. The lack of repeatability within Coupon 2 meso sample properties repeats regardless of build. Therefore, it is expected that any set of samples printed in the orientation found in Coupon 2 will not have repeatable, consistent results.

Table 5-7: Coupon 2 average properties (all printers)

|                                 | <b>Yield Stress</b> | <b>Ultimate Stress</b> | <b>Young's Modulus</b> | <b>Strain to Failure</b> |
|---------------------------------|---------------------|------------------------|------------------------|--------------------------|
| <b>Average</b>                  | 1011.39             | 1000.18                | 201.60                 | 0.18                     |
| <b>Standard Deviation</b>       | 58.25               | 171.75                 | 50.11                  | 0.02                     |
| <b>Coefficient of Variation</b> | 0.058               | 0.172                  | 0.249                  | 0.086                    |

Table 5-8: Coupon 3 average properties (all printers)

|                                 | <b>Yield Stress</b> | <b>Ultimate Stress</b> | <b>Young's Modulus</b> | <b>Strain to Failure</b> |
|---------------------------------|---------------------|------------------------|------------------------|--------------------------|
| <b>Average</b>                  | 1038.18             | 1062.82                | 195.33                 | 0.17                     |
| <b>Standard Deviation</b>       | 34.09               | 37.00                  | 48.47                  | 0.04                     |
| <b>Coefficient of Variation</b> | 0.033               | 0.035                  | 0.248                  | 0.251                    |

## 5.6 Meso Sample Microstructure

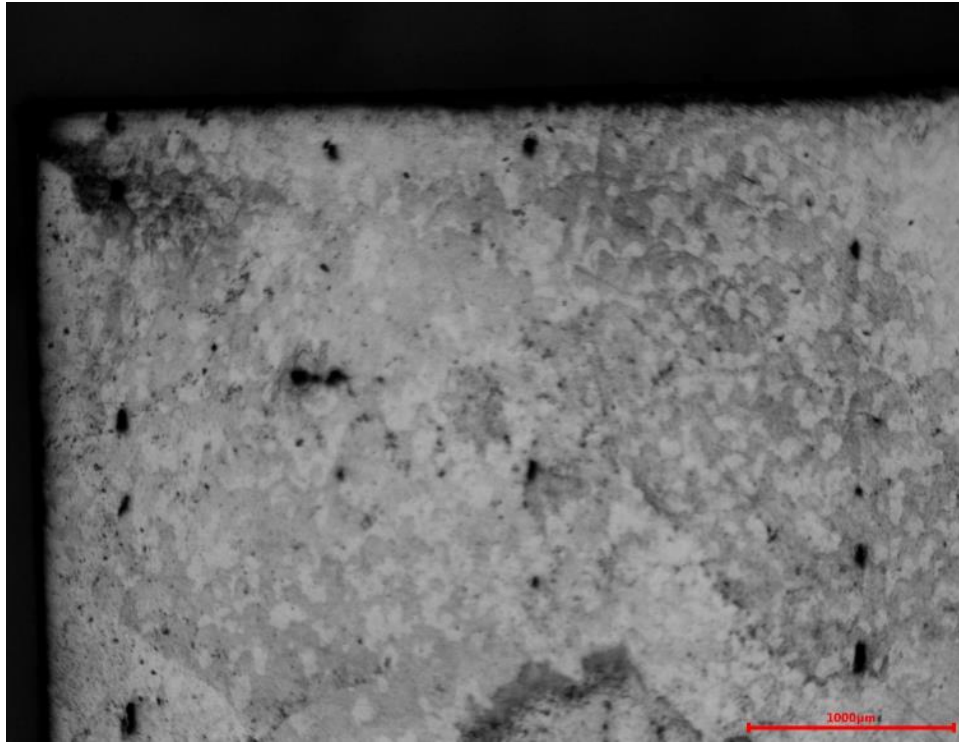


Figure 5-14: Microstructure of meso sample A4-3-2 with copper precipitates and keyhole defects

Figure 5-14 provides a glimpse of the microstructure of meso sample A4-3-2. Note the visible grain boundaries (the lighter round areas) and the evidence of precipitates (tiny dark spots). The precipitate is likely copper since the material was aged at 550 °C, and copper is the primary precipitate at aging temperatures between 480-620 °C, as briefly discussed in Section 2.6.2. Also, note the presence of several keyhole defects.

## 6 Further Analysis of Results

### 6.1 Comparison of Yield Strength by Sample Geometry and Orientation

The yield stress of the zig-zag structures was compared to the yield stress of the meso samples to understand how the sample geometry and orientation impact mechanical properties, specifically yield stress. Therefore, the average Vickers Hardness of the zig-zag structures was converted to yield stress using Equation 6-1 [131]:

$$\sigma_{yield} = \frac{HV}{3} \quad (6-1)$$

The yield stress of narrow and wide zig-zag structures for each Printer was plotted with both Coupon 2 and Coupon 3 meso samples. In each figure, circular series are tensile data, while the triangle is wide base zig zag data, and the X is narrow base zig-zag data. Figure 6-1 through Figure 6-3 show the relationship between the samples.

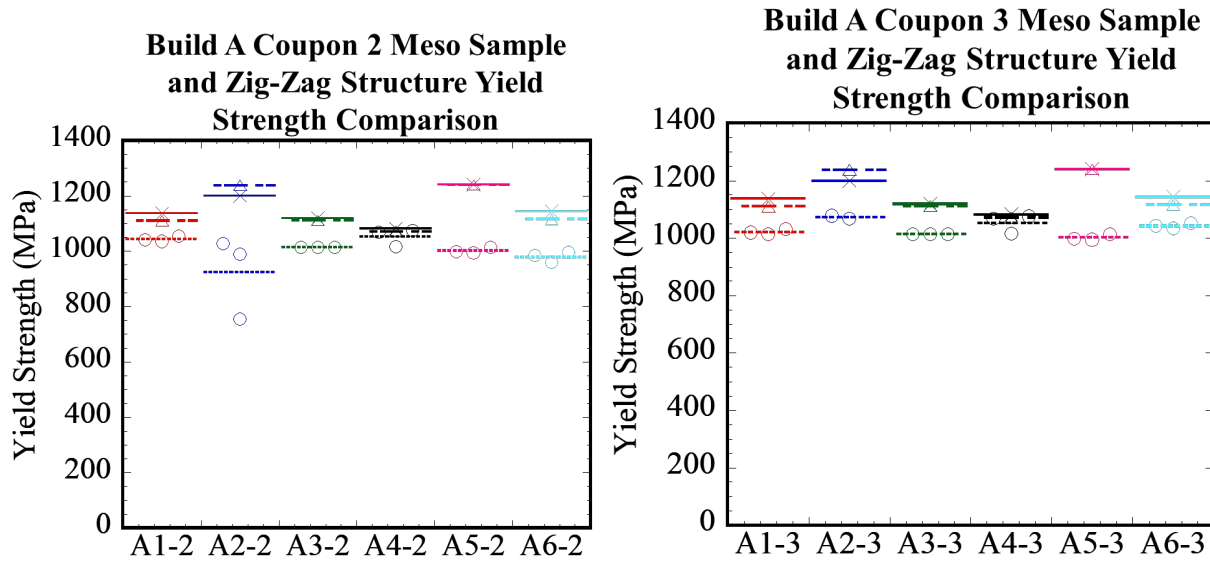


Figure 6-1: Printer A Coupon 2 and Coupon 3 yield stress related to wide and narrow zig-zag yield stress

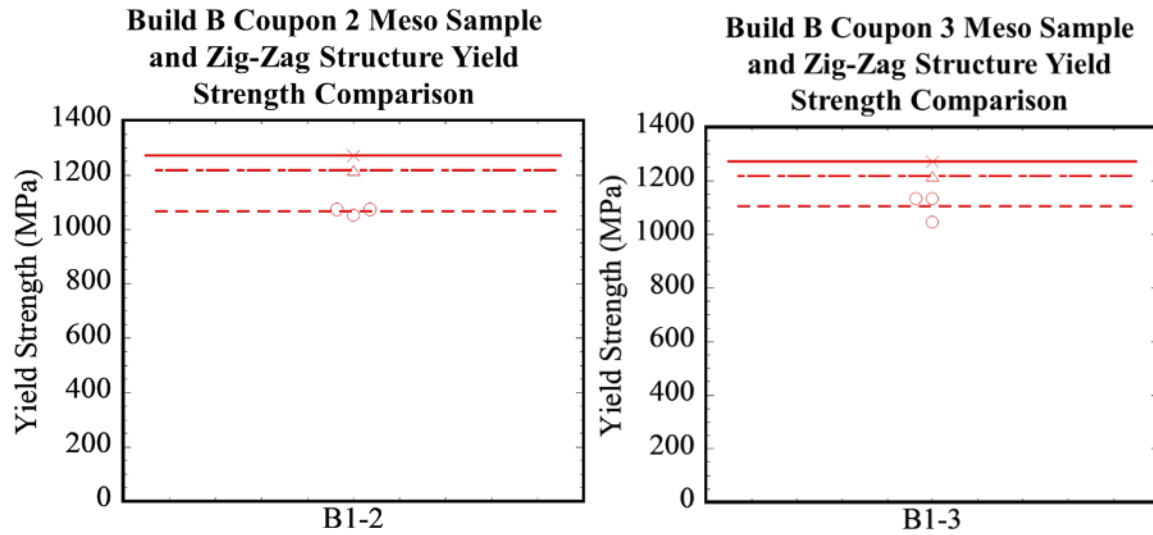


Figure 6-2: Printer B Coupon 2 and Coupon 3 yield stress related to wide and narrow zig-zag yield stress

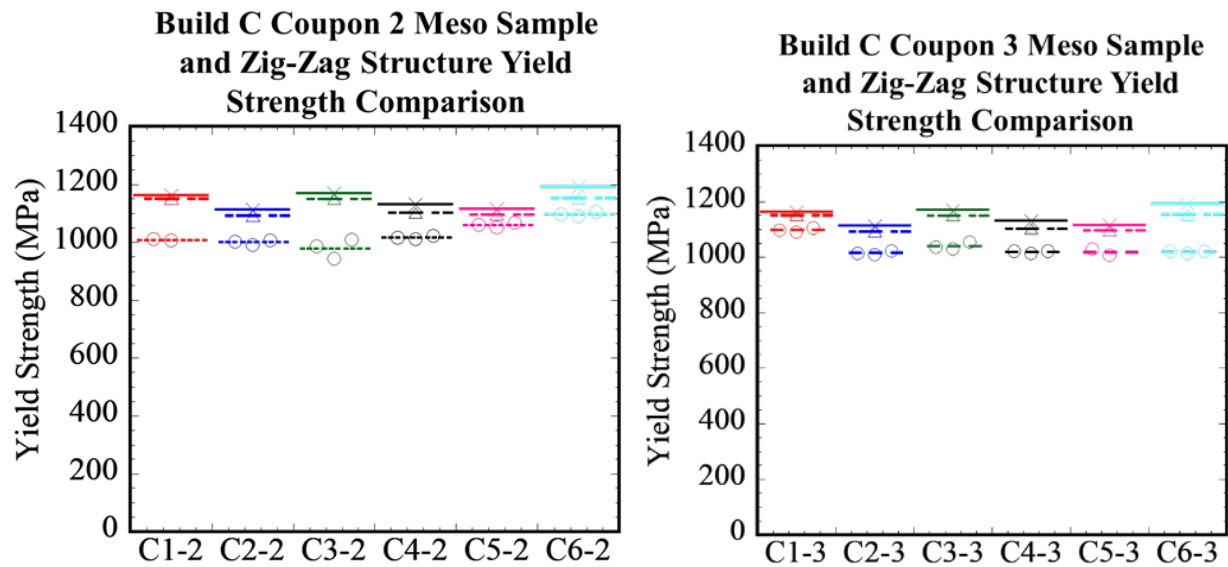


Figure 6-3: Printer C Coupon 2 and Coupon 3 yield stress related to wide and narrow zig-zag yield stress

From each of the figures above, it is clear that the mesosamples had the lowest yield stresses, regardless of coupon or printer. This result is due to the difference in the zig-zag and meso sample geometries. The gauge sections of the meso samples are much more narrow than the narrowest portions of the zig-zag structures. Therefore, heat flows differently through each sample during the printing process and creates variation in the final product and material properties.

## **6.2 Evaluation of Deformation Mechanism**

Fracture mechanics describes how a material with cracks and other defects such as porosity, inclusions, or microcracking will behave. The overall goal of fracture mechanics is to understand how much load a flawed or damaged material can sustain [125]. Fracture mechanics also details the mechanism of material fracture, which aids in the characterization of metals as either ductile or brittle. Therefore, the description of fracture mechanisms in this study's samples is essential for a complete understanding of AM 17-4 PH SS behavior.

### **6.2.1 Mechanism of Ductile Fracture**

As the name suggests, ductile fracture takes place through the grains of a metal with high ductility and toughness. The defining characteristic of ductile fracture is the presence of deformation and necking found after sample fracture. Note that this deformation takes place once a sample loses elasticity and before it fractures. In a traditional tensile test of ductile metal, microvoids nucleate as the material necks. The nucleation or formation of these microvoids results from the metal separation (voids) near grain boundaries. This separation occurs due to high local stress. These microvoids then grow and merge or coalesce towards the center of the gauge section of the sample. The microvoids continue to grow, reducing the contact between the metal surfaces, leading to fracture [125]. Figure 6-4 shows the process of ductile fracture from

voids between grains to final rupture. The fracture surfaces of ductile metals have two distinct features, the flat face and the shear lip. The flat face indicates microvoids' initial nucleation and coalescence while the shear lip creates a cup and cone formation [125]. Figure 6-5 shows a ductile fracture's flat surface, shear lip, and cup cone formation.

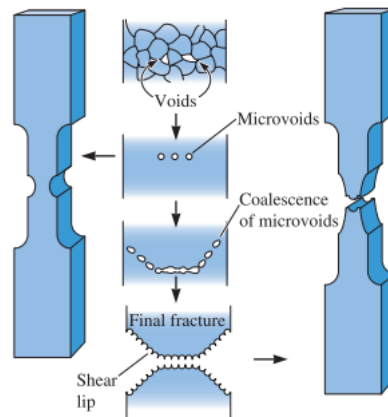


Figure 6-4: The ductile fracture process, from the formation of microvoids to final fracture, resulting in a shear lip. Note the presence of necking in the tensile sample[125]

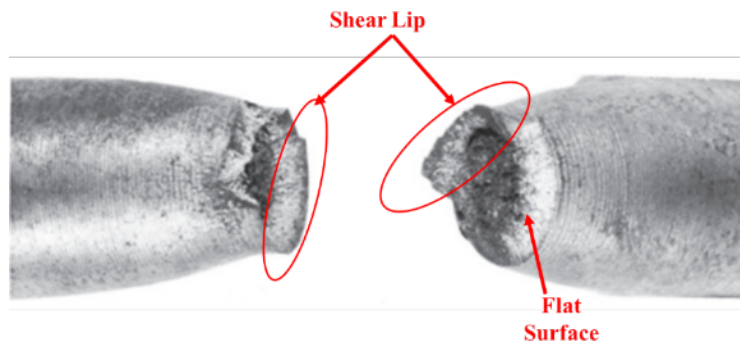


Figure 6-5: Distinct features of ductile fracture- the flat surface, shear lip, and cup and cone formation (modified from [125])

Indications of the microvoids created during ductile fracture, in the form of dimples, exist near the fracture surface. These dimples have an oval shape and are oriented towards the fracture origin. Figure 6-6 shows dimpling on a tested meso sample.

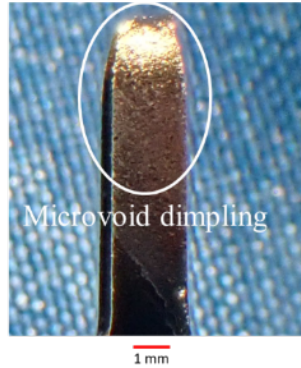


Figure 6-6: Dimpling near the fracture surface of a meso sample (evidence of microvoids)

Note that excluding the presence of a pre-test defect or inclusion, ductile tensile test samples fracture near the gauge section center.

### 6.2.2 *Brittle Fracture Mechanism*

Metals with high strength, low toughness, and low ductility fail through the brittle fracture mechanism. Additionally, if ductile metal experiences low temperatures, has defects, or undergoes high strain rates, it may fail in a brittle manner. Typically, brittle fracture occurs when a material sustains high impact. Note that plastic deformation is either minimal or nonexistent during brittle fracture. Instead, stress concentrations form at defects or flaws in a material [125].

The fracture surface of a brittle material is flat and has a perpendicular orientation to the loading direction if under load. However, if the sample fails from cleavage, the surface is bumpy due to the various orientations of each fractured grain.



Figure 6-7: Fracture surfaces of a brittle fracture. Note the flat fracture surface (modified from [132])

### **6.2.3 Inspection of Study Sample Fracture Behavior**

As mentioned in Section 2.6.1, 17-4 PH SS is known for its high toughness. Therefore, based on the fracture mechanism discussion above, the study meso samples should fracture via the ductile fracture mechanism. As anticipated, most of the meso samples failed via ductile fracture, evidenced by a distinct cup and cone fracture surface. Figure 6-8 through Figure 6-33 except for

*Figure 6-15* show the expected cup and cone fracture surfaces.

*Figure 6-15* has an angled fracture surface without any necking or cup cone formation. This kind of fracture surface may seem brittle. However, closer inspection of the fracture surface shows that the edges of the surface “pull” slightly upward around the edges. Additionally, one edge of each of the samples is flat. Therefore, it is likely that an existing defect caused a premature fracture.

#### **6.2.3.1 Investigation of Specified Meso Sample Builds**

Recall from Section 5.5 that mesosamples from Builds A1, A4, C1, C5, and C6 did not follow the other samples' yield and ultimate strength behavior. In these builds, Coupon 2 samples had higher yield and ultimate strengths than Coupon 3 samples. However, upon further inspection of the fracture surfaces, deep pores are visible in each sample of Coupon 3. These deep pores likely caused premature yield and failure.

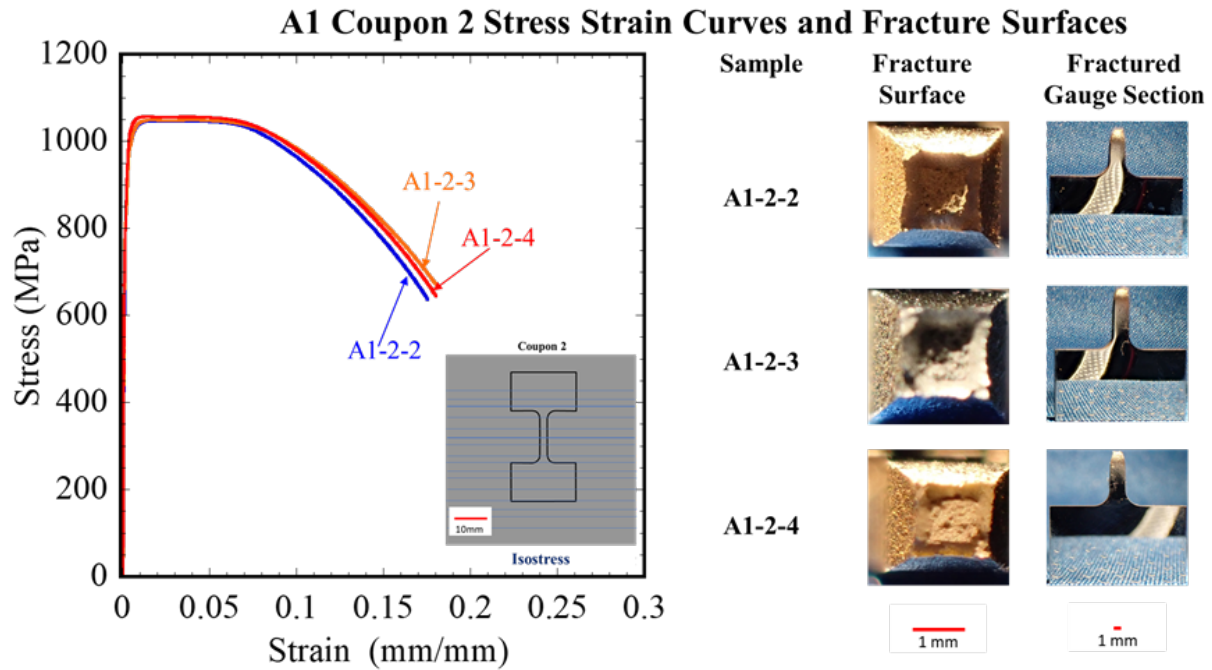


Figure 6-8: Build A1 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

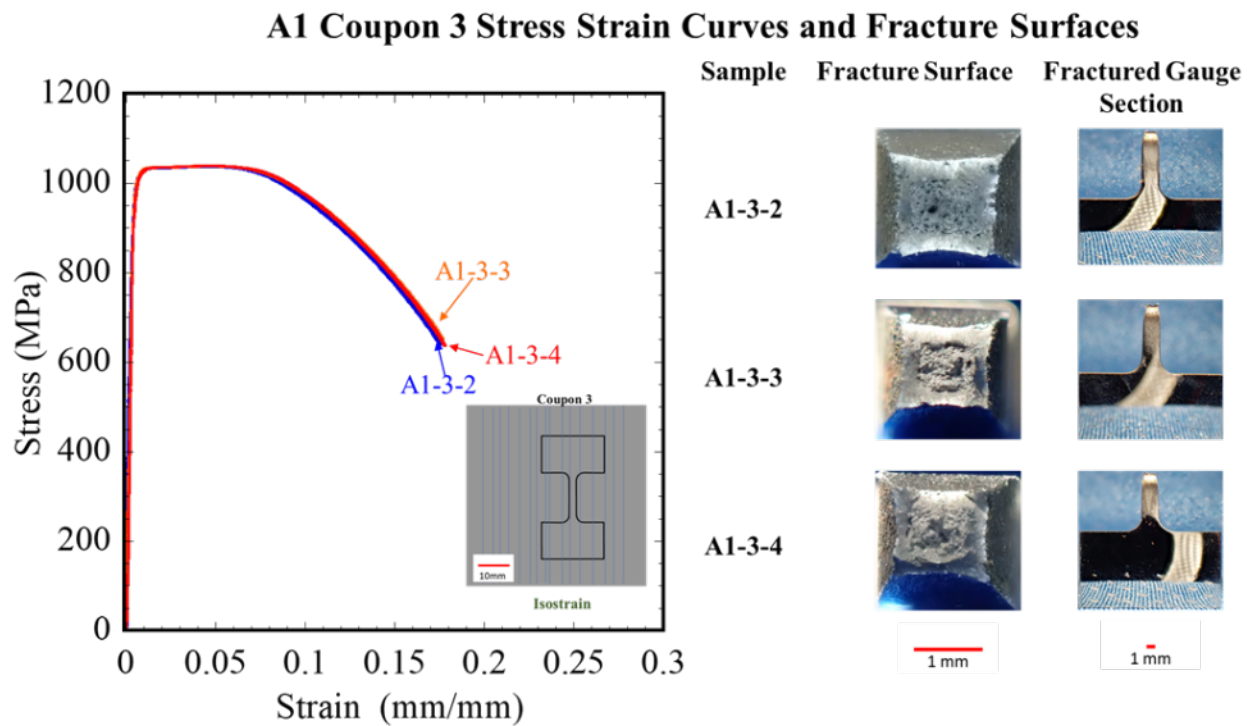


Figure 6-9: Build A1 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

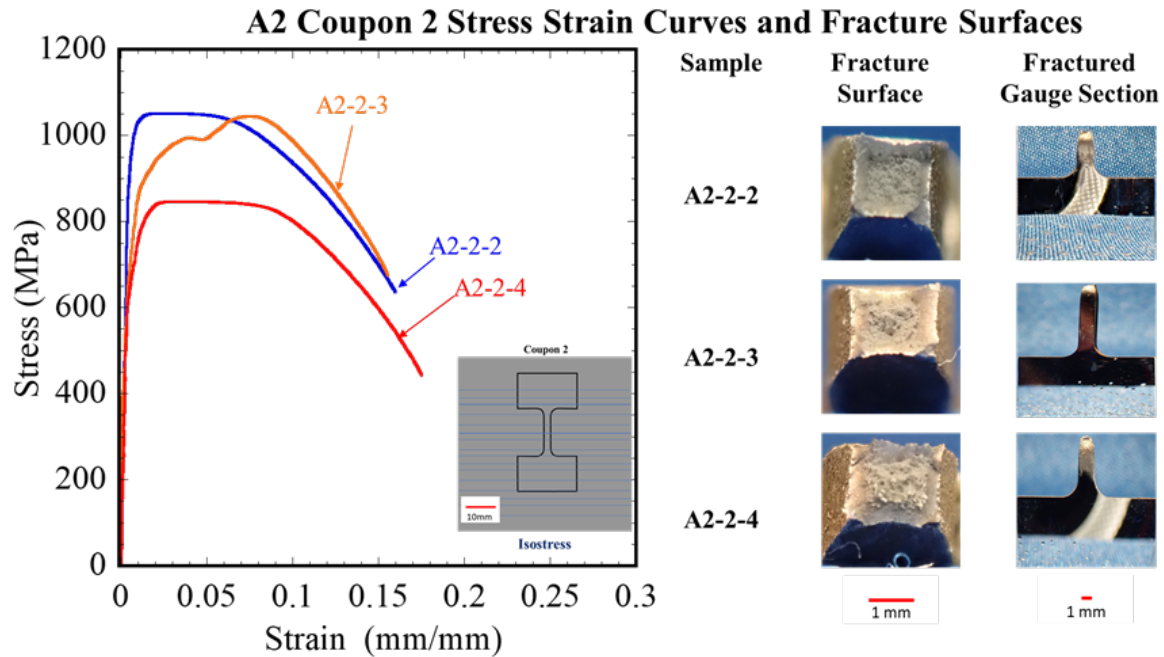


Figure 6-10: Build A2 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

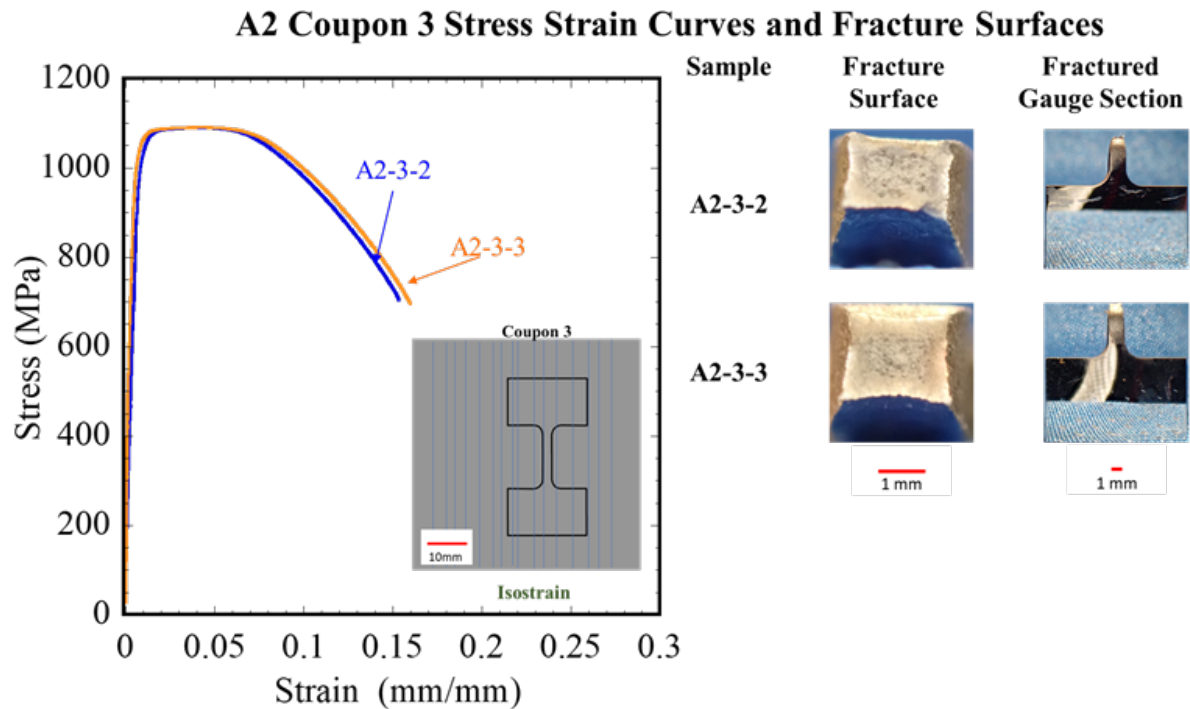


Figure 6-11: Build A2 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

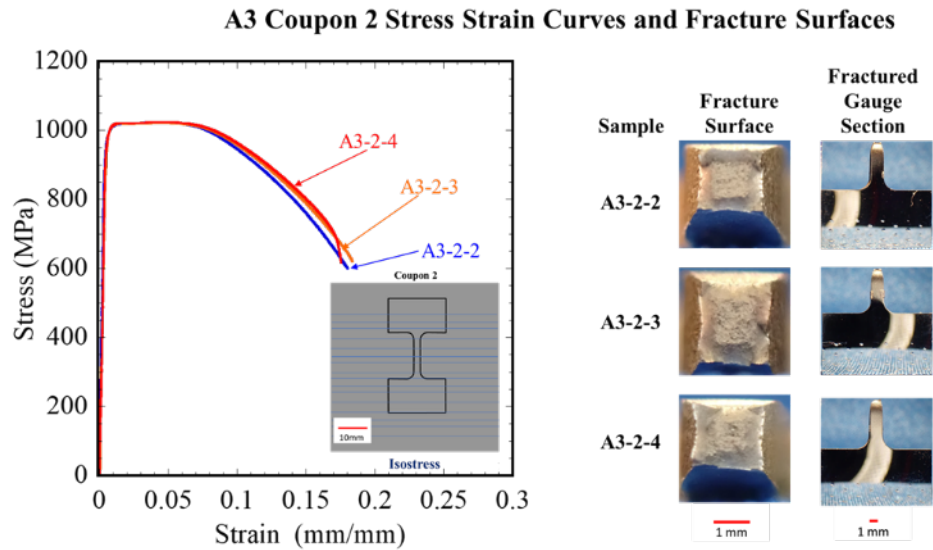


Figure 6-12: Build A3 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

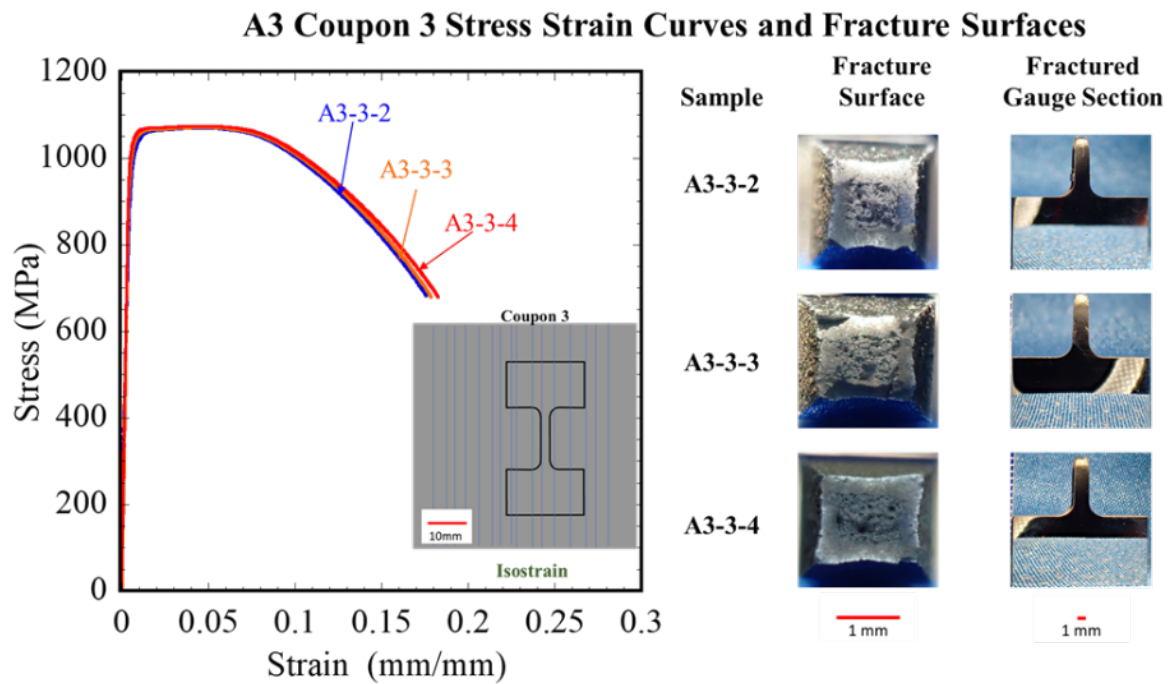


Figure 6-13: Build A3 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

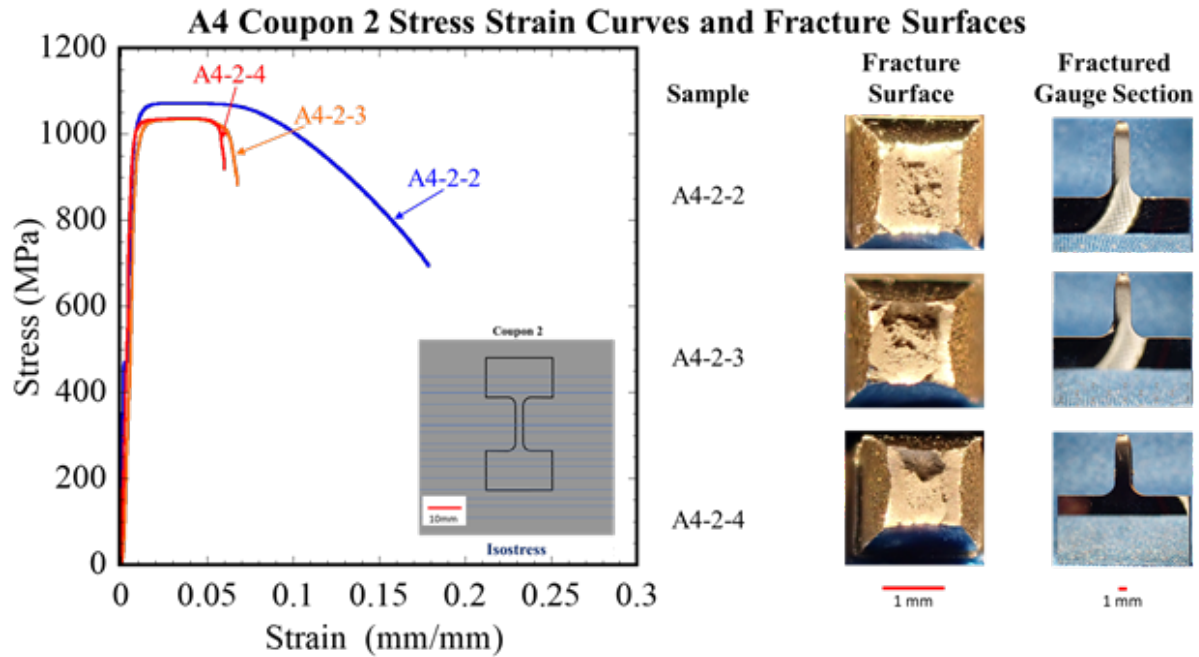


Figure 6-14: Build A4 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

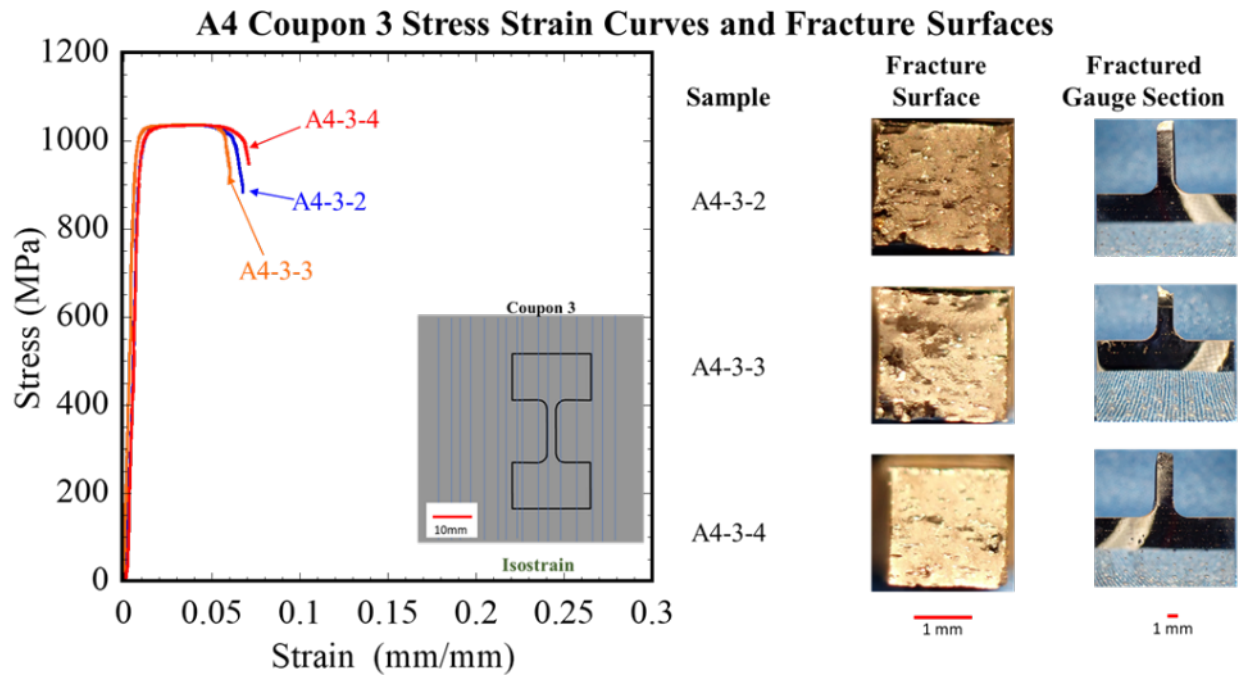


Figure 6-15: Build A4 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

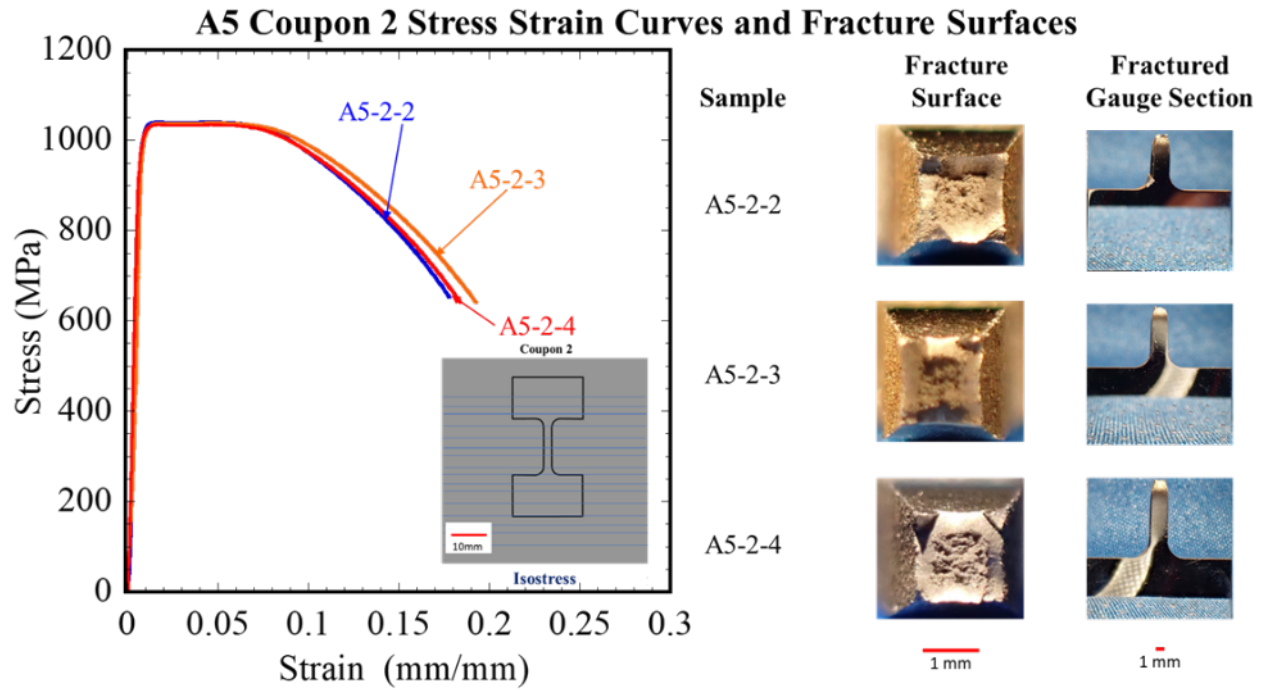


Figure 6-16: Build A5 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

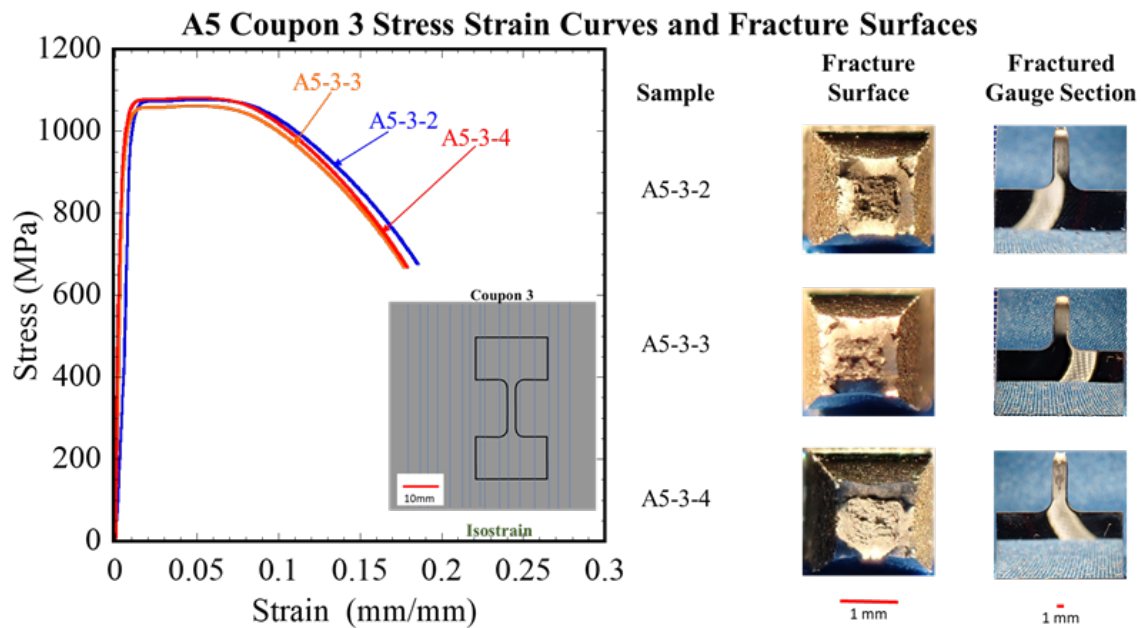


Figure 6-17: Build A5 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

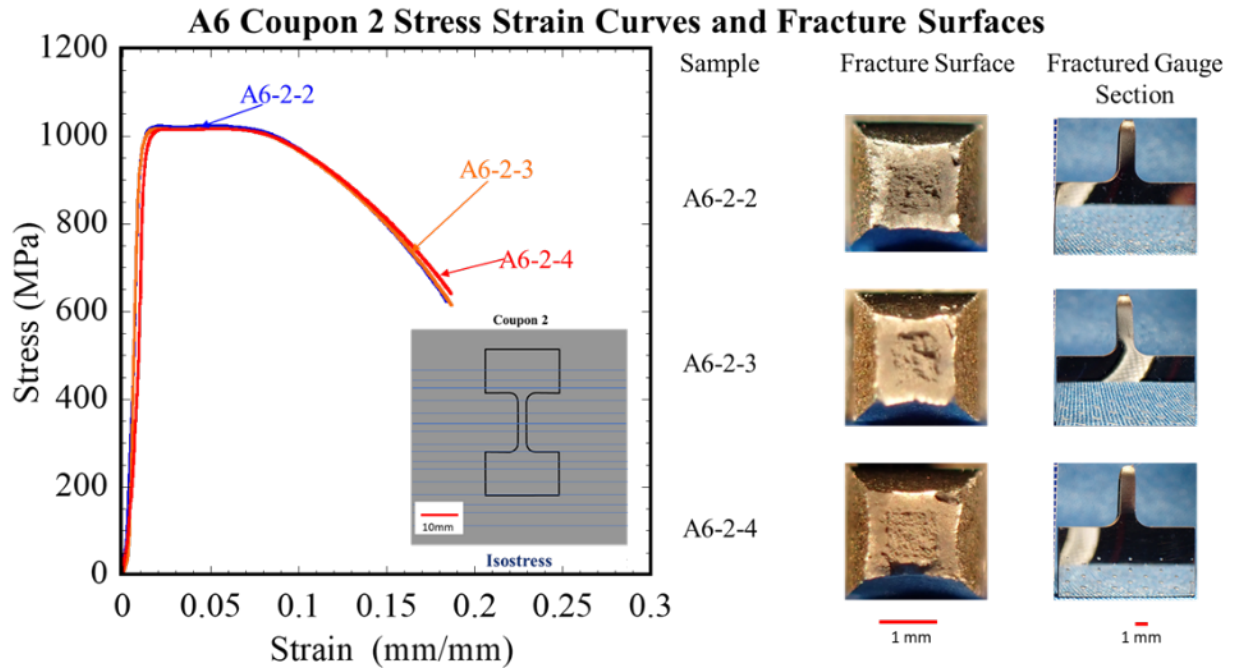


Figure 6-18: Build A6 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

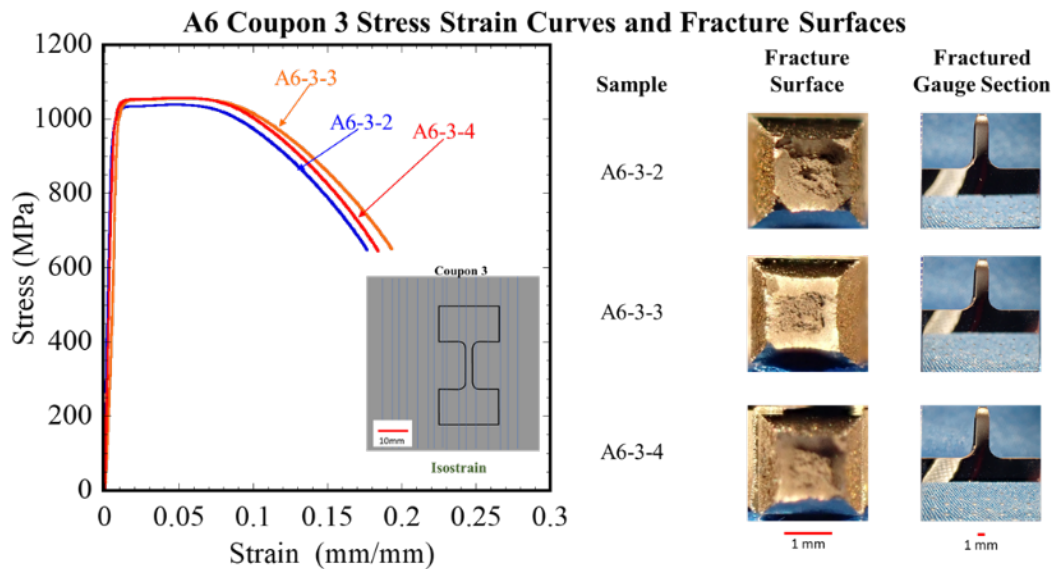


Figure 6-19: Build A6 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

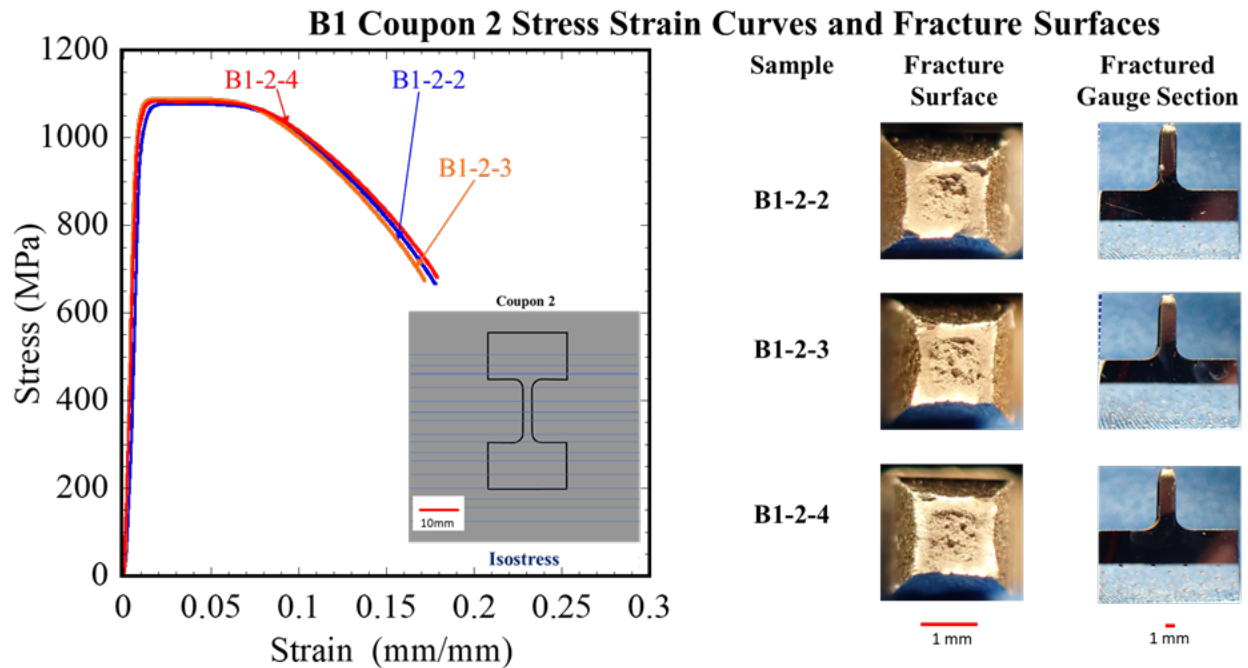


Figure 6-20: Build B1 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

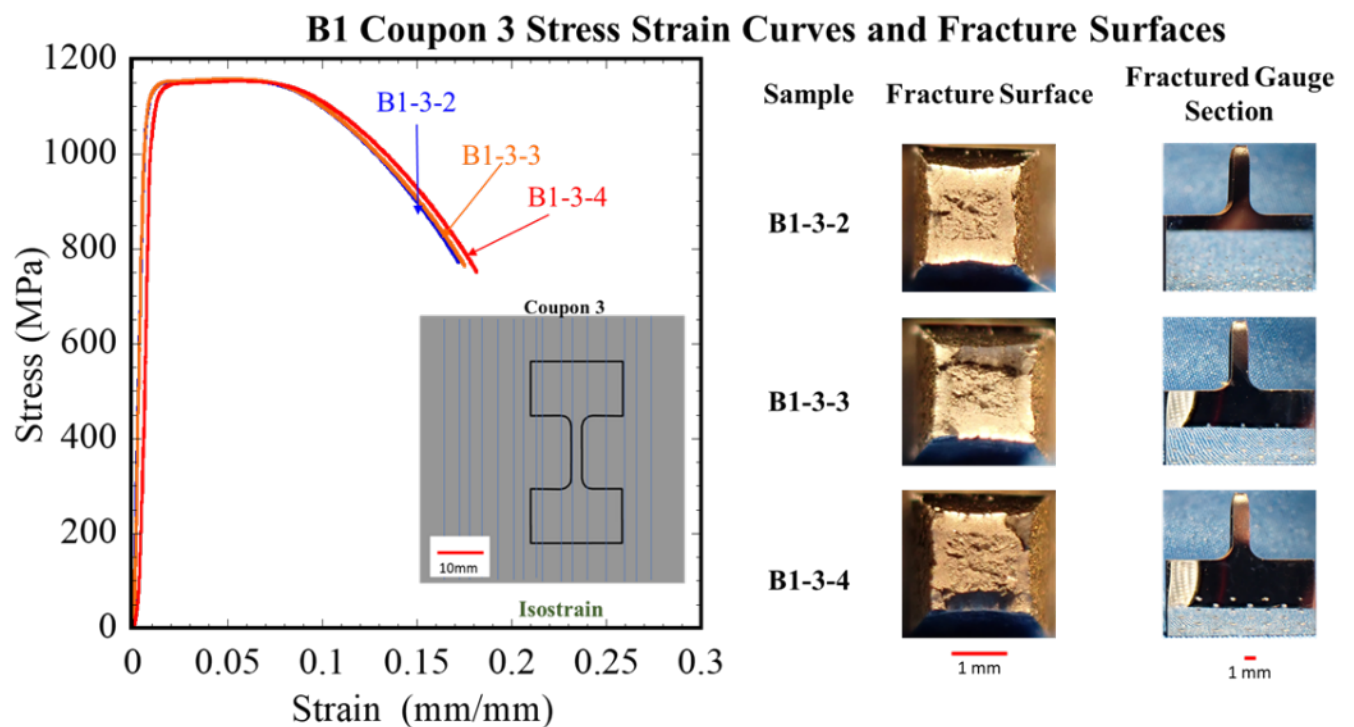


Figure 6-21: Build B1 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

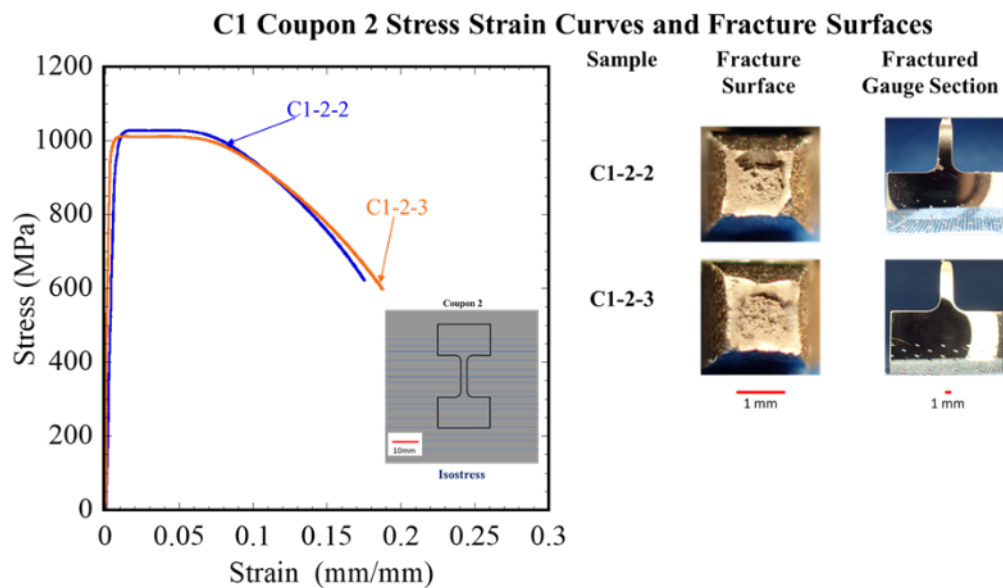


Figure 6-22: Build C1 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

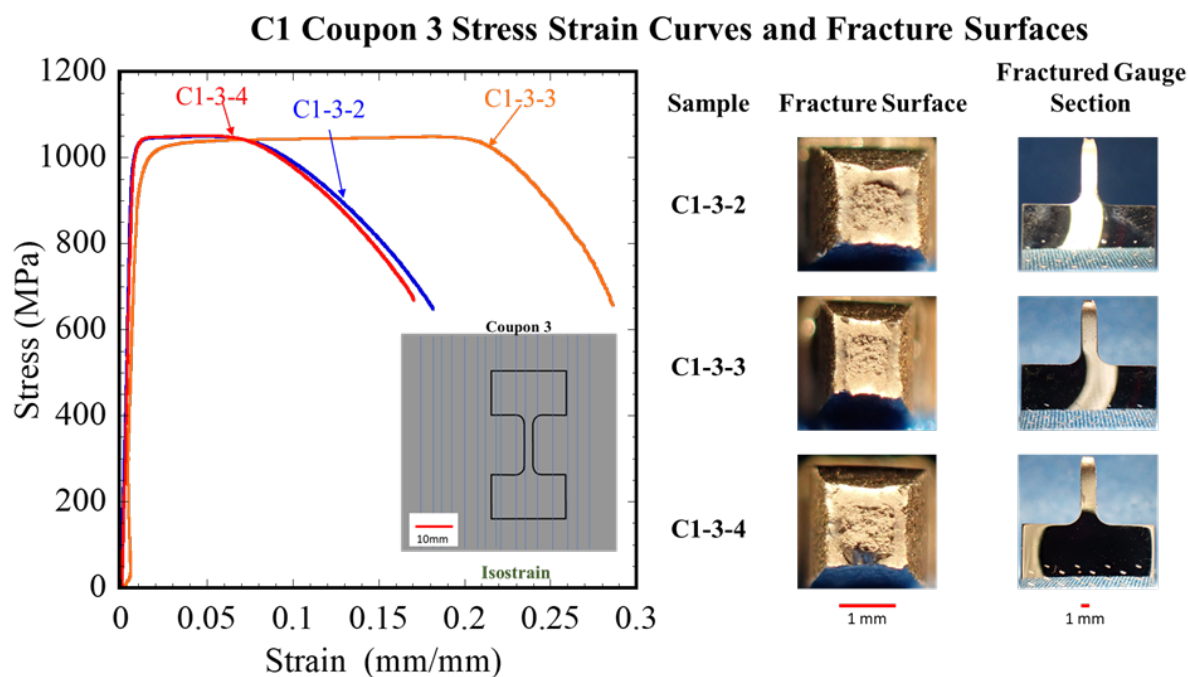


Figure 6-23: Build C1 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

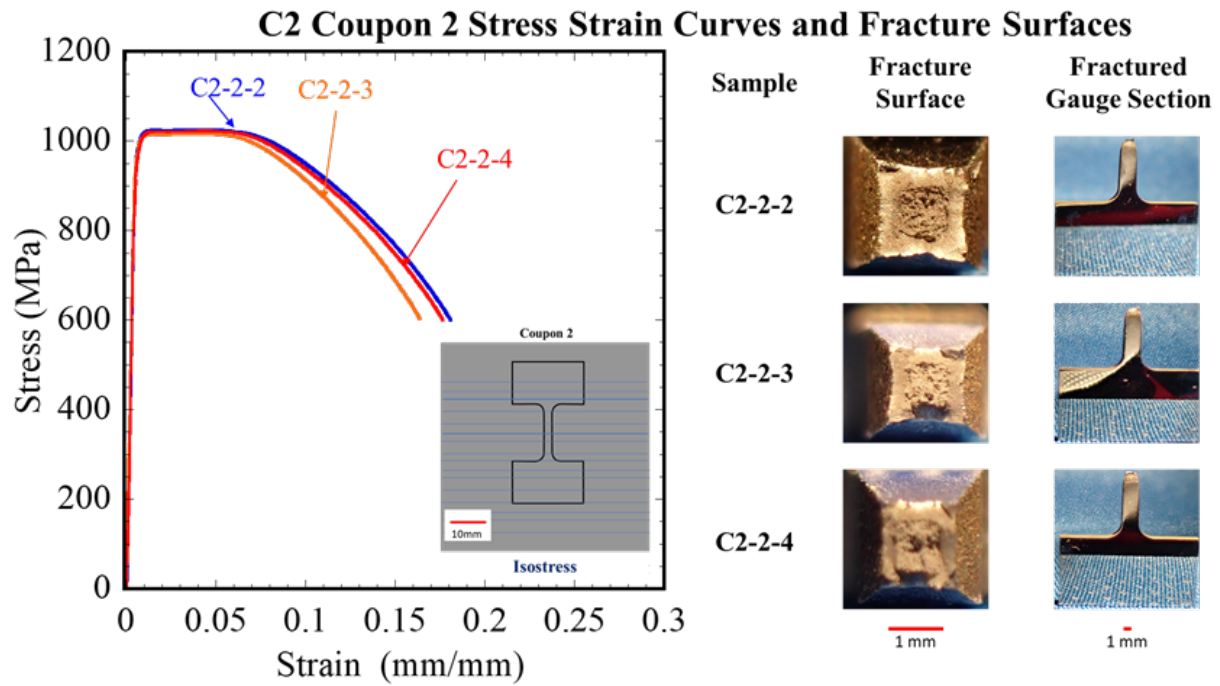


Figure 6-24: Build C2 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

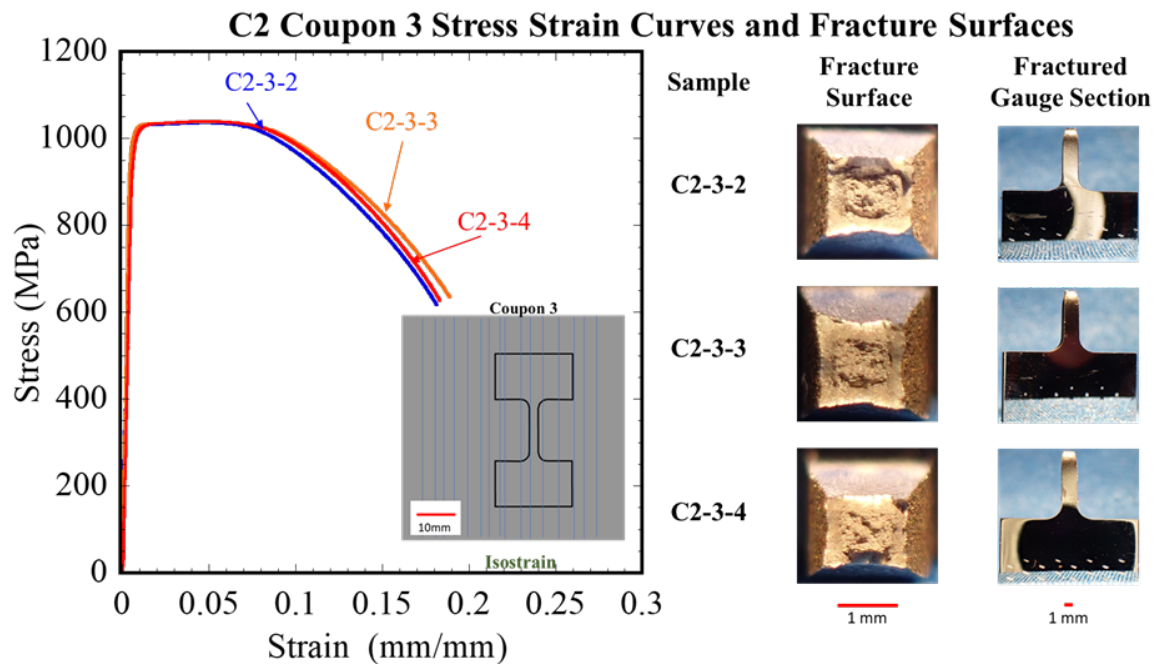


Figure 6-25: Build C2 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

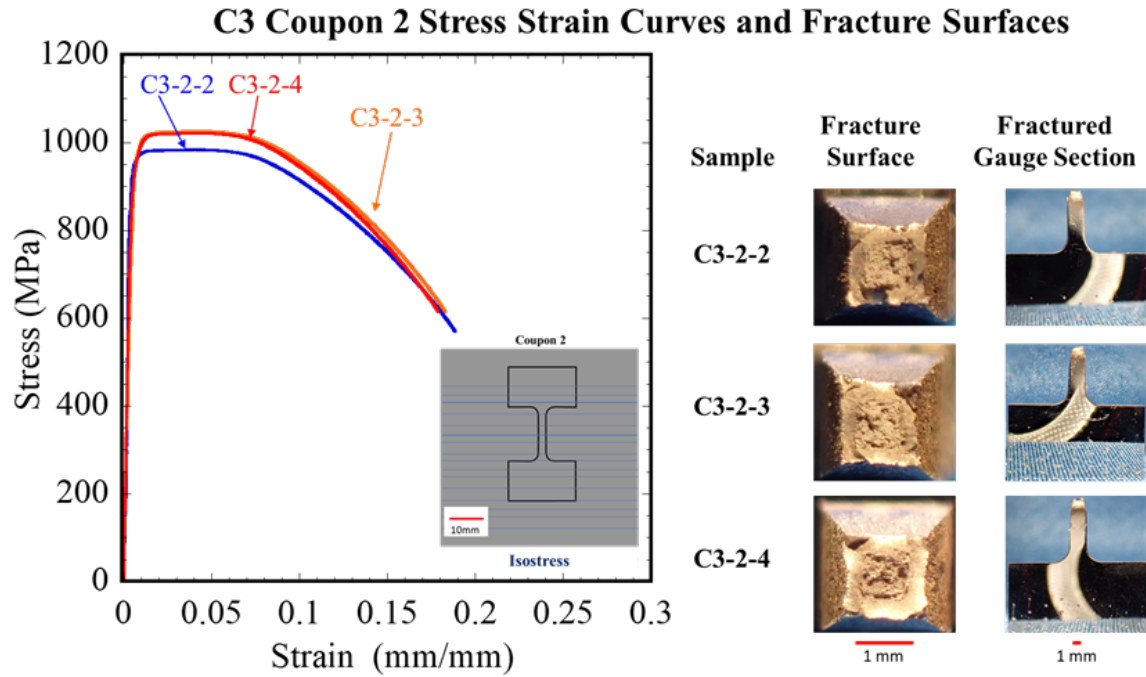


Figure 6-26: Build C3 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

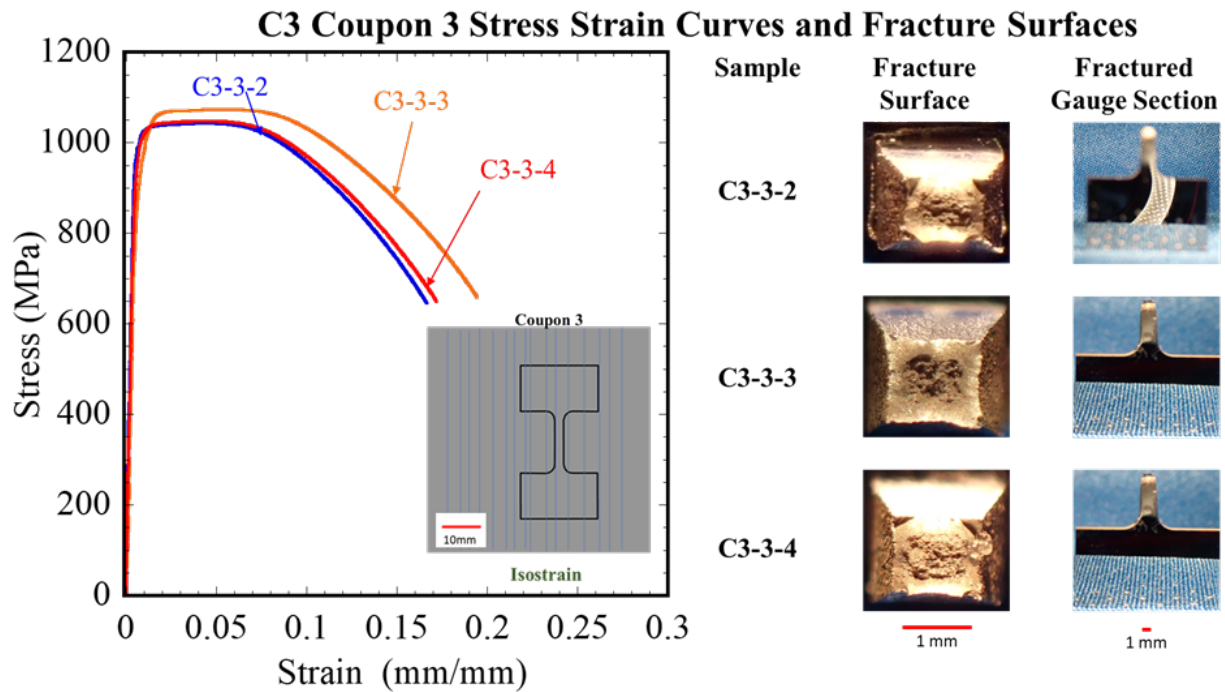


Figure 6-27: Build C3 Coupon 3 stress-strain curves and fracture surface and gauge section of each sample

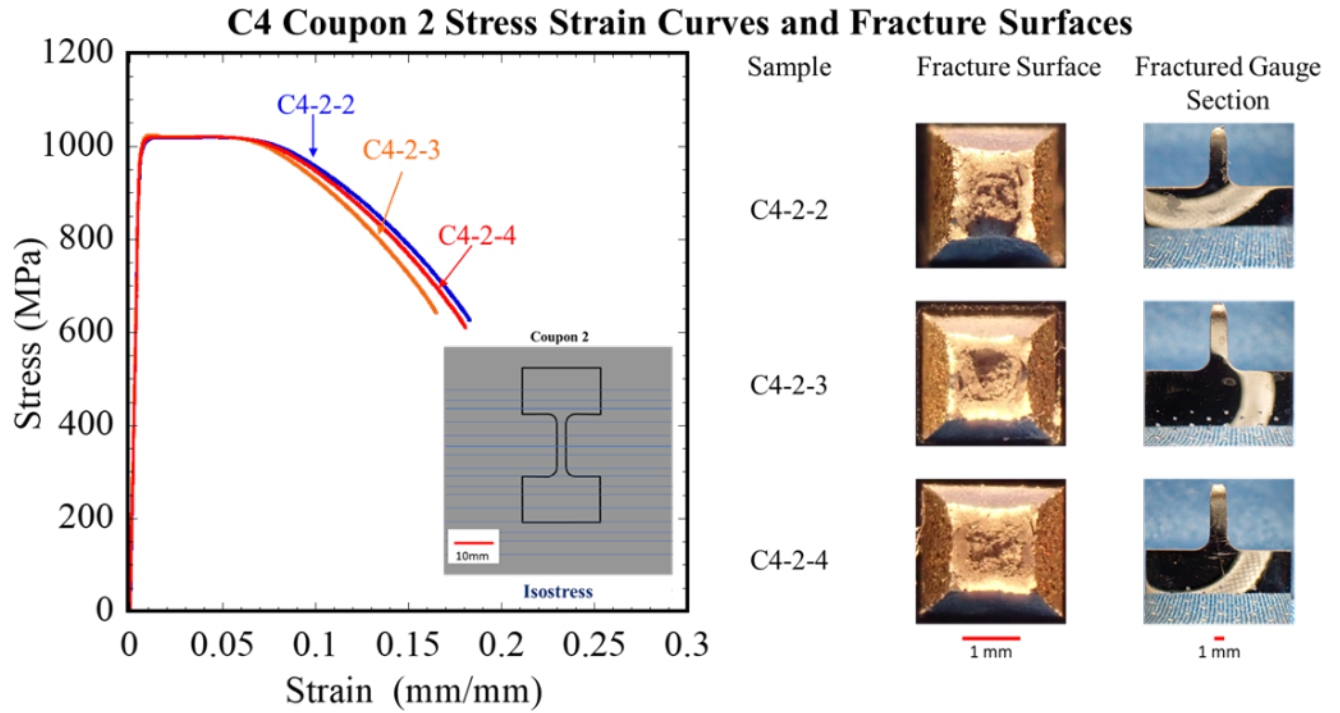


Figure 6-28: Build C4 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

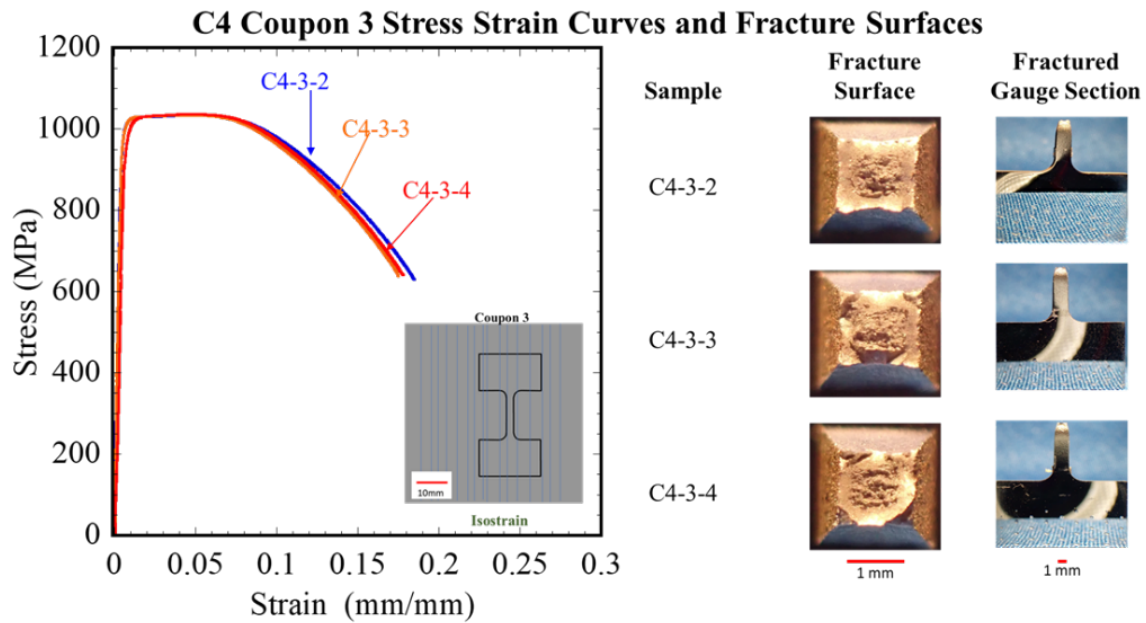


Figure 6-29: Build C4 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

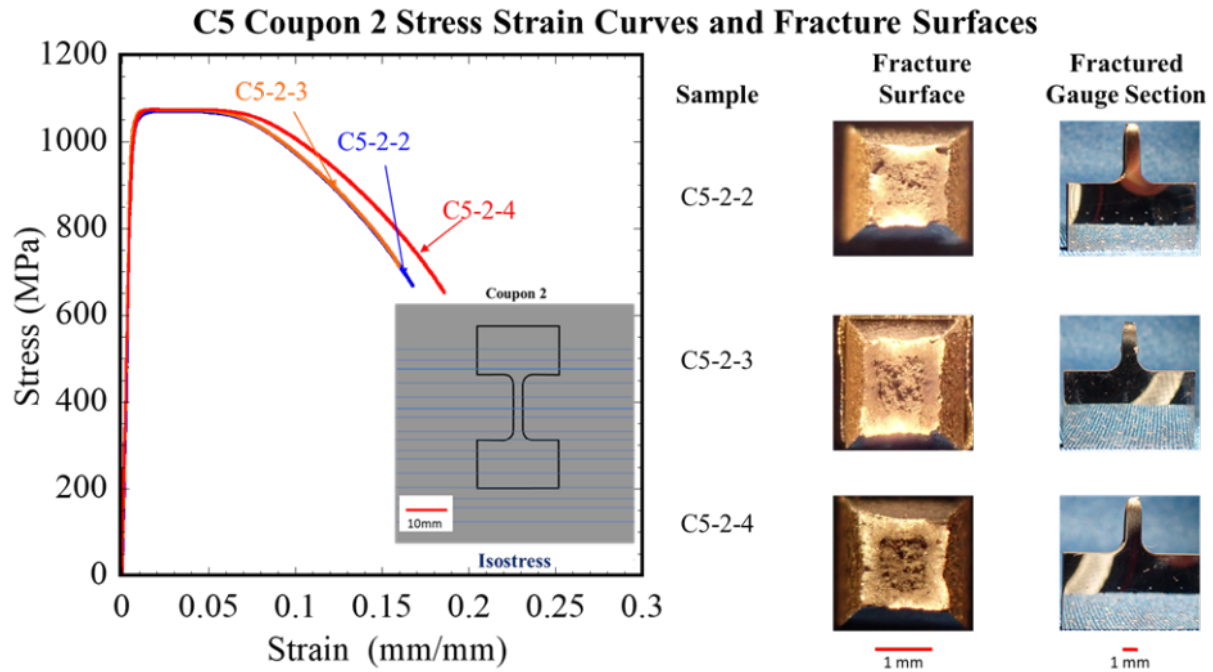


Figure 6-30: Build C5 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

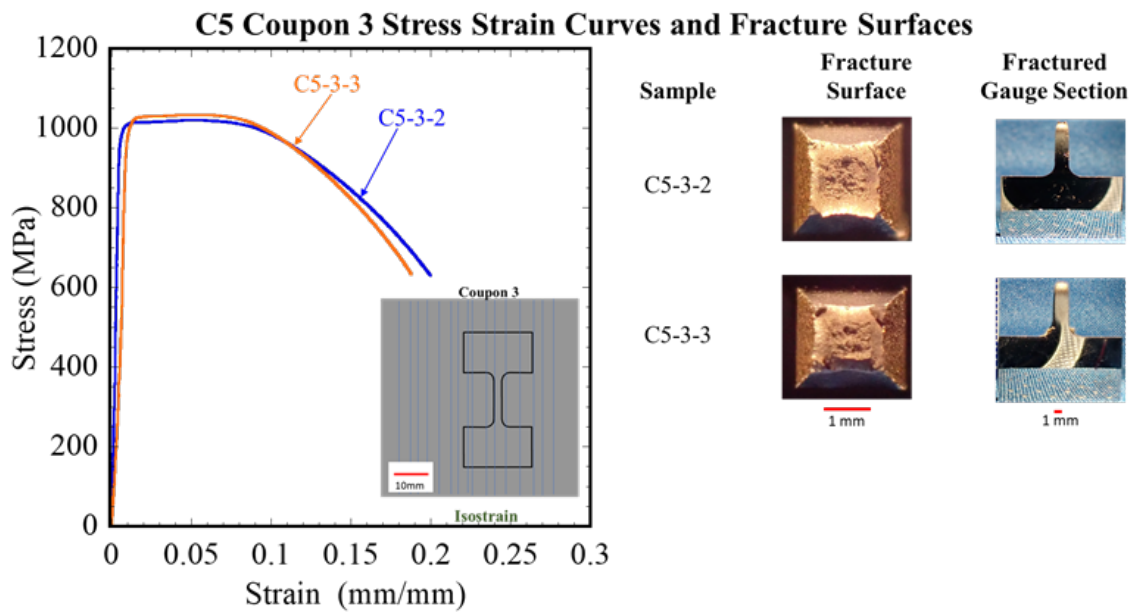


Figure 6-31: Build C5 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

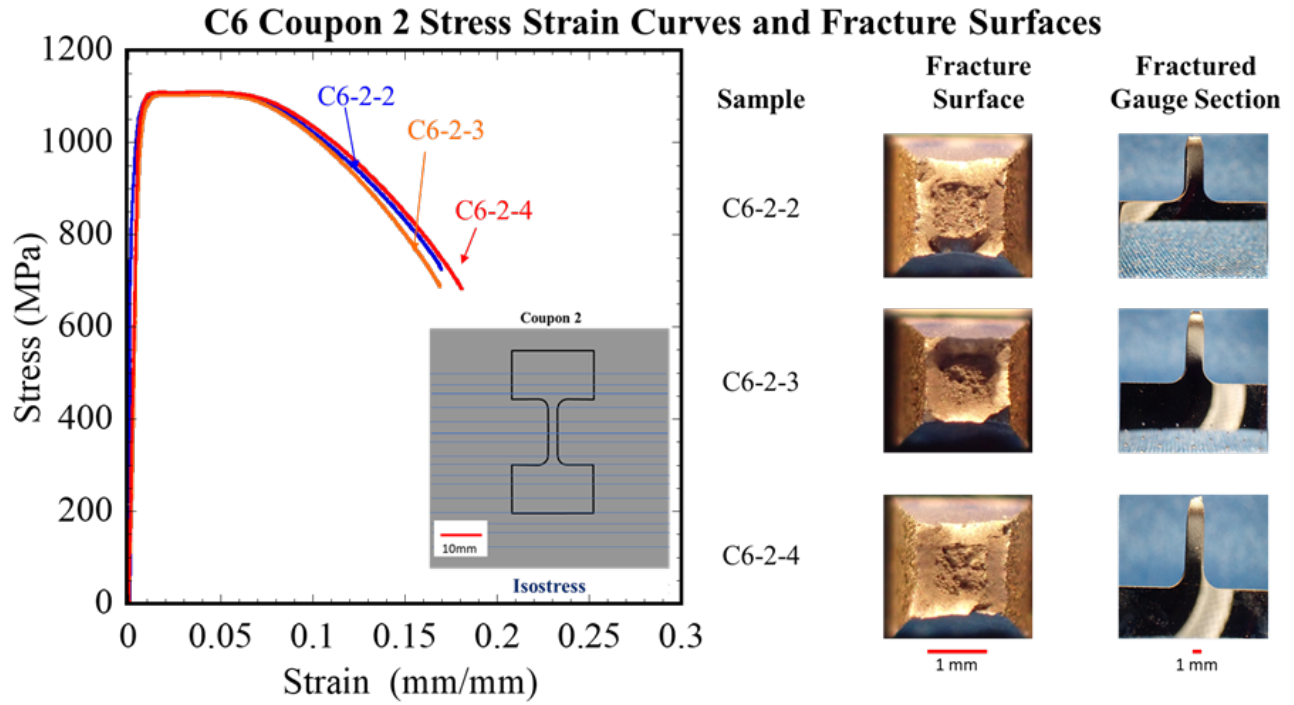


Figure 6-32: Build C6 Coupon 2 stress-strain curves and the fracture surface and gauge section of each sample

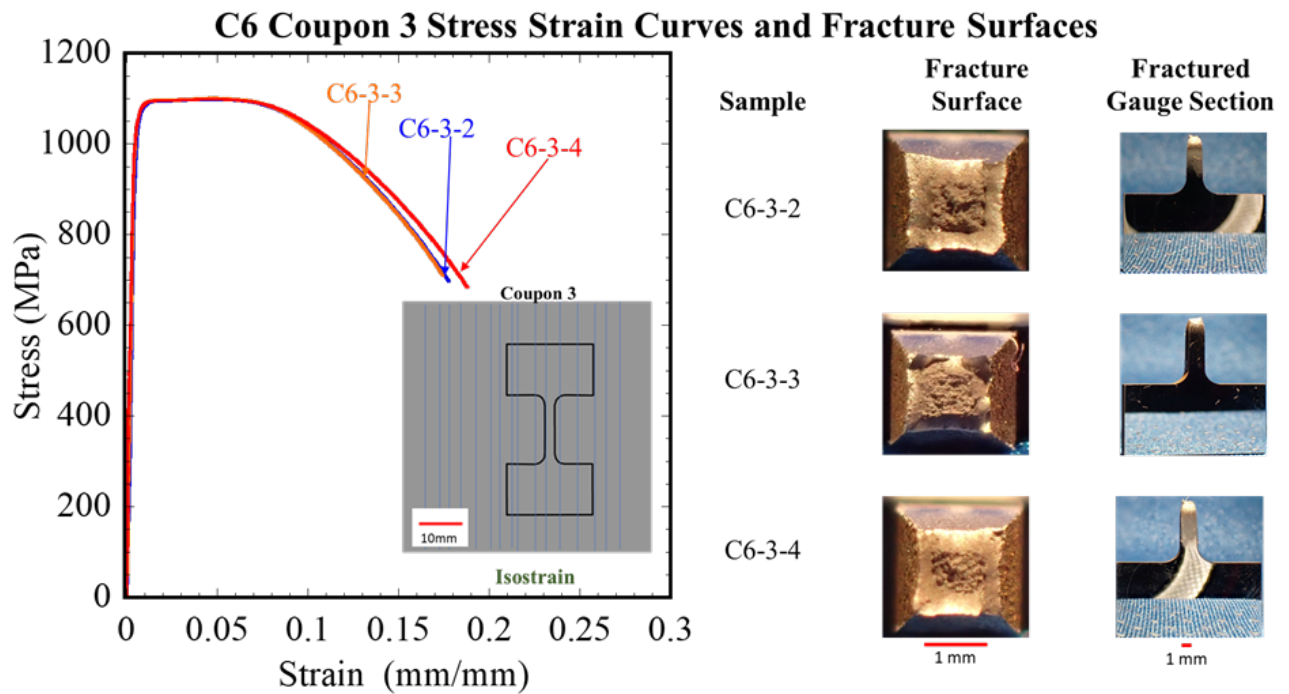


Figure 6-33: Build C6 Coupon 3 stress-strain curves and the fracture surface and gauge section of each sample

In addition to the cup and cone shape, all the samples except those in build A4 have an appreciable area reduction, indicating necking. Area reduction was calculated by approximation of the initial and final areas of the fractured sample. Figure 6-34 shows how the areas were approximated in Microsoft PowerPoint.

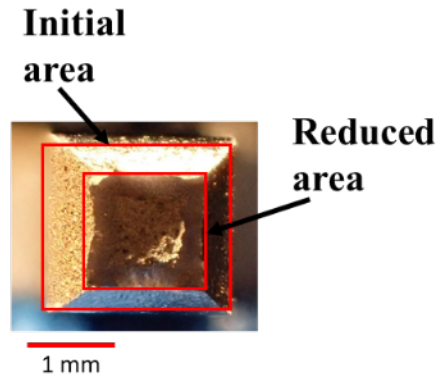


Figure 6-34: Example of initial and reduced areas of meso sample fracture surface

Equation 6-2 is the method used to calculate percent reduction:

$$\% \text{ Reduction} = \frac{\text{Initial Area} - \text{Final Area}}{\text{Initial Area}} \quad (6-2)$$

Table 6-1 lists the average percent reduction per printer by coupon. In addition, the test strain to failure (the failure strain measured by the Instron test machine) was compared to the actual strain to failure for each meso sample to provide a better perspective on the value of percent reduction. Actual strain to failure was calculated using Equation 6-3, where  $A_0$  was the initial meso sample area and  $A$  was the final, reduced meso sample area. Note that the initial and final sample areas are the same values used to calculate the percent reduction of the meso samples.

$$\text{Actual Strain to Failure} = \ln (A_0/A) \quad (6-3)$$

This comparison is valuable because if the actual strain to failure is lower than the test strain to failure, it is known that a void impacted the sample's failure.

Table 6-2 displays the results of this comparison for the three meso samples with abnormal fracture surfaces, A4-3-2, A4-3-3, and A4-3-4.

Table 6-1: Average % reduction by printer and coupon for meso samples with associated statistics

| <b>Build</b> | <b>Average % Reduction</b> | <b>Standard Deviation</b> | <b>Coefficient of Variation</b> |
|--------------|----------------------------|---------------------------|---------------------------------|
| <b>A2</b>    | 42%                        | 0.09                      | 0.22                            |
| <b>A3</b>    | 34%                        | 0.20                      | 0.52                            |
| <b>B2</b>    | 46%                        | 0.10                      | 0.15                            |
| <b>B3</b>    | 35%                        | 0.10                      | 0.33                            |
| <b>C2</b>    | 49%                        | 0.04                      | 0.08                            |
| <b>C3</b>    | 48%                        | 0.09                      | 0.18                            |

Table 6-2: Sample by sample comparison of measured and calculated percent reduction for samples with abnormal fracture surfaces

| <b>Sample</b> | <b>Measured Strain to Failure</b> | <b>Calculated Strain to Failure</b> |
|---------------|-----------------------------------|-------------------------------------|
| <b>A4-3-2</b> | 0.0685                            | 0.000                               |
| <b>A4-3-3</b> | 0.0618                            | 0.000                               |
| <b>A4-3-4</b> | 0.0726                            | 0.000                               |

Note that Table 6-2 confirms the theory that Samples A4-3-2, A4-3-3, and A4-3-4 fractured prematurely due to the presence of a void as all three of these samples have lower calculated percent reduction than measured percent reduction.

### **6.3 Conclusions and Future Work**

This study confirms the repeatability of the EOS M 290 printer for three builds of 17-4 PH SS. Specifically, this study establishes a known lack of repeatability in samples cut perpendicular to the AM print direction. With the exclusion of samples containing defects, gauge sections in line with the printing direction had lower strength than those perpendicular to the printing direction. Additionally, because build geometry changes how heat flows through a part as it is printed, geometry impacts final material properties. Therefore, the geometry and orientation of an AM component affect its strength.

Future work for this study includes examining fracture surfaces using an SEM microscope to understand existing defects or inclusions. Additionally, comparing these results with results of samples without post heat treatment would provide valuable information about the impact of heat treatment of microstructure and mechanical properties on additively manufactured precipitation hardened 17-4 stainless steel.

More importantly, modeling of the flow of heat through each sample type-meso sample, wide-based zig-zag structure, and narrow-based zig-zag structure will provide more concrete information on how the variation of geometry and corresponding variation of heat flow impacts mechanical properties. Finally, it will be essential to repeat this study with different printers to verify the findings based on the EOS M290 printers used in this study.

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## Appendix A. Mechanical Properties of Individual Meso Samples

| Sample | Yield Stress (MPa) | Ultimate Stress (MPa) | Strain to Failure (mm/mm) | Young's Modulus (GPa) | % Reduction |
|--------|--------------------|-----------------------|---------------------------|-----------------------|-------------|
| A1-2-2 | 1040.0             | 1048.1                | 0.166                     | 292.0                 | 54.5%       |
| A1-2-3 | 1035.6             | 1049.8                | 0.190                     | 232.3                 | 44.6%       |
| A1-2-4 | 1053.3             | 1057.7                | 0.181                     | 298.9                 | 45.9%       |
| A1-3-2 | 1031.1             | 1037.7                | 0.177                     | 289.1                 | 42.0%       |
| A1-3-3 | 1017.8             | 1039.1                | 0.178                     | 271.8                 | 32.9%       |
| A1-3-4 | 1013.3             | 1039.0                | 0.179                     | 270.4                 | 47.5%       |
| A2-2-2 | 1027.8             | 1051.9                | 0.160                     | 163.6                 | 35.8%       |
| A2-2-3 | 988.9              | 1045.8                | 0.112                     | 103.9                 | 38.4%       |
| A2-2-4 | 755.6              | 847.4                 | 0.176                     | 137.1                 | 21.9%       |
| A2-3-2 | 1066.7             | 1090.4                | 0.154                     | 124.6                 | 16.9%       |
| A2-3-3 | 1077.8             | 1091.1                | 0.161                     | 161.8                 | 36.2%       |
| A3-2-2 | 1013.9             | 1023.7                | 0.181                     | 263.8                 | 43.3%       |
| A3-2-3 | 1013.9             | 1022.5                | 0.184                     | 202.7                 | 29.8%       |
| A3-2-4 | 1013.9             | 1024.4                | 0.178                     | 247.9                 | 21.5%       |
| A3-3-2 | 1048.6             | 1069.4                | 0.177                     | 199.8                 | 40.6%       |
| A3-3-3 | 1055.6             | 1070.5                | 0.179                     | 226.2                 | 49.8%       |
| A3-3-4 | 1062.5             | 1074.7                | 0.184                     | 219.7                 | 42.5%       |
| A4-2-2 | 1016.7             | 1072.2                | 0.199                     | 289.6                 | 49.8%       |
| A4-2-3 | 1075.0             | 1082.8                | 0.173                     | 218.4                 | 43.4%       |
| A4-2-4 | 1066.7             | 1077.6                | 0.177                     | 142.0                 | 48.6%       |
| A4-3-2 | 995.6              | 1036.0                | 0.069                     | 124.2                 | 0.0%        |
| A4-3-3 | 1017.8             | 1037.9                | 0.062                     | 135.6                 | 0.0%        |
| A4-3-4 | 995.6              | 1035.9                | 0.073                     | 113.9                 | 0.0%        |
| A5-2-2 | 997.8              | 1042.1                | 0.170                     | 205.3                 | 37.6%       |
| A5-2-3 | 1013.9             | 1040.0                | 0.191                     | 162.7                 | 49.0%       |
| A5-2-4 | 993.8              | 1035.5                | 0.177                     | 172.5                 | 44.2%       |
| A5-3-2 | 1020.8             | 1078.2                | 0.186                     | 122.0                 | 46.5%       |
| A5-3-3 | 1041.7             | 1063.0                | 0.171                     | 225.9                 | 45.1%       |
| A5-3-4 | 1069.4             | 1082.9                | 0.171                     | 239.8                 | 55.7%       |
| A6-2-2 | 996.3              | 1024.1                | 0.185                     | 155.5                 | 45.1%       |
| A6-2-3 | 983.3              | 1018.5                | 0.179                     | 170.7                 | 50.0%       |
| A6-2-4 | 1050               | 1058.7                | 0.185                     | 184.5                 | 47.7%       |
| A6-3-2 | 1033.3             | 1040.7                | 0.170                     | 204.0                 | 36.4%       |
| A6-3-3 | 1041.7             | 1057.6                | 0.195                     | 132.5                 | 47.5%       |
| A6-3-4 | 1050.0             | 1058.7                | 0.185                     | 184.5                 | 39.6%       |
|        |                    |                       |                           |                       |             |
| B1-2-2 | 1050.0             | 1078.0                | 0.18                      | 143.7                 | 53.8%       |
| B1-2-3 | 1072.2             | 1090.6                | 0.17                      | 177.9                 | 37.1%       |
| B1-2-4 | 1072.2             | 1084.4                | 0.18                      | 170.2                 | 46.9%       |
| B1-3-2 | 1133.3             | 1156.4                | 0.165                     | 172.9                 | 19.5%       |
| B1-3-3 | 1133.3             | 1158.9                | 0.174                     | 183.6                 | 38.7%       |

|               |        |         |       |       |       |
|---------------|--------|---------|-------|-------|-------|
| <b>B1-3-4</b> | 1044.4 | 1153.7  | 0.182 | 211.2 | 47.1% |
|               |        |         |       |       |       |
| <b>C1-2-2</b> | 1011.1 | 1029.4  | 0.176 | 155.1 | 42.7% |
| <b>C1-2-3</b> | 1005.6 | 1012.9  | 0.189 | 207.2 | 50.7% |
| <b>C1-3-2</b> | 1041.7 | 1050    | 0.183 | 159.6 | 21.4% |
| <b>C1-3-3</b> | 1000   | 1049.4  | 0.287 | 150.5 | 28.1% |
| <b>C1-3-4</b> | 1041.7 | 1052.2  | 0.172 | 176   | 44.4% |
| <b>C2-2-2</b> | 1005.6 | 1026.3  | 0.182 | 194.7 | 45.0% |
| <b>C2-2-3</b> | 1001.7 | 1017.3  | 0.165 | 180.9 | 49.2% |
| <b>C2-2-4</b> | 990    | 1023.2  | 0.178 | 235.7 | 54.9% |
| <b>C2-3-2</b> | 1013.3 | 1036.8  | 0.182 | 149.4 | 51.7% |
| <b>C2-3-3</b> | 1022.2 | 1039.9  | 0.19  | 243.2 | 47.1% |
| <b>C2-3-4</b> | 1008.9 | 1040.5  | 0.185 | 194.4 | 55.5% |
| <b>C3-2-2</b> | 942    | 984.3   | 0.19  | 246.6 | 57.3% |
| <b>C3-2-3</b> | 1008.3 | 1025.5  | 0.184 | 157.5 | 45.3% |
| <b>C3-2-4</b> | 986.2  | 1021.9  | 0.18  | 245.3 | 51.5% |
| <b>C3-3-2</b> | 1027.6 | 1043.7  | 0.168 | 220.4 | 49.7% |
| <b>C3-3-3</b> | 1052.5 | 1074.5  | 0.196 | 157.9 | 47.4% |
| <b>C3-3-4</b> | 1035.9 | 1049    | 0.173 | 200.7 | 45.0% |
| <b>C4-2-2</b> | 1011.1 | 1019.8  | 0.185 | 169.8 | 49.0% |
| <b>C4-2-3</b> | 1022.2 | 1024.1  | 0.166 | 189.9 | 54.8% |
| <b>C4-2-4</b> | 1016.7 | 1021    | 0.182 | 175   | 47.9% |
| <b>C4-3-2</b> | 1020.8 | 1034.3  | 0.169 | 265.3 | 59.4% |
| <b>C4-3-3</b> | 1020.8 | 1035    | 0.176 | 260.9 | 54.5% |
| <b>C4-3-4</b> | 1013.9 | 1037.4  | 0.162 | 195.6 | 54.5% |
| <b>C5-2-2</b> | 1050   | 1069.1  | 0.169 | 206.6 | 55.0% |
| <b>C5-2-3</b> | 1066.7 | 1073.8  | 0.161 | 195   | 43.5% |
| <b>C5-2-4</b> | 1061.1 | 1075.2  | 0.187 | 166.4 | 46.3% |
| <b>C5-3-2</b> | 1006.9 | 1020.7  | 0.202 | 246.7 | 50.5% |
| <b>C5-3-3</b> | 1027.8 | 1034.7  | 0.189 | 109.9 | 53.1% |
| <b>C6-2-2</b> | 1097.2 | 1106    | 0.172 | 249.1 | 44.9% |
| <b>C6-2-3</b> | 1090.3 | 1104.6  | 0.17  | 198.1 | 50.9% |
| <b>C6-2-4</b> | 1104.2 | 1110.8  | 0.182 | 202.7 | 51.2% |
| <b>C6-3-2</b> | 1094.4 | 1099.3  | 0.174 | 243.3 | 42.5% |
| <b>C6-3-3</b> | 1077.8 | 1101.9  | 0.171 | 196.2 | 47.3% |
| <b>C6-3-4</b> | 1094.4 | 1099.99 | 0.178 | 191.1 | 48.7% |

## Appendix B. Comparison of Tested and Calculated % Reduction of Individual Meso Samples

| Sample | Measured % Reduction | Calculated % Reduction |
|--------|----------------------|------------------------|
| A1-2-2 | 0.166                | 0.785                  |
| A1-2-3 | 0.19                 | 0.591                  |
| A1-2-4 | 0.181                | 0.620                  |
| A1-3-2 | 0.177                | 0.545                  |
| A1-3-3 | 0.178                | 0.402                  |
| A1-3-4 | 0.179                | 0.645                  |
| A2-2-2 | 0.16                 | 0.443                  |
| A2-2-3 | 0.112                | 0.484                  |
| A2-2-4 | 0.176                | 0.247                  |
| A2-3-2 | 0.154                | 0.186                  |
| A2-3-3 | 0.161                | 0.449                  |
| A3-2-2 | 0.181                | 0.568                  |
| A3-2-3 | 0.184                | 0.354                  |
| A3-2-4 | 0.178                | 0.242                  |
| A3-3-2 | 0.177                | 0.522                  |
| A3-3-3 | 0.179                | 0.689                  |
| A3-3-4 | 0.184                | 0.554                  |
| A4-2-2 | 0.199                | 0.690                  |
| A4-2-3 | 0.173                | 0.570                  |
| A4-2-4 | 0.177                | 0.666                  |
| A4-3-2 | 0.0685               | 0.000                  |
| A4-3-3 | 0.0618               | 0.000                  |
| A4-3-4 | 0.0726               | 0.000                  |
| A5-2-2 | 0.17                 | 0.472                  |
| A5-2-3 | 0.191                | 0.670                  |
| A5-2-4 | 0.177                | 0.575                  |
| A5-3-2 | 0.186                | 0.625                  |
| A5-3-3 | 0.171                | 0.600                  |
| A5-3-4 | 0.171                | 0.583                  |
| A6-2-2 | 0.185                | 0.601                  |
| A6-2-3 | 0.179                | 0.693                  |
| A6-2-4 | 0.185                | 0.648                  |
| A6-3-2 | 0.17                 | 0.453                  |
| A6-3-3 | 0.195                | 0.648                  |
| A6-3-4 | 0.185                | 0.504                  |
|        |                      |                        |
| B1-2-2 | 0.178                | 0.773                  |
| B1-2-3 | 0.173                | 0.469                  |
| B1-2-4 | 0.178                | 0.633                  |
| B1-3-2 | 0.165                | 0.217                  |

|               |       |       |
|---------------|-------|-------|
| <b>B1-3-3</b> | 0.174 | 0.490 |
| <b>B1-3-4</b> | 0.182 | 0.637 |
|               |       |       |
| <b>C1-2-2</b> | 0.176 | 0.556 |
| <b>C1-2-3</b> | 0.189 | 0.708 |
| <b>C1-3-2</b> | 0.183 | 0.241 |
| <b>C1-3-3</b> | 0.287 | 0.330 |
| <b>C1-3-4</b> | 0.172 | 0.587 |
| <b>C2-2-2</b> | 0.182 | 0.597 |
| <b>C2-2-3</b> | 0.165 | 0.677 |
| <b>C2-2-4</b> | 0.178 | 0.797 |
| <b>C2-3-2</b> | 0.182 | 0.727 |
| <b>C2-3-3</b> | 0.190 | 0.636 |
| <b>C2-3-4</b> | 0.185 | 0.809 |
| <b>C3-2-2</b> | 0.190 | 0.851 |
| <b>C3-2-3</b> | 0.184 | 0.603 |
| <b>C3-2-4</b> | 0.18  | 0.723 |
| <b>C3-3-2</b> | 0.168 | 0.687 |
| <b>C3-3-3</b> | 0.196 | 0.643 |
| <b>C3-3-4</b> | 0.173 | 0.598 |
| <b>C4-2-2</b> | 0.185 | 0.674 |
| <b>C4-2-3</b> | 0.166 | 0.794 |
| <b>C4-2-4</b> | 0.182 | 0.652 |
| <b>C4-3-2</b> | 0.169 | 0.902 |
| <b>C4-3-3</b> | 0.176 | 0.787 |
| <b>C4-3-4</b> | 0.162 | 0.787 |
| <b>C5-2-2</b> | 0.169 | 0.799 |
| <b>C5-2-3</b> | 0.161 | 0.571 |
| <b>C5-2-4</b> | 0.187 | 0.621 |
| <b>C5-3-2</b> | 0.202 | 0.703 |
| <b>C5-3-3</b> | 0.189 | 0.756 |
| <b>C6-2-2</b> | 0.172 | 0.596 |
| <b>C6-2-3</b> | 0.170 | 0.712 |
| <b>C6-2-4</b> | 0.182 | 0.717 |
| <b>C6-3-2</b> | 0.174 | 0.552 |
| <b>C6-3-3</b> | 0.171 | 0.641 |
| <b>C6-3-4</b> | 0.178 | 0.667 |