

MECHANICAL CHARACTERIZATION AND ANALYSIS OF FDM 3D PRINTED COPPER SAMPLES

A Thesis Presented
By
Steven Allen Long

Presented to the Jackson College of Graduate Studies of the University of Central
Oklahoma in Partial Fulfillment of the requirements for the Degree of

MASTER OF SCIENCE IN ENGINEERING PHYSICS – MECHANICAL
ENGINEERING
December 2022

Abstract

Additive manufacturing, specifically 3D printing, technology has revolutionized the design, creation, and building of parts and engineering objects. The fused deposition modeling (FDM) or UV-cured resin-based 3D printings are widely used techniques for rapid prototyping today. The prototypes are typically made of various thermoplastics such as acrylonitrile butadiene styrene (ABS), polylactic acid (PLA), and polyethylene terephthalate glycol (PETG) with each thermoplastic having different properties. However, FDM or resin-based 3D printing cannot manufacture metal parts due to some inherent engineering challenges, such as the requirement of the extremely high melting temperature of the metals, making metal filaments, and involved complex thermal management.

This thesis research has explored and studied the possibility of manufacturing metal parts using an ordinary FDM 3D printer, a specialized kiln, and PLA-Cu filament with 80% - 90% of copper (Cu) contents. The parts were debound and sintered in the kiln to burn off the PLA, which allowed the Cu to bond to create pure Cu parts. The 3D printing flow rates, debinding and sintering times in the kiln were varied to understand the effect of these parameters on the material characteristics and mechanical strength of the printed Cu samples. The ASTM D638 samples were 3D printed at three flow rates: 133%, 135%, and 140% in an FDM printer and debound and sintered at three different times: 3 hour debind 3 hour sinter, 3 hour debind 4 hour sinter, and 3 hour debind 5 hour sinter. The dimensions and weight of the samples were measured before and after sintering. Samples were polished and etched using a 5-step polishing method with different grinding grits (coarse to ultra-fine) and etched with ferric chloride. Grain boundaries and surface qualities of the 3D printed and sintered samples were observed under a high-precision material microscope, stored, and compared with the cold-rolled annealed copper samples. The tensile strength of the samples was evaluated

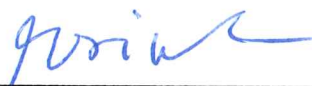
using Universal Testing Machine (UTM). The results show that the 135% 3-hour debind and 4-hour sinter provided the best overall results for the least amount of void within the sample, nicer grain boundaries and mechanical strength. The average stress vs strain value for cold rolled annealed copper is 220.6 Mpa. The results from the tensile testing came out to have a maximum of 39.107 MPa in the 133% flow rate 3 hour debind 4 hour sinter. The second highest stress value came from the 135% flow rate 3 hour debind 4 hour sintering time group at 37.29 Mpa. While the quality and material grain boundaries are comparable with cold-rolled annealed samples, the mechanical characteristics are far off. Nonetheless, the overall aspect of making copper parts from conventional FDM-based 3D printing technology is promising. The findings of this research will contribute to understanding and better utilization of FDM-based 3D printed Cu and general metal parts for future engineering endeavors.

Mechanical Characterization and Analysis of FDM 3D Printed Copper Samples

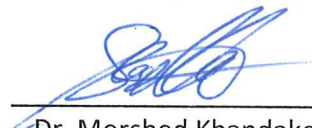
A THESIS

APPROVED FOR THE SCHOOL OF
ENGINEERING

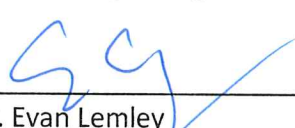
By


Dr. Mohammad Hossan
Committee Chairperson
School of Engineering

12/09/2022
Date


Dr. Morshed Khandaker
Committee Member
School of Engineering

12/9/2022
Date


Dr. Evan Lemley
Committee Member
School of Engineering

12/9/22
Date

TABLE OF CONTENTS

List of Figures.....	VI
List of Tables.....	X
Chapter 1	
1.1. Introduction.....	1
1.2. Evolution of Additive Manufacturing.....	1
1.3. Types of Additive Manufacturing Method and their Characteristics.....	3
1.4. Thesis Research Question.....	5
Chapter 2	
2.1. 3D Metal Printing, Advancement and Challenges.....	8
Chapter 3	
3.1. 3D Printing and Analysis of Copper Samples.....	13
3.2. Material and Methods	
3.2.1. Materials.....	13
3.2.2. FDM 3D printing of Copper Samples.....	15
3.2.3. Debinding and Sintering of Copper Samples.....	15
3.2.4. Polishing of Copper Samples.....	16
3.2.5. Characterization of Samples with Metallurgical Microscopes.....	17
3.2.6. Void Percent Calculation Method Using ImageJ.....	19
3.2.7. Tensile Test with the Universal Testing Machine.....	20
3.2.8. Error and Uncertainty Analysis.....	21
3.3.Results and Discussion	
3.3.1. Effect of Flowrates and sintering times on Dimensional losses.....	23

3.3.2. Effect of Flowrate and sintering times on Mass losses.....	25
3.3.3. Effect of Flowrates and sintering times on Microstructure and Surface porosity.....	26
3.3.4. Effect of Flow rates and Sintering times on Mechanical Strength.....	28
3.3.5. Internal voids and porosity characteristics.....	36
Chapter 4	
4.1. General Conclusion and Discussion.....	37
4.2. Future Works.....	40
4.3. Concluding Thoughts.....	40
References.....	42
Appendix A: Tables.....	A
Appendix B: 133% Flow Rate Images and Graphs.....	G
Appendix C: 135% Flow Rate Images and Graphs.....	L
Appendix D: 140% Flow Rate Images and Graphs.....	Q

List of Figures

Figure 3.1: (a) Crealty CR-10s printing copper samples; (b) Al_2O_3 sintering ballast; (c) Sintering Carbon; (d) Starter kiln; (e) Alumina crucible uncovered within kiln.....	14
Figure 3.2 (a) Buehler MetaServ 250 grinder-polisher; (b) 3D printed specimen holder for polisher; (c) Mockup to produce CNC machined aluminum specimen holder.....	17
Figure 3.3 (a) Nikon MA100 inverted metallographic microscope; (b) Buehler IsoMet Low Speed cutting machine sectioning a sample; (c) Cross-Sectioned 3D Printed Copper Sample.....	18
Figure 3.4 ImageJ Thresholding Method Example [20].....	19
Figure 3.5 (a) Test Resources universal testing machine; (b) Sample on UTM machine within mounting apparatus.....	21
Figure 3.6 CAD Dog Bone Design and Dimensions.....	23
Figure 3.7 (a) 133% sintering time comparison: 3hr, 4hr, and 5hr; (b) 140% sintering time comparison: 3hr, 4hr, and 5hr; (c) 5 hour sintering time comparison between flow rates: 133%, 135%, and 140%.....	24
Figure 3.8 Average mass loss per flow rate.....	25
Figure 3.9 (a) 133% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 133% 3 hour debind 3 hour sinter 50x magnification; (c) 133% 3 hour debind 3 hour sinter 20x magnification void calculation	26
Figure 3.10 The optical microstructure of annealed copper [33].....	28
Figure 3.11 (a) Stress vs Strain curve of 133% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 133% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 133% flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 133% flow rate samples in Newtons.....	29
Figure 3.12 (a) Stress vs Strain curve of 140% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 140% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 140%	

flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 140% flow rate samples in Newtons.....	30
Figure 3.13 (a) Stress vs Strain curve of 135% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 135% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 135% flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 135% flow rate samples in Newtons.....	31
Figure 3.14 Maximum Stress comparison against flow rate and sintering time.....	32
Figure 3.15 Average maximum force per flow rate and sintering time.....	33
Figure 3.15 Annealed Phosphorus Deoxidized Copper Stress vs Strain[32].....	34
Figure 3.17 (a) Sample 1 133% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 133% 3 hour debind 3 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 3 hour sinter.....	36
Figure A1 CAD Dog Bone Design and Dimensions.....	A
Figure B1 (a) 133% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 133% 3 hour debind 3 hour sinter 50x magnification; (c) 133% 3 hour debind 3 hour sinter 20x magnification void calculation.....	G
Figure B2 (a) 133% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 133% 3 hour debind 4 hour sinter 50x magnification; (c) 133% 3 hour debind 4 hour sinter 20x magnification void calculation.....	H
Figure B3 (a) 133% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 133% 3 hour debind 5 hour sinter 50x magnification; (c) 133% 3 hour debind 5 hour sinter 20x magnification void calculation.....	H
Figure B4 (a) Sample 1 133% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 133% 3 hour debind 3 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 3 hour sinter.....	I

Figure B5 (a) Sample 1 133% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 133% 3 hour debind 4 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 4 hour sinter.....	I
Figure B6 (a) Sample 1 133% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 133% 3 hour debind 5 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 5 hour sinter.....	J
Figure C1 (a) 135% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 135% 3 hour debind 3 hour sinter 50x magnification; (c) 135% 3 hour debind 3 hour sinter 20x magnification void calculation.....	L
Figure C2 (a) 135% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 135% 3 hour debind 4 hour sinter 50x magnification; (c) 135% 3 hour debind 4 hour sinter 20x magnification void calculation.....	L
Figure C3 (a) 135% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 135% 3 hour debind 5 hour sinter 50x magnification; (c) 135% 3 hour debind 5 hour sinter 20x magnification void calculation.....	M
Figure C4 (a) Sample 1 135% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 135% 3 hour debind 3 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 3 hour sinter.....	N
Figure C5 (a) Sample 1 135% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 135% 3 hour debind 4 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 4 hour sinter.....	N
Figure C6 (a) Sample 1 135% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 135% 3 hour debind 5 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 5 hour sinter.....	O

Figure D1 (a) 140% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 140% 3 hour debind 3 hour sinter 50x magnification; (c) 140% 3 hour debind 3 hour sinter 20x magnification void calculation.....	Q
Figure D2 (a) 140% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 140% 3 hour debind 4 hour sinter 50x magnification; (c) 140% 3 hour debind 4 hour sinter 20x magnification void calculation.....	Q
Figure D3 (a) 140% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 140% 3 hour debind 5 hour sinter 50x magnification; (c) 140% 3 hour debind 5 hour sinter 20x magnification void calculation.....	R
Figure D4 (a) Sample 1 140% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 140% 3 hour debind 3 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 3 hour sinter.....	S
Figure D5 (a) Sample 1 140% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 140% 3 hour debind 4 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 4 hour sinter.....	S
Figure D6 (a) Sample 1 140% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 140% 3 hour debind 5 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 5 hour sinter.....	T

List of Tables

Table 3.1 Uncertainty Table.....	22
Table A1: 133% Flow Rate 3 Hour Debind 3 Hour Sinter Dimensional Measurements.....	A
Table A2: 133% Flow Rate 3 Hour Debind 4 Hour Sinter Dimensional Measurements.....	B
Table A3: 133% Flow Rate 3 Hour Debind 5 Hour Sinter Dimensional Measurements.....	C
Table A4: 140% Flow Rate 3 Hour Debind 3 Hour Sinter Dimensional Measurements.....	D
Table A5: 140% Flow Rate 3 Hour Debind 4 Hour Sinter Dimensional Measurements.....	E
Table A6: 140% Flow Rate 3 Hour Debind 5 Hour Sinter Dimensional Measurements.....	F

Chapter 1

Background and Thesis Research Questions

1.1 Introduction:

Today, additive manufacturing has been utilized in multiple fields, from aerospace and automotive to bio-medical engineering and applications [1]. The terms “additive manufacturing” and “3D printing” are frequently utilized freely to describe the same thing [2]. In additive manufacturing, a material is added to a build plate in a specific pattern, with more layers added on top of each other and the result being the desired piece. There are many different methods of 3D printing that are used in the present day. All these different methods run off the same basic rules: i) material must be present, ii) energy must be added to the system, and iii) there must be a machine and tool present and active. The material can be anything from a solid (i.e., filament), liquid (i.e., UV reactive resin), powder (i.e., metal powder), or other means that can be added to the existing material on the built platform. Energy must be added to the system for the shape to be changed or held in place. This energy can be heat from an electron beam, electrical resistance, or other means. It can also be UV light in the visible spectrum or as a laser. Energy can also be stored chemically for adhesion or chemical reactions to allow the material to harden to become the final product. For the machine and tool, typically, the tool head lies on a coordinate x,y, and z plane, which is tied to a belt system or a lead screw system. This system allows the head to move in the three planes and make the product.

1.2 Evolution of Additive Manufacturing:

When adding material one layer at a time, additive manufacturing transforms a geometric representation into an actual product [3]. The early additive manufacturing research conducted in

the 1960s aimed to polymerize resin by colliding two laser beams with different wavelengths. Similar techniques were used in 1971 by Wyn Swainson in his patent application for photochemical machining. Japan's Nagoya Municipal Industrial Research Institute's Hideo Kodama pioneered a quick prototyping method requiring only one laser beam. He applied for a patent in 1980; however, Kodama needed more time to progress through the last stages of the Japanese patent application process. He experimented with a 3D production method that uses UV light and photosensitive resin and published papers on his findings in 1980 and 1981. This technology solidifies thin layers of photopolymer to make up a 3D object and is now called stereolithography (SLA). Charles Hull formally discovered stereolithography in 1984, and the technique was patented in 1986. Hull founded 3D Systems and later produced and sold SLA 3D printers in 1988. Around the same time, selective laser sintering (SLS) based 3D printers were invented by Carl Deckard at the University of Texas [4]. SLS is the procedure for melting and fusing powdered materials using a powerful laser. In 1992, Deckard established the Desktop Manufacturing Corporation (DTM Corp), which created the first SLS printer. DTM was eventually purchased by 3D Systems. Decker founded Sinterstation 2000 in 1993, introducing SLS technology into the engineering industry. In 1989 S. Scott and Lisa Crump submitted a patent application for a novel method of fast prototyping known as fused deposition modeling (FDM)[5]. A heated metal or plastic wire is used in FDM, which is extruded from a nozzle. Based on a specified digital model, a computer controls the entire procedure. To ensure that the best layer adhesion is achieved one must keep the layers at a temperature just under the solidification point to ensure that the best layer adhesion is achieved. Electro Optical Systems (EOS) concentrated on and created direct metal laser sintering later in 1989 (DMLS)[6]. To direct the laser to metal powder for liquid phase sintering, DMLS uses computer design models.

EOS later acquired all DTM laser sintering intellectual rights in 2004. In the early 1990s, several different additive manufacturing technologies were investigated. William Masters created an additive manufacturing process that utilized a jet that traveled in a coordinate plane to spray drops of wax substance and make thin cross sections. This process was called "Ballistic Particle Manufacturing." The fixed platform moved to the Z-axis for each layer to be added. In 1995, Michael Feygin submitted a patent to produce laminated objects, which used an automated process for cutting cross-sectional slices from sheet material by following a computerized three-dimensional model.

1.3 Types of Additive Manufacturing Methods and their Characteristics:

As to ASTM Standard F2792, the seven categories of 3D printing methods include material extrusion, direct energy material deposition, binding jetting, material jetting, sheet lamination, powder bed fusion, and vat photopolymerization [3]. Every one of these technologies has a particular use and purpose. The usage of additive manufacturing is expanding to actual items rather than merely for prototypes.

A liquid binding agent is carefully placed during the fast prototyping and 3D printing process known as "binder jetting" to bind powder particles together [7]. The layer is formed using the binder jetting method, which jets chemical binder onto the spreading force. This technique can produce vast quantities of comparable sand-based items, such as casting patterns or raw sintered products. Polymers, metals, sands, ceramics, and hybrids are some of the materials used in this technique. This approach is straightforward, quick, and affordable for the construction capacity. In the directed energy deposition (DEP) method, a material is extruded from a nozzle, similar to FDM. However, the nozzle on DEP is not fixed and can move in the directions of x, y, and z as designed. DEP is frequently used for materials made of metals and metal-based hybrids or for

adding material to upgrade the existing parts [8]. Because of its scalability and variety of features in a single system, it has vastly increased in popularity.

Material extrusion is one of the most widely used and popular additive manufacturing techniques. Polymers and plastics, designing food products or live cell assembly for tissue engineering, and other applications may all be printed in various materials and colors. This process has very cheap costs and may create components or products that are completely functioning. FDM uses heated thermoplastic filament extruded to manufacture parts and models from the bottom to the top through layer-by-layer printing [9].

Drop-by-drop deposition of construction material is known as material jetting. A print head typically releases droplets of photosensitive substance that solidify when exposed to UV light. The pieces produced have a very smooth surface and are quite accurate in their dimensions [3]. Polymers, ceramics, composites, biologicals, and hybrids may all be printed with this technique. Examples of powder bed fusion processes include electron beam melting, selective heat sintering printing, and selective beam sintering. Either an electron beam or laser is utilized to fuse the powder or melt the material. Polymers, metals, ceramics, and composite materials can be used in this method. The selective laser sintering (SLS) is a 3D printing method with variable surface finishes, high precision, and operating speed. In SLS, powdered polymers are sintered or melted together using a strong laser. For selective heat sintering (SHS) to function, layers of powdered thermoplastic are heated using a thermal printhead.

The 3D printing technique known as sheet lamination combines different material sheets to make a part, component, or whole object. This technique is known as laminated object manufacturing (LOM) [10]. Full-color printing may be done with this procedure in a manageable and reasonably priced manner. Additionally, the trash can be recycled. An ultrasonic additive

manufacturing (UAM) technique is also used in joining metal layers cut from foil material without features to form an object or a component.

Vat polymerization, which involves curing photo-reactive polymers with a laser, light, or ultraviolet light, takes place inside a vat instead of being sprayed or extruded. Digital light processing (DLP) or stereolithography (SLA) technologies are used in vat polymerization-based 3D printing. The success of the SLA depends on the photoinitiator, exposure times, light sources, material dyes, and pigments [11]. DLP and SLA are comparable; however, DLP uses a different light source. DLP exposes the entire surface of the photopolymer vat in a single pass using a standard light source, like an arc lamp. In SLA, the resin in the vat is selectively cured using a UV laser and directed light. While DLP produces products far more quickly than SLA, both have good details and a smooth surface.

1.4 Thesis Research Question:

Despite the popularity of FDM and resin-based 3D, the conventional 3D metal printer primarily uses powder bed fusion, binder jetting, and selective laser melting methods due to technical challenges involved in the FDM-based metal extrusion. Recently the potential filament-based 3D printing of metal parts has become research of great interest. Metal parts are printed using a regular FDM 3D printer with specialty manufactured filament consisting of high amounts of desired metal (80% to 90%) and small quantities of thermoplastic polymer (20 % to 10%) such as polylactic acid (PLA). After printing, the parts are debound and sintered. The debinding process in which the plastic is removed. In the sintering, the remaining metal particles are brought just below their melting point, in which they become attracted to and stick to one another. However, it is uncertain how different 3D printing factors, as well as debinding and sintering process parameters, affect the material characteristics as well as the mechanical

strength of the created 3D metal objects. Hence the goals of this thesis are to study and analyze the effect of

1. extrusion rate on the mechanical and material properties of copper samples
2. sintering times on the mechanical and material properties of copper samples

The following deliverables are assessed to evaluate the goal of the thesis:

1. Fabricate ASTM standard dog-bone copper samples using FDM 3D printer with 133% flow rate
2. Fabricate ASTM standard dog-bone copper samples using FDM 3D printer with 135% flow rate
3. Fabricate ASTM standard dog-bone copper samples using FDM 3D printer with 140% flow rate
4. Debind and sintering 3D printed copper samples of 133%, 135% and 140% rates with 3 hrs sintering time
5. Debind and sintering 3D printed copper samples of 133%, 135% and 140% rates with 4 hrs sintering time
6. Debind and sintering 3D printed copper samples of 133%, 135% and 140% rates with 5 hrs sintering time
7. Record and compare dimensional change of the samples before and after various sintering times
8. Record and compare weight change of the copper samples before and after various sintering times

9. Perform polishing and etching of the sintered copper samples with standard polishing and etching protocols of the copper samples
10. Observe, capture and compare the grain boundaries of copper samples as a functions of 3D printing flow rates and sintering time
11. Estimate and compare void percentage on the top surfaces and internal x-sectional area of the copper samples for the multiple process parameters
12. Perform and analyze the tensile strength of 3D printed and sintered copper samples of the various parameters.

Chapter 2

Literature Review

2.1 3D metal Printing, Advancement and Challenges:

When it comes to 3D printing metals, there are a few methods that are most used: electron beam melting (EBM), direct metal laser sintering (DMLS), and selective laser melting (SLM). In DMLS, a base material (i.e., metal powder) is bonded together by sintering in which the grains are not fully melted together [12]. The material is not brought to its melting point, but the powder comes to a state where it is almost sticky, and this state allows the powder to bond to itself. According to Jiménez and their team, it is unquestionably true that no technology at this time completely satisfies all of the needs of the job standards in the building and architectural industries [12]. From this statement, the sintered product is not nearly as strong as its melted counterpart. In SLM 3D printing, the powder is fully melted together. This is a much more extreme method than DMLS. SLM requires more heat for this process to take place but results in a stronger part than what can be achieved with DMLS.

It's crucial to pay attention to details when working with additive manufacturing, such as the surface roughness, minimum feature size, and geometric precision of an item. Once a part has been created, it is machined into its final form that is dimensionally accurate, and any surface imperfections are removed [13]. Depending on the settings employed, the powder bed fusion (PBF) generally has the best resolution, with LM having a somewhat greater resolution than EBM [13]. In most cases, much like with FDM additive manufacturing, smaller features and

smaller layer thicknesses increase the deposition rate or printing rate. PBF methods are capable of printing metal mesh as well as foam-like structures with high resolution.

There tends to be an issue with porosity, cracking, delamination, and swelling while handling metal additive manufacturing. During powder atomization of the powder feedstock, gas pores may form in a spherical shape. In most cases, porosity formation is kept at a minimum to avoid cracks and defects. Cracking, delamination, and swelling are the defects that normally appear because of imperfect heating, such as over or under-heating. Cracking tends to appear during solidification or subsequent heating, while macroscopic cracks are from defects like porosity. Delamination is from a lack of layer adhesion due to improper melting. If the temperature is too high, the melt pool has a tendency to move outwards, causing swelling in the layer [13].

Beamless metal additive manufacturing techniques are becoming more and more common.

Beamless metal additive manufacturing techniques are exactly as the name suggests. They do not use a beam such as a laser or electrons to melt the metal. Liquid metal jetting, wire and arc additive manufacturing, and shape deposition manufacturing utilize beam-based technologies but use a different energy source than a laser or electron beam. Layer bonding is accomplished via bulk sintering in the furnace through the use of binder jetting, material extrusion, nanoparticle inkjet printing, selective inhibition sintering, aerosol jet, and 3D screen printing [14]. In contrast to beam-based techniques, binder jetting has a greater print resolution and surface polish.

Extrusion-based additive manufacturing significantly lowers production costs. Comparing extrusion-based metal additive manufacturing to beam-based systems, one benefit is that the green pieces are sintered after printing, leaving little to no residual heat stress.

Recently Hao Lu et al. studied FDM based metal printing and their mechanical properties for aluminum, stainless steel, bronze, and copper. In their thermogravimetric analysis (TGA), it

involved measuring the sample's mass over time while the temperature rises at a predetermined pace. The thermal stability and rate of degradation of the printed metals were also included in TGA studies. For this particular experiment, 50 mg of the substance under investigation was poured into the sample pan and heated to 700 deg C at a steady rate of 10 C/min. However, the aluminum samples were heated to 500 deg C to protect their equipment. Thermomechanical analysis is another test that was run. TMA calculates the amount of material deformation in a controlled temperature environment. The materials were put through a compression test by Hao et al. The test pieces are solid cylinders with a 2:1 L/D ratio. Aluminum, bronze, copper, high-carbon steel, and standard PLA are employed in that experiment. Stress-strain curves were used to display the results of compression experiments. When comparing the compression behavior of metal filament with conventional PLA, it can be seen that the presence of metal inside the material reduces the product's mechanical strength. Researchers examined the tensile characteristics of metal filament specimens and looked at various printing factors, such as the kind of material used, the layer thickness, and the sintering temperature. The layer height used to print the unsintered metal specimens was 0.1 mm. The sintered copper samples were printed using layers that were 0.1, 0.2, and 0.3 mm thick. The samples were sintered between 1050⁰C and 1065⁰C. An overall perspective of the specimens' micron-scale structure was provided using optical microscopy. Two distinct metal and polymer phases were seen in their samples before sintering. The Cu particles are visible with the dark hue against the brighter polymer background because a significant portion of the area is filled with PLA.

The distribution of the copper particles inside the substance was not uniform. The polymer broke down after sintering, left the copper and spaces behind. The gas produced by the thermal breakdown of PLA during the sintering or printing process might be to blame for the observed

voids. This explains why the Cu-PLA has a lower tensile strength than components made using conventional techniques. 1065⁰C was the sintering temperature. SEM technology was also utilized to investigate the structure's intricacies.

Cristina-Elisabeta PELIN et al. studied the mechanical properties of 3D printed metals. In general, powder with a certain particle size based on the production method is the raw material for the additive manufacture of metals [16]. The diameters can vary between 15 and 45 micrometers for laser sintering and 45 and 106 micrometers for electron beam sintering. Maraging steel was the metallic substance utilized in laser 3D printing. Maraging is known as martensite-hardening steel with a high limit of extension and little deformation.

The laser-printed samples were examined using optical and scanning electron microscopy (SEM) from a morphostructural perspective. Images of the failure site were captured using both morphostructural analysis methods following destructive mechanical testing. Optical examination revealed the failure mode resulting from the broken 3D printing's overlapping layers. SEM images revealed an irregular, discontinuous structure that resembles a sponge in several ways. Following the mechanical tension test, plastic deformation in the printing direction was seen for all three cylindrical samples, suggesting a ductile rupture of the substance. Final testing revealed that the direction of printing had a negligible impact on mechanical qualities. This demonstrates that the sample was not anisotropic [16].

Terry et al. studied the hardness and porosity characteristics of filament-based 3D printed Cu samples. In their study, they examined the porosity and hardness of printed and sintered samples. These samples were printed, debound, sintered, and then mounted and polished. The porosity is examined at different magnification values. An image processing software was utilized to calculate the percentage of pores. For the hardness testing, they utilized the Rockwell Hardness

method. The results of their study state that the discrepancies between the sample sizes before and after sintering can be maintained under 5%. However, the accuracy was reduced due to the influence of oxidation. The microstructural data showed that the porosity could be reduced to as low as 4.3%. The porosity in the samples depends on the powder bonding quality [15]. In their hardness testing, the standard Rockwell Hardness value is 106 for a similar specimen. This value was used in calculating the percent difference between the standard and the printed copper. The difference was approximately 57.98% or 44.68. This shows that copper is much softer than traditional copper. In their testing, they got various numbers that show that the filament does not make a bond uniformly.

Chapter 3

Methods, Results and Discussion

3.1 3D Printing and Analysis of Copper Samples:

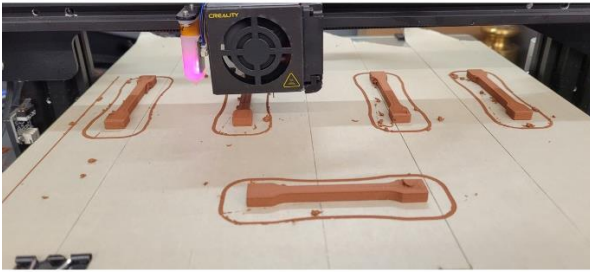
Copper filament from The Virtual Foundry in Stoughton, Wisconsin was utilized in this study. The copper was chosen due to its lower melting point, malleability, and electrical and thermal conductivity. These attributes allow copper for many engineering applications, such as rocket engine liners, refrigerant transport tubing, heat exchangers, and wiring in electric motors [18]. The copper filament is approximately 87-90% copper and 10% PLA and a proprietary binder [19]. The density of the filament is 4.7 g/cc [19]. This thesis research aimed to explore and study the effect of various process parameters involved in 3D printed and sintered processes to fabricate solid copper parts and their mechanical properties. This is so that the material can be utilized in suitable engineering and structural applications.

3.2 Materials and Methods:

3.2.1 Materials:

For this study, the following materials and equipment were used: Creality CR-10s 3D printer by Creality out of Shenzhen, China, with an all metal hot end, hardened steel 0.6mm nozzle, and dual gear extruder, computer-controlled kiln from Sapphire3D from out of Schaumburg, Illinois, copper Filament, Al₂O₃ Sintering Refractory Ballast, Sintering Carbon, Alumina Sintering Crucible, white painters tape, Elmer's glue stick, Crucible tongs, Test Resources Universal Testing Machine out of Shakopee, Minnesota, Pneumatic clamp x2, 321 Stainless Steel Tool

Wrap, and ferric chloride. The stainless steel foil, hardened steel nozzle, extruder, and ferric chloride were purchased from Amazon.com, Inc.



(a)



(b)



(c)



(d)



(e)

Figure 3.1 (a) CreaLITY CR-10s printing copper samples; (b) Al_2O_3 sintering ballast ; (c) Sintering Carbon; (d) Starter kiln (e) Alumina crucible uncovered within kiln.

3.2.2 FDM 3D printing of copper Samples:

Three different test groups were formed from the same base settings on the 3D printer, but only one parameter changed with each group. The flow rate varied at 133%, 135%, and 140% in each group. The print settings go as follows: 230°C on the hot end, 30 mm/s print speed, and 60°C on the build plate. The build plate also had a layer of blue painter's tape and Elmer's glue stick to promote bed adhesion and allow for a barrier for sufficient part removal. The dog bone style specimen was created in the Solidworks CAD software based out of Massachusetts, with ASTM D638 standard, and it was printed five pieces at a time, as shown in figure 3.1a.

3.2.3 Debinding and Sintering of copper Samples:

Within these test groups, a sub-group was formed. Each would undergo varying debinding and sintering times in the kiln, with the temperature remaining consistent. Group one underwent a 3 hour debind and 3 hour sintering time. Group 2 underwent a 1-hour increase in sintering time, i.e., 4 hours of sintering time. Group 3 had an increase in sintering time by 2 hours from the recommended time, i.e., 5 hours of sintering time. Al_2O_3 sintering refractory ballast was placed in the crucible, and the 3D-printed copper dog bone samples were buried in the Al_2O_3 . The sintering ballast was tamped down, and at least 25 mm of space was left at the top for the sintering carbon that would be added later. A key element was that there was at least 15mm between the copper samples and the crucible walls and the top of the refractory. Then the crucible with the model inside was placed within the kiln. The temperature was ramped up at a rate of 200°C per hour to 2040C and held at 204°C for 2 hours. Next, the temperature was ramped up in the furnace to 482°C at a rate of 111.2°C per hour and held at 482°C for 3 hours. When the debind cycle was completed, the crucible was removed using heat-resistant gloves and crucible tongs. For the sintering cycle, the entire 25mm space remaining on the top of the

sintering ballast was filled with sintering carbon using a non-metal scoop, stainless steel foil was tightly placed atop, and the crucible was placed back in the kiln. The sintering process was done in three different segments of the temperature cycle. First, the temperature in the kiln was ramped up to 482°C at a rate of 200°C per hour; second, the temperature was ramped up to 1018°C at a rate of 179°C per hour; and third, the temperature was ramped up to 1074°C at 28°C per hour and held at this temperature for 3 hours. Finally, the kiln was left to cool down naturally, and the sintering process was complete.

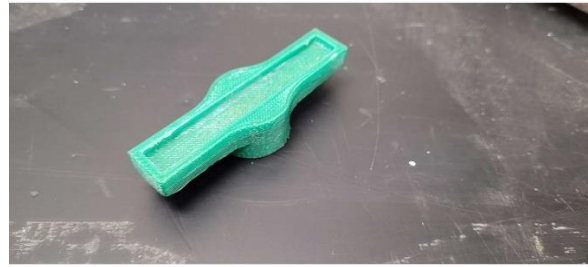
3.2.4 Polishing of copper Samples:

For the polishing of the copper parts after the sintering process was taken place. A 4-step process of polishing was conducted with the various grits sandpaper with Buehler polishing machine as follows: Using a 240-grit SiC pad at 240-300 rpm with a load of 5-6 Newtons until all surfaces were at the same plane and printing and sintering damage was removed. Next, a 9- μ m diamond paste was used on an Ultra-Pol cloth at 120-150 rpm with a load of 5-6 Newtons for 5 minutes to remove scratches and crack marks. Then a 3- μ m diamond paste on a Trident cloth at 120-150 rpm with a load of 5-6 Newtons for 3 minutes was used to further refine of the surfaces. Finally, 1- μ m diamond paste on a Trident cloth at 120-150 rpm with a load of 5-6 Newtons for 2 minutes was used ultra-fine polishing of the surfaces.

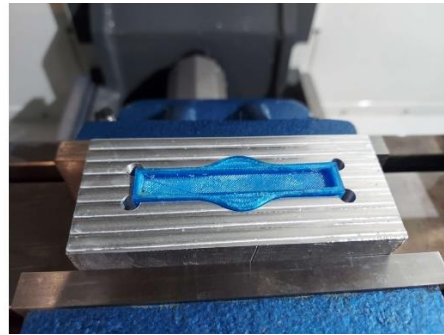
Next was the etching procedure. The copper samples were polished as stated above. Then the samples are submerged into the ferric chloride solution for 5 minutes with constant agitation and then submerged in water to dilute the acid. The sample was then taken to the microscope for imaging.



(a)



(b)



(c)

Figure 3.2 (a) Buehler MetaServ 250 grinder-polisher; (b) 3D printed specimen holder for polisher; (c) Mockup to produce CNC machined aluminum specimen holder

3.2.5 Characterization of Samples with Metallurgical Microscopes:

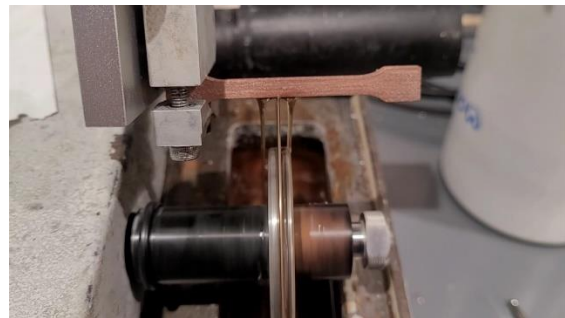
The images were captured using a Nikon MA100 inverted metallographic microscope. The images were taken at 5x, 10x, 20x, 50, and 100x magnification. The images were put into the image processing software ImageJ that was developed by Wayne Rasband out of Bethesda, Maryland. Using the software, the histogram data was utilized to calculate the percentage of voids or impurities in the parts. The voids or impurities show up as black spots or dark-colored areas against the copper. The computer program ImageJ is utilized to determine the percentage of copper that is on the surface of the samples. First, the image was converted into an 8-bit grayscale. Then it was thresholded using the binary function. The scale is created from the on-

image scale and converted into a value for the program to interpret. Finally, the image is measured using the analyze function, and the percentage of white pixels that make up the image is calculated.

In order to record the cross-sectional void of the samples, samples were cut in half. To do this, a Buehler IsoMet Low-Speed cutting machine was utilized. The machine was run at setting seven which is approximately 200 RPM utilizing IsoCut Fluid as the coolant. A 75g weight was added to the cutting arm to help reduce chatter and uneven cutting on the samples.



(a)



(b)



(c)

Figure 3.3 (a) Nikon MA100 inverted metallographic microscope; (b) Buehler IsoMet Low Speed cutting machine sectioning a sample; (c) Cross-Sectioned 3D Printed Copper Sample

3.2.6 Void Percent Calculation Method Using ImageJ:

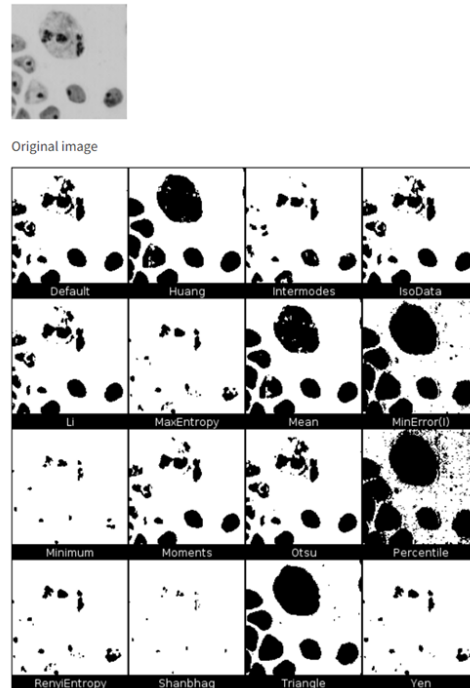


Figure 3.4 ImageJ Thresholding Method Example[20]

The default threshold method was utilized through ImageJ and determined to be the most accurate for this use case. The default method is a derivative of the IsoData method, which is told in the calculation to either ignore black pixels or ignore white pixels. Using the initial threshold, the IsoData method sorts the image into object and background and averages the pixels at or below the threshold. The average of the two values is calculated, then the threshold is incremented and repeated until the threshold is greater than the composite average [21]. The Intermodes method is described as using iterative smoothing to make two local maxima in which they are added together and divided by two [22]. This method is not useful for images with peaks or valleys. Huang's method minimizes the measurement of fuzziness from the calculation, which

would completely eliminate anything that is not on the surface of the sample [23]. Li's minimum cross entropy thresholding ends up counting pixels near the center gray level containing a value of 128 [24]. The method is not ideal if there is a lack of data or the gradient is zero. If there is a sudden drop-off between black and white pixels, it does not handle that well. The minimum error threshold method assumes the object and gray pixel values are normally distributed, which is not the case in our sample [25]. Otsu's threshold clustering algorithm finds the threshold that minimizes the difference between the intra-class variance, which is the sum of variance between the two classes [26]. The percentile method considers 0.5 foreground pixels which are very messy and not accurate for our use case [27]. The ReyniEntropy method is very similar to the MaxEntropy method, which was not suitable [28]. Shanbhag's method views an image of two fuzzy sets corresponding with a gray-level function. This method eliminates greyscale around or within the object. The triangle method is unable to determine the skewness of the data and considers that a maximum peak remains near one end of the histogram and searches toward the other end. The algorithm looks for which side of the maximum peak of data and searches for the threshold within that range of the data [30]. Yen's thresholding method is based on two factors – i) the difference between the original images and ii) the number of bits needed to represent the thresholded image. A cost function is generated from this information that determines the number of gray levels and how they should be classified as threshold values [31]. The method almost completely removes the gray in the example image, which may be useful for biology but not for our use case either. Therefore, the default auto-threshold method was determined to be one of the most accurate methods and utilized in the analysis.

3.2.7 Tensile Test with the Universal Testing Machine:

The universal testing machine was set up with pneumatic clamping jaws to grip the sample. It was found that the samples were small enough that the jaws could not grip them correctly. Therefore, a spacer was designed and 3D printed. After the initial setup of the machine took place by adding the clamps and positioning the clamps at the correct distance, the load cell was zeroed. The sample was then placed within the clamps and captured. The stroke was also zeroed at this point. Then at a speed of 1 mm/s, the stroke was increased until the sample was broken/failed.



(a)



(b)

Figure 3.5 (a) Test Resources universal testing machine; (b) Sample on UTM machine within mounting apparatus

3.2.8 Statistical and Uncertainty Analysis:

The error and uncertainty within the estimation are characterized by standard deviation based on the mean values, contrasts between measured and calculated, and test vulnerability. For the

physical measurements of this study the were repeated under the same conditions every time.

The standard deviation was calculated from these values utilizing the following equation:

$$\sqrt{\frac{\sum(x - y)^2}{(n - 1)}}$$

In this x is the sample mean average and n is the sample size.

The tools utilized in the study has an uncertainty and error characterizing them. The values are shown below in **Table 3.1**

<i>Parameter</i>	<i>Uncertainty</i>
<i>Porosity</i>	< 0.25%
<i>Yield strength</i>	< 0.4%
<i>Maximum tensile strength</i>	< 0.4%
<i>Dimensional measurement accuracy</i>	< 1.25%

Table 3.1 Uncertainty Table

3.3 Results and Discussion:

At the beginning of the study, it was originally thought that the sintering carbon was enough to keep oxygen out of the crucible and keep the samples from coming in contact with oxygen during the sintering process. Through analysis of the many samples created, the large amount of surface crystallization was not normal and extra steps were required to prevent the oxidation from occurring. A stainless-steel foil is typically utilized in heat-treating metals to cover the crucible. Stainless steel 321 was the alloy of foil used in this study. This foil was proven in testing to reduce the amount of oxygen that would make its way into the crucible, with multiple factors pointing to this. After the sintering process of the parts, there was a large quantity of

carbon left over on the top of the sintering ballast as well as the crystallization on the exterior of the parts was eliminated.

3.3.1 Effect of Flowrates and sintering times on Dimensional losses:

The below results utilize the measurements from the documentation taken of the size of each sample before and after the debind and sintering process. In **Figure 3.6** the dimensions are shown and labeled within Solidworks.

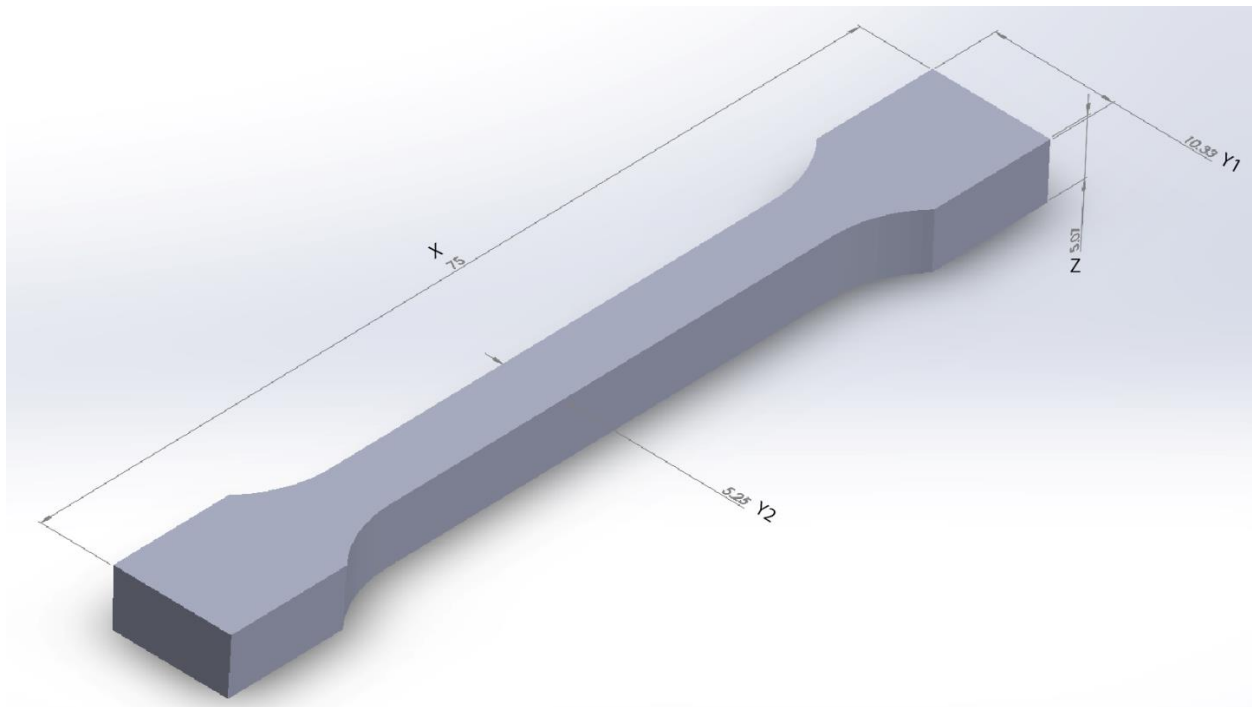


Figure 3.6 CAD Model of ASTM Dog Bone Sample



Figure 3.7 (a) 133% sintering time comparison: 3hr, 4hr, and 5hr; (b) 140% sintering time comparison: 3hr, 4hr, and 5hr; (c) 5 hour sintering time comparison between flow rates: 133%, 135%, and 140%.

On the samples, Y1 is the width of the largest part of the dogbone, while Y2 is the width of the narrow portion of the dogbone. Z is the thickness of the sample. With the samples placed

vertically in the crucible, it is surprising that the percentage of shrinkage in the X position (or length) is between 2% and 6%. Y1 shrinks between 7% and 9%, while Y2 shrinks approximately the same amount. The Z axis has the broadest range observed out of any other axis, of 4% and 9%. The shrinking on the Z, Y1 and Y2 axis being the highest was unexpected. The highest shrinking was expected along the X axis due to the positioning within the crucible. It can be inferred that these have the highest amount of shrinkage due to the length not being able to shift as much. The ballast holds the samples in place tightly and due to their shape, the ballast cannot be moved out of the way for it to shrink. Therefore other axis must compensate for this effectively giving up much more of their material to preserve the initial length.

3.3.2 Effect of Flowrate and sintering times on Mass losses:

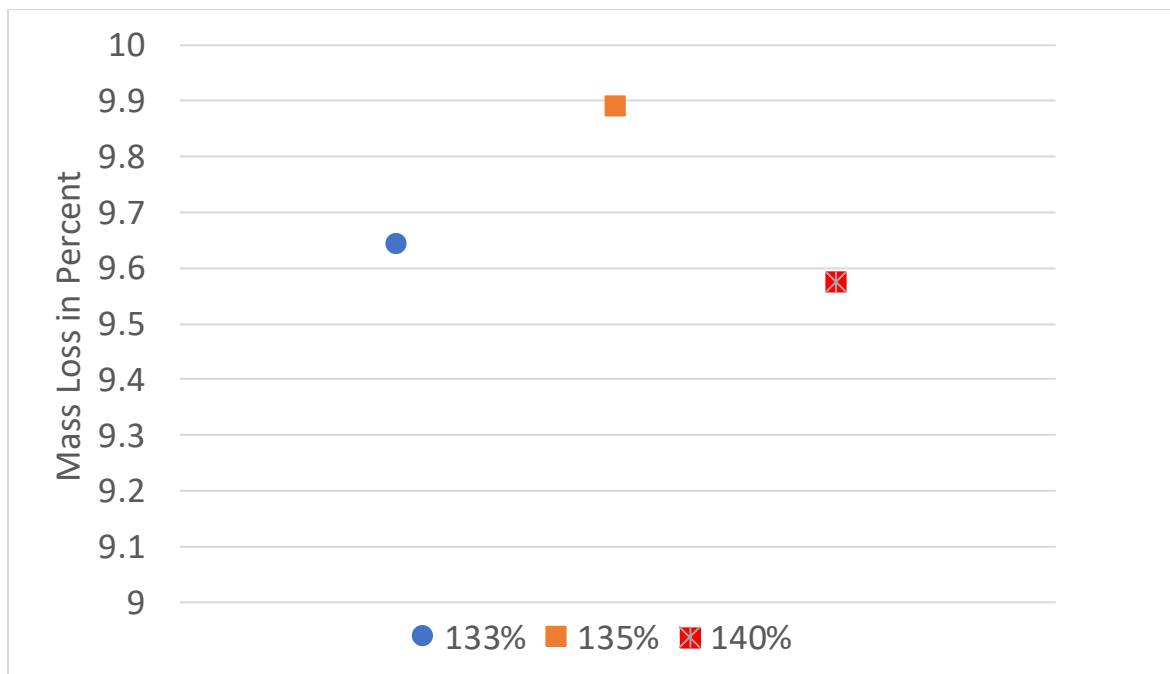


Figure 3.8 Average mass loss per flow rate

The tested filament contains 90% copper and 10% PLA blend. The results above show that approximately 10% of the material is removed and is within an acceptable margin of error. While analyzing the mass loss of each sample, it became apparent that the difference between sintering times and flow rates was within an error of each other. The variance between each sample was minuscule and only gave an outline of whether the material was fully sintered. This can be ascertained from the knowledge that the filament contains approximately 10% PLA and, in each trial, the mass lost is equivalent to 10% within a reasonable degree.

3.3.3 Effect of Flowrates and sintering times on Microstructure and Surface porosity:

Through imaging with the microscope, we could see the copper sample's grain structure and surface density.

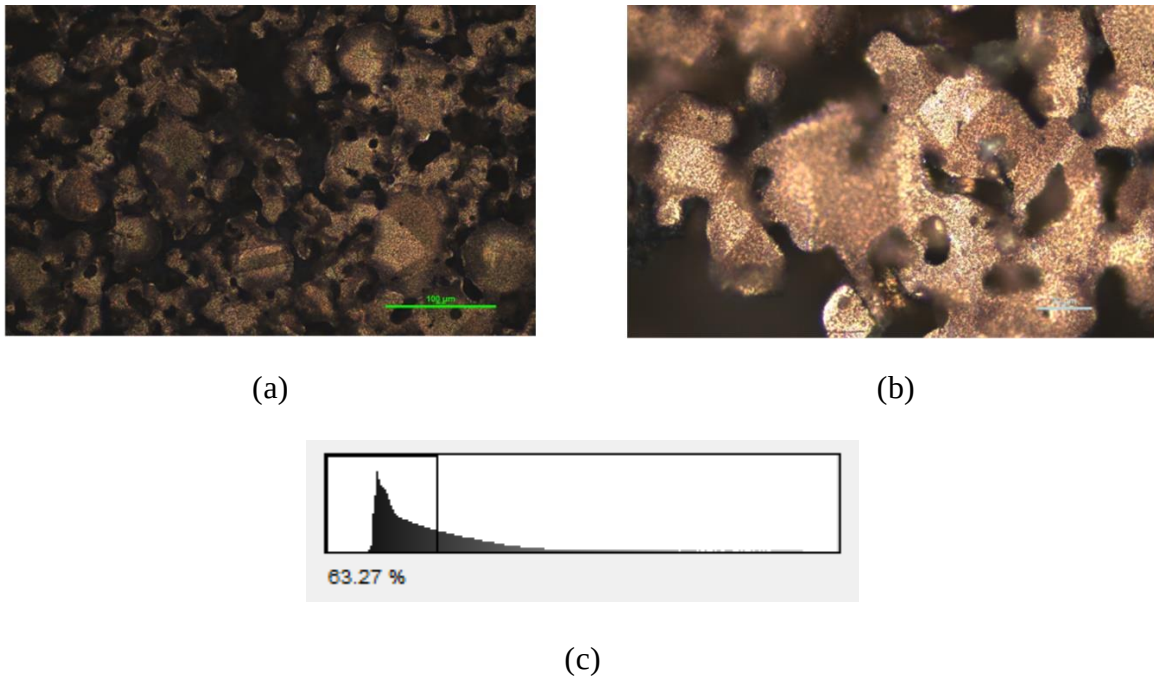


Figure 3.9 (a) 133% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 133% 3 hour debind 3 hour sinter 50x magnification; (c) 133% 3 hour debind 3 hour sinter 20x magnification void calculation

The images above and in the appendix show the approximate percentage of copper within a 192x120um area. On almost all of the samples other than the 133% flow rate, the amount of copper on the surface increased as sintering time increased. The 133% flow rate samples decreased as the sintering time increased. The 133% 3-hour debind 3-hour sinter sample measured 63.27% surface void. The 133% 3-hour debind 4-hour sinter measured 77.60%, and the 133% 3-hour debind 5-hour sinter measured 79.47% surface void. The 135% flow rate group measured at 68.18%, 30.28%, and 64.77% for the 3 hours debind 3-hour sinter, 3 hours debind 4-hour sinter, and 3 hours debind 5-hour sinter, respectively. Lastly, the 140% flow rate group measured 71.47%, 43.80%, and 11.12% for the respective debind and sintering times. The large surface void area has multiple factors involved. One of which is the initial grinding process. The samples cannot all be sanded for the same amount of time due to its intended purpose of removing surface impurities. As long as the sample appears to be completely copper, and any surface oxidization is removed it proceeds through the rest of the process. Another factor is the idea that a material is being removed in the debinding process. PLA is actively burned off at a controlled rate, and during the sintering process the material is attracted to itself causing a shrinking. During the shrinking process it may not be uniform which would cause peaks and valleys on the surface of the sample up to a certain unknown depth.

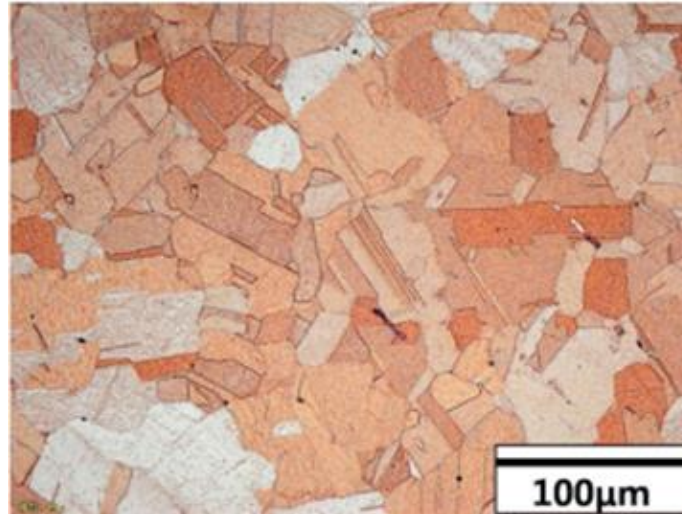
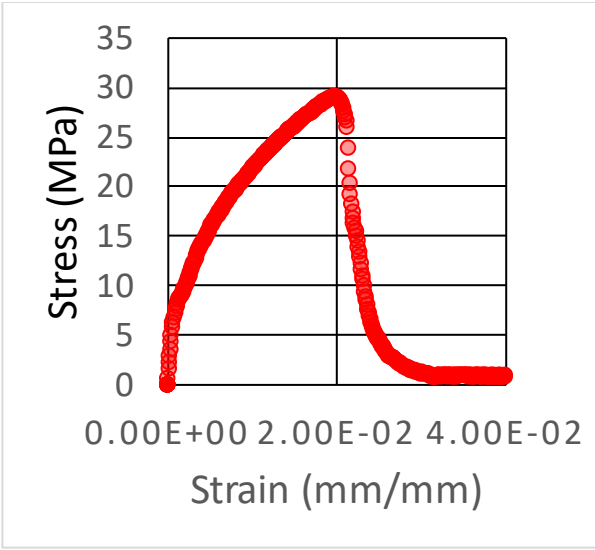


Figure 3.10 The optical microstructure of annealed copper [33]

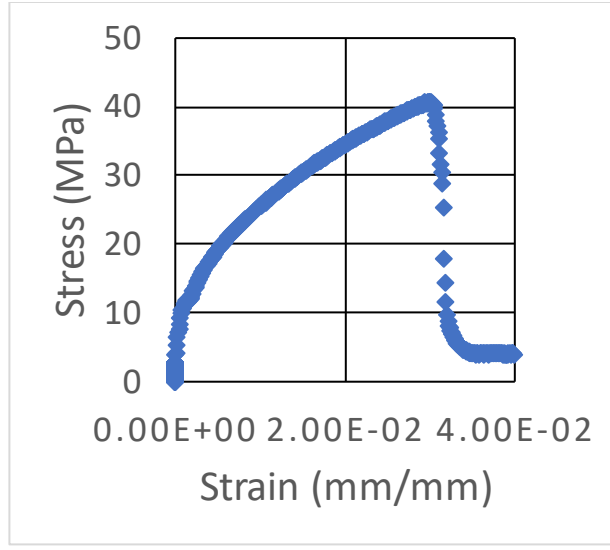
The grain structure between the images captured of the 3D printed samples and the referenced images above is very similar to the annealed copper. There is a possibility that what is observed as the grain structure is micro-cracks developed in the cooling of the sample post sintering process or even during the polishing process.

3.3.4 Effect of Flow rates and Sintering times on Mechanical Strength:

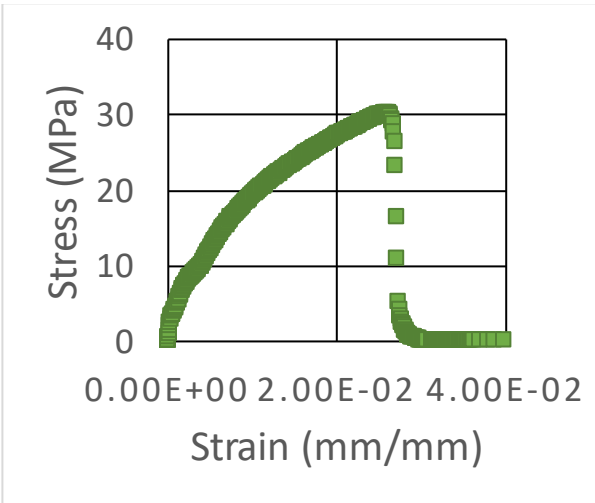
This translated into strength testing as well, with the granular structure of the parts being more consistent than before. The results from the new procedures are shown below in **Figure 3.11** through **Figure 3.13** in stress vs. strain graphs.



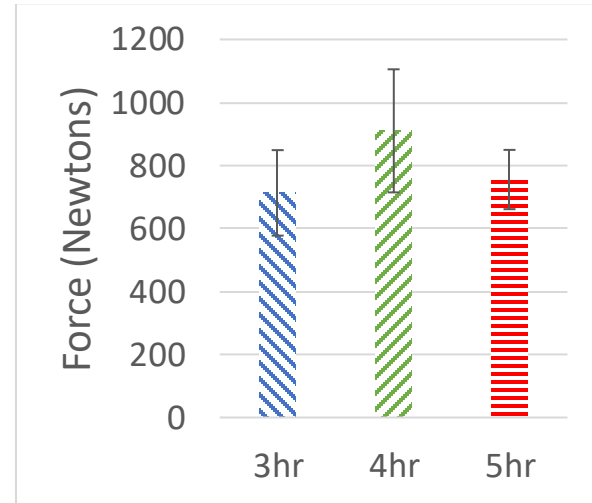
(a)



(b)

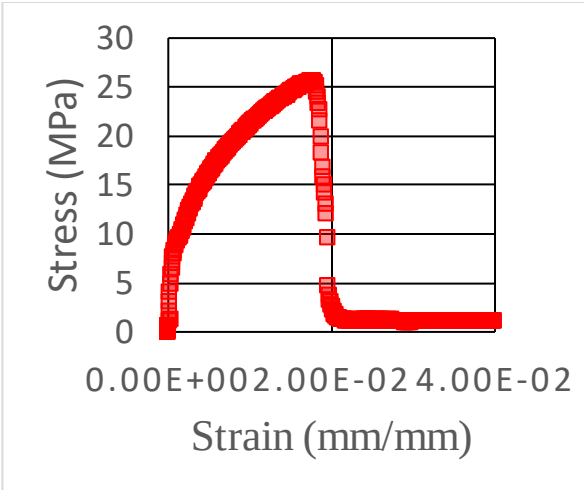


(c)

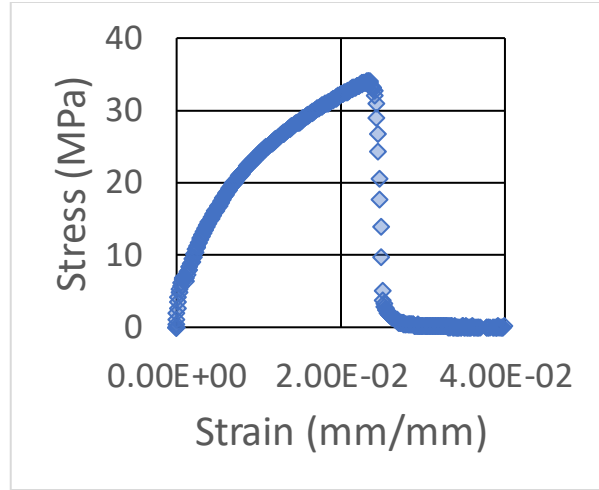


(d)

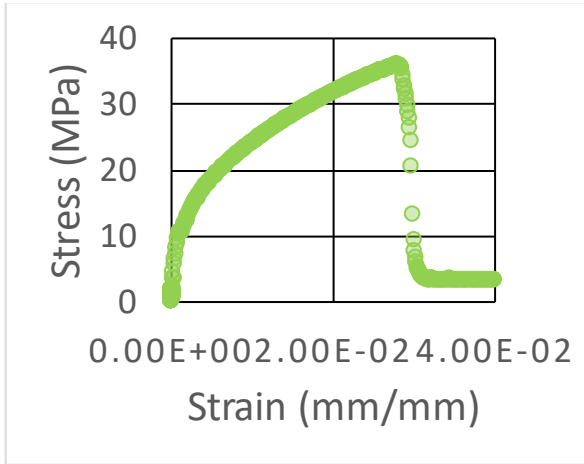
Figure 3.11 (a) Stress vs Strain curve of 133% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 133% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 133% flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 133% flow rate samples in Newtons; the cross-sectional area of the samples were 25 mm^2 .



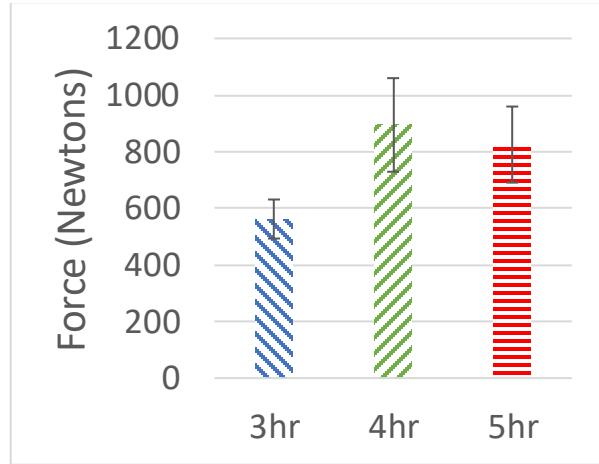
(a)



(b)

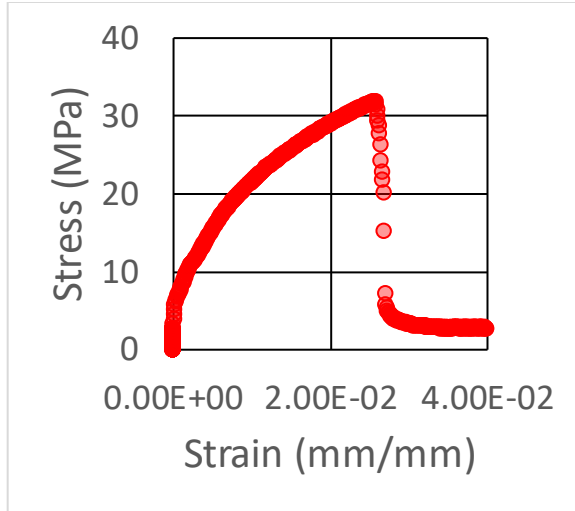


(c)

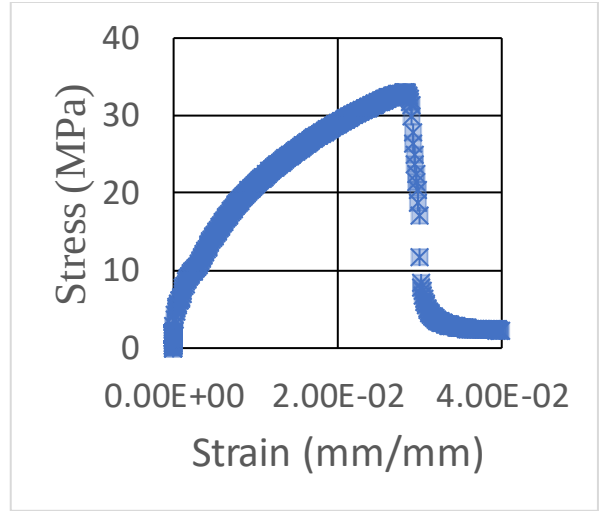


(d)

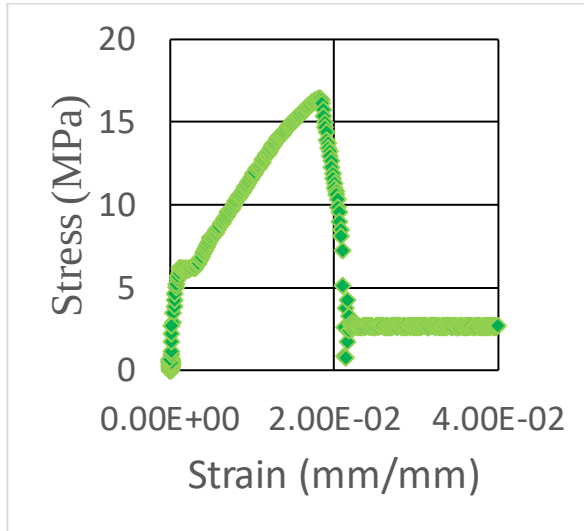
Figure 3.12 (a) Stress vs Strain curve of 135% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 135% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 135% flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 135% flow rate samples in Newtons; the cross-sectional area of the samples were 25 mm^2 .



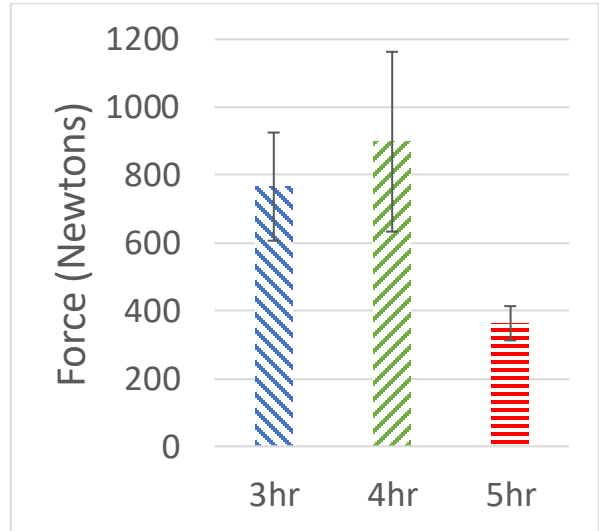
(a)



(b)



(c)



(d)

Figure 3.13 (a) Stress vs Strain curve of 140% flow rate 3 hour debind 3 hour sinter sample; (b) Stress vs Strain curve of 140% flow rate 3 hour debind 4 hour sinter sample; (c) Stress vs Strain curve of 140% flow rate 3 hour debind 5 hour sinter sample; (d) Average maximum force applied to the 140% flow rate samples in Newtons; the cross-sectional area of the samples were 25 mm^2 .

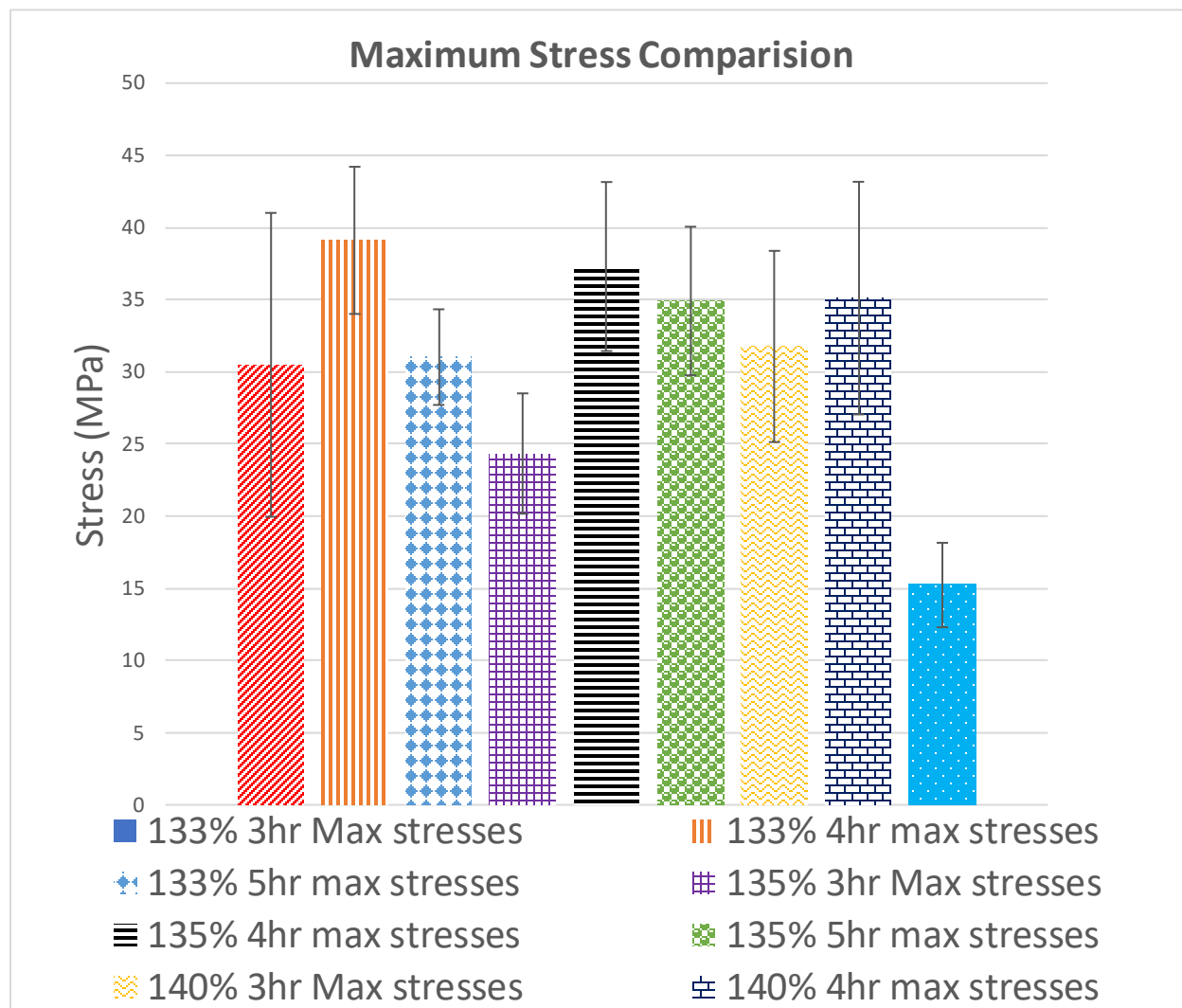


Figure 3.14 Maximum Stress comparison against flow rate and sintering time

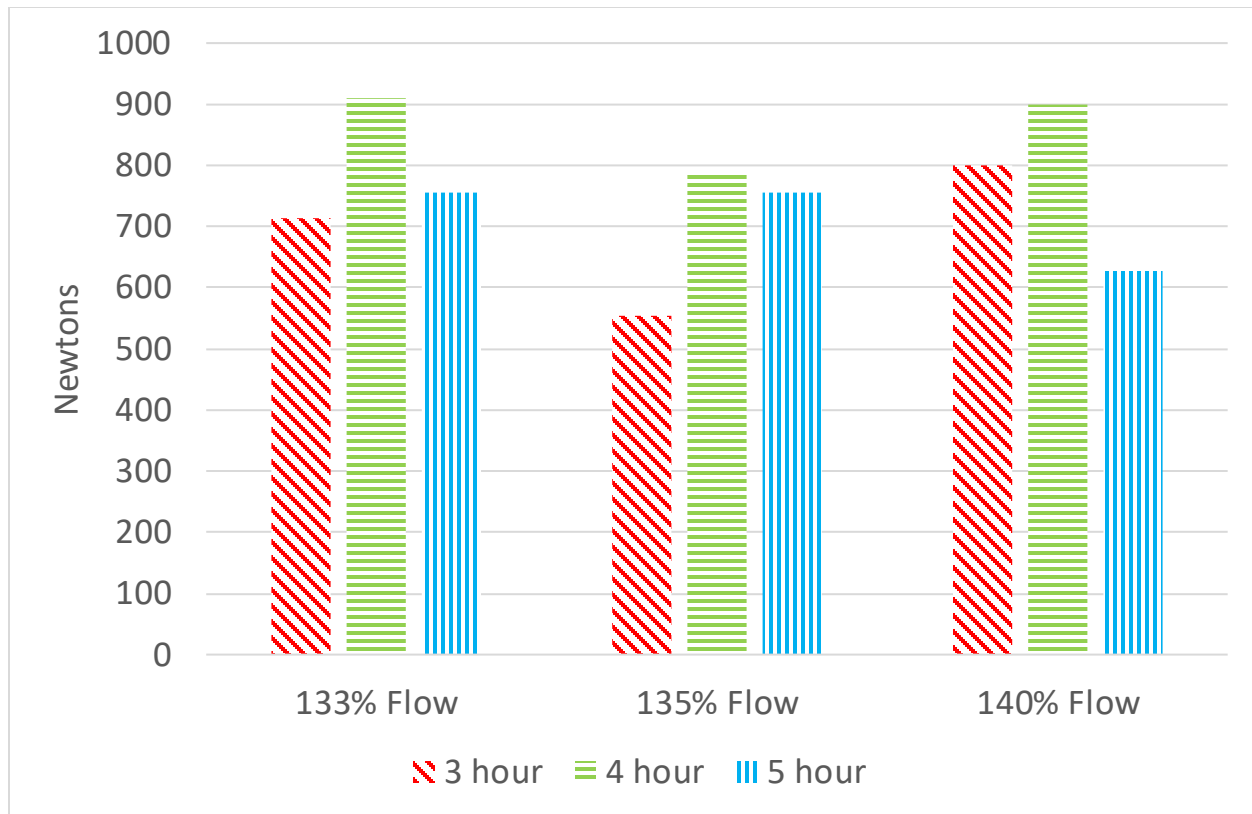


Figure 3.15 Average maximum force per flow rate and sintering time

While the strength was more consistent, it was not comparable to standard annealed copper which is shown below in **Figure 3.16**.

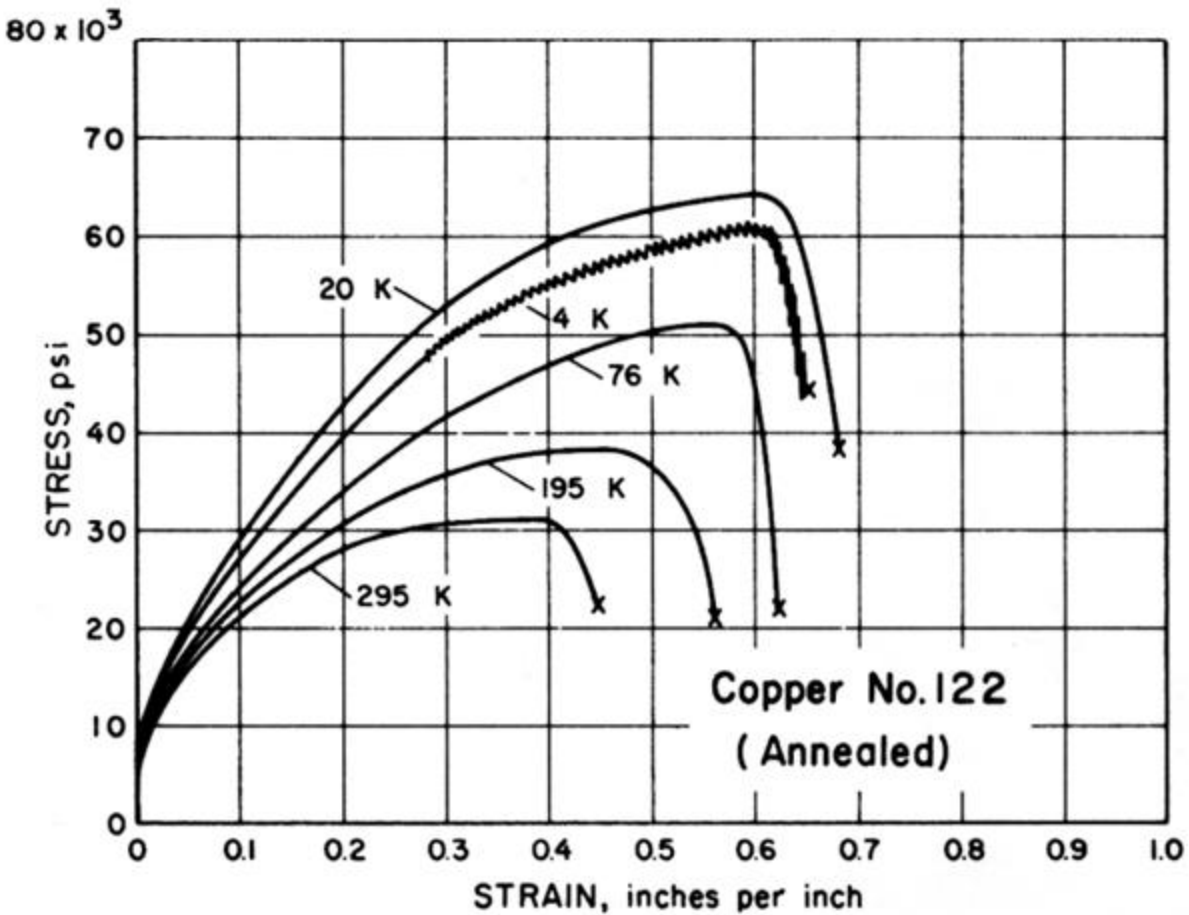


Figure 3.16 Annealed Phosphorus Deoxidized Copper Stress vs Strain in [32]

The stress shown in **Figure 3.16** for the annealed copper at 295 K or 21.85 degrees Celsius maxes out at approximately 3200 psi or 220.6 MPa. This is much greater than any of the results from the study conducted. From the results shown in the figures above, it is clear that the 133% flow rate with a 3-hour debind and a 4-hour sintering procedure had higher average results in the stress vs. strain test. Its average stress was 39.107 MPa. The next highest maximum stress value is with the 135% 3-hour debind 4-hour sinter sample set with 37.2929 MPa. Surprisingly, increasing the sintering time to 5 hours decreased the maximum stress to 31.017 MPa with a

133% flow rate and 34.905 MPa with a 135% flow rate. The 133% 3-hour debind 3-hour sinter only had a stress value of 30.488 MPa. The next best for the 135% flow rate group was the 3-hour debind 5-hour sinter with an average max stress of 34.905 MPa. Finally, the lowest average stress is the 3-hour debind 3-hour sinter group with 24.339 MPa. For the 140% flow rate group, the 3 hours debind 3-hour sinter and the 3-hour debind 4-hour sinter are very similar. The 3 hours debind 3-hour sinter group came in at 31.756 MPa, and the 3-hour debind 4-hour sinter measured 35.107 MPa. The 140% 3-hour debind 5-hour sinter group performed terribly. It only had a stress value of 15.223 MPa. These values can be observed in Figure 3.9 above. In all cases except for one, the 5-hour sinter did not help the samples at all and actually reduced their strength. This loss in strength is possibly from an increase in missing material or void within the parts. This increase in void is later confirmed in the following section where the samples are cross-sectioned. It is also hypothesized that if the debinding is not completed and the PLA is burnt off, then it will do so during the sintering process causing the copper particles to not bond with one another. When conducting a Student T-Test, it is shown that the comparison between the flow rate for the 133% flow rate is insignificant, but the 3-hour to the 4-hour comparison of the 135% flow rate shows significance. Statistical significance is also shown in the 140% 3 hours to 5 hours and the 4-hour to 5-hour comparison. In comparing the flow rates and sintering times, the only two groups that show any statistical significance is the 133% flow rate to 140% flow rate 5-hour sintering time and the 135% flow rate to 140% flow rate 5-hour sintering time. Otherwise, there is no statistical significance between any group.

3.3.5 Internal voids and porosity characteristics:

In a cross-sectional analysis, the copper has multiple voids within the samples. These voids cause problems with the strength of the samples, which can explain some of the stories of what happened. ImageJ is utilized in the analysis of how much of a void exists. The images are below.

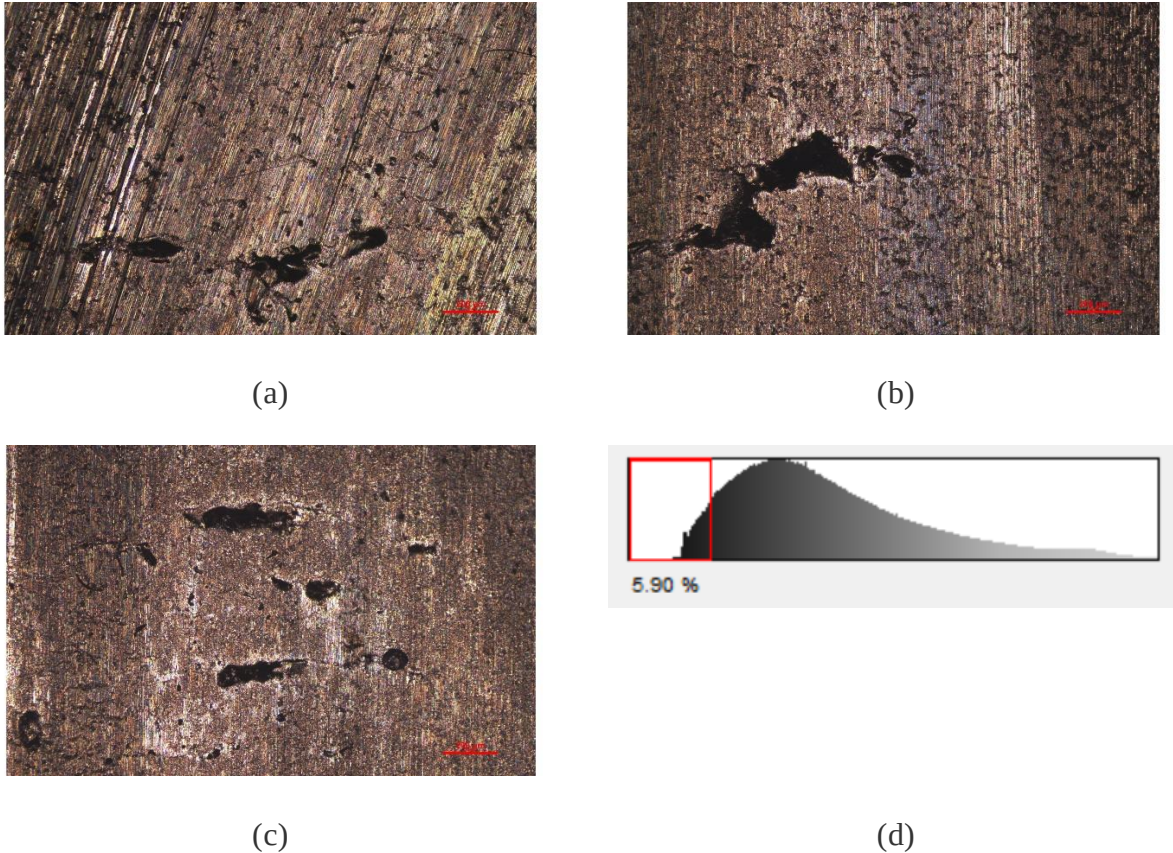


Figure 3.17 (a) Sample 1 133% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 133% 3 hour debind 3 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 3 hour sinter

The 133% 3-hour debind 3-hour sinter has an average of 3.74% void percentage. The 133% 3-hour debind 4-hour sinter has an average of 3.23% void percentage. The 133% 3-hour debind 5-hour sinter has an average of 1.79% void percentage. The 135% 3-hour debind 3-hour sinter has

an average of 3.73% void percentage. The 135% 3-hour debind 4-hour sinter has an average of 1.43% void percentage. The 135% 3-hour debind 5-hour sinter has an average of 9.46% void percentage. The 140% 3-hour debind 3-hour sinter has an average of 2.11% void percentage. The 140% 3-hour debind 4-hour sinter has an average of 2.85% void percentage. Lastly, the 140% 3-hour debind 5-hour sinter has an average 11.54% void percentage. This makes the highest flow rate and sintering time the worst for having an internal void; as the sintering time increases consistently, the internal void percentage increases. It appears that the middle ground for our samples at 4 hours of sintering time has the lowest void percentage, with the only exception being the 140% flow rate samples. The large void found within the samples is thought to come from a combination of a couple theories. The debinding process of the samples could have not been completed before starting the sintering process. With this happening the PLA is not slowly evacuated from the parts causing large voids as it is boiled off at a very rapid rate. When it is boiled off at such a rapid rate, the copper particles are not as dense as they previously were making it difficult for them to stick to one another and combine back into one piece. This also could explain why the tensile strength was reduced as the sintering time increased and was reduced as well with the increase of material with the increase of flow rate.

Chapter 4

Conclusion and Future Works

4.1 General Conclusion and Discussion:

Additive manufacturing and 3D printing technology allow for the construction of complex geometries; thus, parts that were not able to be constructed by using conventional machining and material subtraction-based manufacturing methods are now possible. 3D printing allows for the optimization of designs for structural integrity and other parameters within the industries such as aerospace and automotive [34]. Traditional methods of manufacturing limit what is possible; therefore, there is a need for an out-of-the-box type of manufacturing. For example, through traditional CNC manufacturing, the size of the tool head limits what can be done when working with resources. Along with this, there are high costs, risks, and unforeseen requirements when it comes to traditional metal additive manufacturing. Examples of this are the metal powders used in the process are flammable and explosive, requiring proper disposal and needing to place parts in a kiln to reduce residual stress [35, 36]. This is where FDM metal additive manufacturing is helpful. It has the potential to fill the gap that is left by other methods of manufacturing metal parts.

During the testing process, three flow rates were tested alongside three sintering times, totaling nine different testing groups. The flow percentages utilized were 133%, 135%, and 140%, and the sintering times utilized were 3 hours, 4 hours, and 5 hours. While randomly testing these samples, one sample was removed from each of the nine groups and polished using a Buehler planetary grinding machine. After these steps were completed, the part was etched by submersion using ferric chloride for five minutes, rinsed off with water to stop the etching process, and then examined using a metallurgy microscope. The grain boundary was observed

and captured. The samples are then pulled apart using the Universal Testing Machine, the tensile strength is recorded, and stress versus strain is calculated from this value. Using a Buehler low-speed reciprocating saw, the samples are cross-sectioned and imaged utilizing the metallurgy microscope again. ImageJ uses the default threshold method to calculate the void within the sample.

After analyzing the results, the following conclusions are made:

From the samples, the following conclusions can be made:

- a. Conventional FDM 3D printer can successfully print copper samples using hybrid Cu-PLA filament with 80%-90% copper content
- b. Successfully fabricated ASTM standard dog-bone copper samples using FDM 3D printer with 133%, 135% and 140% flow rate.
- c. Successfully debound and sintered 3D printed copper samples of 133%. 135% and 140% rates with 3 hrs sintering time.
- d. Successfully debound and sintered 3D printed copper samples of 133%. 135% and 140% rates with 4 hrs sintering time.
- e. Successfully debound and sintered 3D printed copper samples of 133%. 135% and 140% rates with 5 hrs sintering time.
- f. Dimensional loss of the samples depends on the orientation of samples in the crucible during the debinding and sintering process. Dimensional losses are not uniform in each direction (x,y,z) of the samples. The maximum losses happen in vertical orientational direction as the material can flow due to the gravity.
- g. With the samples placed vertically in the crucible, it is surprising that the percent of shrinkage in the X position (or length) is between 2% and 6%. Y1 shrinks between 7% and 9%

while Y2 shrinks approximately the same amount. The Z axis has the broadest range observed out of any of the other axes, of 4% and 9%.

h. The weight loss of the sample resembles the percentage content of the Cu in the Cu-PLA filament. 3D printing flow rate does not have a significant impact on weight loss. This was observed with the most mass loss being approximately 9.8% with the 135% flow rate group. The least mass lost is approximately 9.58% with the 140% flow rate group. This data is within the margin of error and could stem from a non-homogeneous mixture between the copper and the PLA within the filament.

i. Regular conventional polishing method was successfully applied for 3D printed Cu samples.

j. No significant variation of the grain structure of the 3D printed Cu samples were observed. However, the sintering time and flow rate have significant influence in making perfect grain structure. The grain boundaries are very similar between every group and align with the annealed copper.

k. More void areas are found at the surface of the samples compared to the inner cross sectional area of the samples. However, the void area depends on the flow rate and sintering time. The 140% flow rate 3 hour debind and 3 hour sinter had the lowest void percentage on the surface of the sample at 28.56%. The 140% flow rate 3 hour debind 5 hour sinter had the highest surface void percentage at 63.41%.

l. The inner core of the sample has less voids and is more solid. The 135% flow rate 3 hour debind and 4 hour sinter sample had the lowest internal void calculation. The 140% 3 hour debind 5 hour sinter had the highest internal void percentage at 11.54%.

m. In the tensile testing the samples underperformed when matched against traditional annealed copper. The maximum stress experienced is from the 133% flow rate 3 hour debind 4 hour sinter group with 39.107 MPa. The lowest stress observed occurred with the 140% flow rate 3 hour debind 5 hour sinter at 15.223 MPa.

4.2 Future Works:

While many things about this method of metal additive manufacturing are unknown, that just shows the importance of further research. For example, the printing parameters and 3D printer nozzle size could influence the strength and porosity. Also, changing the debinding and sintering times outside of what was tested could become useful. Experimenting until the ideal debinding and sintering time for an internal area would be extremely useful for larger parts and ensuring they have been debound and sintered properly. Conducting a 3-point bending test, and torsion test, as well as testing samples with different printing orientations, might lead to other experimental findings. New discoveries can be made when conducting more tests, allowing for a deeper scientific understanding of this type of technology.

4.3 Concluding Thoughts:

3D metal printing using FDM 3D printer is recently developed with a low upfront cost and a substantial cost reduction over time. This development is groundbreaking, but it may have some hidden downfalls. These potential downfalls revealed in this study include a reduction in rigidity, tensile strength, and much more when compared to a billet or known solid part. While the quality and material grain boundaries are comparable with cold-rolled annealed samples, the mechanical characteristics are far off. Nonetheless, the overall aspect of making copper parts from conventional FDM-based 3D printing technology is promising. The findings of this research will

contribute to understanding and better utilization of FDM-based 3D printed Cu and general metal parts for future engineering endeavors.

.

References:

- [1] P. D. Murugan *et al.*, "A current state of metal additive manufacturing methods: A review," *Materials Today: Proceedings*, 2021.
- [2] W. Gao *et al.*, "The status, challenges, and future of additive manufacturing in engineering," *Computer-Aided Design*, vol. 69, pp. 65-89, 2015.
- [3] N. Shahrubudin, T. C. Lee, and R. Ramlan, "An overview on 3D printing technology: Technological, materials, and applications," *Procedia Manufacturing*, vol. 35, pp. 1286-1296, 2019.
- [4] A. Su and S. J. Al'Aref, "History of 3D printing," in *3D Printing applications in cardiovascular medicine*: Elsevier, 2018, pp. 1-10.
- [5] V. G. Gokhare, D. Raut, and D. Shinde, "A review paper on 3D-printing aspects and various processes used in the 3D-printing," *Int. J. Eng. Res. Technol*, vol. 6, no. 06, pp. 953-958, 2017.
- [6] M. Khaing, J. Fuh, and L. Lu, "Direct metal laser sintering for rapid tooling: processing and characterisation of EOS parts," *Journal of Materials Processing Technology*, vol. 113, no. 1-3, pp. 269-272, 2001.
- [7] M. Kamran and A. Saxena, "A comprehensive study on 3D printing technology," *MIT Int J Mech Eng*, vol. 6, no. 2, pp. 63-69, 2016.
- [8] D. Svetlizky *et al.*, "Directed energy deposition (DED) additive manufacturing: Physical characteristics, defects, challenges and applications," *Materials Today*, vol. 49, pp. 271-295, 2021.
- [9] P. Dudek, "FDM 3D printing technology in manufacturing composite elements," *Archives of metallurgy and materials*, vol. 58, no. 4, pp. 1415--1418, 2013.
- [10] I. Gibson, D. W. Rosen, and B. Stucker, "Sheet lamination processes," in *Additive manufacturing technologies*: Springer, 2010, pp. 223-252.
- [11] A. Pîrjan and D.-M. Petroşanu, "The impact of 3D printing technology on the society and economy," *Journal of Information Systems and Operations Management*, vol. 7, no. 2, pp. 360-370, 2013.
- [12] M. Jiménez, L. Romero, I. A. Domínguez, M. d. M. Espinosa, and M. Domínguez, "Additive manufacturing technologies: an overview about 3D printing methods and future prospects," *Complexity*, vol. 2019, 2019.
- [13] W. J. Sames, F. List, S. Pannala, R. R. Dehoff, and S. S. Babu, "The metallurgy and processing science of metal additive manufacturing," *International materials reviews*, vol. 61, no. 5, pp. 315-360, 2016.
- [14] M. Vaezi, P. Drescher, and H. Seitz, "Beamless metal additive manufacturing," *Materials*, vol. 13, no. 4, p. 922, 2020.
- [15] H. Lu, "Preliminary Mechanical Characterization of the Low-Cost Metal 3D Printing," Tennessee Technological University, 2020.
- [16] C.-E. Pelin, G. M. Stoican, A. Stefan, M. V. Pricop, S. Ilina, and G. Pelin, "Mechanical properties of 3D printed metals," *INCAS Bulletin*, vol. 13, no. 1, pp. 123-129, 2021.
- [17] S. Terry, I. Fidan, and K. Tantawi, "Dimensional analysis of metal powder infused filament-low cost metal 3D printing," in *2019 International Solid Freeform Fabrication Symposium*, 2019: University of Texas at Austin.
- [18] J. R. Davis, *Copper and copper alloys*. ASM international, 2001.
- [19] The Virtual Foundry, "SAFETY DATA SHEET Copper Filamet™," ed. Online, 2021.
- [20] ImageJ Wiki. "Auto Threshold." <https://imagej.net/plugins/auto-threshold> (accessed December 4, 2022).

- [21] T. Ridler and S. Calvard, "Picture thresholding using an iterative selection method," *IEEE trans syst Man Cybern*, vol. 8, no. 8, pp. 630-632, 1978.
- [22] J. M. Prewitt and M. L. Mendelsohn, "The analysis of cell images," *Annals of the New York Academy of Sciences*, vol. 128, no. 3, pp. 1035-1053, 1966.
- [23] L.-K. Huang and M.-J. J. Wang, "Image thresholding by minimizing the measures of fuzziness," *Pattern recognition*, vol. 28, no. 1, pp. 41-51, 1995.
- [24] C. H. Li and C. Lee, "Minimum cross entropy thresholding," *Pattern recognition*, vol. 26, no. 4, pp. 617-625, 1993.
- [25] J. Kittler and J. Illingworth, "Minimum error thresholding," *Pattern recognition*, vol. 19, no. 1, pp. 41-47, 1986.
- [26] N. Otsu, "A threshold selection method from gray-level histograms," *IEEE transactions on systems, man, and cybernetics*, vol. 9, no. 1, pp. 62-66, 1979.
- [27] W. Doyle, "Operations useful for similarity-invariant pattern recognition," *Journal of the ACM (JACM)*, vol. 9, no. 2, pp. 259-267, 1962.
- [28] J. N. Kapur, P. K. Sahoo, and A. K. Wong, "A new method for gray-level picture thresholding using the entropy of the histogram," *Computer vision, graphics, and image processing*, vol. 29, no. 3, pp. 273-285, 1985.
- [29] A. G. Shanbhag, "Utilization of information measure as a means of image thresholding," *CVGIP: Graphical Models and Image Processing*, vol. 56, no. 5, pp. 414-419, 1994.
- [30] G. W. Zack, W. E. Rogers, and S. A. Latt, "Automatic measurement of sister chromatid exchange frequency," *Journal of Histochemistry & Cytochemistry*, vol. 25, no. 7, pp. 741-753, 1977.
- [31] M. Sezgin and B. Sankur, "Survey over image thresholding techniques and quantitative performance evaluation," *Journal of Electronic imaging*, vol. 13, no. 1, pp. 146-165, 2004.
- [32] C. D. A. Inc. "Standards & Properties: Mechanical Properties of Copper and Copper Alloys at Low Temperatures." https://www.copper.org/resources/properties/144_8/ (accessed December 4, 2022).
- [33] J. Yang and W. Kim, "Examination of high-temperature mechanisms and behavior under compression and processing maps of pure copper," *Journal of Materials Research and Technology*, vol. 9, no. 1, pp. 960-968, 2020.
- [34] S. Li, S. Yuan, J. Zhu, C. Wang, J. Li, and W. Zhang, "Additive manufacturing-driven design optimization: Building direction and structural topology," *Additive Manufacturing*, vol. 36, p. 101406, 2020.
- [35] S. P. Moylan, J. A. Slotwinski, A. Cooke, K. Jurrens, and M. A. Donmez, "Lessons learned in establishing the NIST metal additive manufacturing laboratory," *NIST technical note*, 2013.
- [36] C. Li, Z. Liu, X. Fang, and Y. Guo, "Residual stress in metal additive manufacturing," *Procedia Cirp*, vol. 71, pp. 348-353, 2018.

Appendix A: Tables and Relevant Information

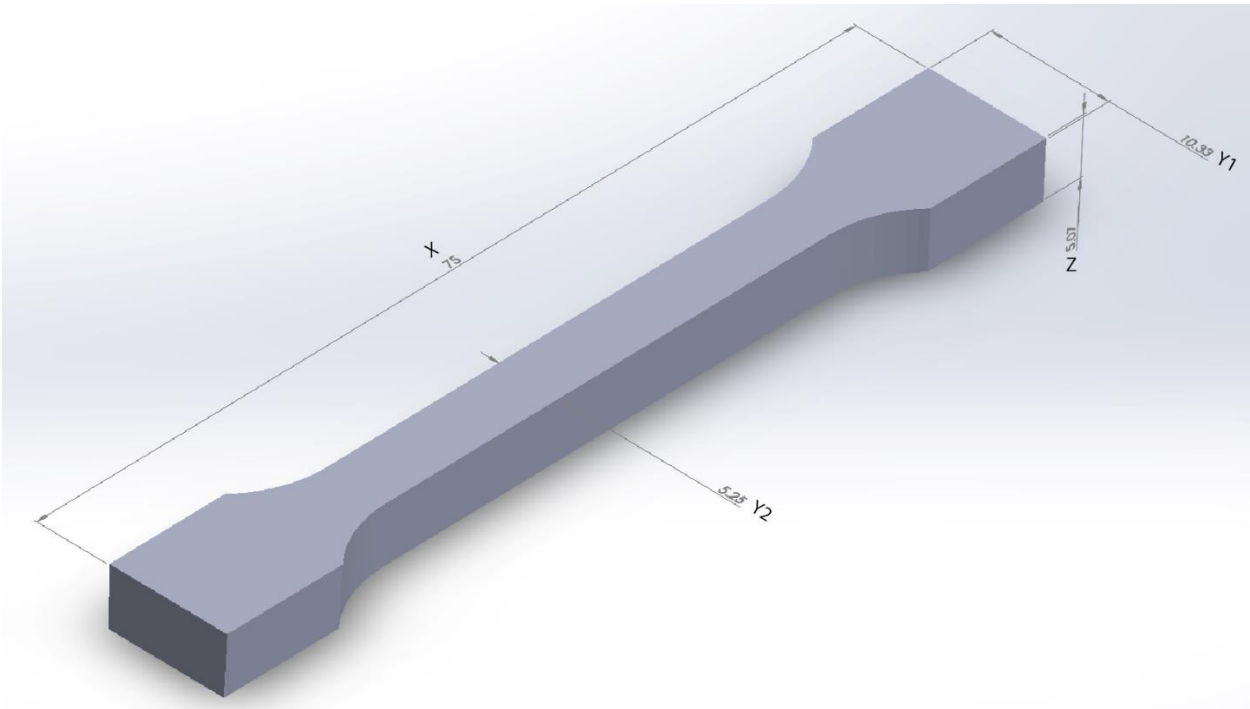


Figure A1

Sample #	x (mm)	y1 (mm)	y2 (mm)	z (mm)
1	75.34	10.64	5.5	5.23
2	75.37	10.65	5.57	5.22
3	75.32	10.66	5.5	5.07
4	75.38	10.74	5.59	5.25
5	75.29	10.61	5.54	5.13
AVERAGE	75.34	10.66	5.54	5.18
Post Sintering				
Sample #	x	y1	y2	z
1	71.82	9.85	5.31	5.25

2	71.96	9.86	5.09	5.1
3	70.58	9.72	5.01	4.83
4	71.26	10	5.15	4.76
5	71.18	9.72	5.02	4.64
AVERAGE	71.36	9.83	5.116	4.916
DIFFERENCE	3.98	0.83	0.424	0.264
PERCENT	5.282718344	7.786116323	7.653429603	5.096525097
SHRINK				

Table A1 133% Flow Rate 3hr Debind 3hr Sinter

133% Flow Rate 3hr Debind 4hr Sinter

Sample #	X (mm)	y1 (mm)	y2 (mm)	z (mm)
1	75.22	10.58	5.67	5.16
2	75.27	10.6	5.68	5.32
3	75.4	10.64	5.57	5.3
4	75.3	10.68	5.56	5.11
5	75.24	10.68	5.57	5.25
AVERAGE	75.286	10.636	5.61	5.228
Post Sintering				
Sample #	x	y1	y2	z
1	71.6	9.74	4.9	4.69
2	72.25	9.87	5.05	4.84
3	70.71	9.33	4.88	5.15
4	71.75	9.68	4.98	5.22

5	72.47	9.87	5.13	4.9
AVERAGE	71.756	9.698	4.988	4.96
DIFFERENCE	3.53	0.938	0.622	0.268
PERCENT	4.68878676	8.819104927	11.08734403	5.126243305
SHRINK				

Table A2 133% Flow Rate 3hr Debind 4hr Sinter

133% Flow Rate 3hr Debind 5hr Sinter

Sample #	X (mm)	y1 (mm)	y2 (mm)	z (mm)
1	75.13	10.54	5.63	5.23
2	75.24	10.61	5.78	5.36
3	75.2	10.56	5.58	5.25
4	75.16	10.62	5.65	5.37
5	75.17	10.64	5.58	5.19
AVERAGE	75.18	10.594	5.644	5.28
Post Sintering				
Sample #	x	y1	y2	z
1	72	9.75	5.07	4.8
2	71.65	10.01	5.17	4.82
3	72.17	9.63	5.01	5.04
4	72.52	9.62	5.1	4.93
5	72.69	9.76	5.01	4.99
AVERAGE	72.206	9.754	5.072	4.916

<i>DIFFERENCE</i>	2.974	0.84	0.572	0.364
<i>PERCENT</i>	3.955839319	7.929016424	10.13465627	6.893939394
<i>SHRINK</i>				

Table A3 133% Flow Rate 3hr Debind 5hr Sinter

140% Flow Rate 3hr Debind 3hr Sinter

<i>Sample #</i>	X (mm)	y1 (mm)	y2 (mm)	z (mm)
1	75.5	10.86	5.76	5.26
2	75.48	10.85	5.73	5.55
3	75.59	10.85	5.74	5.22
4	75.47	10.79	5.7	5.27
5	75.49	10.74	5.72	5.32
<i>AVERAGE</i>	75.506	10.818	5.73	5.324
<i>Post Sintering</i>				
<i>Sample #</i>	x	y1	y2	z
1	72.97	9.98	5.32	4.92
2	72.49	9.74	5.09	4.87
3	73.19	9.99	5.28	4.87
4	73.22	10.03	5.22	5.03
5	72.44	10.02	5.07	4.61
<i>AVERAGE</i>	72.862	9.952	5.196	4.86
<i>DIFFERENCE</i>	2.644	0.866	0.534	0.464

<i>PERCENT</i>	3.501708473	8.005176558	9.319371728	8.71525169
<i>SHRINK</i>				

Table A4 140% Flow Rate 3hr Debind 3hr Sinter

140% Flow Rate 3hr Debind 4hr Sinter

<i>Sample #</i>	X (mm)	y1 (mm)	y2 (mm)	z (mm)
1	75.55	10.75	5.73	5.59
2	75.27	10.74	5.67	5.17
3	75.45	10.81	5.71	5.53
4	75.55	10.72	5.72	5.56
5	75.57	10.78	5.72	5.23
<i>AVERAGE</i>	75.478	10.76	5.71	5.416
<i>Post Sintering</i>				
<i>Sample #</i>	x	y1	y2	z
1	72.05	9.85	5.14	5.59
2	71.4	9.7	5.05	4.96
3	72.66	10.2	5.27	4.85
4	72.61	10.12	5.3	5.36
5				
<i>AVERAGE</i>	72.18	9.9675	5.19	5.19
<i>DIFFERENCE</i>	3.298	0.7925	0.52	0.226
<i>PERCENT</i>	4.369485148	7.365241636	9.106830123	4.17282127
<i>SHRINK</i>				

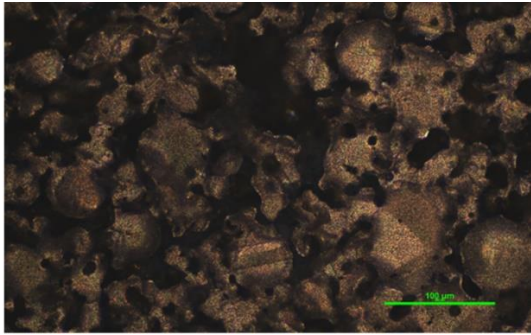
Table A5 140% Flow Rate 3hr Debind 4hr Sinter

140% Flow Rate 3hr Debind 5hr Sinter

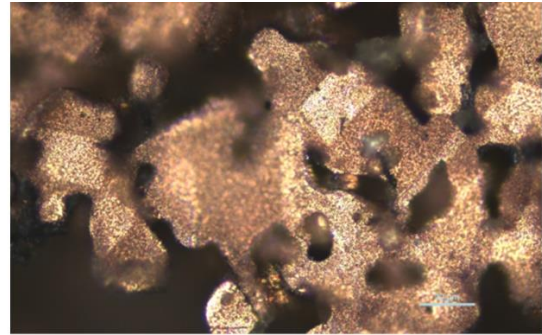
<i>Sample #</i>	<i>X (mm)</i>	<i>y1 (mm)</i>	<i>y2 (mm)</i>	<i>z (mm)</i>
<i>1</i>	74.91	10.28	5.3	4.66
<i>2</i>	74.89	10.31	5.32	4.64
<i>3</i>	74.91	10.31	5.45	4.66
<i>4</i>	74.92	10.33	5.17	4.66
<i>5</i>	74.9	10.3	5.3	4.64
<i>AVERAGE</i>	74.906	10.306	5.308	4.652
<i>Post Sintering</i>				
<i>Sample #</i>	<i>x</i>	<i>y1</i>	<i>y2</i>	<i>z</i>
<i>1</i>	73.39	9.51	5.11	4.41
<i>2</i>	73.07	9.73	5.02	4.53
<i>3</i>	72.61	9.03	5.01	4.34
<i>4</i>	72.25	9.54	4.77	4.32
<i>5</i>	72.65	9.49	4.66	4.36
<i>AVERAGE</i>	72.794	9.46	4.914	4.392
<i>DIFFERENCE</i>	2.112	0.846	0.394	0.26
<i>PERCENT</i>	2.819533816	8.208810402	7.422758101	5.588993981
<i>SHRINK</i>				

Table A6 140% Flow Rate 3hr Debind 5hr Sinter

Appendix B: 133% Flow Rate Images and Graphs



(a)

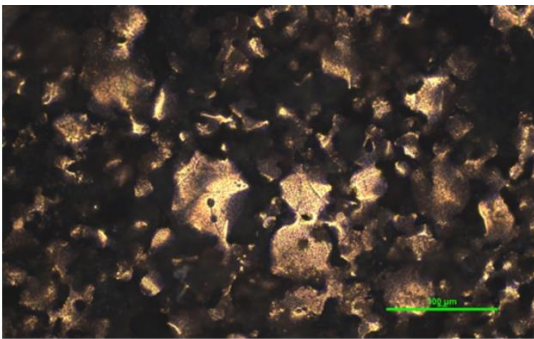


(b)

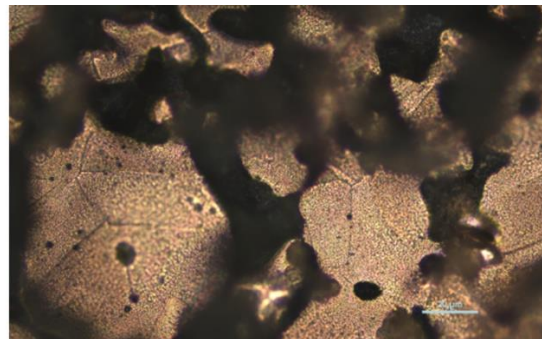


(c)

Figure B1 (a) 133% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 133% 3 hour debind 3 hour sinter 50x magnification; (c) 133% 3 hour debind 3 hour sinter 20x magnification void calculation



(a)

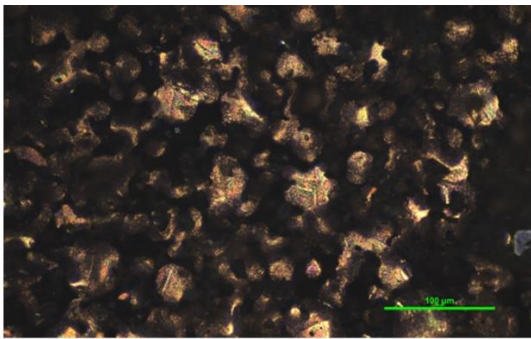


(b)

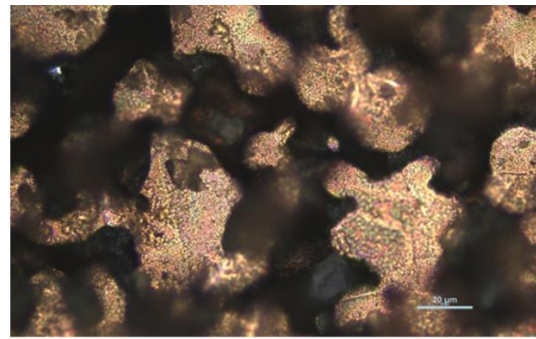


(c)

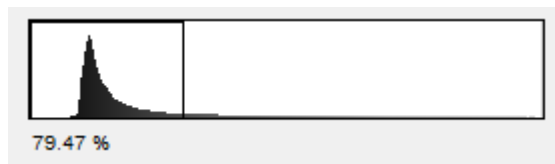
Figure B2 (a) 133% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 133% 3 hour debind 4 hour sinter 50x magnification; (c) 133% 3 hour debind 4 hour sinter 20x magnification void calculation



(a)

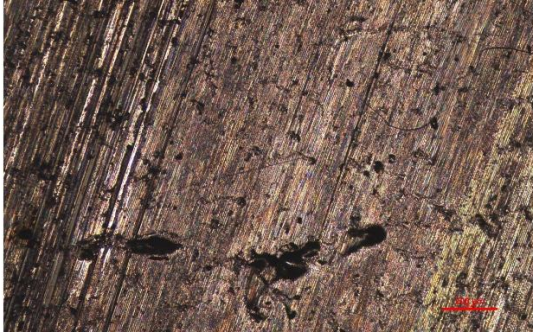


(b)

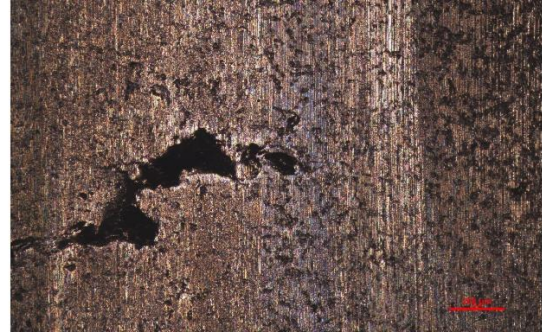


(c)

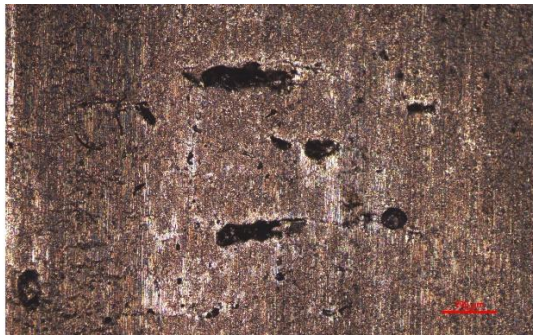
Figure B3 (a) 133% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 133% 3 hour debind 5 hour sinter 50x magnification; (c) 133% 3 hour debind 5 hour sinter 20x magnification void calculation



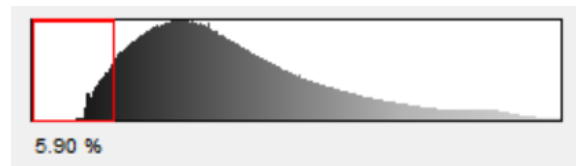
(a)



(b)

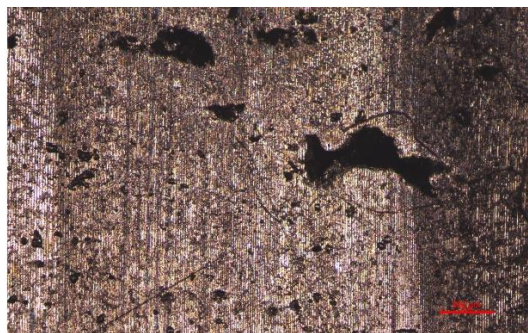


(c)

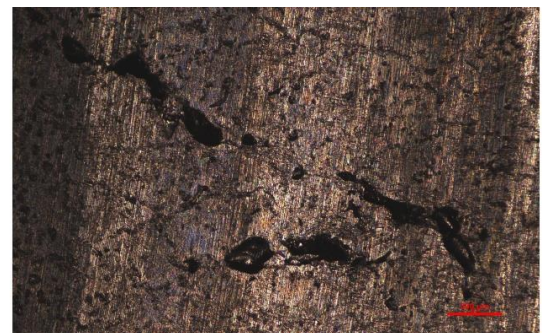


(d)

Figure B4 (a) Sample 1 133% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 133% 3 hour debind 3 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 3 hour sinter



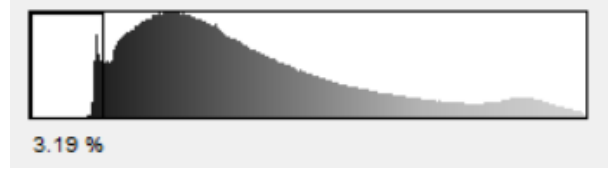
(a)



(b)

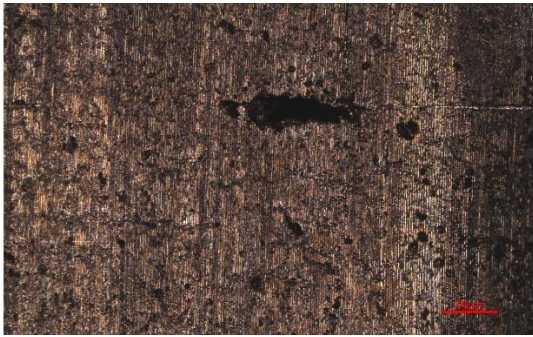


(c)

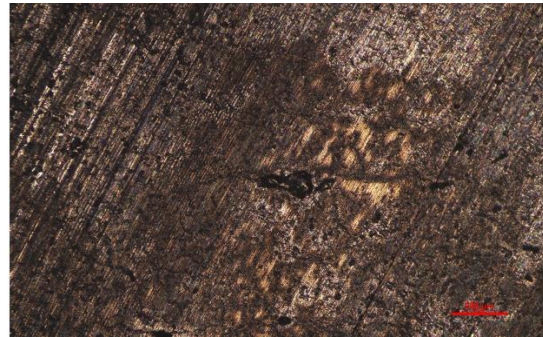


(d)

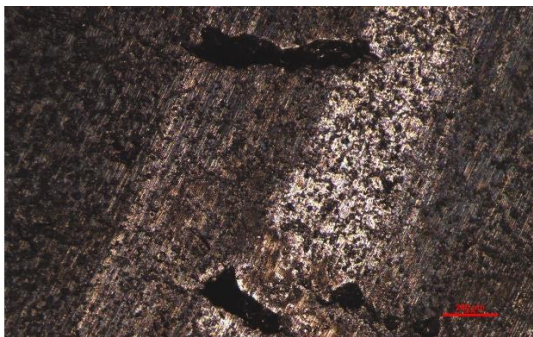
Figure B5 (a) Sample 1 133% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 133% 3 hour debind 4 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 4 hour sinter



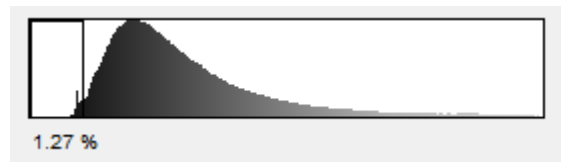
(a)



(b)



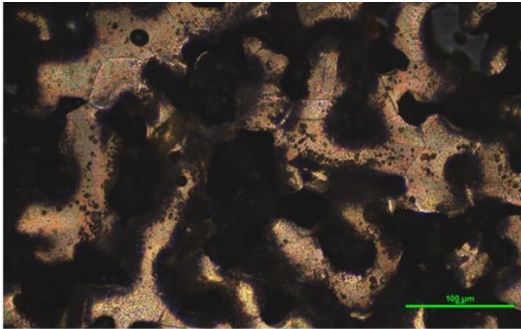
(c)



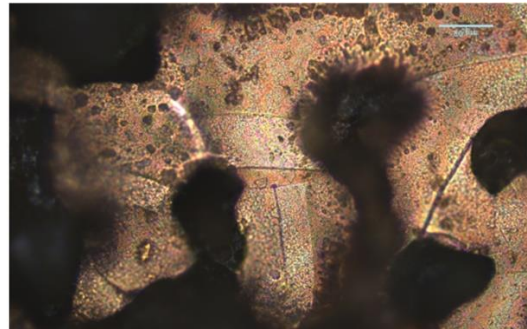
(d)

Figure B6 (a) Sample 1 133% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 133% 3 hour debind 5 hour sinter; (c) Sample 3 133% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 133% flow rate 3 hour debind 5 hour sinter

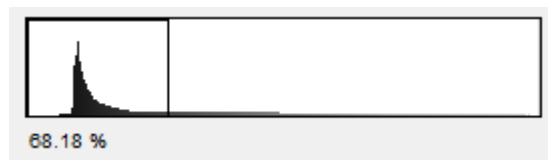
Appendix C: 135% Flow Rate Images and Graphs



(a)

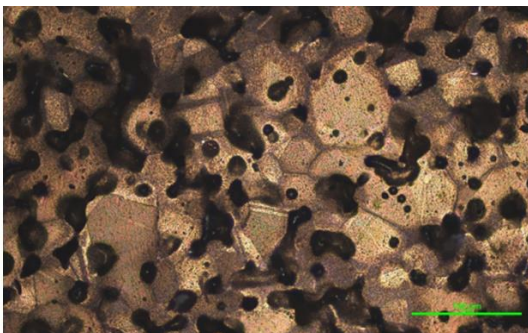


(b)

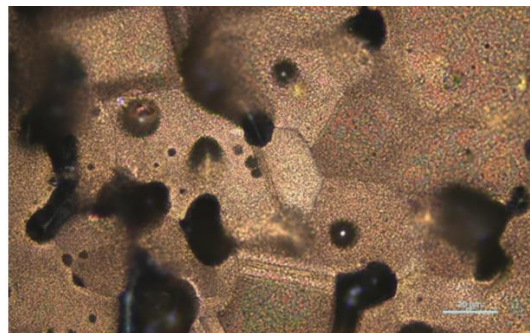


(c)

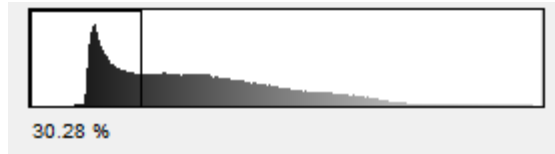
Figure C1 (a) 135% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 135% 3 hour debind 3 hour sinter 50x magnification; (c) 135% 3 hour debind 3 hour sinter 20x magnification void calculation



(a)

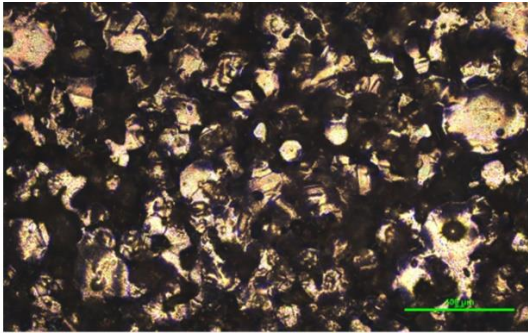


(b)

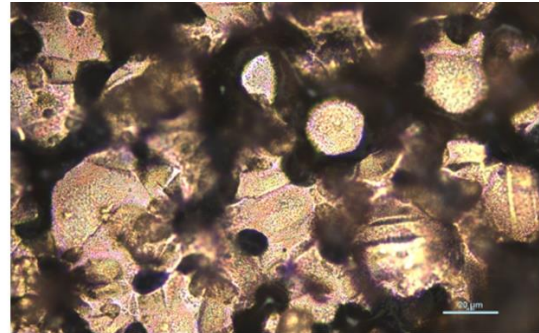


(c)

Figure C2 (a) 135% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 135% 3 hour debind 4 hour sinter 50x magnification; (c) 135% 3 hour debind 4 hour sinter 20x magnification void calculation



(a)

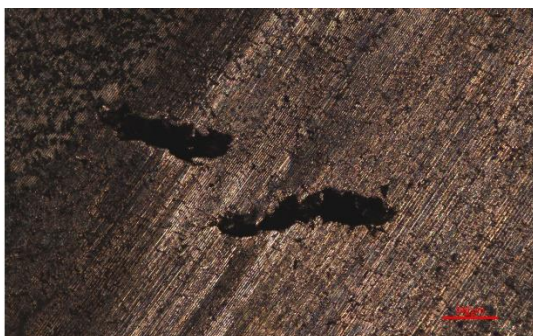


(b)

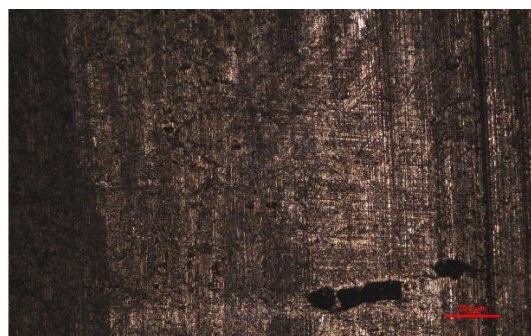


(c)

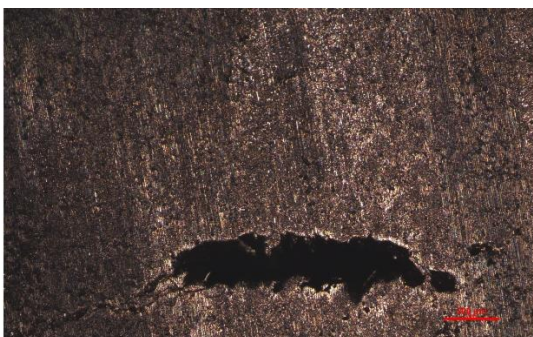
Figure C3 (a) 135% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 135% 3 hour debind 5 hour sinter 50x magnification; (c) 135% 3 hour debind 5 hour sinter 20x magnification void calculation



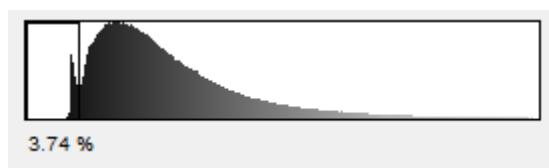
(a)



(b)



(c)



(d)

Figure C4 (a) Sample 1 135% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 135% 3 hour debind 3 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 3 hour sinter



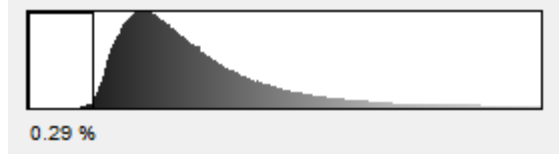
(a)



(b)



(c)

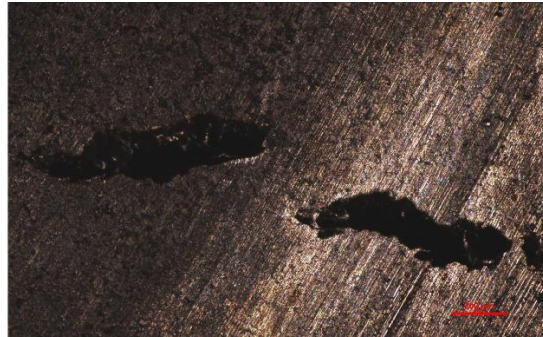


(d)

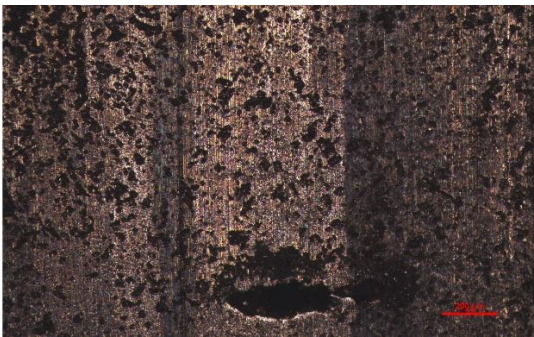
Figure C5 (a) Sample 1 135% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 135% 3 hour debind 4 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 4 hour sinter



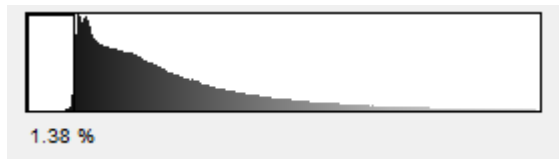
(a)



(b)



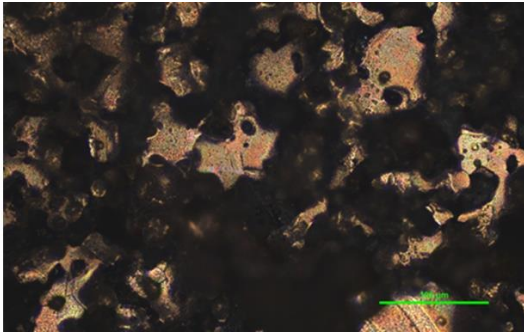
(c)



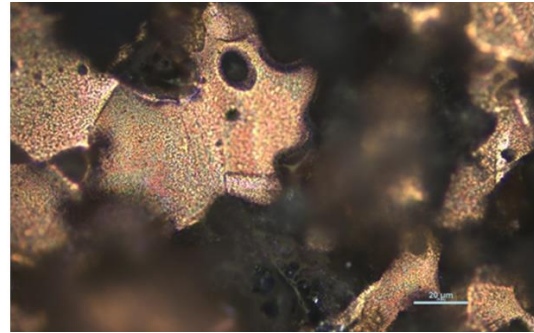
(d)

Figure C6 (a) Sample 1 135% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 135% 3 hour debind 5 hour sinter; (c) Sample 3 135% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 135% flow rate 3 hour debind 5 hour sinter

Appendix D: 140% Flow Rate Images and Graphs



(a)

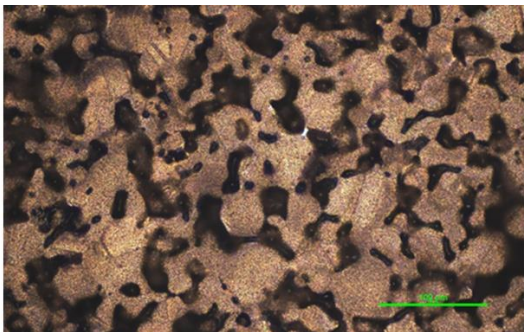


(b)

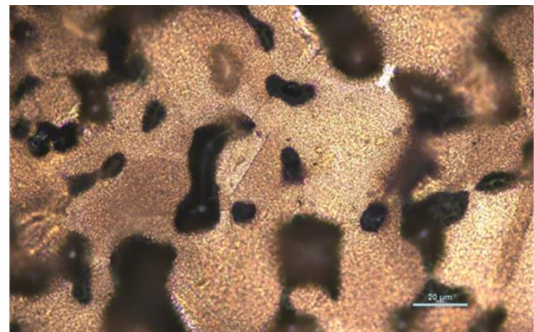


(c)

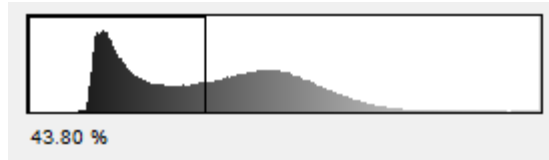
Figure D1 (a) 140% flow rate 3 hour debind 3 hour sinter 20x magnification; (b) 140% 3 hour debind 3 hour sinter 50x magnification; (c) 140% 3 hour debind 3 hour sinter 20x magnification void calculation



(a)

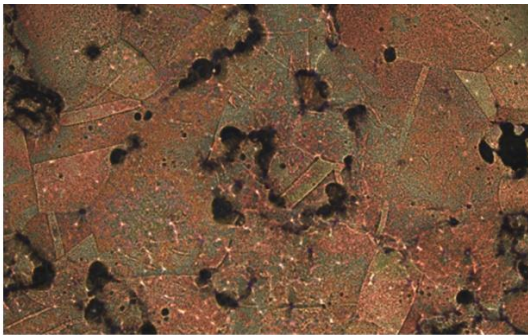


(b)

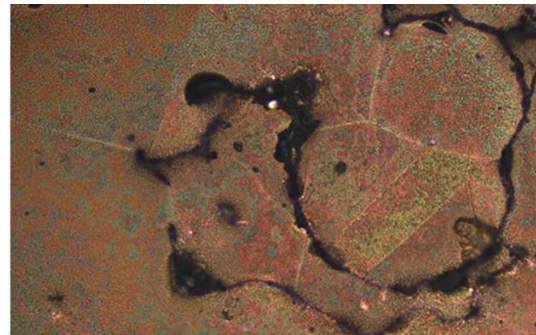


(c)

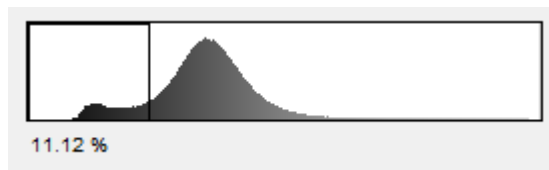
Figure D2 (a) 140% flow rate 3 hour debind 4 hour sinter 20x magnification; (b) 140% 3 hour debind 4 hour sinter 50x magnification; (c) 140% 3 hour debind 4 hour sinter 20x magnification void calculation



(a)



(b)

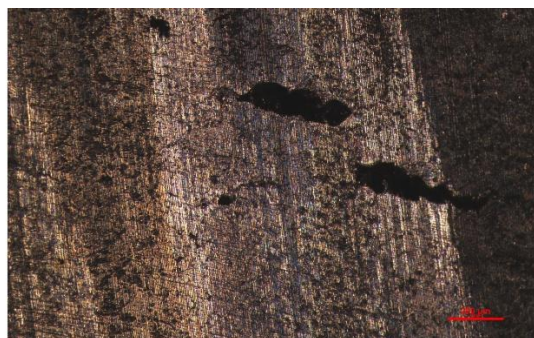


(c)

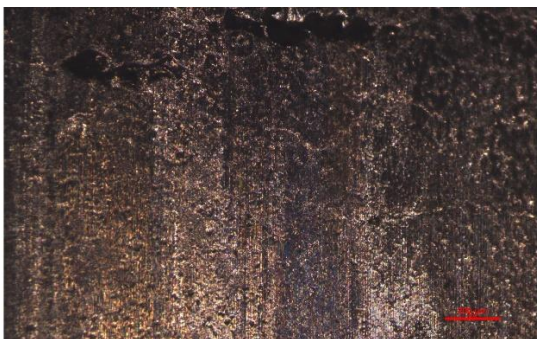
Figure D3 (a) 140% flow rate 3 hour debind 5 hour sinter 20x magnification; (b) 140% 3 hour debind 5 hour sinter 50x magnification; (c) 140% 3 hour debind 5 hour sinter 20x magnification void calculation



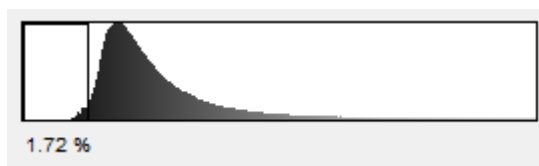
(a)



(b)



(c)

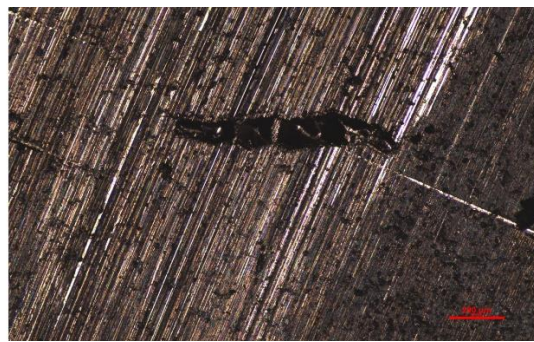


(d)

Figure D4 (a) Sample 1 140% flow rate 3 hour debind 3 hour sinter; (b) Sample 2 140% 3 hour debind 3 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 3 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 3 hour sinter



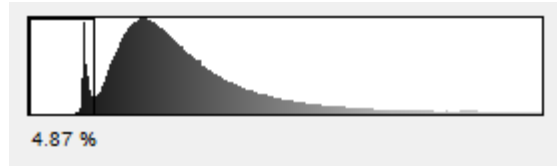
(a)



(b)

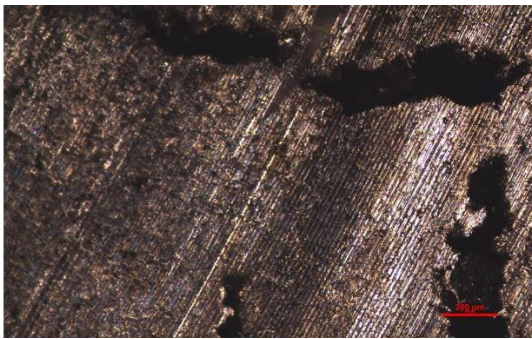


(c)

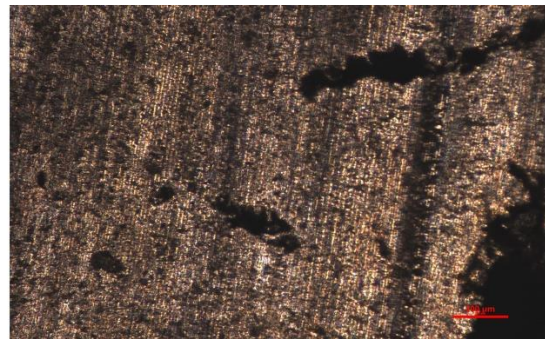


(d)

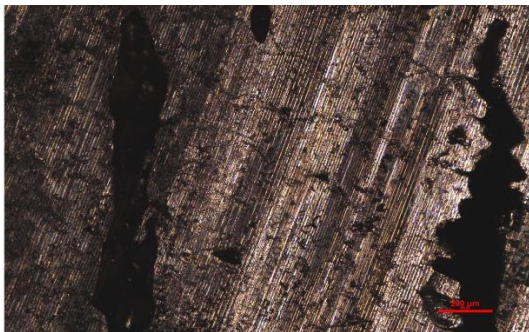
Figure D5 (a) Sample 1 140% flow rate 3 hour debind 4 hour sinter; (b) Sample 2 140% 3 hour debind 4 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 4 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 4 hour sinter



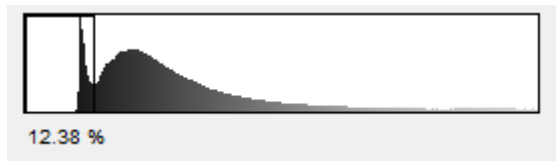
(a)



(b)



(c)



(d)

Figure D6 (a) Sample 1 140% flow rate 3 hour debind 5 hour sinter; (b) Sample 2 140% 3 hour debind 5 hour sinter; (c) Sample 3 140% flow rate 3 hour debind 5 hour sinter; (d) Void percentage for sample 1 140% flow rate 3 hour debind 5 hour sinter