Investigation of Additively Manufactured Molybdenum-tungsten-Rhenium Alloys

Randolph T. Abaya
INVESTIGATION OF ADDITIVELY MANUFACTURED MOLYBDENUM-TUNGSTEN-RHENIUM ALLOYS

THESIS

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THESIS

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Abstract

The process of creating metal components through additive manufacturing is changing the way different industries can avoid the shortcomings of traditional metal production. Metals such as tungsten, molybdenum, and rhenium have many advantages for different applications, especially when alloyed together. In this study, an additively manufactured alloy containing 70% molybdenum, 25% tungsten, and 5% rhenium (70Mo-25W-5Re) is tested for its strength, ductility, hardness, and porosity.

The 70Mo-25W-5Re alloy is printed through Laser Powder Bed Fusion (LPBF) under different conditions such as printing speed and printing atmosphere. Additionally, the effects of post printing heat treatment are conducted to understand the advantages to its property changes. The printed alloys are subject to flexural loading and its physical characteristics are tested and observed. The alloy is found to be stronger at slower printing speeds which corresponds to a greater input energy density. Additionally, heat treatments acted to improve strength but had little effect on porosity or hardness.

The benefits of the 70Mo-25W-5Re alloy have a potential for real world applications due to its ease in production. The findings of this research demonstrated how readily alloys of these elements can be studied by leveraging additive manufacturing and post processing heat treatments. This technique will encourage research into different combinations of the constituent elements to find promising compositions in the alloy space.
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I. Introduction

Tungsten (W), also known as wolfram in other countries, is one of the most heat resistant and densest metals found in nature. This metal was originally recognized as the mineral wolframite from Peter Woulfe, a chemist from England in the 18th century. A couple years later, the tungsten we know today was discovered by Jose and Fausto d’Elhuyar of Spain when they refined the mineral through the process of separating the tungsten from the metal oxide, wolframite. Due to its strength and resistance to high temperature, tungsten is widely used in alloys that require these properties such as drill bits, welding, and even spacecraft operations [1]. Tungsten alone, however, has its faults. It is brittle, which means that to improve its application for dynamic uses, it must be mixed with other metals [2]. This is where metals such as molybdenum and rhenium provide utility.

Like tungsten, molybdenum (Mo) is known for its high melting point. This metal, whose name first derived from the Greek word “molybdos” for lead, was first discovered by the Swedish chemist Carl Welhelm Scheele and refined to its pure form first by Peter Jacob Hjelm a few years later. Molybdenum is commonly used for alloying to increase an alloy’s hardness, strength, and resistance to heat and corrosion. These properties make molybdenum valuable for applications such as engine parts, drills, and heating elements. Interestingly, molybdenum also has a role for biological applications [3]. Given that molybdenum is lighter than tungsten, it becomes advantageous to alloy the two together.
to reduce the density of the alloy while leveraging the beneficial effect of solid solution strengthening.

Rhenium (Re) is an important metal for alloying due to its ability to improve ductility and tensile strength. Rhenium, named after the famous Rhine River, was originally speculated to exist since the creation of the periodic table by Dimitri Mendeleev. Its discovery dates to the early 20th century by German scientists Walter Noddack, Otto Berg, and Ida Noddack. Like tungsten and molybdenum, rhenium has a high melting point. With a characteristic like this, rhenium would serve its purpose in equipment such as filaments, grid heaters, and nuclear reactors [4]. With the special qualities molybdenum and rhenium provides, it becomes beneficial for these metals to be alloyed with tungsten.

1.1 Additive Manufacturing

The commercial use of additive manufacturing did not make an appearance until 1987 when the company 3D Systems came up with the process of using ultraviolet light to shape and process a liquid polymer that reacts to that light. This process, known as stereolithography (SL), continued to develop and became widely used for different applications such as prototyping. Additive manufacturing for metals did not become available until the arrival of selective laser sintering (SLS). This process, which came from the DTM corporation, involves using a high-powered laser to heat the metallic grains. The metallic grains would then melt and fuse together to form a solid product. With SLS, the speed and direction at which the laser moves can create unique shapes out
of different metals [5] [6]. These early examples of additive manufacturing helped pave the way with manufacturing unique parts made of different materials.

1.2 Research Outline

1.2.1 Problem Statement

The relatively low cost and ease of additive manufacturing has potential to improve its production across all kinds of applications. The issue that stands, however, is what other metals should be considered when producing a tungsten alloy. Two metals that are considered in the present research are both molybdenum and rhenium. Molybdenum has properties associated with a high melting point and a low thermal expansion coefficient. Rhenium is also associated with having a high melting point as well as high hardness as seen in Table 1.1.

<table>
<thead>
<tr>
<th>Property</th>
<th>W</th>
<th>Mo</th>
<th>Re</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Number</td>
<td>74</td>
<td>42</td>
<td>75</td>
</tr>
<tr>
<td>Atomic Mass, (g/mol)</td>
<td>183.8</td>
<td>95.9</td>
<td>186.2</td>
</tr>
<tr>
<td>Crystal Structure</td>
<td>bcc</td>
<td>bcc</td>
<td>hcp</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>19.25-19.35</td>
<td>10.1-10.3</td>
<td>21.00-21.02</td>
</tr>
<tr>
<td>Melting Temperature (°C)</td>
<td>3410-3420</td>
<td>2607-2622</td>
<td>3157-3181</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td>1670-3900</td>
<td>380-2100</td>
<td>1000-2500</td>
</tr>
<tr>
<td>Yield Strength (MPa)</td>
<td>1350-3500</td>
<td>170-2000</td>
<td>280-2350</td>
</tr>
<tr>
<td>Young's Modulus of Elasticity (GPa)</td>
<td>340-405</td>
<td>315-343</td>
<td>461-471</td>
</tr>
<tr>
<td>Hardness (MPa)</td>
<td>4500-8500</td>
<td>1500-6500</td>
<td>2600-7500</td>
</tr>
</tbody>
</table>

Current alloys have mostly focused on the combination of tungsten and molybdenum and tungsten and rhenium. Meanwhile, the tungsten, molybdenum, rhenium alloy has been rarely studied in this form due to the cost of rhenium. Even fewer is the analysis of additively manufactured alloys of tungsten, molybdenum, and rhenium. Due
to the ease of additive manufacturing compared to traditional alloying, it is worth looking into the creation of such an alloy and analyzing its properties. Understanding its properties could lead to an advantage of quickly and efficiently producing high temperature air and space vehicle structures.

1.2.2 Research Questions

Designing air and space vehicles efficiently will require an understanding of the process and materials used in creating components. If one were to use an additively manufactured alloy of molybdenum and tungsten with a small fraction of rhenium, there needs to be some knowledge on what methods can best replicate a material that can meet mission needs. This leads to the following questions:

- Can a mixture of elemental powders produce a homogeneous mixture through additive manufacturing? Does this affect the alloy’s properties?
- How does the molybdenum, tungsten, rhenium alloy compare to the more widely used molybdenum-tungsten alloy? What additional procedures outside of additive manufacturing settings can be done to further improve the alloy’s properties?
- What are the effects of heat treatment when applied to a printed alloy? Will factors such as length of heat treatment and temperature play a significant role?

1.2.3 Scope and Methodology

All printed alloys are created on the same additive manufacturing machine. Part of the experimental design includes varying printing speed and the composition of the shield gas atmosphere in which the samples are printed. However, all other conditions remain consistent. A total of three samples are printed per unique printing parameter
combination. This is done to get an average of the samples properties in case there are any outliers. To analyze the stress and strain of each sample, all samples go through a three-point bend test where the applied load is recorded. This data with the dimensions of the sample is used to calculate the strength and strain at fracture of the alloy. After all samples are analyzed for its strength and ductility, its microstructure is studied and its hardness, porosity, and homogeneity are measured on the same equipment and software.

All samples are printed through laser powder bed fusion, specifically with the GE Additive/Concept Laser MLab 200R Cusing. The bend test for these samples is conducted on the MTS Acumen Electromechanical Test System. Additional data gathering equipment used is the scanning electron microscope for microstructure observations and the QATM Qness machine for hardness testing. The software used includes the EDAX team software for homogeneity analysis and the Zeiss software for porosity analysis.

### 1.2.4 Assumptions

Oxidation from storage may influence the quality of the printed sample. The longer a powder is stored, the more of an influence oxidation can have, thus weakening the final product of the sample [8]. In this study, the printed samples come from differently stored batches of metal powders. However, the production of these samples took place in a fairly short period of time. Therefore, it is assumed that differences due to storage will not be a factor in this research.
Other assumptions include the powder bed thickness being the same and printing parameter specifications being consistent. All samples printed are also set to have a 50 micron hatch spacing with 20 micron layer thickness.

1.2.5 Impacts

Selecting the ideal powder composition, printing speed, and post printing heat treatment process to produce a strong, heat resistant alloy can come a long way with varying Air Force and Space Force missions. The combination of tungsten, molybdenum, and rhenium has potential in supporting nuclear thermal propulsion. During the GE-710 program, a combination of this alloy is seen to have advantages such as high strength, melting point, and ductility in addition to low oxygen permeability. However, higher bond stresses caused by the fuel matrix and alloy during thermal cycling poises as an issue [9]. Comparing the results of these alloys to other additively manufactured alloys can open other research opportunities related to air and space vehicle designs. The results of the heat-treated tungsten, molybdenum, rhenium alloy from this research can also inspire an additional variety of methods such as changing the ratio of each metal powder or heat treatment process.
II. Background

2.1 Tungsten and Molybdenum

Tungsten and molybdenum are highly sought-after materials for different industries. Physical and chemical properties of tungsten and molybdenum such as their high melting points, high tensile strengths, high electrical and thermal conductivities, and high wear resistance makes these materials able to serve many purposes [10] [11]. The biggest difference is that the density of tungsten is much higher than that of molybdenum. All these different properties make tungsten versatile in many applications such as its use in lighting, high temperature equipment, welding, aviation, space aviation, and even in sports and leisure [12]. Like tungsten, molybdenum is commonly used in aviation and aerospace. Its resistance to high temperatures also makes molybdenum a common metal for the nuclear power industry [11]. Understanding the metallurgy and production can provide an insight to how it benefits different purposes and what promises it can hold when it is improved upon.

2.1.1 Crystallographic Properties

There are three crystallographic modifications of tungsten: α-tungsten, β-tungsten, and γ-tungsten. The α-tungsten form has a body centered cubic crystal structure and is the most stable of the three. The β-tungsten form is metastable which means at a certain temperature above 600 °C, the β-tungsten form can convert to the α-tungsten form. As for γ-tungsten, it is a crystal structure of face centered cubic. They are only found in the
sputtered layers and, like $\beta$-tungsten, will convert to the $\alpha$-tungsten form when heated above a certain temperature [12].

At its solid state, molybdenum is crystallized in the body-centered cubic structure where it is at its most stable form. At higher temperatures and pressures, molybdenum is calculated to be in the face-centered cubic structure. Although it is possible for molybdenum to obtain a hexagonal close-packed structure, it would not be stable [13]. The different geometries of these crystal structures can be seen in Figure 2.1. Since both tungsten and molybdenum have similar crystal structures, there should be no complications in combining both metals through alloying.

Figure 2.1 Bravais Crystal Lattice [14]

2.1.2 Alloying

Despite the advantages in strength and extreme heat resistance, tungsten alone is not sufficient to accomplish some missions. In colder temperatures, tungsten remains brittle and poses a problem when its strength is required in an arctic environment. Tungsten is also one of the densest metals on earth, making it difficult for air and space
operations. Alloying other metals with tungsten is a practical solution to circumventing the inherent disadvantages of tungsten.

Tungsten generally reacts well with groups 4 to 7 in the periodic table. This group includes both rhenium and molybdenum [15]. The addition of rhenium into the tungsten and molybdenum alloy is a topic worth studying. Rhenium can improve both tensile strength and ductility. Additionally, rhenium has interesting properties at high temperatures such as increased strength and fatigue fracture prevention [4]. Studies even show that tungsten-rhenium alloys are stronger and harder than pure tungsten over ranging temperatures as seen in Figure 2.2.

![Figure 2.2 Tensile properties over a range of temperatures for tungsten (W) and alloy with rhenium (Re) [16]](image-url)
Molybdenum is a metal known for its ductility. Mechanically, molybdenum can resist high stress and high strain as well as high temperatures due to having the fifth highest melting point compared to other metals. Despite being generally softer and weaker than tungsten and rhenium, molybdenum is less dense than these two metals. This makes it a more suitable metal that could be applied to air and space applications [17].

2.1.3 70% Molybdenum 25% Tungsten 5% Rhenium Alloy (70Mo-25W-5Re)

In this research, the properties of an alloy containing 70 % molybdenum, 25% tungsten, and 5% rhenium is evaluated. Given the advantage of being a lighter metal compared to tungsten and rhenium, molybdenum is the metal that dominates in presence within the alloy. Figure 2.3 shows the density chart of different combinations of tungsten, molybdenum, and rhenium where density is calculated with equation 2.1 where W is the material and ρ is the density.
An alloy that mostly contains rhenium is subject to be very dense as seen from the chart. Rhenium may be dense, but its addition to the alloy has been proven to provide benefits to the alloy’s characteristics such as improved ductility and toughness. Instead of omitting the metal entirely, a small percentage is added to the alloy for research.

In addition to density, the ratio of tungsten, molybdenum, and rhenium is chosen to avoid detrimental intermetallic phases during its alloying process. If the alloy were to experience these phases, it risks being a less tough and more corrosive metal. Detrimental
intermetallic phases are more likely to occur at higher concentrations of rhenium as seen in Figure 2.4.

![Tungsten, molybdenum, rhenium isothermal ternary diagram at 1600 °C](image)

**Figure 2.4 Tungsten, molybdenum, rhenium isothermal ternary diagram at 1600 °C**

From the isothermal ternary diagram, most of the detrimental intermetallic phases occur when there is a higher concentration of rhenium in the alloy. Molybdenum is able to tolerate the presence of greater concentrations of rhenium without formation of intermetallic phases than tungsten. This motivates the alloy compositions to be comprised primarily of molybdenum rather than tungsten.

Even though molybdenum dominates in concentration of the alloy, there is little concern with how it will blend with tungsten. Figure 2.5 exhibits the binary alloy of tungsten and molybdenum across an array of temperatures. What this figure shows is the
phases that are present depending on the alloy composition and temperature. In this figure, a 0% mass percent of molybdenum indicates a 100% mass percent of tungsten. Below the shaded region is the solid state of the two elements and above that region is the liquid phase. From this graph, the molybdenum and tungsten alloy are an isomorphous system in the shaded region. This means that since both metals are mixable with each other in the liquid state and solid state, there is no expectation of undesirable phases forming.

Figure 2.5 Molybdenum and tungsten binary phase diagram
Given what is known about W-Re alloys and W-Mo alloys and their benefits, it is worth understanding the benefits of what a combination of the three metals can provide. Of course, there are numerous combinations of concentrations that can be done. As a start, a study on a 70Mo-25W-5Re alloy can set an idea of what characteristics and promises the material can provide.

2.2 Traditional Processing

Tungsten and molybdenum ores have been mined and processed for centuries, but only began being used for engineering purposes by the mid-19th century [18]. The process of obtaining a functional form of tungsten starts like most metals with mining ores of its naturally stable oxide, WO₃, in mines. For molybdenum, the metal is most commonly found as its stable sulfide, MoS₂. These materials then go through the process of transforming from compound raw material into pure metals for manufacturing use.

2.2.1 Powder Production

The production of tungsten powder is crucial for the formation of tungsten materials. The shape and size of the powder can have an influence on the ease of the compacting and sintering process. Therefore, it is important to carefully process the powder to save time with the development of solid tungsten. To produce tungsten powder, the element itself must first be isolated. Tungsten trioxide (WO₃) is the starting compound when it comes to powder production. Hydrogen is used to isolate the tungsten element by a reduction reaction with the oxide. This gas is important since it is used to react with the oxide in the compound.
Using either push-type furnaces or rotary furnaces, the WO₃ material is heated at high temperatures with the hydrogen to produce tungsten powder. These powders typically range from 0.1 μm to 100 μm in grain diameters. These different grain sizes are affected by the temperature applied, the method of heating, and the quality of the hydrogen used [12].

Like tungsten, having a careful production of molybdenum powders is also important. The powder production of molybdenum runs a similar process with isolating and heating the raw material to create molybdenum powders. Molybdenum being mined starts off as MoS₂, and it must be converted to technical molybdic oxide (MoO₃) by extracting the sulfur. This process is possible through heating the raw material in furnaces with multiple stages. The reaction with outside oxygen and heat results in technical molybdic oxide. This compound is further refined into pure molybdic oxide through sublimation. In the final process, like tungsten, molybdenum is extracted to its purest form by introducing hydrogen and heat through a rotary furnace. What is left is pure molybdenum powder [19].

2.2.2 Powder Metallurgy

In the traditional method of producing tungsten, powder metallurgy is commonly used. The two main steps are compaction and sintering. The first step of compaction is the process of taking the tungsten powder and compacting it either through the die pressing method or through isostatic pressing. Die pressing involves using a hydraulic or mechanical press while isostatic pressing involves putting the tungsten powder in a mold and subjugating it through hydrostatic pressure. Compacting the tungsten powder is
difficult overall due to the rigidity of the powder. Nevertheless, it is a step required to set up sintering. The sintering process, or heat treatment, of the compacted powder is the step that densifies the tungsten powder into a solid metal. In this process, the temperature and pressure determine how dense the final product will become. The sintering process can either be a direct sintering process or an indirect sintering process. The differences between the processes are the sintering time and temperature. The size of the final product also drives which method of sintering is possible [12].

Molybdenum powders can also go through a similar process of compacting and sintering. The compacting technique and how well the powder is sintered can determine how much of the mechanical properties of molybdenum are retained [20].

2.2.3 Problems with Traditional Metallurgy

The production of tungsten has been refined over the years since its discovery. Despite the current method of compacting and sintering being common, problems still exist in this method. If one desires to have tungsten produced at a specific shape, the fabrication of that shape would require a complex process of adjusting temperatures and formation. The upper and lower temperature limit of the shaping process must be followed, otherwise cracks and splits can develop [12].

2.3 Additive Manufacturing of Metals

Additive manufacturing is a method of creating parts by layers. The formation of these layers and the shape they make is dependent on a 3D model created on a computer. Early development of additive manufacturing involved the creation of parts made of plastic due to its characteristics that make it easy to fabricate. It has since then evolved to
create metal parts through the processes of selective laser sintering (SLS), electron beam melting (EBM), and laser engineering net shaping (LENS). Generally, these processes use a similar approach of using a high-powered laser or electron beam to fully melt a bed of powdered metal. This process binds the powder together and forms a solid metal object. With the ability of the powder bed to move, specific shapes can be created [21].

2.3.1 Advantages of Additive Manufacturing

Additive manufacturing has numerous advantages in the industrial world already. Advantages such as its efficiency, mass customization, on-demand manufacturing, and the ability to modify a design without significant time or cost penalties help push manufacturers to pursue additive manufacturing. In the aerospace industry, parts are already being produced through additive manufacturing across different models of commercial and military aircraft [22].

Tungsten and molybdenum are refractory metals due to their high melting point. The flaw with refractory metals, however, is that they are often subject to aggressive oxidation at high temperatures. Through additive manufacturing, the atmosphere can be controlled in the chamber the alloy is built in to reduce oxidation. Additive manufacturing can also save costs when working with these refractory metals. Through traditional methods, the production of parts with complicated designs becomes expensive through the process of subtracting and joining metals. Further, machining on these hard metals also incurs more cost due to cutting tool wear. Working with these refractory powders is a difficult and laborious process, especially when it comes to sintering and shaping. With additive manufacturing, the shaping is directly done in the machine. The
part being produced rarely needs to be bent or punched to create a specific shape. With the incentives of fewer restrictions to a part design and a shorter lead time, additive manufacturing gains attention from different industries [22] [23].

### 2.3.2 Additive Manufacturing Limitations

Despite the ease of working with additive manufacturing, the final product is still bound to different flaws. Flaws include porosity, surface roughness, and cracking during and after the printing process.

#### 2.3.2.1 Porosity

Porosity is a common byproduct of metals produced through additive manufacturing. One cause of porosity is a lack of careful control in keyhole mode melting. During keyhole melting, the energy density of the laser beam aimed at the powder bed causes the metal to evaporate. This process leaves a cavity that increases the laser’s absorption and allows the laser to reach deeper into the powder bed. If the cavity is unstable, it collapses leaving behind pores such as the ones seen in Figure 2.6 [24]. Porosity can also form from trapped gas. During the atomization process, non-metals such as inert gases do not dissolve with metals in a liquid state. By the time the metal solidifies, the gas entrapped in the metal leaves a void inside the printed part. The machine settings influences porosity sizes as well. Laser power and speed determines how well the fusion between powders can occur [25]. Figure 2.7 demonstrates different pores created from various printing speed and laser power.
Figure 2.6 Metallographic cross section of porosity from keyhole mode melting [24]

Figure 2.7 (a) Porosity effected by printing speed (I) 250 (II) 500 (III) 750 and (IV) 1000 mm/s [26], (b) Porosity effected by laser power (I) 90 (II) 120 and (III) 180 W [27]
2.3.2.2 Surface Roughness

Printed components from additive manufacturing are bound to be produced with rough surfaces. As layer thickness increases, the surface roughness increases with it. Although common, rough surfaces need to be resolved since they will reduce the strength of the printed part. The rough surfaces stem from inadequate melting and balling. Inadequate melting can occur when the laser produces too little heat to completely fuse the metal powder. What is typically left is a powder particle fused to a solid surface. For the balling phenomenon to occur, the print speed is set too high. This high print speed causes balls of the particles to form at the edge of the solidified path that the laser left behind. These small balls of metal form because of the Rayleigh Instability, a phenomenon where liquids break up into smaller parts, which occurs during the metal’s liquid phase [25]. Figure 2.8 demonstrates surface roughness as caused by inadequate melting and balling.

![Figure 2.8](image)

**Figure 2.8** (a) SEM image of solid powders on build surface [28], (b) balling effect [29]
2.3.2.3 Cracking

In additive manufacturing parts, solidification cracking, liquidation cracking, and delamination can occur. Solidification cracking typically appears along the grain boundaries of a part. The cause of solidification cracking is the stress from the solidifying powder layer. The difference in temperatures between the layers causes this stress, and if the stress is higher than the strength of the solidifying metal, cracking will occur. In the partially melted zones, liquidation cracking can occur. This cracking appears when solidification shrinkage in the partially melted zones cause a tensile force. The force from this phenomenon causes the cracking. For delamination to occur, the yield strength of the metal must be less than the residual stress. When this happens, layers become separated since the layers are not fused [25].

2.3.3 Resolving Additive Manufacturing Limitations

Given the limitations that metallic additive manufacturing inherently comes with, there are multiple methods in overcoming these shortfalls. Porosity may not be totally unavoidable, but it can be minimized. Post processing methods such as hot isostatic pressing can internally close the pores. This method, however, is both expensive and time consuming [25]. From Figure 2.8, a slower printing speed with a higher laser power can minimize the porosity in the alloy. Surface roughness can be resolved by machining or etching the alloy until it is smooth, but it becomes impractical and difficult for complex shapes and parts where surface roughness can exist internally.

The strength of the material produced is significantly affected by porosity and rough surfaces, but heat treatment can be a solution to make up for this loss. In some
research on other alloys, such as titanium and aluminum, the temperature and length of time of the heat treatment can heavily influence the properties of the alloy. In most cases, heat treatment improved the strength of the material as well as decreased hardness [30] [31]. Even though heat treatment has no quantifiable effect on reducing pore size, it does influence an alloy’s microstructure and hardness [32].

2.4 Summary

This research will investigate the properties of 70Mo-25W-5Re alloys produced through additive manufacturing. Properties such as stress, strain, porosity count, and hardness are evaluated. There is plenty of research and evaluations on Mo-W and W-Re alloys. However, little research has been conducted on this specific alloy, and there are promises of this alloy having physical properties that are useful for air and space applications. Additive manufacturing has the potential to be more beneficial than traditional tungsten and molybdenum metallurgy given how easy, quickly, and cost effective it is to produce an alloy. If the additively manufactured alloy proves to be on par with other alloys used for air and space applications, it has the potential for saving time and cost in the production of specific tungsten alloyed parts.
III. Methodology

3.1 Printing Process

The additive manufacturing equipment used to produce the samples studied in this research is the GE Additive/Concept Laser MLab 200R Cusing(R) as seen in Figure 3.1. This machine has a 100 x 100 x 100 mm build envelope with a focus diameter of approximately 50 μm and a maximum build speed of 7 m/s. This machine is also capable of accepting gases such as argon and nitrogen, which can be used during the printing process.

Figure 3.1 GE Additive/Concept Laser MLab 200R Cusing(R) [33]
The printing process involves the mixing of molybdenum, tungsten, and rhenium powders where the result is a 70% composition of molybdenum, 25% composition of tungsten, and 5% composition of rhenium. Another set of samples, studied in a parallel research effort, involves a composition of 70% molybdenum and 30% tungsten (Mo-30W). The Mo-30W samples are not the subject of this research but outcomes from that effort influenced experiment design for this research. The conditions of interest when printing the samples focuses on these main factors: the gas it was printed with and the speed the samples were printed. The conditions in which these samples were printed include a set of samples printed under argon gas and a set of samples printed with argon-3% hydrogen gas. The Mo-30W samples were printed with both argon-hydrogen and argon gas while the molybdenum, tungsten, rhenium samples were only printed in argon gas. The speed in which these samples were printed include 100 mm/s, 200 mm/s, 225 mm/s, 400 mm/s, 600 mm/s, and 800 mm/s. These samples were printed with 50 micron hatch spacing and 20 micron layer thickness. Two types of samples were printed under this print speed, and they were vertical bars and 45-degree bars as seen in Figure 3.2. These bars were roughly 18 mm long with a width of approximately 4 mm and thickness of 2 mm.
After understanding the characteristics of a broad range of printing speeds, the focus then shifted to comparing samples at 100 mm/s, 200 mm/s, 300 mm/s and 400 mm/s. Samples of these different printing speed also underwent heat treatment. This step was added to understand the effect heat treatment made on the sample’s microstructure. These samples were then heat treated at a temperature of 1600 °C for 4, 8, 12, and 24 hours. Another set of samples were heat treated at 2000 °C for 12 hours and 2200 °C for 6 hours.

3.2 Bend Test Preparation

Prior to the bend test, the samples must be ground to remove the residual powder that remains from the printing process. This is done to understand the strength and strain of the alloy that has been purely bonded together. Performing a bend test of a non-ground
sample will consider the strength of unbonded metal powders. To focus on the properties of the fused powder, the length of the samples must be ground off.

The samples are ground using the Buehler Ecomet 300 Grinder and Polisher (Figure 3.3) with a rotational setting of 260 revolutions per minute (rpm). The sandpaper used to grind the samples is a 240-grit silicon carbide (SiC) sandpaper. While the machine runs, the samples are applied to the surface of the sandpaper by manually applying force. Once the surface of the sample is smooth, it is washed with isopropyl alcohol and dried with nitrogen gas. The result of the grinding procedure can be seen in Figure 3.4.

![Figure 3.3 Buehler Ecomet 300 Grinder and Polisher](image-url)
The final step prior to the bend test is all samples are baked with the OmegaLux LMF 3550 Benchtop Muffler Furnace as seen in Figure 3.5. These samples are baked for 1 hour at a temperature of 120°C. This is done so that all liquids that are left from the grinding operation are evaporated. By the end of the baking process, the samples are free of effects from residual water and alcohol.
3.3 Bend Test Procedure

After the samples’ length, width, and thickness are measured, the bend test is conducted. This test is conducted with the MTS Acumen Electromechanical Test System as seen in Figure 3.6. The samples are bent by a three-point contact process. The sample is set onto the machine by lying flat on two points. The separation between these two points is 14 mm. The third point is then applied to the top surface of the sample. This point will lightly touch the sample, and the axial distance of this point also gets recorded. Once the machine runs, the third point slowly applies an axial force on the sample. This
point continues to apply force until the sample breaks. The recorded data from this machine includes the axial displacement, axial force, and time it took for the machine to run.

Figure 3.6 MTS Acumen Electromechanical Test System [36]
3.4 Data processing

The output data of the bend test machine gives the axial displacement of the sample (in mm), the axial force applied to the sample (in kN), and the time it took for the machine to run (in seconds). Using this data and the samples’ measurements, the samples’ bending stress (Equation 3.1) and strain (Equation 3.2) can be determined. The breaking stress (in MPa) is calculated by the following equation:

\[
\sigma = \frac{3FL}{2wt^2}
\]  

(3.1)

where, \( F \) is the axial force (in kN), \( L \) is the length between out contact points (in mm), \( w \) is the width of the sample (in mm), and \( t \) is its thickness of the sample (in mm). The bending strain is calculated by the equation:

\[
\varepsilon = \frac{6Dt}{L^2}
\]  

(3.2)

where \( D \) is the axial displacement (in mm) recorded by the MTS machine. From these two formulas, the MATLAB code will calculate the stress and strain of each sample as well as the average strength and strain at fracture of each sample condition based on printing speed and heat treatment time.

3.5 Carbon Puck

Some samples after the bend test are mounted in a carbon metallographic puck. The purpose of doing this allows observation of the sample’s cross-sectional surface. Analysis such as a hardness test and pore count can be done once these pucks are ground out to a smooth surface. The set up for this involves applying the broken samples into the
puck machine and applying carbon powder into the machine. The machine used to mount
the samples into the carbon puck is the MetLab Metpress A as seen in Figure 3.7. This
machine uses electrohydraulic pressure and heat to solidify the carbon powder with the
test samples into a single uniform mold. The result after the machine runs is a mold of the
carbon and samples in the shape of a puck as seen in Figure 3.8.

![Figure 3.7 MetLab Metpress A](image)

Once the puck is made, the next step is to grind it. This is done in order to
minimize scratches. Minimizing scratches is important because it allows some equipment
to focus on the pores of the cross-sectional area. Using the grinding machine from Figure
3.3, the machine is set to 200 rpm for 1 minute. This time, grinding is accomplished with
the machine rather than manually. The puck is gradually sanded with increasing grit
counts, rinsed with alcohol, and dried with nitrogen gas until the scratches are minimized.
3.6 Data Analysis

Data such as fracture observations, hardness, porosity, and homogeneity are obtained using different equipment and software.

3.6.1 Fracture Surface Observations

Observations of fracture surfaces are conducted using the scanning electron microscope. Specifically, the Tescan Maia3 SEM shown in Figure 3.9 is used to capture images of the sample fracture surfaces. Samples are inserted in the machine and put in a vacuum environment. Through different settings, fracture surfaces can be imaged at large magnifications. The field views that are taken in this research vary in the micrometer scale.
3.6.2 Hardness Testing

The hardness testing of the printed samples is conducted through the Vickers hardness test. This widely used method of testing involves using a diamond indenter with the shape of a 136° pyramid. This diamond applies a user inputted load to the surface. What is left is a square indent that is measured, and that measurement determines a unitless hardness value. The hardness value can be determined through equation 3.3 where \( P \) is the force applied and \( d \) is the length of imprint left by the indenter [39].

\[
HV = 1.8544 \frac{P}{d^2}
\]  

(3.3)
The machine used to find each sample’s hardness is the QATM Qness Hardness Tester as seen Figure 3.10. The settings applied on this equipment are 1 kg loads with 10 indents on specimens printed at 100 mm/s.

![Figure 3.10 QATM Qness Hardness Tester](image)

**Figure 3.10 QATM Qness Hardness Tester [40]**

### 3.6.3 Porosity Data

Factors such as entrapped gas and fast printing speeds can contribute to the porosity of a sample. To evaluate the porosity present in each sample, the carbon puck must have a smooth surface so that scratches are not mistaken as pores. Images of these surfaces are then taken with the Axiocam 503 mono camera and put through the Zeiss software. This software takes an image of the sample’s surface and counts the number of pores in the sample and the total area of the combined pores.
3.6.4 Homogeneity Analysis

From looking at the energy dispersive x-ray spectroscopy (EDS) result, one can have a better understanding on how well each element of the alloy has mixed with each other. EDS works by taking advantage of each element’s unique atomic structure. An electron gun is used to excite the elements which leads to these elements emitting x-rays. These x-rays are measured and compiled to produce a spectrum of different wavelength peaks which are used to identify different elements. The software used for EDS is conducted on the EDAX team software using a standard quality scan with a working distance of 10 mm, a magnitude of 250x, and a 256x200 resolution.
IV. Analysis and Results

The first examination that will take place is the analysis of the bend test. After taking the axial force, axial displacement, and dimensions of the sample and processing the data, the breaking stress versus the percent strain can be produced. This data is useful in discovering how different printing conditions effect the samples strength and ductility. Next, the microstructure of the bent samples is observed through the scanning electron microscope. This observation can provide insight into what caused each sample to fracture as well as what possible physical trends are present at strength and strain failure. The hardness testing, porosity analysis, and homogeneity analysis are also conducted to provide insight on the alloy’s internal structural properties.

4.1 Bend Test Results

The initial experiment looks at minimizing the oxygen content during the printing process. This is from the fact that oxygen has a negative influence in the grain boundaries as well as causing porosity [8]. In order to minimize oxygen, the printing process introduces two gaseous atmospheres: argon and argon-hydrogen. A comparison of the overall strength and percent strain at fracture of the samples built in these two atmospheres is shown in Figure 4.1.
Figure 4.1 Average % Strain vs Breaking Stress per Print Speed of 70% Mo 30% W under Argon-Hydrogen and Argon Gas

From the results, the samples printed in the argon hydrogen gas outperformed the samples printed in argon gas. This could be from the argon hydrogen environment performing better with mitigating the oxygen content compared to the argon environment. Another interesting comparison to look at is the printing speeds. The results can be seen in Table 4.1.
From observing the samples of both vertical and 45-degree samples, the results clearly shows that the slower printing speeds outperforms the faster speeds. With a slower printing speed, there is more time for the metal powders to diffuse in their molten state and form a homogeneous mixture. Better mixing and homogeneity will result in a more consistent microstructure which eliminates weak points of discontinuities of strength and ductility in the microstructure.

These previous results all came from a mixture of 70% molybdenum and 30% tungsten. The next alloy that is to be analyzed is the 70% molybdenum, 25% tungsten, and 5% rhenium alloy. For this print, the samples are printed primarily through an argon-hydrogen atmosphere since it produced better strength and percent strain results. The results of the breaking stress vs percent strain of all samples can be seen in Figure 4.2. Figure 4.3 and Table 4.2 features the average breaking stress vs percent strain of each printing speed.
Figure 4.2 % Strain vs Breaking Stress of 70% Mo 25% W 5% Re

Figure 4.3 Average % Strain vs Breaking Stress per Print Speed of 70% Mo 25% W 5% Re
Table 4.2 Average Breaking Stress (MPa) vs % Strain of 70% Mo 25% W 5% Re

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<th>Non Heat Treated 45 Average</th>
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<td>800</td>
<td>146.252</td>
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* Indicates insufficient number of samples to generate standard deviation

From comparing the two alloys printed in argon-hydrogen, the 70% Mo 25% W 5% Re alloy has a lower breaking stress value than the 70% Mo 30% W alloy when comparing by printing speed. Apart from the 70% Mo 30% W printed at 100 mm/s, the overall ductility of the 70% Mo 25% W 5% Re alloy is higher. This could be due to the difference in homogenous microstructure. The 70% Mo 30% W alloy printed at 100 mm/s has a more consistent stress strain response, meaning there are less weak spots to weaken it. The difference in strength between the two types of alloys could be from the difference in the alloy’s metallic mixture. Rhenium is generally lower in tensile and yield strength than tungsten. By removing some tungsten and replacing it with rhenium, there is an expectation that the strength will decrease. The breaking stress and percent strain from Table 4.2 are the properties of the sample straight out of the additive manufacturing machine. An additional heat treatment process, however, changes the composition of the sample. Figure 4.4 reveals the average breaking stress and percent strain of the 70% Mo 25% W 5% Re samples that underwent a heat treatment for 0, 4, 8, 12, and 24 hours. Table 4.3 shows the average stress and strain of the heat-treated samples per
printing speed. A comparison of the stress and strain as heat treatment time increases can be seen in Figure 4.5 and Figure 4.6 for vertical samples and Figure 4.7 and Figure 4.8 for 45-degree samples.

![Figure 4.4 Average % Strain vs Breaking Stress per Print Speed of Heat Treated 70% Mo 25% W 5% Re](image1)

![Figure 4.5 Average % Strain vs Breaking Stress per Print Speed of Heat Treated 70% Mo 25% W 5% Re](image2)
Table 4.3 Average Breaking Stress (MPa) vs % Strain of Heat Treated 70% Mo 25% W 5% Re

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* Indicates insufficient number of samples to generate standard deviation
Figure 4.5 Stress vs Heat Treatment Time per vertical print speed

Figure 4.6 Strain vs Heat Treatment Time per vertical print speed
Figure 4.7 Stress vs Heat Treatment Time per 45° print speed

Figure 4.8 Strain vs Heat Treatment Time per 45° print speed
From just heat treating the samples for 4 hours, the stress and strain significantly improved. The average strength jumped up to an average of 100 MPa and the average percent strain improved by a factor of 0.3 in some cases. However, the average strain also drops by a factor of 0.2 in other cases. In general, the breaking strength is increased, and maximum strain decreased with longer heat treatments. An example of a significant improvement is seen comparing the 45-degree sample printed in 400 mm per second and heat treated for 24 hours (Table 4.3) with the non-heat-treated 45-degree sample printed with a speed of 100 mm per seconds (Table 4.2). The average breaking stress jumps from 290.23 MPa to 415 MPa. The only case of a sample not improving in strength is the case of a vertically 4-hour heat treated sample printed at 200 mm/s as seen in Figure 4.5. Ultimately this value caused a large standard deviation of 120 which brings down the average value of all samples in that category.

The amount of time the samples are heat treated did not have a clear correlation in affecting the strength. For example, a sample printed at 100 mm/s and heat treated for 4 hours had a similar stress and strain to another sample printed at 100 mm/s and heat treated for 8, 12, and 24 hours. Looking at the standard deviation of these same samples show that the distance from the mean is similar for 8, 12, and 24 hours while the 4-hour heat treatment sample has a much higher deviation from the mean. At minimum, heat treating the printed sample for at least 4 hours changed the strength of the material. Besides the percent strain of the 100 mm/s sample in Figure 4.6, the percent strain appears to do a slight decrease after post heat treatment.

In order to have a better understanding on the effects of heat treatment, some samples were also treated at differing temperatures. All samples heat treated at a different
temperature are printed with argon gas. Vertical and 45-degree samples are printed at 100 mm per second and 400 mm per second. The 45-degree samples and some of the vertical samples are then heat treated for 12 hours at 2000 °C. The rest of the vertical samples are heat treated for 6 hours at 2200 °C. The result of their average stress strain values can be seen in Figure 4.10 and Table 4.4. The stress strain is found in Figure 4.9. A comparison of the different heat treatment temperatures can be found in Figure 4.11, Figure 4.12, and Figure 4.13.

Figure 4.9 % Strain vs Breaking Stress of Heat Treated 70% Mo 25% W 5% Re at 2000 °C and 2200 °C
Figure 4.10 Average % Strain vs Breaking Stress per Print Speed of Heat Treated 70% Mo 25% W 5% Re at 2000 °C and 2200 °C

Table 4.4 Average Breaking Stress (MPa) vs % Strain of Heat Treated 70% Mo 25% W 5% Re

<table>
<thead>
<tr>
<th>Speed (mm/s)</th>
<th>Stress (MPa)</th>
<th>Stress SD</th>
<th>Strain</th>
<th>Strain SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>418.87</td>
<td>115.23</td>
<td>0.79</td>
<td>0.15</td>
</tr>
<tr>
<td>400</td>
<td>311.14</td>
<td>43.99</td>
<td>0.54</td>
<td>0.12</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>Stress SD</th>
<th>Strain</th>
<th>Strain SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>2000C 100 mm/s V</td>
<td>543.33</td>
<td>48.47</td>
<td>2.14</td>
</tr>
<tr>
<td>2000C 400 mm/s V</td>
<td>417.40</td>
<td>162.03</td>
<td>0.73</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Speed (mm/s)</th>
<th>Stress (MPa)</th>
<th>Stress SD</th>
<th>Strain</th>
<th>Strain SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>350.63</td>
<td>80.09</td>
<td>0.58</td>
<td>0.04</td>
</tr>
<tr>
<td>400</td>
<td>240.90</td>
<td>18.97</td>
<td>0.41</td>
<td>0.01</td>
</tr>
</tbody>
</table>

12h Heat Treated 2000C Vertical Average

12h Heat Treated 2000C 45 Average
Figure 4.11 Strain vs stress for vertical samples heat treated at 1600 °C and 2000 °C

Figure 4.12 Strain vs stress for 45° samples heat treated at 1600 °C and 2000 °C
When comparing the values of samples heat treated at high temperatures with samples heat treated at a relatively lower temperature, there is a noticeable slight decrease in stress and strain value as the temperature of heat treatments go up. The only outlier is the comparison of the 45-degree samples where the stress and strain increased as the temperature went up. Considering that the samples are relatively close in stress and strain values, it is worth investigating the properties of this alloy printed with hydrogen and heat treated at these higher temperatures.

4.2 Scanning Electron Microscope (SEM) results

The SEM is used to analyze the samples at a microscopic level. At this magnitude, it becomes easier to observe the fracture surface of different materials. In this experiment, the fracture surface of the different printing speeds and heat treatments are
observed. The fracture surface of the argon sample shows evidence on the sample’s weaker nature. Figure 4.14 shows a significant amount of molybdenum oxide indicated by the bright spots. This formation as discussed in Chapter 2 is known to weaken molybdenum alloys.

![Figure 4.14 Molybdenum Oxide formed at the fracture surface](image)

Another interesting observation is comparing surface images of samples printed at different speeds. Figure 4.15 shows the fracture surface of a non-heat-treated sample printed in both 100 mm per second and 600 mm per second.
Both samples are made from the same mixture ratio, and both are printed from the same hydrogen batch. The difference between their creation is the printing speed. From the figure above, it is noticeable to see that the cracks are longer in the faster printing speed than the slower printing speed. A longer fracture indicates that there is some weakness in the fusing between the metal powders during the printing process. This fracture is evident that the slow printing speed leads to a greater strength and percent strain as discussed in section 4.1 and as seen in Table 4.1.

Another byproduct of a slower printing speed can be seen in Figure 4.16 where the samples are taken at a 1000x magnitude.
From the faster printing speed, the shape of the partially melted particles is more noticeable when compared to the slower printing speed. On the right side of Figure 4.16, there are some noticeable bumps and grooves that have a rounded shape. The left side of the figure has some rounded shapes as well, but not as frequent as the one on the right. With a faster printing speed, there is less time for heating leading to less time for the metallic powders to be melted together. What is left is a product of partially melted particles which significantly affect the homogeneity and therefore the strength of the material as noted from Table 4.2 from section 4.1.

As mentioned in section 4.1, the amount of time a sample is heat treated did not significantly affect the strength. Through SEM observations, the fracture surfaces are similar across samples with similar printing speeds despite being heat treated for a different amount of time. Like the non-heat-treated samples, the printing speed played a
bigger factor in strength, and it can be seen through the size of the cracks and number of particles left unmelted. Samples such as the ones seen in Figure 4.17 show similar features such as unmelted particles despite being heat treated at different times. These observations lead to the conclusion that 1600°C is an insufficient temperature to develop significant microstructural evolution in this alloy.

Figure 4.17 70Mo-25W-5Re fracture surface of the 300 mm/s sample heat treated at (a) 4 hours (b) 8 hours (c) 12 hours (d) 24 hours highlighting unmelted particles
4.3 Vickers Hardness Test

The Vickers Hardness test is a method in determining the hardness of a material. This measurement is determined by how much resistance a material has to deformation. The hardness test involves a machine applying pressure onto the surface of a material and applying an indentation onto the surface. The size of the indent is measured, and that measurement determines the hardness value (HV) of the material. The average hardness value for the 100 mm per second samples can be seen in Table 4.5.

Table 4.5 Average Hardness Value of various heat treated 100 mm/s samples

<table>
<thead>
<tr>
<th></th>
<th>Mean Value</th>
<th>Range</th>
<th>Hardness Min</th>
<th>Hardness Max</th>
<th>Standard dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 hours</td>
<td>219.4</td>
<td>44</td>
<td>197</td>
<td>241</td>
<td>12.62</td>
</tr>
<tr>
<td>4 hours</td>
<td>177.3</td>
<td>67</td>
<td>137</td>
<td>204</td>
<td>18.64</td>
</tr>
<tr>
<td>8 hours</td>
<td>177.9</td>
<td>41</td>
<td>157</td>
<td>198</td>
<td>13.45</td>
</tr>
<tr>
<td>12 hours</td>
<td>174.2</td>
<td>23</td>
<td>161</td>
<td>184</td>
<td>7.04</td>
</tr>
<tr>
<td>24 hours</td>
<td>172</td>
<td>41</td>
<td>151</td>
<td>192</td>
<td>13.17</td>
</tr>
</tbody>
</table>

From the results, the hardness stayed consistent across heat treated specimens. The hardness value of the heat-treated samples stayed consistent with a value of approximately 175. The drastic change in hardness occurs when the non-heat-treated specimen goes through heat treatment. The hardness dropping from heat treatment may indicate that the temperature was sufficient to lead to recovery of the material. Recovery is a process by which strain is able to diffuse out of the crystal lattice which would impact the residual stress in the grains and give a lower value of hardness. This process described means that the 1600 °C heat treatment acted like a stress relief, but not sufficiently high to cause recrystallization or homogenization of the unmixed microstructure. Further evidence of the recovery phase can be seen in section 4.5 through
the EDS data. Homogenization is not accomplished, so the only other explanation of the lowered hardness value is recovery.

4.4 Porosity Analysis

The amount of porosity found in an additively manufactured alloy is an important factor when understanding its strength. A highly porous metal is subject to fracture when experiencing a large amount of force. Therefore, it is of interest that these metals remain dense with minimal pores in order to avoid catastrophe when it is used as a part. The porosity of the carbon pucks is first analyzed through the Axiocam 503 mono camera. Figure 4.18 shows an example of what the porosity under the microscope looks like.
Figure 4.18 Porosity of 70Mo-25W-5Re under a 4-hour treatment with printing speeds of (a) 100 mm/s, (b) 200 mm/s, (c) 300 mm/s, (d) 400 mm/s
The Zeiss software is used to count the number of pores and measure the pore’s area on the surface of the samples. Figure 4.19 shows an example of the porosity count and Table 4.6 shows the porosity count and total pores area for each heat-treated sample of differing printing speeds.

![Figure 4.19 Pore count of a non-heat-treated sample printed at 100 mm per seconds](image)

Figure 4.19 Pore count of a non-heat-treated sample printed at 100 mm per seconds
As expected, the most noticeable factor of porosity is printing speed. As seen from Table 4.6, porosity tended to have a steady increase as the printing speed increases. When comparing the porosity results of the non-heat-treated alloys with the heat-treated alloys, it is easy to conclude that heat treatment alone does not decrease porosity. Instead, it appears to increase it. For porosity to noticeably change, pressure must also be included during the heat treatment process. Process like hot isostatic press were not conducted in this research.

4.5 Energy Dispersive X-ray Spectroscopy (EDS) results

The EDS result of the alloy shows that the overall concentration has stayed consistent with the alloy concentration of tungsten, molybdenum, and rhenium. The printing speed and length of time on heat treatment showed that the overall concentration profile of the material did not change. The concentration of molybdenum stayed roughly around 70%, but the tungsten concentration ranged from 26 to 34% while rhenium averaged less than 4% in concentration for samples heat treated at 1600 °C, 2000 °C, and
2200 °C. Table 4.7 displays the average concentration of samples printed at different speeds and heat treated at different times. Table 4.8 displays additional concentrations for different heat treatment temperatures. Figure 4.20 shows the EDS spectra of one of the samples. No other elements were detected in the spectra such as hydrogen or oxygen, but these elements can be difficult to impossible to quantify well with the process because of the nature of the technique.

**Table 4.7 Average Concentration of Mo, W, and Re at different printing speeds and heat treatment time**

<table>
<thead>
<tr>
<th>Heat Treatment (hours)</th>
<th>Speed (mm/s)</th>
<th>Mo Average (Weight%)</th>
<th>W Average (Weight%)</th>
<th>Re Average (Weight%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>100</td>
<td>70.45</td>
<td>26.98</td>
<td>2.57</td>
</tr>
<tr>
<td>4</td>
<td>200</td>
<td>72.22</td>
<td>25.06</td>
<td>2.72</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>71.84</td>
<td>25.67</td>
<td>2.50</td>
</tr>
<tr>
<td>4</td>
<td>400</td>
<td>68.44</td>
<td>28.49</td>
<td>3.07</td>
</tr>
<tr>
<td>8</td>
<td>100</td>
<td>67.34</td>
<td>29.78</td>
<td>2.88</td>
</tr>
<tr>
<td>8</td>
<td>200</td>
<td>69.88</td>
<td>27.05</td>
<td>3.08</td>
</tr>
<tr>
<td>8</td>
<td>300</td>
<td>72.09</td>
<td>25.63</td>
<td>2.27</td>
</tr>
<tr>
<td>8</td>
<td>400</td>
<td>69.94</td>
<td>27.45</td>
<td>2.61</td>
</tr>
<tr>
<td>12</td>
<td>100</td>
<td>72.38</td>
<td>25.11</td>
<td>2.51</td>
</tr>
<tr>
<td>12</td>
<td>200</td>
<td>68.26</td>
<td>28.75</td>
<td>2.99</td>
</tr>
<tr>
<td>12</td>
<td>300</td>
<td>76.56</td>
<td>21.53</td>
<td>1.93</td>
</tr>
<tr>
<td>12</td>
<td>400</td>
<td>69.42</td>
<td>27.63</td>
<td>2.95</td>
</tr>
<tr>
<td>24</td>
<td>100</td>
<td>69.90</td>
<td>27.62</td>
<td>2.48</td>
</tr>
<tr>
<td>24</td>
<td>200</td>
<td>67.87</td>
<td>29.33</td>
<td>2.81</td>
</tr>
<tr>
<td>24</td>
<td>300</td>
<td>74.16</td>
<td>23.54</td>
<td>2.30</td>
</tr>
<tr>
<td>24</td>
<td>400</td>
<td>67.40</td>
<td>29.23</td>
<td>3.37</td>
</tr>
</tbody>
</table>
Figure 4.20 Characteristic X-ray spectrum of 70Mo-25W-5Re printed at 100 mm/s and heat treated for 4 hours

Table 4.8 Concentration of Mo, W, and Re at different printing speeds, heat treatment time, and heat treatment temperatures

<table>
<thead>
<tr>
<th>Temperature (Celsius)</th>
<th>Heat Treatment (hours)</th>
<th>Speed (mm/s)</th>
<th>Mo (Weight%)</th>
<th>W (Weight%)</th>
<th>Re (Weight%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N/A</td>
<td>0</td>
<td>100</td>
<td>61.78</td>
<td>34.03</td>
<td>4.19</td>
</tr>
<tr>
<td>N/A</td>
<td>0</td>
<td>400</td>
<td>71.01</td>
<td>25.77</td>
<td>3.22</td>
</tr>
<tr>
<td>2000</td>
<td>12</td>
<td>100</td>
<td>74.09</td>
<td>22.58</td>
<td>3.33</td>
</tr>
<tr>
<td>2000</td>
<td>12</td>
<td>400</td>
<td>70.55</td>
<td>28.62</td>
<td>0.83</td>
</tr>
<tr>
<td>2200</td>
<td>6</td>
<td>100</td>
<td>67.87</td>
<td>29.27</td>
<td>2.86</td>
</tr>
<tr>
<td>2200</td>
<td>6</td>
<td>400</td>
<td>65.53</td>
<td>34.47</td>
<td>0</td>
</tr>
</tbody>
</table>

The complete homogeneity of some of these samples have not occurred since there are multiple phases instead of one uniform phase. The faster the printing speed, the less homogenous the alloy becomes, and the more phases appear. Figure 4.21 shows the progressive appearance of these phase as printing speed increases.
Figure 4.21 Phase diagram of 70Mo-25W-5Re printed at (a) 100 mm/s (b) 200 mm/s (c) 300 mm/s (d) 400 mm/s and heat treated for 4 hours

The concentration of the different phases remains consistent, but the presence of these phases increases as printing speed increases. For perfect homogenization to occur,
the colors would have to be consistent in these figures. Figure 4.21 only displayed the
phase diagram of the samples heat treated at 1600 °C. Comparing the different heat
treatment temperature shows an increase in homogenization. Figure 4.22 shows the
different phases of tungsten at increasing heat treatment temperatures. At 1600 °C, the
homogenization of tungsten appears to be like the sample that did not underwent heat
treatment. However, homogenization appears to become more apparent at higher
temperatures. At 2000 °C, homogenization of tungsten improves since there are less
bright spots indicating phases. Further improvement in homogenization occurs at a higher
temperature of 2200 °C. This indicates that even though 1600 °C had no significant effect
in improving homogenization, there is potential for homogenization to improve at higher
temperatures.
4.6 Summary

The bend test results provided valuable insight with how printing speed, shield gas composition, and heat treatment impacted strength and ductility. The Mo-30W alloys printed in argon-hydrogen gas came out to be stronger than alloys printed in argon. The analysis on the printing speed also proved that the slower printing speed provided samples with a higher strength and ductility. The average strength and percent strain of the 70Mo-25W-5Re also improves after undergoing heat treatment. The fracture surfaces observed through the SEM further proved how printing speed and shield gas composition affects the physical structures of the alloy. The observations through the SEM also
revealed that there are no significant microstructural changes developed from heat
treatment at 1600 °C. The results of the hardness test provided evidence in the change of
composition distribution through heat treatment. Comparing the hardness of the heat
treated and non-heat-treated results indicate the material underwent a recovery process
during heat treatment at 1600 °C. The porosity analysis is used to further prove the
impacts of printing speed on porosity and the ineffectiveness of heat treatment reducing
porosity. The EDS result showed that perfect homogeneity does not exist within the
alloy. The faster printing speeds did show more phases than slower ones, but heat
treatment did not significantly change the composition distribution and homogeneity of
the alloy. Overall, the different parameter combinations prior to the bend test have proved
that printing speeds, shield gas composition and heat treatment impact the different
material properties of the alloy.
V. Conclusions and Recommendations

5.1 Shortcomings

The conclusions that are reached from this research are limited to the properties tested or analyzed. Due to the high cost of rhenium powders, the number of duplicate samples for each unique printing and heat-treating scenario is small. Having more trials can help improve an understanding of different trends that each heat-treatment style can provide. More sample replicates can help identify what trials are outliers and what trends are common.

Given the length of time to conduct this experiment, there is a limitation on how many differing heat-treated samples can be produced. Heat treatments at temperatures of 1600 °C, 2000 °C, and 2200 °C were chosen for this research, but having smaller increments in temperature may also help identify if there is a clear relationship between heat treatment temperature and strength and ductility.

5.2 Conclusions

This research has covered an analysis of the additively manufactured 70Mo-25W-5Re alloy. This alloy is observed and analyzed after being printed at different speeds and gaseous environments. Additionally, the effects of heat treating the additively manufactured alloy is observed and compared to other traditional tungsten alloys such as tungsten-molybdenum. Across the board, a slower printing speed that is printed with argon-hydrogen has proved to produce an alloy with a higher strength and percent strain value than that of an alloy printed with a faster speed with argon. The strength for the 70Mo-25W-5Re alloys also improved when they undergo heat treatment. Generally, the
length of time of heat treatment had no discernable impact to how much strength the alloy gains. Based on comparing the length of heat treatment time to stress, there has been no consistent trend on how much stronger or weaker an alloy gets the longer it is heat treated. Aside from the vertical samples printed at 100 mm/s, the ductility of the alloy has decreased with heat treatment. Like the case of the alloy’s strength, there is no discernable relationship between heat treatment time and stress. Despite the effect heat treatment has done to the alloy’s strength, results from other tests and evaluations show that the length of time that a sample is heat treated had no quantifiable impact in changing the alloy’s hardness or porosity count. Recovery, indicated by hardness measurements, occurred very quickly at 1600 °C.

With the observations made with different heat treatment temperatures, the results are mixed. The ductility and strength improved significantly with the 45-degree sample printed at 100 mm/s and heat treated at 2000 °C. However, in other cases, the strength and ductility of some samples heat treated at 2000 °C are either worse or on par with samples heat treated at 1600 °C. The same goes with comparing samples heat treated at 1600 °C with samples heat treated at 2200 °C.

This research answered the questions laid out in Chapter 1. A mixture of elemental powders do not produce a completely homogeneous mixture through additive manufacturing, which affects the alloy’s properties. For the 70Mo-25W-5Re alloys, heat treatment will also further increase the strength. The temperature the samples are heat treated show noticeable effects with the strength of the alloy. However, length of heat treatment time has no significance in changing the alloy’s porosity, hardness, or homogeneity.
5.3 Future Recommendations

This work focused mostly on one combination of tungsten, molybdenum, and rhenium. There is an abundance of combinations that can be done with the study of these three metals in the alloy system. The only limit is figuring out combinations that avoid detrimental intermetallic phases. The properties of different alloy combinations can yield different results, and it is possible that there is an alloy combination that is more beneficial in terms of strength and density compared to 70Mo-25W-5Re. The heat treatment of the alloy in this research also focused on temperatures of 1600 °C, 2000 °C, and 2200 °C. It will be an interesting case study to see whether there is a trend between an alloy’s strength and hardness with a higher or lower heat treatment temperature. Additional testing such as a high temperature bend test can provide a better understanding of how well this alloy can be used in a practical sense. Since tungsten alloys are used for its high heat resistance, it would be interesting to see how the 70Mo-25W-5Re alloy will fare and what strengths it maintains in an extremely hot environment. The EDS results only shows the metallic composition and phases of each alloy. However, this method is limited to detecting heavier elements due to the nature of its technique. Methods such as inert gas fusion can provide useful insights in future research. The process is useful in detecting lighter elements such as oxygen and hydrogen. Detecting these elements in the alloys can provide insight in determining how much of an influence these gasses have on strength, ductility, and homogeneity. There are many research paths when it comes to discovering a valuable alloy produced through additive manufacturing
Appendix A. Matlab Code

A.1 Stress Strain Data Processing for 1600 °C Heat Treated and Non-Heat-Treated Samples

```
1 %%%%%%%%%%%%%%%%%
2 %
3 % 70% Molybdenum 25% Tungsten 5% Rhenium manufactured by selective laser melting
4 % 4-8-12-24hr Vacuumed Heated
5 % 100-200-300-400 Print Speed
6 % Vertical-45deg Orientation
7 % Bend Test Data
8 %
9 % Author: Maj Ryan Kemnitz
10 % Revised: Lt Jae Yu - 11 October 2021
11 %
12 %%%%%%%%%%%%%%%%%
13 %
14 close all;clear all; clc
15
16 % 4hr Heat Vertical
17 cd 'C:\70Mo20W5Re Heat Treated\4h\4h Vertical'
18 B = dir('**/*.txt');
19
20 for i = 1:length(B)
21    file = [B(i).folder,'\',B(i).name];
22    temp = importdata(file);
23
24    A{i} = -temp.data;
25
26    first_loc = find(A{i}(:,2)>2e-3,1);
27    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);
```
last_loc = find(A{i}(:,1)>0.5,1);
if isempty(last_loc) == true
    last_loc = length(A{i}( :,1));
end

C{i} = A{i}(first_loc:last_loc-3,:);

[val(i,1),loc] = max(C{i}(:,2));
max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;300;300;400;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-4h-Measurements-v.xlsx');
widths = dims(:,1);
thicks = dims(:,2);

real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
grind_newton=val;
real_strain = 6*max_disp.*thicks/14^2;

MOWRe4hrV_all = [speeds real_strain real_stress];
for i = 1:length(unique_speeds)
    o = (i-1)*3+1;
    p = i*3;
    locs = find(speeds==unique_speeds(i));
    average_stress(i) = mean(real_stress(locs));
    average_strain(i,1) =100*mean(real_strain(locs));
end

end
EV = 200./(0.020*0.050*unique_speeds);

MOWRe4hrV = [unique_speeds EV average_stress' average_strain];

hold on
plot(real_strain*100,real_stress,'s','LineWidth',2); grid on

%% 4hr Heat 45 deg
clearvars -except MOWRe4hrV MOWRe4hrV_all

cd 'C:\70Mo20W5Re Heat Treated\4h\4h 45'
B = dir('**/*.txt');

for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = -temp.data;
    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);
    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end
    C{i} = A{i}(first_loc:last_loc-3,:);
    [val(i,1),loc] = max(C{i}(:,2));
max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;300;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-4h-Measurements-45.xlsx');
widths = dims(:,1);
thicks = dims(:,2);

real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
real_strain = 6*max_disp.*thicks/14^2;
MOWRe4hr45_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
o = (i-1)*3+1;
p = i*3;
locs = find(speeds==unique_speeds(i));
average_stress(i) = mean(real_stress(locs));
average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);
MOWRe4hr45 = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'o','LineWidth',2)
clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all

cd 'C:\70Mo20W5Re Heat Treated\8h\8h Vertical'

B = dir('**/*.txt');

for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end

    C{i} = A{i}(first_loc:last_loc-3,:);
    [val(i,1),loc] = max(C{i}(:,2));
    max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;300;400;400;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-8h-Measurements-v.xlsx');
widths = dims(:,1);
thicks = dims(:,2);
real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
grind_newton=val;
real_strain = 6*max_disp.*thicks/14^2;

MOWRe8hrV_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
    o = (i-1)*3+1;
    p = i*3;
    locs = find(speeds==unique_speeds(i));
    average_stress(i) = mean(real_stress(locs));
    average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);

MOWRe8hrV = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'s','LineWidth',2)

%% 8hr Heat 45 deg

clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all MOWRe8hrV MOWRe8hrV_all

cd 'C:\70Mo20W5Re Heat Treated\8h\8h 45'
B = dir('**/*_.txt');
for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
temp = importdata(file);

A{i} = -temp.data;

first_loc = find(A{i}(:,2)>2e-3,1);
A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

last_loc = find(A{i}(:,1)>0.5,1);
if isempty(last_loc) == true
    last_loc = length(A{i}(:,1));
end

C{i} = A{i}(first_loc:last_loc-3,:);

[val(i,1),loc] = max(C{i}(:,2));
max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;300;400;400];
unique_speeds = unique(speeds);

dims = importdata('MoWRe-8h-Measurements-45.xlsx');
widths = dims(:,1);
thicks = dims(:,2);

real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
real_strain = 6*max_disp.*thicks/14^2;

MOWRe8hr45_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
\[ o = (i-1) \times 3 + 1; \]
\[ p = i \times 3; \]
\[ \text{locs} = \text{find(speeds==unique\_speeds(i))}; \]
\[ \text{average\_stress}(i) = \text{mean(real\_stress(locs))}; \]
\[ \text{average\_strain}(i,1) = 100 \times \text{mean(real\_strain(locs))}; \]

\[ \text{end} \]

\[ \text{EV} = 200.0 / (0.020 \times 0.050 \times \text{unique\_speeds}); \]

\[ \text{MOWRe8hr45} = [\text{unique\_speeds} \ \text{EV} \ \text{average\_stress}' \ \text{average\_strain}]; \]

\[ \text{clearvars} \ -\text{except} \ \text{MOWRe4hrV} \ \text{MOWRe4hrV\_all} \ \text{MOWRe4hr45} \ \text{MOWRe4hr45\_all} \ ... \]
\[ \text{cd 'C:\70Mo20W5Re Heat Treated\12h\12h Vertical'} \]
\[ \text{B} = \text{dir('**/*.txt')}; \]

\[ \text{figure} \]
\[ \text{hold on} \]
\[ \text{for} \ i = 1: \text{length(B)}; \]
\[ \text{file} = [\text{B(i).folder,}\ \backslash,\ \text{B(i).name}]; \]
\[ \text{temp} = \text{importdata(file)}; \]
\[ \text{A(i)} = -\text{temp.data}; \]
\[ \text{first\_loc} = \text{find(A(i)(:,2)>2e-3,1)}; \]
\[ \text{A(i)(:,1)} = \text{A(i)(:,1)}-\text{A(i)(first\_loc,1)}; \]
last_loc = find(A{i}(:,1)>0.5,1);
if isempty(last_loc) == true
    last_loc = length(A{i}(:,1));
end

C{i} = A{i}(first_loc:last_loc-3,:);
plot(C{i}( :,1),C{i}( :,2))
[val(i,1),loc] = max(C{i}( :,2));
max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;300;400;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-12h-Measurements-v.xlsx');
widths = dims(:,1);
thicks = dims(:,2);
real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
grind_newton=val;
real_strain = 6*max_disp.*thicks/14^2;
MOWRe12hrV_all = [speeds real_strain real_stress];
for i = 1:length(unique_speeds)
o = (i-1)*3+1;
p = i*3;
locs = find(speeds==unique_speeds(i));
average_stress(i) = mean(real_stress(locs));
average_strain(i,1) =100*mean(real_strain(locs));
EV = 200./(0.020*0.050*unique_speeds);

legend('100(1)','100(2)','100(3)','200(1)','200(2)','200(3)','300(1)','300(2)','300(3)','400(1)','400(2)','400(3)')

hold off

figure

MOWRe12hrV = [unique_speeds EV average_stress' average_strain];

hold on

plot(real_strain*100,real_stress,'s','LineWidth',2)

%% 12hr Heat 45 deg

clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all ...

MOWRe8hrV MOWRe8hrV_all MOWRe8hr45 MOWRe8hr45_all MOWRe12hrV MOWRe12hrV_all

cd 'C:\70Mo20W5Re Heat Treated\12h\12h 45'

B = dir('**/*.txt');

for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = -temp.data;
    first_loc = find(A{i}(;2)>2e-3,1);
    A{i}(;1) = A{i}(;1)-A{i}(first_loc,1);
last_loc = find(A{i}(:,1)>0.5,1);
if isempty(last_loc) == true
    last_loc = length(A{i}(:,1));
end

C{i} = A{i}(first_loc:last_loc-3,:);

[val(i,1),loc] = max(C{i}(:,2));
max Disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;300;400;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-12h-Measurements-45.xlsx');
widths = dims(:,1);
thicks = dims(:,2);
real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
real_strain = 6*max_disp.*thicks/14^2;
MOWRe12hr45_all = [speeds real_strain real_stress];
for i = 1:length(unique_speeds)
o = (i-1)*3+1;
p = i*3;
locs = find(speeds==unique_speeds(i));
average_stress(i) = mean(real_stress(locs));
average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);

MOWRe12hr45 = [unique_speeds EV average_stress' average_strain];

hold on
plot(real_strain*100,real_stress,'o','LineWidth',2)

%% 24hr Heat Vertical

clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all ...
MOWRe8hrV MOWRe8hrV_all MOWRe8hr45 MOWRe8hr45_all ...
MOWRe12hrV MOWRe12hrV_all MOWRe12hr45 MOWRe12hr45_all
cd 'C:\70Mo20W5Re Heat Treated\24h\24h Vertical'
B = dir('**/*.txt');
figure
hold on
for i = 1:length(B);
    file = [B(i).folder,'\',B(i).name];
temp = importdata(file);
A{i} = -temp.data;
first_loc = find(A{i}(:,2)>2e-3,1);
A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);
last_loc = find(A{i}(:,1)>0.5,1);
if isempty(last_loc) == true
    last_loc = length(A{i}(:,1));
end

end
C{i} = A{i}(first_loc:last_loc-3,:);  
plot(C{i}(:,3),C{i}(:,1))  
[val(i,1),loc] = max(C{i}(:,2));  
max_disp(i,1) = C{i}(loc,1);  
end  
speeds = [100;100;100;200;200;300;300;400;400];  
unique_speeds = unique(speeds);  
dims = importdata('MoWRe-24h-Measurements-v.xlsx');  
widths = dims(:,1);  
thicks = dims(:,2);  
real_stress = 3*val*1000*14./(2*widths.*thicks.^2);  
grind_newton=val  
real_strain = 6*max_disp.*thicks/14^2;  
MOWRe24hrV_all = [speeds real_strain real_stress];  
for i = 1:length(unique_speeds)  
    o = (i-1)*3+1;  
    p = i*3;  
    locs = find(speeds==unique_speeds(i));  
    average_stress(i) = mean(real_stress(locs));  
    average_strain(i,1) =100*mean(real_strain(locs));  
end  
legend('1','2','3','4','5','6','7','8','9','10')  
EV = 200./(0.020*0.050*unique_speeds);  
hold off  
figure
MOWRe24hrV = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'s','LineWidth',2)

%% 24hr Heat 45 deg
clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all ...
MOWRe8hrV MOWRe8hrV_all MOWRe8hr45 MOWRe8hr45_all ...
MOWRe12hrV MOWRe12hrV_all MOWRe12hr45 MOWRe12hr45_all ...
MOWRe24hrV MOWRe24hrV_all

cd 'C:\70Mo20W5Re Heat Treated\24h\24h 45'
B = dir('**/*.txt');

for i = 1:length(B);
    file = [B(i).folder,'\',B(i).name];
temp = importdata(file);
A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end

    C{i} = A{i}(first_loc:last_loc-3,:);
    [val(i,1),loc] = max(C{i}(:,2));
     max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;200;300;300;400;400;400];
unique_speeds = unique(speeds);

dims = importdata('MoWRe-24h-Measurements-45.xlsx');
widths = dims(:,1);
thicks = dims(:,2);

real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
real_strain = 6*max_disp.*thicks/14^2;

MOWRe24hr45_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
    o = (i-1)*3+1;
    p = i*3;
    locs = find(speeds==unique_speeds(i));
    average_stress(i) = mean(real_stress(locs));
    average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);

MOWRe24hr45 = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'o','LineWidth',2)

83
clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45 MOWRe4hr45_all ...
MOWRe8hrV MOWRe8hrV_all MOWRe8hr45 MOWRe8hr45_all ...
MOWRe12hrV MOWRe12hrV_all MOWRe12hr45 MOWRe12hr45_all ...
MOWRe24hrV MOWRe24hrV_all MOWRe24hr45

cd 'C:\70Mo20W5Re Heat Treated\Non Heat Treated\Vertical'
B = dir('**/*.txt');

for i = 1:length(B);
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end

    C{i} = A{i}(first_loc:last_loc-3,:);

    [val(i,1),loc] = max(C{i}(:,2));
    max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;200;200;225;225;225;400;400;600;600;600;600;800;800];
unique_speeds = unique(speeds);
dims = importdata('MoMeasurement_V.xlsx');
widths = dims(:,1);
thicks = dims(:,2);

real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
real_strain = 6*max_disp.*thicks/14^2;

MOWRe0hrV_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
    o = (i-1)*3+1;
    p = i*3;
    locs = find(speeds==unique_speeds(i));
    average_stress(i) = mean(real_stress(locs));
    average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);

MOWRe0hrV = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'o','LineWidth',2)

%% 0hr Heat 45
clearvars -except MOWRe4hrV MOWRe4hrV_all MOWRe4hr45
MOWRe4hr45_all ...
cd 'C:\70Mo20W5Re Heat Treated\Non Heat Treated\45'

B = dir('**/*.txt');

for i = 1:length(B);
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);

    A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1) - A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end

    C{i} = A{i}(first_loc:last_loc-3,:);
    [val(i,1),loc] = max(C{i}(:,2));
    max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;200;200;400;400;400;600;600;600;800;800;800];
unique_speeds = unique(speeds);

dims = importdata('MoMeasurement_45.xlsx');
widths = dims(:,1);
thicks = dims(:,2);
553  real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
554  real_strain = 6*max_disp.*thicks/14^2;
555
556  MOWRe0hr45_all = [speeds real_strain real_stress];
557
558  for i = 1:length(unique_speeds)
559      o = (i-1)*3+1;
560      p = i*3;
561      locs = find(speeds==unique_speeds(i));
562      average_stress(i) = mean(real_stress(locs));
563      average_strain(i,1) =100*mean(real_strain(locs));
564
565  end
566
567  EV = 200./(0.020*0.050*unique_speeds);
568
569  MOWRe0hr45 = [unique_speeds EV average_stress' average_strain];
570  hold on
571  plot(real_strain*100,real_stress,'o','LineWidth',2)
572
573  title('70% Molybdenum 25% Tungsten 5% Rhenium Strain vs Stress')
574  xlabel('Strain (dimensionless)')
575  ylabel('Stress (Newton/milimeter^2)')
576  legend('MoWRe 4hr V','MoWRe 4hr 45','MoWRe 8hr V','MoWRe 8hr 45','MoWRe 12hr V','MoWRe 12hr 45','MoWRe 24hr V','MoWRe 24hr 45','MoWRe 0hr V','MoWRe 0hr 45')
577
578  hold on
579  plot(real_strain*100,real_stress,'o','LineWidth',2)
580
87
A.2 Stress Strain Processing for 2000 °C and 2200 °C Heat Treated Samples
% 70% Molybdenum 25% Tungsten 5% Rhenium manufactured by 
selective laser melting

% Heat Treated in 2000C and 2200C
% 100-400 Print Speed
% Vertical-45deg Orientation
% Bend Test Data
%
% Author: Maj Ryan Kemnitz
% Revised: Lt Jae Yu - 11 October 2021
%
%%%%%%%%%%%%%%
%
close all;clear all; clc

% 2000C 12hr Heat Vertical
cd 'C:\70Mo20W5Re Heat Treated\Other HT RT\2000C_12hr_v'
B = dir('**/*.txt');

for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end
end
34 \[ C(i) = A(i)\{\text{first\_loc:last\_loc-3,:}\}; \]
35
36 \[ [\text{val(i,1),loc]} = \text{max}(C(i)(:,2)); \]
37 \[ \text{max\_disp(i,1)} = C(i)\{\text{loc,1}\}; \]
38 \end
39
40 \text{speeds} = [100;100;100;400;400;400];
41 \text{unique\_speeds} = \text{unique(speeds)};
42 \text{dims} = \text{importdata('MoWRe-2000C-Measurements-v.xlsx')};
43 \text{widths} = \text{dims(:,1)};
44 \text{thicks} = \text{dims(:,2)};
45
46 \text{real\_stress} = 3*\text{val*1000*14}/(2*\text{widths.*thicks.^2});
47 \text{grind\_newton} = \text{val};
48 \text{real\_strain} = 6*\text{max\_disp.*thicks/14^2};
49
50 \text{MOWRe2000C\_V\_all} = [\text{speeds real\_strain real\_stress}];
51 \text{for i} = 1:length(\text{unique\_speeds})
52 \text{o} = (i-1)*3+1;
53 \text{p} = i*3;
54 \text{locs} = \text{find(speeds==unique\_speeds(i))};
55 \text{average\_stress}(i) = \text{mean(real\_stress(locs))};
56 \text{average\_strain}(i,1) = 100*\text{mean(real\_strain(locs))};
57 \end
58
59 \text{EV} = 200./(0.020*0.050*\text{unique\_speeds});
60
61
62 \text{MOWRe2000C\_V} = [\text{unique\_speeds EV average\_stress' average\_strain}];
63 \text{hold on}
plot(real_strain*100,real_stress,'s','LineWidth',2); grid on

%% 2000C 12hr Heat 45 deg

clearvars -except MOWRe2000C_V MOWRe2000C_V_all

cd 'C:\70Mo20W5Re Heat Treated\Other HT RT\2000C_12h_45'

B = dir('**/*.txt');

for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);

    A{i} = -temp.data;

    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);

    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end

    C{i} = A{i}(first_loc:last_loc-3,:);

    [val(i,1),loc] = max(C{i}(:,2));
    max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;400;400;400];
unique_speeds = unique(speeds);
 dims = importdata('MoWRe-2000C-Measurements-45.xlsx');
 widths = dims(:,1);
 thicks = dims(:,2);

 real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
 real_strain = 6*max_disp.*thicks/14^2;

 MOWRe2000C_45_all = [speeds real_strain real_stress];

 for i = 1:length(unique_speeds)
   o = (i-1)*3+1;
   p = i*3;
   locs = find(speeds==unique_speeds(i));
   average_stress(i) = mean(real_stress(locs));
   average_strain(i,1) =100*mean(real_strain(locs));

 end

 EV = 200./(0.020*0.050*unique_speeds);

 MOWRe2000C_45 = [unique_speeds EV average_stress' average_strain];

 hold on
 plot(real_strain*100,real_stress,'o','LineWidth',2)

 clearvars -except MOWRe2000C_V MOWRe2000C_V_all MOWRe2000C_45
 MOWRe2000C_45_all
 cd 'C:\7Mo20W5Re Heat Treated\Other HT RT\2200C_6h_v'
 B = dir('**/*.txt');
for i = 1:length(B)
    file = [B(i).folder,'\',B(i).name];
    temp = importdata(file);
    A{i} = temp.data;
    first_loc = find(A{i}(:,2)>2e-3,1);
    A{i}(:,1) = A{i}(:,1)-A{i}(first_loc,1);
    last_loc = find(A{i}(:,1)>0.5,1);
    if isempty(last_loc) == true
        last_loc = length(A{i}(:,1));
    end
    C{i} = A{i}(first_loc:last_loc-3,:);
    [val(i,1),loc] = max(C{i}(:,2));
    max_disp(i,1) = C{i}(loc,1);
end

speeds = [100;100;100;400;400;400];
unique_speeds = unique(speeds);
dims = importdata('MoWRe-2200C-Measurements-v.xlsx');
widths = dims(:,1);
thicks = dims(:,2);
real_stress = 3*val*1000*14./(2*widths.*thicks.^2);
grind_newton=val;
real_strain = 6*max_disp.*thicks/14^2;
MOWRe2200C_V_all = [speeds real_strain real_stress];

for i = 1:length(unique_speeds)
    o = (i-1)*3+1;
    p = i*3;
    locs = find(speeds==unique_speeds(i));
    average_stress(i) = mean(real_stress(locs));
    average_strain(i,1) =100*mean(real_strain(locs));
end

EV = 200./(0.020*0.050*unique_speeds);

MOWRe2200C_V = [unique_speeds EV average_stress' average_strain];
hold on
plot(real_strain*100,real_stress,'s','LineWidth',2)

title('70% Molybdenum 25% Tungsten 5% Rhenium Strain vs Stress')
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('MoWRe 2000C 12h V','MoWRe 2000C 12h 45','MoWRe 2200C 6h V')

figure
plot(MOWRe2000C_V(:,4),MOWRe2000C_V(:,3),'s','LineWidth',2)
hold on
plot(MOWRe2000C_45(:,4),MOWRe2000C_45(:,3),'o','LineWidth',2)
plot(MOWRe2200C_V(:,4),MOWRe2200C_V(:,3),'s','LineWidth',2)
grid on
title('70% Mo 25% W 5% Re Average Strain vs Stress')
A.3 Heat Treatment Temperature vs Stress and Strain Code

clc;clear all;close all

cd 'C:\70Mo20W5Re Heat Treated\Stress Strain Value\All Stress Strain\Vertical'

MoWRe1600_100_v=importdata('100mms at 1600C v.xlsx');
MoWRe1600_200_v=importdata('200mms at 1600C v.xlsx');
MoWRe1600_300_v=importdata('300mms at 1600C v.xlsx');
MoWRe1600_400_v=importdata('400mms at 1600C v.xlsx');

MoWRe2000data=importdata('MoWRe 2000C 12hr V all.xlsx'); %12 hour HT only
MoWRe2000_100_v=MoWRe2000data(1:3,2:3);
MoWRe2000_400_v=MoWRe2000data(4:6,2:3);
MoWRe2200data=importdata('MoWRe 2200C 6hr V all.xlsx'); % 6 hour HT only
MoWRe2200_100_v=MoWRe2200data(1:3,2:3);
MoWRe2200_400_v=MoWRe2200data(4:6,2:3);

cd 'C:\70Mo20W5Re Heat Treated\Stress Strain Value\All Stress Strain\45'
MoWRe1600_100_45=importdata('100mms at 1600C 45.xlsx');
MoWRe1600_200_45=importdata('200mms at 1600C 45.xlsx');
MoWRe1600_300_45=importdata('300mms at 1600C 45.xlsx');
MoWRe1600_400_45=importdata('400mms at 1600C 45.xlsx');

MoWRe2000data=importdata('MoWRe 2000C 12hr 45 all.xlsx'); % 12 hour HT only
MoWRe2000_100_45=MoWRe2000data(1:3,2:3);
MoWRe2000_400_45=MoWRe2000data(4:6,2:3);

%% Stress plots

% Vertical Stress for 1600C
figure()
subplot(2,2,1)
plot(MoWRe1600_100_v(:,1),MoWRe1600_100_v(:,3),'o','LineWidth',2)
title('100 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,2)
plot(MoWRe1600_200_v(:,1),MoWRe1600_200_v(:,3),'o','LineWidth',2)
title('200 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,3)
plot(MoWRe1600_300_v(:,1),MoWRe1600_300_v(:,3),'o','LineWidth',2)
title('300 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,4)
plot(MoWRe1600_400_v(:,1),MoWRe1600_400_v(:,3),'o','LineWidth',2)
title('400 mm/ss')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

sgtitle('Stress vs Heat Treatment Time per Vertical Print Speed')

%45 degree Stress at 1600C
figure()
subplot(2,2,1)
plot(MoWRe1600_100_45(:,1),MoWRe1600_100_45(:,3),'o','LineWidth',2)
title('100 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,2)
plot(MoWRe1600_200_45(:,1),MoWRe1600_200_45(:,3),'o','LineWidth',2)
title('200 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,3)
plot(MoWRe1600_300_45(:,1),MoWRe1600_300_45(:,3),'o','LineWidth',2)
title('300 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

subplot(2,2,4)
plot(MoWRe1600_400_45(:,1),MoWRe1600_400_45(:,3),'o','LineWidth',2)
title('400 mm/ss')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Stress (N/mm^2)')
axis([0 24 0 700])

sgtitle('Stress vs Heat Treatment Time per 45 Print Speed')
%% Strain Plots

figure()

subplot(2,2,1)
plot(MoWRe1600_100_v(:,1),MoWRe1600_100_v(:,2)*100,'o','LineWidth',2)
title('100 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Strain')
axis([0 24 0.4 1.9])

subplot(2,2,2)
plot(MoWRe1600_200_v(:,1),MoWRe1600_200_v(:,2)*100,'o','LineWidth',2)
title('200 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Strain')
axis([0 24 0.4 1.9])

subplot(2,2,3)
plot(MoWRe1600_300_v(:,1),MoWRe1600_300_v(:,2)*100,'o','LineWidth',2)
title('300 mm/s')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Strain')
axis([0 24 0.4 1.9])

subplot(2,2,4)
plot(MoWRe1600_400_v(:,1),MoWRe1600_400_v(:,2)*100,'o','LineWidth',2)
```
130   title('400 mm/ss')
131   grid on
132   xlabel('Heat Treatment Time (hours)')
133   ylabel('Strain')
134   axis([0 24 0.4 1.9])
135
136   sgtitle('Strain vs Heat Treatment Time per Vertical Print Speed')
137
138   %45 degree strain at 1600C
139   figure()
140   subplot(2,2,1)
141   plot(MoWRe1600_100_45(:,1),MoWRe1600_100_45(:,2)*100,'o','LineWidth',2)
142   title('100 mm/s')
143   grid on
144   xlabel('Heat Treatment Time (hours)')
145   ylabel('Strain')
146   axis([0 24 0.4 1.9])
147
148   subplot(2,2,2)
149   plot(MoWRe1600_200_45(:,1),MoWRe1600_200_45(:,2)*100,'o','LineWidth',2)
150   title('200 mm/s')
151   grid on
152   xlabel('Heat Treatment Time (hours)')
153   ylabel('Strain')
154   axis([0 24 0.4 1.9])
155
156   subplot(2,2,3)
157   plot(MoWRe1600_300_45(:,1),MoWRe1600_300_45(:,2)*100,'o','LineWidth',2)
158   title('300 mm/s')
```
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Strain')
axis([0 24 0.4 1.9])

subplot(2,2,4)

plot(MoWRe1600_400_45(:,1),MoWRe1600_400_45(:,2)*100,'o','LineWidth',2)
title('400 mm/ss')
grid on
xlabel('Heat Treatment Time (hours)')
ylabel('Strain')
axis([0 24 0.4 1.9])

sgtitle('Strain vs Heat Treatment Time per 45 Print Speed')

%% 2000C and 2200C
figure()
subplot(2,1,1)

plot(MoWRe1600_100_v(10:12,2)*100,MoWRe1600_100_v(10:12,3),'o','LineWidth',2)
hold on
plot(MoWRe2000_100_v(:,1)*100,MoWRe2000_100_v(:,2),'s','LineWidth',2)
title('100 mm/s')
grid on
axis([0.4 2.5 0 700])
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('1600 C','2000 C')

subplot(2,1,2)
plot(MoWRe1600_400_v(10:12,2)*100,MoWRe1600_400_v(10:12,3),'o','LineWidth',2)
hold on
plot(MoWRe2000_400_v(:,1)*100,MoWRe2000_400_v(:,2),'s','LineWidth',2)
title('400 mm/s')
grid on
axis([0.4 2.5 0 700])
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('1600 C','2000 C')
sgtitle('Strain vs Stress Vertical 12h')

%45 degree
figure()
subplot(2,1,1)
plot(MoWRe1600_100_45(10:12,2)*100,MoWRe1600_100_45(10:12,3),'o','LineWidth',2)
hold on
plot(MoWRe2000_100_45(:,1)*100,MoWRe2000_100_45(:,2),'s','LineWidth',2)
title('100 mm/s')
grid on
axis([0.4 3.5 0 700])
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('1600 C','2000 C')

subplot(2,1,2)
plot(MoWRe1600_400_45(10:12,2)*100,MoWRe1600_400_45(10:12,3),'o','LineWidth',2)
hold on
plot(MoWRe2000_400_45(:,1)*100,MoWRe2000_400_45(:,2),'s','LineWidth',2)
title('400 mm/s')
grid on
axis([0.4 2.4 0 700])
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('1600 C','2000 C')

sgtitle('Strain vs Stress 45 degree 12h')

%2200C

figure()
subplot(2,1,1)
plot(MoWRe1600_100_v(4:6,2)*100,MoWRe1600_100_v(4:6,3),'o','LineWidth',2)
hold on
plot(MoWRe1600_100_v(7:9,2)*100,MoWRe1600_100_v(7:9,3),'o','LineWidth',2)
plot(MoWRe2200_100_v(:,1)*100,MoWRe2200_100_v(:,2),'s','LineWidth',2)
title('100 mm/s')
grid on
axis([0.4 2.5 0 700])
xlabel('Strain (dimensionless)')
ylabel('Stress (Newton/milimeter^2)')
legend('1600 C 4hr','1600 C 8 hr','2200 C 6 hr')
239
240    subplot(2,1,2)
241
242    plot(MoWRe1600_400_v(3:5,2)*100,MoWRe1600_400_v(3:5,3),'o','LineWidth',2)
243
244    plot(MoWRe1600_400_v(6:8,2)*100,MoWRe1600_400_v(6:8,3),'o','LineWidth',2)
245
246    plot(MoWRe2200_400_v(:,1)*100,MoWRe2200_400_v(:,2),'s','LineWidth',2)
247
248    title('400 mm/s')
249
250    grid on
251
252    axis([0.2 2.4 0 700])
253
254    xlabel('Strain (dimensionless)')
255
256    ylabel('Stress (Newton/milimeter^2)')
257
258    legend('1600 C 4hr','1600 C 8 hr','2200 C 6 hr')
259
260    sgtitle('Strain vs Stress Vertical')
Bibliography


# Investigation Of Additively Manufactured Molybdenum-Tungsten-Rhenium Alloys

## Abstract
The process of creating metal components through additive manufacturing is changing the way different industries can avoid the shortcomings of traditional metal production. Metals such as tungsten, molybdenum, and rhenium have many advantages for different applications, especially when alloyed together. In this study, an additively manufactured alloy containing 70% molybdenum, 25% tungsten, and 5% rhenium (70Mo-25W-5Re) is tested for its strength, ductility, hardness, and porosity. The 70Mo-25W-5Re alloy is printed through Laser Powder Bed Fusion (LPBF) under different conditions such as printing speed and printing atmosphere. Additionally, the effects of post printing heat treatment are conducted to understand the advantages to its property changes. The printed alloys are subject to flexural loading and its physical characteristics are tested and observed. The alloy is found to be stronger at slower printing speeds which corresponds to a greater input energy density. Additionally, heat treatments acted to improve strength but had little effect on porosity or hardness. The benefits of the 70Mo-25W-5Re alloy have a potential for real world applications due to its ease in production. The findings of this research demonstrated how readily alloys of these elements can be studied by leveraging additive manufacturing and post processing heat treatments. This technique will encourage research into different combinations of the constituent elements to find promising compositions in the alloy space.