Towards understanding material characteristics through the additive manufacturing arc

Alden Watts
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Towards understanding material characteristics through the additive manufacturing arc

by

Alden Watts

A thesis submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Materials Science and Engineering

Program of Study Committee:
Peter Collins, Major Professor
   Richard LeSar
   Frank Peters

The student author, whose presentation of the scholarship herein was approved by the program of study committee, is solely responsible for the content of this thesis. The Graduate College will ensure this thesis is globally accessible and will not permit alterations after a degree is conferred.

Iowa State University

Ames, Iowa

2019

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DEDICATION

This document is dedicated to naps and milkshakes, two of the best things in life.
# TABLE OF CONTENTS

ACKNOWLEDGEMENTS ........................................................................................................ V

ABSTRACT ........................................................................................................................ VI

CHAPTER 1.  INTRODUCTION .................................................................................. 1

CHAPTER 2.  LITERATURE REVIEW ...................................................................... 3
  2.1 Additive Manufacturing ..................................................................................... 3
    2.1.1 Introduction .................................................................................................. 3
    2.1.2 The additive manufacturing process ....................................................... 4
    2.1.3 The LENS™ system .................................................................................. 6
  2.2 Titanium .............................................................................................................. 8
    2.2.1 Introduction .................................................................................................. 8
    2.2.2 Basic properties ......................................................................................... 9
    2.2.3 Titanium alloys ......................................................................................... 11
    2.2.4 Titanium production and processing ...................................................... 13

CHAPTER 3.  METHODOLOGY AND EXPERIMENTAL ....................................... 18
  3.1 Powders and Builds .......................................................................................... 18
  3.2 Powder Characterization ................................................................................. 22
    3.2.1 Scanning auger for oxide layer ............................................................... 22
    3.2.2 Particle size distribution ........................................................................... 23
    3.2.3 Flow ......................................................................................................... 23
    3.2.4 Powder morphology ................................................................................ 25
    3.2.5 Differential scanning calorimetry ............................................................ 25
  3.3 Build Characterization ...................................................................................... 25
    3.3.1 Preliminary microtensile ......................................................................... 25
    3.3.2 Microtensile of the LENS™ build parts .................................................. 26
    3.3.3 SEM imaging of the LENS™ build parts ................................................. 27
    3.3.4 EBSD scans of the LENS™ build parts .................................................... 28

CHAPTER 4.  RESULTS AND DISCUSSION ......................................................... 31
  4.1 Powder Characterization ................................................................................. 31
    4.1.1 Scanning auger for oxide layer ............................................................... 31
    4.1.2 Particle size distribution ........................................................................... 32
    4.1.3 Flow ......................................................................................................... 33
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ABSTRACT

The directed energy powder feed additive manufacturing (AM) process fabricates components with good quality, accuracy, and efficiency. However, this is a complex process and without careful consideration can lead to discontinuities in the final product such as porosity, cracks, or unfavorable microstructure. These material discontinuities may occur due to the limited understanding of the relationship between AM source materials and the evolution of the composition and microstructure during the deposition process. This makes it vital to study and correlate the composition, morphology, and characteristics of both the source material and of the final printed parts. In this research the properties of Ti-6Al-4V powders are investigated using methods such as scanning auger microscopy, scanning electron microscopy, various flow measurement methods, differential scanning calorimetry, and electron backscatter diffraction. The properties of LENS™ depositions using these powders are then investigated using tensile testing, scanning electron microscopy, and electron backscatter diffraction.
CHAPTER 1. INTRODUCTION

To my knowledge, in the beginning there was nothing for some amount of time before The Big Bang spread matter through the universe. From this matter stars were born and then died in cycles to spread their star stuff for new stars to be born. From this process titanium was created and spread through the universe. Additive Manufacturing (AM) is by comparison a new invention. Some of the first patents related to AM come from the late 1970′s[1] into the mid 1980′s[2], where it was primarily used in small-scale, research-oriented settings. A brief timeline of the history of AM can be seen in Figure 1.1.

Over the past decade research into this field has boomed, however much of this research has been limited in scope. As materials scientists we are taught early about the “materials tetrahedron,” which has the four corners of structure, processing, properties, and performance. Much of the AM research that has been done has focused on how certain processing parameters, such as material feed rate and heat source motion speed, effect the properties and performance of the final build. This work will strive to investigate each step in the process as thoroughly as possible so as to gain a better understanding of the AM paradigm and its effects on the final product.

It will begin by examining the powders selected for use in the experiments. Attempting to correlate synthesis processes, morphology, flow rates, and chemistry of the powders. It will then move on to characterization of the AM built parts, attempting to correlate the performance and properties of the parts back to the properties of the powders.
This thesis will cover the “inputs” and “outputs” of the AM process, the selection of the powder stock used for the builds and the characterization of the final product. The material chosen for this research was the alloy Ti-6Al-4V. This is because it is one of the most heavily researched materials in not only the AM field, but in general, mainly due to its heavy usage in many different industries because of its outstanding mix of desirable properties. This thesis will begin with a discussion and review of AM and titanium and its alloys, will move on to outline the experimental procedures and methodology used, and conclude with a discussion of the results and conclusions of those experiments.

Figure 1.1 Timeline of the AM process an illustration of the research boom of the 2000's.
CHAPTER 2. LITERATURE REVIEW

2.1 Additive Manufacturing

2.1.1 Introduction

Compared to many other manufacturing processes AM is relatively new, it has only been around for about 30 years. Despite its relative newness, much research has been conducted into AM and the excitement and expectation surrounding it continues to increase. This is because AM has the potential to revolutionize the way many industries view and produce their products. Using AM a part can be shaped to an extremely close approximation of its final geometry with little to no need for final machining compared to other production methods, greatly reducing material waste and processing time[3, 4].

There is such a wide variety of different processes that have been developed to perform AM that a generic definition would be of some use to begin to appreciate the process. Collins put forth this definition that encapsulates all possible processes: “a process in which a local heat source melts both a small volume of the supporting architecture (e.g., substrate or previously deposited material) and an additional volume of new material while the heat source is in constant motion relative to the supporting architecture and newly deposited material[5].” This process can be seen visually in Figure 2.1. From this definition we can see three of the four primary features of an AM system: the heat source, the preform, and the reference for motion. There is also a fourth defining feature of an AM system that is informed by the methods chosen for the first three, which is the build atmosphere.
2.1.2 The additive manufacturing process

The most common heat sources in AM are electron beam, laser, and plasma. Electron beam systems require a vacuum for the electron beam to operate. Electron beam may be used with both a wire feed system or a powder bed. Unlike electron beam, laser systems have more freedom in their build atmosphere, with the decision often based on the properties of the material deposited. In both cases it has been seen that AM can give microstructures and properties on par with cast and wrought products with the proper post-build processing such as heat treatments or hot isostatic pressing[3, 6, 7].

The use of plasma as a heat source in AM is essentially an applied version of plasma arc welding, an arc is formed in the torch and contained by a flow of Ar gas.
blown over the torch head. This is still a relatively unexplored method and so far has been restricted to wire feed systems[8].

There are in essence two types of precursor material, wire and powder. Wire is used as a precursor material by having a system in place to continuously feed the wire into the melt pool formed by the heat source. Powder as a precursor material has two different sub-types. First is powder bed, wherein there is a source of powder that is spread over the build area, the heat source is then used to selectively melt the areas of the powder bed that are necessary for that layer before a brush or wiper recovers the build area with a new layer of powder for the next build layer. The second way powders are used as a preform are in powder blown systems. These involve the blowing of powder directly into the melt pool. In the powder blown and wire feed methods the rate of preform feed is important as there is a delicate balance between feeding too much material and too little due to the effects of overbuilding and underbuilding[9].

In powder based systems the powder morphology is important. If powders are too rough or non-uniform in shape then they may not pack well in the powder bed case or flow well in the powder blown case. This can lead to an increase in defects in the final microstructure due to overbuilding or underbuilding[10].

There are three methods for reference of motion. The first method is where the heat source is in motion and the build area is stationary. The second method is where the build area is in motion and the heat source is stationary. The final method is a hybrid of the first two methods, where both the heat source and build area are both in motion.
The build atmosphere is a choice mostly made based on the needs of the material being worked with. As previously mentioned, electron beam systems require a vacuum, however laser and plasma based systems do not necessarily need to be under vacuum or even an inert atmosphere for some materials.

2.1.3 The LENS™ system

For the purposes of this work I will be focusing on the Laser Engineered Net Shaping (LENS™) system built by Optomec. The LENS™ system was originally conceived by a collaboration between Sandia National Laboratory and Pratt and Whitney, but was licensed to Optomec in 1997[11]. This system consists of a laser that is brought into final focus within a glove box, allowing for the use of an inert gas atmosphere for the build to reduce the likelihood that there is oxygen, hydrogen, or other gaseous elements in the atmosphere that could be scavenged by the melt pool. It has a series of nozzles that blow a supply of powder into the melt pool. The build is secured to a platform within the glove box that moves in the XY plane, while the laser and powder feed nozzles move in the Z direction as the build height increases[12].

![Figure 2.2 An example of a LENS system (left)[13], an example of the build head in a LENS system (right)[14].](image)
Figure 2.2 shows the LENS$^\text{TM}$ system[13] and the build head as it would be seen during a deposition[14].

When performing a build in the LENS$^\text{TM}$ there are 5 parameters that are important to consider. The first is the power of the laser. This will determine how much energy is transferred into the build, which can have a significant effect on whether the powders are able to melt and re-solidify adequately. The second is the speed at which the laser moves across the build. The third is the build hatch width, which measures the space between each pass of the laser on a build layer. The fourth is the layer thickness. The final parameter is the powder feed rate, which is important for making sure that the build is not overbuild or underbuilt.

The first four of the parameters are commonly used together to create the energy density parameter, which is commonly used as a metric in the study of AM samples. Energy density is obtained from the following equation[9]:

$$E = \frac{P}{vlh}$$  \hspace{1cm} \text{Eq. 1}

where P is the laser power, v is the laser travel speed, l is the layer thickness, and h is the hatch width. The energy density can be an excellent metric for determining the occurrence of certain defects, such as porosity or lack of fusion layers[15].

Many of the properties of a finished build are determined by the thermal history of the build. Because of the AM process a specific section of a build may be heated past certain transformation temperatures multiple times during the build process. This unique property of the AM process will cause fluctuations in the cooling rate and thermal
gradients that may lead to non-equilibrium microstructures in the finished build[16, 17]. It can also lead to a build up of residual stress in the part, which can be deleterious to the mechanical properties of the final part[18].

To alleviate some of the problematic properties that the AM process can leave in a material there is often a final heat treatment performed as a final step. This can be done either at an elevated temperature, or at an elevated temperature and pressure. This step helps to relieve some of the stress and to homogenize the microstructure in the built parts[19]. There has also been work done towards fine tuning the processing variables such that the final microstructure is more desirable [20-22].

2.2 Titanium

2.2.1 Introduction

The first steps towards the discovery of titanium were taken in 1791 by a British reverend named William Gregor in Cornwall. He found a strange magnetic black sand near a river which we now know this to be the mineral ilmenite. He managed to remove the iron content with a magnet and purify the remains, but was unable to identify the substance. The story continues in 1795 in Berlin, where a German chemist named Martin Heinrich Klaproth was able to isolate titanium oxide from a piece of rutile he had obtained from Hungary. He named this new element titanium after the Greek Titans of myth.

It was more than century later, in 1910, when Matthew Albert Hunter further advanced titanium metallurgy in America by isolating titanium by heating titanium tetrachloride with Na within a steel container. The product titanium was brittle and small
in quantity, and the process was not yet considered feasible for widespread production. The true breakthrough came in the 1930's when Wilhelm Justin Kroll, a scientist from Luxembourg now widely considered to be the “father of titanium,” developed the Kroll process. Using this process titanium tetrachloride was reduced with Na or Mg in an inert atmosphere. This process produced usable, pure titanium that looked porous and spongy, giving the produce the name “titanium sponge.” The Kroll process has remained mostly unchanged to the present and is still in wide use for titanium production[23, 24].

2.2.2 Basic properties

Titanium has a hexagonal close packed (HCP) crystal structure at room temperature. This is commonly referred to as the α phase. At elevated temperatures titanium undergoes an allotropic transformation to a body-centered cubic (BCC) crystal structure, known as the β phase (Figure 2.3). This allotropic α to β phase transformation occurs at 882±2°C. The β transus temperature is important when

![Crystal structure of both the alpha and beta phases](image)
discussing titanium metallurgy as it is the central feature for the processing of many different alloys where heat treating or mechanical processing at very specific temperatures in relation to the β transus is necessary to engender the desired properties[23].

Alloying elements are broken down into two main categories, α stabilizers and β stabilizers. Common α stabilizers include Al, O, N, and C. Al is a substitutional element for titanium whereas O, N, and C are interstitial. When alloyed with Ti all four elements raise the β transus temperature, stabilizing the α phase.

The β stabilizers are further broken down into two groups, β isomorphous and β eutectic, named for their phase diagrams. β isomorphous elements include V, Mo, and Ta and have a higher solubility in Ti than the β eutectic elements, and with a high enough concentration these elements will stabilize the β phase of Ti to room temperature. β eutectoid forming elements include Fe, Mn, Cr, and Co among others.

![Phase diagram showing the effect of beta stabilizing element concentration](image)
Care must be taken when adding β eutectoid elements as even very low concentrations can lead to the formation of intermetallic phases.

There are also some elements that have effectively no impact on the transition temperature. These elements slightly lower the transition temperature but then at higher concentrations will then raise the transition temperature. Zr, Hf, and Sn all fall into this category of neutral elements[23, 24].

2.2.3 Titanium alloys

Based upon the alloying elements there are three major alloy classifications: α, α+β, and β alloys. These three classifications are broken down by the amount of the β phase present in the alloy as shown in Figure 2.4. An alloy is an α alloy if it has only a small amount of β stabilizing element added to it. The various grades of commercially (CP) titanium are also often grouped into this category. There is also the “near α” classification, that is sometimes used to separate out a certain portion of the alloys in the α+ β region which tend to have desirable properties at high temperature. The line between α+ β and β alloys is more concrete, with alloys achieving at least β phase metastability upon quenching to room temperature being considered β alloys. Special mention should be given the α+β alloy Ti-6Al-4V, which accounts for more than 50% of all titanium alloy usage worldwide[23].

Titanium and many titanium alloys are perhaps most well known for their excellent specific strength and corrosion resistance, especially when compared to other structural alloys such as steels and aluminum alloys. The excellent corrosion resistance of titanium is caused by a TiO₂ layer that quickly passivates the material when titanium
is exposed to oxygen[24]. This layer protects from many deleterious environments such as chlorides, hypochlorites, sulfates, sulfites, and many acidic environments. Exceptions to this exist with sulfuric, hydrochloric, phosphoric acids. Hydrofluoric acid in particular will readily attack titanium and dissolve the oxide layer, for this reason hydrofluoric acid mixtures are often used to chemically etch, mill, and pickle titanium[25, 26].

One other property that all titanium alloys share is that their properties dependence on their processing history and composition[24]. The final microstructure in any titanium alloy will be heavily dependent on the type and scale of processing used in the production process for that specific product. Given how dependent properties are on microstructure this means that two samples of the same titanium alloy may have different properties if they were processed in different ways. This can be seen in Figure 2.5 Tensile elongation vs cooling rate for 3 different Ti-6Al-4V processing routes[24].
2.5 with the tensile elongation of Ti-6Al-4V using different processing routes and cooling rates. We can see specifically that even at similar cooling rates the lamellar and bimodal elongations are significantly different[24].

2.2.4 Titanium production and processing

Traditionally, titanium products are largely produced in two ways: casting and forging. In recent years AM has been investigated as a production method, a topic that is covered in greater detail elsewhere in this thesis. Casting involves the melting of titanium sponge and any alloying stock, the liquid metal is then poured into a mold to the desired final shape. This casting is then machined down further to remove any surface defects due to the casting process. In forging the melted material is cast into an ingot or bulk mold. This material is then shaped through thermomechanical processing into the desired final shape[24, 27-29].

Generally, titanium alloys go through roughly the same processing steps (Figure 2.6). The primary differences will be the temperatures each step are taken at and the

![Figure 2.6 An example of a processing route used in lamellar α+β alloys, highlighting the role that the temperature plays in the deformation phase and the cooling rate plays in the recrystallization phase[24].](image)
heating or cooling rate used to get to and from those temperatures. The first processing step is homogenization. This step involves heat treating the alloy at a certain temperature in order to ensure the microstructure and chemistry of the alloy are homogenous throughout. The next step is deformation, where the alloy will be deformed into a shape, often a sheet or bar. This process breaks up the grains formed during homogenization and will give them a preferential texture. The third step is recrystallization. This step is similar to the homogenization step, but is typically performed at a lower temperature, below the β transus, or it may be skipped entirely depending on the desired end microstructure. The purpose of this step is to relieve the stress and regrow the grains from the deformation step and the time spent in recrystallization is key in determining the grain size in the final microstructure. The final step is annealing or aging. This step is another heat treatment step and is performed at an even lower temperature than recrystallization to allow certain microstructural features to preferentially form in the final microstructure[24].

For CP titanium and α alloys most of the processing that is done is to obtain a desirable texture or grain size in the final microstructure, rolling into sheet or bar stock for example, to take advantage of the anisotropy of the HCP α phase microstructure. Because of the low amount of alloying additions in α alloys they do not respond as greatly to thermomechanical processing as β or α + β alloys, which are both heavily processed to achieve the final microstructure. For this same reason, α alloys are also more weld-able than β or α + β alloys. Their microstructures are simpler and the fewer alloying additions leads to less phase segregation, which can be deleterious for weld strength. One other unique property of CP titanium and α alloys are their cold-
workability. These two properties, their ability to be welded and cold-worked, are the two major advantages that CP titanium and α alloys have over β alloys and the more widespread α + β alloys[24, 30].

In contrast to CP titanium and α alloys, where the alloys would be homogenized, deformed into shape, and then possibly recrystallized, many titanium alloys respond well to thermomechanical processing and the final properties of these alloys vary greatly depending on the processing route taken during production. There are four main processing routes taken for β alloys: β annealed, β processed, through-transus processed, and bimodal. In general, the α phase prefers to nucleate as a continuous layer at the β grain boundaries. This is particularly important in the β annealed route. Each step in these processes has a different effect on the final properties of the alloy depending on the temperature it is performed at in relation to the β transus, the heating or cooling rate of the step, as well as the length of time the step is performed[31].

Like β alloys, α + β alloys respond well to thermomechanical processing and the final properties of these alloys are heavily dependent on the microstructure that is given by the chosen processing route. There are three primary microstructures possible in α + β alloys, these are: lamellar, bimodal, and fully equiaxed[24].

The lamellar microstructure is characterized by many small platelets or laths of α phase that are suspended in a matrix of β phase, which is primarily located between α grains or laths. One of the most important steps in the formation of the lamellar microstructure is the cooling rate during the recrystallization step as this determines the size of the α laths and α colonies, which are important factors in determining slip length. Smaller slip lengths lead to better yield strength, so it may be advantageous to increase
the cooling rate to decrease the size of the α colonies. This relationship is not limited to
the lamellar microstructure, all three of the α + β microstructures benefit from this effect
in their own ways[32].

The critical feature in the bimodal microstructure is the β grain size. The
important processing parameters for this microstructure are the cooling rate from the β
phase field during the homogenization step, the deformation step, and the annealing
temperature in the α + β phase field during the recrystallization step. By slowing the
cooling rate during homogenization large β grains are allowed to grow within the
microstructure, these beta grains are then deformed but not broken during the
deflection step, and then by annealing high within the α + β phase field, just below the
β transus, α lamellae are allowed to grow within the large β grains. By finely controlling
these steps the result is α grains in a matrix of α + β lamellae[24].

One other consideration when dealing with the bimodal microstructure is the
issue of the alloying element partitioning effect where alloying elements will partition into
separate phases. In α + β alloys this happens with α stabilizers preferring the α phase
and β stabilizers preferring the β phase. As the alloy is cooling down the α stabilizing
elements will preferentially partition into the alpha phase as it is nucleating, this will
leave the beta phase rich in the β stabilizing elements but very poor in the α stabilizing
elements. In the bimodal microstructure this means that the α lamellae that are growing
within the β grains during the recrystallization step are going to be poor in the α
stabilizing elements compared to other α areas. This phenomenon is not strictly limited
to the bimodal microstructure, as it has been noted in other microstructures produced
via AM[33].
As the name suggests the fully equiaxed microstructure consists of equiaxed α grains within a β matrix, however unlike in the bimodal microstructure there is no consideration given in processing to enlarging the β grains. There are two processing routes used to get the fully equiaxed microstructure, the first being similar to that used in the bimodal microstructure but to slow down the cooling rate in the recrystallization step to allow for the α grains to grow rather than the α lamellae in the bimodal microstructure. The second processing route used to create the fully equiaxed microstructure is to perform the recrystallization step at a low enough temperature that the α phase is formed fully equiaxed from the deformed microstructure[32].

As with the fully lamellar and bimodal microstructures the fully equiaxed microstructure’s properties are driven largely by the slip length, which is driven by the α grain size[24, 34, 35]. This is similar to the behaviors of some fully lamellar α alloys, such as CP titanium, where α colony size is a driving factor in the properties[24].
CHAPTER 3. METHODOLOGY AND EXPERIMENTAL

3.1 Powders and Builds

In order to investigate any differences caused by powder manufacturing methods in AM builds, a total of 15 Ti-6Al-4V powders from 5 different manufacturers were purchased for use in the project. Each manufacturer used a different process that involved melting the source material via some applied heat source and then rapid cooling of the molten material to form powder. The complete list of powders used in this work can be found in Table 3.1.

The AP&C and Puris powders were produced via plasma wire atomization. In this process a wire is fed into a sealed, high purity Ar atmosphere and superheated by plasma torches. The resulting melt is then rapidly cooled and forms into a fine powder, which is then removed from the chamber to reduce the amount of satellite particles that can attach to newly formed particles (Figure 3.1)[36].

The Hoeganaes powders were produced via the Electrode Induction Gas Atomization (EIGA) process. This process is performed under an inert atmosphere and

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Sizes</th>
<th>Grades</th>
<th>Production Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP&amp;C</td>
<td>+10/-45µm</td>
<td>5</td>
<td>Plasma Wire Atomization</td>
</tr>
<tr>
<td></td>
<td>+45/-106µm</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>Hoeganaes</td>
<td>+10/-45µm</td>
<td>5</td>
<td>EIGA</td>
</tr>
<tr>
<td></td>
<td>+50/-100µm</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>Praxair</td>
<td>+20/-43µm</td>
<td>5</td>
<td>VIM-AGA</td>
</tr>
<tr>
<td></td>
<td>+20/-45µm</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td></td>
<td>+53/-106µm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Timet*</td>
<td>-/-45µm</td>
<td>-</td>
<td>PREP</td>
</tr>
<tr>
<td></td>
<td>+45/-106µm</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Puris</td>
<td>+45/-109</td>
<td>5</td>
<td>Plasma Wire Atomization</td>
</tr>
</tbody>
</table>

* - Timet did not have the capability in their facilities to certify what the lower bounds of their powder size would be, nor did they provide information about the grade of powder they manufactured.
involves a large rod or bar of material is used as an electrode and fed into a series of induction coils while being constantly rotated. The resulting melt is then moved through a series of gas nozzles to atomize the melt and allow it to cool into powder (Figure 3.1)[37].

The Praxair powder was produced via Vacuum Induction Melt Argon Gas Atomization (VIM-AGA)(Figure 3.2). In this process the material is first melted in a

![Figure 3.1 Illustrations of the Plasma Wire Atomization (left)[34] and EIGA (right)[35] methods.](image1)

![Figure 3.2 Illustrations of the VIM-AGA (left)[37] and PREP (right)[38] methods.](image2)
vacuum induction furnace and then poured into a series of gas jets to atomize the powder. This is similar to the EIGA process, however, because the EIGA process involves melting the material directly into the gas jets there is no interaction with any ceramic liners like with the VIM-AGA process, which sometimes leads to contamination of the produced powders[37-39].

The Timet powder was produced via the Plasma Rotating Electrode Process (PREP)(Figure 3.2). This process is similar to the EIGA process but the source material is melted via plasma instead of electrode induction[37, 40].

From the 15 purchased powders four were chosen to make initial builds for investigation. All such chosen powders were in the +45/-106 size range to accommodate the recommendations set by Optomec for use in LENS™ systems. The grade 23 powders from Hoeganaes and Praxair as well as both AP&C grades were chosen to give an overview of both different powder manufacturers as well as different powder grades. From each chosen powder two builds were deposited, one at a power that had been investigated previously, and one at a significantly lower power in order to investigate any effect energy density may play in the final microstructure and properties of the builds. All other parameters were the same across all 8 builds in order to investigate any variation in the finished build that may be caused by the difference in energy density between the power settings used. All builds were conducted using the Optomec LENS™ MR-7. A list of build parameters can be found in Table 3.2.
Table 3.2 Parameters used in the LENS depositions.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Low Power</th>
<th>High Power</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>250 W</td>
<td>330 W</td>
</tr>
<tr>
<td>Speed</td>
<td>25 in/min</td>
<td>25 in/min</td>
</tr>
<tr>
<td>Powder Feed</td>
<td>3 g/min</td>
<td>3 g/min</td>
</tr>
<tr>
<td>Hatch Width</td>
<td>0.381 mm</td>
<td>0.381 mm</td>
</tr>
<tr>
<td>Layer Thickness</td>
<td>0.254 mm</td>
<td>0.254 mm</td>
</tr>
<tr>
<td>Energy Density</td>
<td>206 J/mm$^3$</td>
<td>293 J/mm$^3$</td>
</tr>
</tbody>
</table>

Each build used a grade 2 CP titanium build plate to ensure bonding of the build material to the build plate. The builds had a footprint of 1 in. by 2 in. and were ¾ in. tall (Figure 3.3). The laser scan path was a simple back and forth strategy, however it was rotated by 45° in following best practice for allow for an even deposition of material.
Following the build the deposited blocks were removed from the build plates using a high speed saw in Metals Development.

3.2 Powder Characterization

3.2.1 Scanning Auger Microscopy for oxide layer thickness

Scanning Auger Microscopy was performed on the powders using the JEOL JAMP-7830F Auger Microprobe. The primary purpose of these experiments was to determine the oxide layer thickness of the various powders. This was achieved by measuring the levels of O₂ in the powder particles as they were etched away by an argon ion beam, when the measured O₂ levels evened out (blue line in Figure 3.4) it was assumed the oxide layer had been transited and the oxide layer thickness was calculated using the etching rate and the elapsed time. For each powder multiple sites were investigated across 5 to 8 particles and the results were averaged. The etching

![Figure 3.4 Readout of C, Ti, and O levels in the scanning auger runs.](image)
rate of TiO$_2$[26] was used to approximate the etching rate of the oxide layer of the Ti-6Al-4V powders to determine the oxide layer thickness based on the etching time.

3.2.2 Particle size distribution

In order to confirm the particle size distribution data provided by the manufacturers two methods were implemented to measure the particle size distribution of the as received powders.

First, the powders were fed into a Microtrac automated particle size distribution analyzer. The powders were suspended in deionized water and then passed in front of a laser and the subsequent laser diffraction angles were used to measure the size of the particles.

Second was direct measurement using scanning electron microscope (SEM). The powders were mounted in a conductive Bakelite puck and then gently ground down using 600 grit SiC abrasive discs in order to get a cross sectional image of the powders. The images were taken at the Sensitive Instruments Facility (SIF) at Ames Lab on the FEI Teneo LoVac FE-SEM using an accelerating voltage of 15 kV, current of 0.8 nA, and a working distance of approximately 10 mm. Using these images and the Materials Image Processing and Automated Reconstruction (MIPAR) image analysis software it was possible to obtain measurements of the size and spheroidicity.

3.2.2 Flow

Similar to the particle size distribution measurements, two separate methods were used to measure the flow rates of the powders.
The first method was the Hall Flow standard. All tests were conducted under ambient conditions. For each powder 50 g was measured out and placed into a Hall Flowmeter (Figure 3.5) funnel and the time taken for the powder to completely empty out of the funnel was measured. In the event that a powder would not flow, one light tap to the funnel was permitted to allow the powder to start flowing in accordance with ASTM B213-17. Each powder was measured 6 times and averaged to come to the final measurement.

The second method was conducted using the LENS™ to weigh how much powder would flow through the system per minute using different RPM settings. This was achieved by loading the hoppers on the LENS™ with powder as normal, however the line that delivered the powder into the glovebox chamber of the LENS™ was disconnected at the solenoid valve right before the glovebox chamber and was instead funneled into a plastic bag. This method was only performed on the four powders chosen for initial LENS™ depositions, and each powder was allowed to run for 3
minutes per RPM setting on the *LENS*™. These bags were then weighed to determine how much grams of powder are flowing through the system per minute.

### 3.2.4 Powder morphology

To investigate the surface morphology of the as received powders SEM imaging was performed. Samples were prepared by placing a tab of carbon adhesive tape onto a 12.7 mm aluminum mounting stub and then gently pressing a small amount of powder onto the carbon tape in order to avoid any damage to the powders during the mounting process. The SEM analysis was performed using the same conditions as noted previously.

### 3.2.5 Differential scanning calorimetry

Differential Scanning Calorimetry (DSC) was performed on the powders and LENS builds using the Nietzsch STA449 F3 Jupiter auto-changing differential scanning calorimeter. The samples were placed in alumina crucibles and the experiments were performed under a helium atmosphere with a heating rate of 10°C/min.

### 3.3 Build Characterization

#### 3.3.1 Preliminary microtensile

Due to constraints of the available load cell and grips for tensile testing requiring a non-standard dimension for the planned tensile tests it was decided to perform a series of preliminary tensile tests on wrought Ti-6Al-4V plate. The plate was given to the Ames Lab machine shop to be electron discharge machined (EDM) into flat dog-
bone shapes. A series of 9 microtensile tests were then conducted on the Zwick Roell tensile machine using a 2.5 kN load cell (Figure 3.6).

3.3.2 Microtensile of the LENS\textsuperscript{TM} built parts

To get tensile specimens machined from the LENS\textsuperscript{TM} builds they were sectioned on a low-speed diamond saw and then portions were sent to the Ames Lab machine shop to be machined into tensile specimens via electrical discharge machining (EDM) (Figure 3.7). These specimens were then ground down using 400 and 600 grit SiC grinding pads in order to remove the EDM recast layer. Roughly 50 mµ were removed from each side in order to ensure the recast layer was completely removed\cite{41}. These were then tested using the Zwick Roell tensile testing machine using a 2.5 kN load cell. For each build 6 tensile test were performed and the results averaged to obtain information about the mechanical properties of each build.
3.3.3 SEM imaging of the LENS™ built parts

In order to examine the microstructure of the LENS™ builds SEM imaging was performed on a sample from each build. A section was sliced from the center of each build parallel to the ZY plane using a low-speed diamond saw. This section was then further cut down into three pieces from the edges and center of the builds (Figure 3.8). These pieces were mounted in a conductive Bakelite mounting powder and taken from 400 to 1200 grit SiC grinding pads. The samples were then polished using 0.04 µm colloidal silica for 30 minutes, etched using Kroll’s Reagent for 10 seconds, and then polished again using 0.04 µm colloidal silica for 30 minutes. Finally, the samples were placed on a Buehler VibroMet 2 vibratory polisher for 16 hours at 60% maximum
vibration amplitude. Imaging was performed using an FEI Teneo LoVac FE-SEM using an accelerating voltage of 10 kV and current of 0.8 nA at a working distance of approximately 10 mm. For each build, locations from within nine areas were imaged according to Figure 3.9 in order to determine any difference in microstructure due to the location in the sample in the ZY plane.

3.3.4 EBSD scans of the LENS™ built parts

Electron backscatter diffraction (EBSD) was performed on a sample from each build to ascertain the texture of the builds. Samples were prepared by sectioning a portion parallel the ZY plane using a low-speed diamond saw. The samples were then mounted to a flat stage using crystal bond and taken from 400 to 1200 grit SiC grinding pads. The samples were then polished using 0.04 µm colloidal silica for 30 minutes, etched using Kroll’s Reagent for 10 seconds, and then polished again using 0.04 µm
colloidal silica for 30 minutes. Finally, the samples were placed on a Buehler VibroMet 2 vibratory polisher for 16 hours at 60% maximum vibration amplitude. After this the samples were cleaned and mounted onto a 12.7 mm aluminum mounting stub using conductive silver paint.

The EBSD scans were performed using the FEI Teneo LoVac FE-SEM’s Oxford EBSD detector using an accelerating voltage of 20 kV, current of approximately 2.0 nA, and a step size of 0.30 µm[42].

After these scans were completed the samples were cleaned using acetone to remove the conductive silver paint and then re-etched using Kroll’s Reagent for 13 seconds in order to preform optical microscopy to confirm the shapes of the grains.
Throughout this chapter there have been references to numerous machines and pieces of equipment used in the various experiments for this thesis. I have included a table for these here with additional information about their locations.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>LENS™</td>
<td>Metals Development</td>
</tr>
<tr>
<td>JEOL JAMP 7830 F Scanning Auger Microscope</td>
<td>Wilhelm Hall</td>
</tr>
<tr>
<td>Nietzsch STA 449 F3 Jupiter Auto-changing DSC</td>
<td>Wilhelm Hall</td>
</tr>
<tr>
<td>Zwick Roell tensile machine</td>
<td>Metals Development</td>
</tr>
<tr>
<td>FEI Teneo LoVac FE-SEM</td>
<td>Sensitive Instruments Facility</td>
</tr>
</tbody>
</table>
4.1 Powder Characterization

4.1.1 Scanning Auger Microscopy for oxide layer thickness

The measured values showed some variance between powder manufacturer, and some minor variance between grades of the same manufacturer, but the differences were generally negligible. As Figure 4.1 demonstrates, the values tended to range between 5nm and 8nm. There were three major outliers to these results, the Hoeganaes +10/-45 grades 5 and 23 powders had an oxide layer thickness of
approximately 14 nm and 11 nm respectively, and the Puris powder had an oxide layer thickness of approximately 17 nm.

4.1.2 Particle size distribution

In both methods the results showed that the particle size distribution of all powders was completely in line with the manufacturers reported values.

Additionally, Table 4 shows the mean eccentricity of each powder, used as a measure of powder spheroidicity. The closer a powder is to 1 according to this method, the less spherical the powder is, and a value of 0 would indicate a perfectly spherical particle. Each manufacturer had their powders fall into a similar range, with the Hoeganaes and Praxair powders being roughly 0.5, whereas the AP&C powders were roughly at 0.3. This means that the AP&C powders were significantly more spherical than the Hoeganaes and Praxair powders, only the Timet +45/-106 powder compared in terms of spheroidicity.

Table 4.1 Mean powder eccentricity values obtained via MIPAR analysis.

<table>
<thead>
<tr>
<th>Powder</th>
<th>Mean Eccentricity</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP&amp;C +10/-45 Gr 5</td>
<td>0.36</td>
</tr>
<tr>
<td>AP&amp;C +10/-45 Gr 23</td>
<td>0.33</td>
</tr>
<tr>
<td>AP&amp;C +45/-106 Gr 5</td>
<td>0.3</td>
</tr>
<tr>
<td>AP&amp;C +45/-106 Gr 23</td>
<td>0.32</td>
</tr>
<tr>
<td>Hoeganaes +10/-45 Gr 5</td>
<td>0.5</td>
</tr>
<tr>
<td>Hoeganaes +10/-45 Gr 23</td>
<td>0.45</td>
</tr>
<tr>
<td>Hoeganaes +50/-100 Gr 5</td>
<td>0.53</td>
</tr>
<tr>
<td>Hoeganaes +50/-100 Gr 23</td>
<td>0.44</td>
</tr>
<tr>
<td>Praxair +20/-45 Gr 5</td>
<td>0.49</td>
</tr>
<tr>
<td>Praxair +20/-43 Gr 23</td>
<td>0.56</td>
</tr>
<tr>
<td>Praxair +53/-106 Gr 5</td>
<td>0.5</td>
</tr>
<tr>
<td>Praxair +53/-106 Gr 23</td>
<td>0.5</td>
</tr>
<tr>
<td>Timet -/-45</td>
<td>0.42</td>
</tr>
<tr>
<td>Timet +45/-106</td>
<td>0.29</td>
</tr>
<tr>
<td>Puris +45/-109 Gr 5</td>
<td>0.4</td>
</tr>
</tbody>
</table>
4.1.3 Flow

Under the Hall Flow standard most of the +10/-45 μm powders did not flow at all. Only the AP&C +10/-45 μm grade 23 and the Timet -/-45 μm powders flowed, and each of these showed a significantly higher flow time than the larger powder of their type. Among the +45/-106 μm powders all of them flowed. There was no significant difference between the different grades in the flow time, however it was seen that the AP&C and Timet powders had significantly better flow times than the Praxair and Hoeganaes powders (Figure 4.2). This correlates well with the spheroidicity data, where it was seen that the AP&C and Timet powders were significantly more spherical than the Hoeganaes or Praxair powders.

Figure 4.2  Flow rates for various powders. The chart on the left shows the flow rates under the Hall Flow standards, the chart on the right shows the flow rates as measured in the LENS™.
However, in the tests done using the LENS™ it was observed that the Hoeganaes, Praxair, and AP&C grade 23 powders all had similar measurements of about 6 grams per minute. Whereas the AP&C grade 5 powder had a significantly lower measurement of 4.4 grams per minute, at odds with the Hall Flow measurement where the two grades of AP&C powder were only separated by 0.2 seconds.

4.1.4 Powder morphology

As can be seen in Figure 4.3, the AP&C powders largely had the same characteristics across powder size and grade. All were mostly smooth and spherical with some sections where the martensitic microstructure of the powders were noticeable on the surface, as well as some satellite particles that were mostly significantly smaller than the host particle. There were some occurrences of either misshapen or deformed particles. These mostly fell into two categories: those that were oblong but still mostly smooth, and those that appeared to be deformed due to impact by another particle while still cooling during the synthesis process.

As can be seen in Figure 4.4, the Hoeganaes powders were rough and spherical. Like the AP&C powders there was a significant amount of satellite particles, but the Hoeganaes satellites were larger, rougher, and in the grade 23 powders they were mostly flat or oblong shaped. Special mention should be given to the +50/-100 grade 23 powder which showed an extreme amount of satellite particles of varying sizes and morphology. There were also instances of completely misshapen particles with little to no spheroidicity. Additionally, there appeared to be particles that were either hollow or had holes punctured in them by collisions with other particles, especially in the two larger powders.
Figure 4.3  SEM images of the AP&C powder morphologies. a) +10/-45 Grade 5, b) +45/-106 grade 5, c) +10/-45 grade 23, d) +45/-106 grade 23.

Figure 4.4  SEM images of the Hoeganaes powder morphologies. a) +10/-45 Grade 5, b) +50/-100 grade 5, c) +10/-45 grade 23, d) +50/-100 grade 23.
Figure 4.5 SEM images of the Praxair powder morphologies. a) +20/-45 Grade 5, b) +53/-106 grade 5, c) +20/-45 grade 23, d) +53/-106 grade 23.

Figure 4.6 SEM images of the Timet and Puris powder morphologies. a) Timet -/-45, b) Timet +45/-106, c) Puris +45/-109 grade 5.
As seen in Figure 4.5, the Praxair powders were rough and somewhat spherical. There was a higher incidence of non-spherical particles compared to the AP&C and Hoeganaes powders. There were some satellite particles similar to those in the AP&C and Hoeganaes powders, however many of them were more solidly connected to the host particle than in the other powders where the satellites appeared to be only lightly tacked on. Similar to the Hoeganaes powder there were some particles that seemed to be hollow or have had holes punctured in them.

As seen in Figure 4.6a and b, the Timet powders were similar in surface morphology to the AP&C powders. They were mostly smooth and spherical, however they had very few satellite particles. There were portions of the particles where the martensitic microstructure of the particle was visible on the surface. There were a few instances of misshapen particles and particles that were deformed while still partially cooled.

As seen in Figure 4.6c, the Puris powder was mostly round and rough. There was satellite particles there were similar to those in the Praxair powders, where they were more solidly attached than in the other powders. Additionally there were many large and oblong satellite “layers” and misshapen or cracked particles.

4.1.5 Differential scanning calorimetry

Examination of the heating curves shows that while there is some variance in the magnitude of the endothermic peaks associated with the α to β transition temperature, each set of low and high energy density builds has the peaks at the same temperature range (Figure 16). However, the powders tended to have their peaks shifted up to
slightly higher temperatures, except in the Hoeganaes powder where the transition happened at a lower temperature. It was also the trend that the lower energy density builds took more heat in order to begin the transition than the higher energy density builds. The perturbations in the AP&C Grade 23 powder curve are likely caused by an environmental anomaly at the time of testing, but the overall shape of the curve is still apparent.

Figure 4.7 DSC heating curves for the builds and selected powders during the alpha/beta transition temperature.
4.2 Build Characterization

4.2.1 Builds

Of note about the LENS™ builds in general is that for the duration of this work with the builds lack of fusion layers were found at all heights throughout each as they were sectioned to create the various samples for use in experimentation (Figure 4.8). Closer examination of Figure 3.8 near the bottom middle of the build will reveal that lack of fusion layers there were beginning to show.
4.2.2 Preliminary microtensile

There was a notable variance in the 9 microtensile tests that were performed on the wrought Ti-6Al-4V. The averages for yield strength and ultimate tensile strength were both in line with prior data on wrought Ti-6Al-4V, however the percent elongation of the runs were less than that of literature values of Ti-6Al-4V and the elastic moduli were significantly higher in the microtensile tests than values found in literature (Figure 4.9)[23, 25]. The disparities in the elastic modulus could be caused by the small sample
geometry in relation to the average grain size in the samples or by some problem with
the Zwick Roell machine.

4.2.3 Microtensile of the LENS\textsuperscript{TM} built parts

It was found that there was little difference in the average yield strength or
ultimate tensile strength between the high and low energy density builds, typically less
than 5\%, or between builds done using different powders. However, there was a
significant difference in both the elongation to fracture and the elastic modulus, both
between high and low energy density and between powder types (Figure 4.10 and
Figure 4.11. The average elongation at fracture of the samples varied between 1% and 5%. Between the low energy density and higher energy density builds all but the Praxair samples varied significantly, by 30% to 40%. The Praxair builds only varied between the low energy density and high energy density samples by 10%. The average elastic modulus had the same problem as in the preliminary microtensile experiments where there was a high amount of scatter in the experimental data. Even when averaged the data skewed very high, about 140 GPa, with only the high energy density Praxair build coming close the accepted value of 113 GPa. These results are consistent with the values obtained for the yield strength and ultimate tensile strength from other research into the tensile properties of non-heat-treated Ti-6Al-4V parts produced via AM[43].
4.2.4 SEM imaging of the LENS™ built parts

All of the builds exhibited the same microstructures. These were primarily the classical α martensite that Ti-6Al-4V is known for. Examples of this can be seen in Figures 4.12, 4.13, and 4.14 for the AP&C grade 5 HED build. There were some interesting features that were noted in some sections of the microstructure. In Figure 4.15 we can see examples of both the classical α martensitic microstructure (circled in blue) as well as an odd feature noticed in the builds (circled in red). It was observed that there were some faulted α laths in the builds. This could have been caused by the stress induced by the β to α transition. Alternatively, it may have been induced by necessary β growth due to alloying element partitioning during cooling.

There was also evidence of some β phase precipitation in the α laths (Figure 4.16). This has been observed before in Ti₃Mo[14] and in similarly non-heat-treated AM Ti-6Al-4V[43].

Additionally there was no observed porosity in the samples outside of the layers where the lack of fusion defects were noted on the bulk samples. Analysis of the average lath widths via MIPAR yielded no discernable pattern for any differences in lath widths across locations within a sample or between different builds.
Figure 4.12 SEM images of the AP&C grade 5 HED builds across 3 different latitudes for the left portion of the build. Top, middle, and bottom in order from the top.
Figure 4.13 SEM images of the AP&C grade 5 HED builds across 3 different latitudes for the center portion of the build. Top, middle, and bottom in order from the top.
Figure 4.14  SEM images of the AP&C grade 5 HED builds across 3 different latitudes for the left portion of the build. Top, middle, and bottom in order from the top.
Figure 4.15  SEM image of a portion of the AP&C grade 5 HED build from the bottom right portion of the build.
Figure 4.16 SEM image of the AP&C grade 5 HED build from the top right portion of the build.
4.2.5 EBSD scans of the LENS™ built parts

In many of the samples there appeared to be columnar prior-β grains that showed strong texturing effect between these grains. The one exception being the AP&C Grade 23 HED build, which appeared to have well equiaxed grains (Figures 4.17 – 4.24). Further investigation by optical microscopy confirmed this observation. The AP&C Grade 23 HED build had an equiaxed grain structure throughout the sample, whereas the other 7 builds all had columnar prior-β grains throughout as has been reported in many other works.[9, 18, 33, 44]

However, in all samples there appeared to be preferential texturing in the basal (0001) plane. The texture appeared to be deflected roughly 30-45° off of center, which may have been caused by lattice deformation during the β to α transition during cooling. In all other planes there seemed to be a more even texture.
Figure 4.17 EBSD map of the AP&C Grade 5 HED build
Pole Figures

Builds: AP&C L Site 1 Map Data
Ti (6/mmm)
Complete data set
774014 data points
Equal Area projection
Upper hemisphere
Half width: 10°
Cluster size: 10°
Exp. densities (mud):
Min = 0.00, Max = 20.67

Figure 4.18 EBSD map of the AP&C Grade 5 LED build
Figure 4.19 EBSD map of the AP&C grade 23 HED build
Figure 4.20  EBSD map of the AP&C grade 23 LED build
Figure 4.21 EBSD map of the Hoeganaes HED build
Figure 4.22 EBSD map of the Hoeganaes LED build
Figure 4.23 EBSD map of the Praxair HED build
Figure 4.24  EBSD map of the Praxair LED build
Figure 4.25 Optical images of the AP&C grade 5 HED (left) and LED (right) builds

Figure 4.26 Optical images of the AP&C grade 23 HED (left) and LED (right) builds
Figure 4.27  Optical images of the Hoeganaes HED (left) and LED (right) builds

Figure 4.28  Optical images of the Praxair HED (left) and LED (right) builds
CHAPTER 5. CONCLUSIONS AND FUTURE WORK

- A broad approach was applied to the investigation of the properties of the AM of Ti-6Al-4V. By characterizing the material at each step in the AM process a better understanding of the effects of each step were gained.

- It was shown the effect that powder selection can have on the final build properties, especially the surface morphology and powder sizes. We observed that the smaller powders either did not flow or flowed exceptionally slowly compared to the larger powders. Additionally it was seen that the powders with the smoothest surface morphology and most spheroidicity, the AP&C and Timet powders, also had the best flow rates under the Hall Flow standard. This did not necessarily transfer to flow in the LENS however, as the AP&C grade 5 powder had a markedly lower flow rate compared to the other powders tested in such a manner.

- It was also seen that the yield and ultimate tensile strength of the LENS builds met or exceeded both the bench marks set by the preliminary tests and known literature values. However there was a marked decrease in the elongation to failure. Although both of these results have been seen in previous work into the subject.

- While the microstructure of the LENS builds was the typical α martensite, it was seen through EBSD that there was some preferential texturing in the Z direction of the builds, possibly as an artifact of the columnar β grain growth. There was also some interesting features in
some of the α laths that may be due to either stress from the processing route or alloying element partitioning.

- It was shown that the powder production method, grade, or the power settings used in the LENS™ did not have noticeable effects on the microstructures or mechanical properties of the final built parts.
- There is, however, much work to be done, especially in the areas of flow, mechanical properties, and heat treatments. The AM field would greatly benefit from a flow test that was designed specifically for it, as right now it is relying on methods that were not created with AM in mind. Due to the great deal of variance in the individual tensile tests more data, either from more tensile tests or from a secondary method, would help with clarifying the tensile properties of the builds. And finally, the logical next step is to characterize the builds after a series of heat treatments and annealing steps have been performed.
REFERENCES


