Effect of oxygen concentration in build chamber during laser metal deposition of Ti-64 wire

EYVIND ENGBLOM
Abstract

Additive manufacturing of titanium and other metals is a rapidly growing field that could potentially improve component manufacturing through optimization of geometries, less material waste and fewer process steps. Although powder-based additive manufacturing processes have so far been predominant, methods using a wire as feedstock has gained popularity due to faster deposition rates and lower porosity in deposited material. The titanium alloy Ti-6Al-4V accounts for the majority of aerospace titanium alloy consumption and as titanium is a precious and expensive resource, reducing material waste is an important factor.

Laser metal deposition with wire (LMD-w) is currently used in production at GKN Aerospace in Trollhättan. One important process parameter is the oxygen level in the chamber during deposition as titanium is highly reactive with oxygen at process temperatures. Oxygen enrichment of titanium can cause embrittlement and reduced fatigue life due to formation of alpha-case, an oxygen enriched region directly beneath the surface. The oxygen level in the chamber is controlled through extensive use of protective inert gas which is a costly and time-consuming practice. The objective of this thesis was to study how elevated oxygen levels in the chamber would affect surface oxidation, chemical composition, tensile properties and microstructure.

Two different sample geometries were built with Ti-6Al-4V wire at an oxygen level of 100, 500 and 850 ppm. The subsequent analysis was based around microstructural features, alpha-case formation, chemical composition in surface layers, and tensile tests. Results showed that elevated oxygen levels in the build chamber did not degrade the chemical composition or tensile properties with regard to aerospace specifications. However, significant layers of alpha-case were found in all samples indicating that subsequent processing such as machining or etching is needed.
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# List of Abbreviations

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<th>Abbreviation</th>
<th>Full Form</th>
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<tbody>
<tr>
<td>AM</td>
<td>Additive manufacturing</td>
</tr>
<tr>
<td>LMD</td>
<td>Laser metal deposition</td>
</tr>
<tr>
<td>LMD-w</td>
<td>Laser metal deposition with wire</td>
</tr>
<tr>
<td>LMD-p</td>
<td>Laser metal deposition with powder</td>
</tr>
<tr>
<td>SLM</td>
<td>Selective laser melting</td>
</tr>
<tr>
<td>EBM</td>
<td>Electron beam melting</td>
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<tr>
<td>DMLSS</td>
<td>Direct metal laser sintering</td>
</tr>
<tr>
<td>LENS</td>
<td>Laser engineered net shaping</td>
</tr>
<tr>
<td>WAAM</td>
<td>Wire arc additive manufacturing</td>
</tr>
<tr>
<td>TRL</td>
<td>Technology readiness level</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate tensile strength</td>
</tr>
<tr>
<td>YS</td>
<td>Yield Strength</td>
</tr>
<tr>
<td>ICP-EA</td>
<td>Inductively coupled plasma-element analysis</td>
</tr>
<tr>
<td>IGFA</td>
<td>Inert gas fusion analysis</td>
</tr>
<tr>
<td>RA</td>
<td>Reduction of area</td>
</tr>
<tr>
<td>CP</td>
<td>Commercially pure</td>
</tr>
<tr>
<td>A-case/α-case</td>
<td>Alpha-case</td>
</tr>
</tbody>
</table>
1. Introduction

New manufacturing methods to optimize component manufacturing are under development. These methods are based on the idea that material is additively manufactured in layers. This contrasts to subtractive manufacturing where components are formed by removing material from a large piece casting or forging. Subtractive manufacturing is time-consuming and in the worst cases, more than 90% of the starting material is machined. Compared to more traditional manufacturing methods, additive manufacturing will allow for improved optimization of geometries, fewer manufacturing steps and less wasted material.

Additive Manufacturing using laser and wire (LMD-w) has been developed at GKN Aerospace in Trollhättan for over 10 years and is used in production today. One important process parameter is the oxygen concentration in the chamber during the build sequence. Titanium alloys are highly reactive with oxygen at elevated temperatures, creating a brittle surface layer and could potentially contaminate the material out of specification, affecting the microstructure and thereby part performance. Oxygen concentration is controlled through the use of protective inert gas and extensive use is expensive and adds processing time.

The focus of this thesis is evaluating the effect of oxygen content in the build chamber during manufacturing of titanium components using LMD and Ti-6Al-4V wire. An experimental study was performed to evaluate the effect of oxygen concentration during manufacturing. Two different geometries were built at three different target oxygen concentration levels (100, 500 and 850 ppm). An important objective was to identify a suitable requirement on oxygen concentration and material properties, which will be a trade-off between the economic perspective and part performance.

1.1 Research question

The research question can be summarized in the following points:

- Quantify impact on oxygen concentration level on surface oxidization, chemical composition of bulk material, microstructure and mechanical performance.
- Identify suitable oxygen requirements for applications with or without subsequent processing (e.g. machining or etching).
2. Titanium: alloys, microstructure and oxidation

Aerospace has historically been a large consumer of titanium metal alloys and commercially pure (CP) titanium metal with applications in military, civil and space industry. Titanium metal is primarily used in these industries for structural applications, due to its specific strength (strength/weight ratio) and ability to maintain mechanical properties at elevated temperatures (up to 300-500 °C depending on the alloy). Titanium is also known to resist corrosion, mainly because of the thin surface layer of TiO₂, referred to as the oxide scale, that protects the bulk of the material.

2.1. Alloys and alloying elements

Commercial titanium-based metals and alloys consist mainly of two crystal structures: α (HCP) and β (BCC). The α-phase is stable at room temperature for pure titanium and is the major constituent in most alloys. Titanium alloys are divided into different groups depending on the alloying elements and whether these elements stabilize the α- or the β-phase, Figure 1 and Table 1.

The different groups are:

- **α-alloys**
  - All alloys that are fully (or nearly 100%) α-phase, including all forms of CP (commercially pure) titanium.
- **Near α-alloys**
  - Consists of mostly α with smaller fractions of β
- **α + β alloys**
  - Alloys containing a mixed microstructure of α and β.
- **Near β-alloys**
  - Consists of mostly β with smaller fractions of α
- **β-alloys**
  - Structure consists entirely of β-phase, although fractions of α-phase are sometimes present.

![Figure 1: Common alloying elements and their effect on the titanium phase diagram [1].](image-url)
Table 1: Common alloying and interstitial elements and their effects [2].

<table>
<thead>
<tr>
<th>Alloying element</th>
<th>Effect</th>
<th>Compositional range (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>Alpha stabilizer</td>
<td>2-7</td>
</tr>
<tr>
<td>V</td>
<td>Beta stabilizer</td>
<td>2-20</td>
</tr>
<tr>
<td>Mo</td>
<td>Beta stabilizer</td>
<td>2-20</td>
</tr>
<tr>
<td>Cr</td>
<td>Beta stabilizer</td>
<td>2-12</td>
</tr>
<tr>
<td>Zr</td>
<td>Neutral</td>
<td>2-8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Interstitial element</th>
<th>Max solubility in CP-Ti α (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>Alpha stabilizer 14.2 [3]</td>
</tr>
<tr>
<td>N</td>
<td>Alpha stabilizer 7.6 [4]</td>
</tr>
</tbody>
</table>

Categorizing alloys by their effect on phase stabilization has its benefits but can also be imprecise. For instance, α-alloys are defined as such because the alloy is in the α-phase region of the phase diagram at room temperature. This contrasts to the β-alloys category. Most β-alloys are metastable since they, at room temperature, are located in the α+β phase region. The defining characteristic for β-alloys is that they cannot undergo a martensitic transformation through quenching from the β phase field, which α+β alloys can, Figure 2. Alloys located in the β-phase region are virtually non-existent for commercial use.

![Figure 2: Schematic section through a β isomorphous phase diagram displaying categorization of titanium alloys [1].](image)
The most commonly used titanium alloy is the \( \alpha + \beta \) alloy Ti-6Al-4V, which is the main focus of this thesis. The Ti-6Al-4V alloy represents \( \sim 50\% \) of world titanium metal consumption and \( \sim 80\% \) of all titanium used within aerospace [5]. The aluminum and vanadium serve to stabilize the \( \alpha \)-phase and \( \beta \)-phase, respectively. The Ti-6Al-4V alloy exists in two alloy types: regular and extra-low-interstitials (ELI), the latter named with reference to its lower oxygen concentration. The Ti-6Al-4V alloy has gained popularity due to its good balance of strength, ductility, fatigue properties and fracture toughness. However, the maximum service temperature for Ti-6Al-4V is low (\( \sim 300 \) °C); therefore, its utility within aerospace is mostly restricted to colder parts of the aircraft and engine such as the fan case and embossments (bosses).

2.2. Interstitial elements and \( \alpha \)-case

The amount of interstitial elements in titanium alloys greatly impact the tensile properties. Increasing the content of oxygen, nitrogen and carbon can significantly increase the tensile strength and hardness of the material while making it less ductile. Therefore, it is necessary to strike a balance between strength and ductility, apparent in the AMS4954 specification for welding wire where the tolerable oxygen content lies between 0.12-0.18 wt. %. Severe degradation of ductility can occur over relatively small changes in interstitial content [1] [2] [5].

Another reason why the interstitial content must be controlled is the formation of alpha(\( \alpha \))-case, denoted by A (uppercase) or \( \alpha \) (lowercase). A-case is defined as an oxygen enriched region of \( \alpha \)-phase typically formed between the oxide scale and the bulk material. A-case growth in the material results in embrittlement and severe degradation of fatigue properties, ductility and fracture toughness. However, the definition of \( \alpha \)-case is somewhat imprecise; there is no exact compositional limit in terms of oxygen content. The \( \alpha \)-case layer is typically measured using optical microscopy with requirements on the maximum allowed thickness. Despite the lack of a precise definition, there are some characteristics that define \( \alpha \)-case.

The tell-tale signs of \( \alpha \)-case is producing a distinctive microstructural region near the surfacedirectly underneath the oxide scale and increased hardness due to increased interstitial content. When preparing microstructural samples for \( \alpha \)-case measurement, Ti-6Al-4V is in most cases etched in two steps with Kroll’s reagent followed by ammonium fluoride, which shows the \( \alpha \)-case as a whitened region contrasting against the darker region of the bulk material. As oxygen stabilizes the \( \alpha \)-phase and has the highest solubility in \( \alpha \)-phase of common interstitial elements (14.2 wt. %, highlighted in see Figure 3), it is often seen as the sole driver for \( \alpha \)-case formation. The creation of the \( \alpha \)-case layer occurs due to the diffusion of oxygen from the surface, which increases both the amount of \( \alpha \)-phase in the near-surface region and the interstitial oxygen content.
In addition an interstitial element worthy of consideration is nitrogen, as the element is also an $\alpha$-stabilizer and has a relatively high solubility (7.6 wt. %, highlighted in Figure 4) in the $\alpha$-phase.

Reducing oxygen and nitrogen content results in a desired hardening effect on the material; however, aerospace components have clear limits for interstitial content. According to specifications AMS4999A (directly deposited and annealed Ti-6Al-4V) and AMS4954 (Ti-6Al-4V welding wire that is used as feedstock), the overall compositional requirement for oxygen is between 0.12-0.2 wt. percent whereas for nitrogen the upper limit is 0.05 wt. percent for the final products [6] [7]. This sets a
clear limit for tolerable uptake of interstitial elements for all subsequent processing (welding, heat treatments, additive manufacturing etc.).

Alpha-case is of great importance for mechanical properties, yet there are more aspects to consider as oxidation and nitridation of titanium at elevated temperatures involves complex mechanisms. Oxidation and nitridation of titanium at elevated temperatures involves complex mechanisms. Therefore, in addition to α-case formation, there are several other aspects to consider such as contamination of the oxide scale. In an article by Birhan Sefer et al, submitted for publication in Corrosion Science Journal in late 2017, titanium alloys Ti-6Al-4V and Ti-6Al-2Sn-4Zr-2Mo were heat treated at 500-700 °C at varying exposure times. The latter alloy has a service temperature of 450 °C, making it more suitable for hotter applications than Ti-6Al-4V. In this study, it was found that both alloys developed layers of α-case which followed increased temperature and exposure time and that Ti-6Al-2Sn-4Zr-2Mo was comparatively more resistant to α-case formation. Another finding was the formation of Al₂O₃ in parallel layers inside the TiO₂ oxide scale. Again, Ti-6Al-2Sn-4Zr-2Mo proved more resistant than Ti-6Al-4V, as can be seen in Figure 5. Contamination of the oxide scale in this way facilitates inward diffusion of oxygen and thus the growth of the α-case layer.

In a study performed on a different α+β alloy (Ti-6Al-2Sn-4Zr-2Mo) Raghuveer Gaddam et al measured α-case thickness by optical microscopy, hardness and electron probe micro-analysis (EPMA) [9]. Concentration profiles of normalized oxygen concentration from the EPMA showed that the near-surface region contained a higher oxygen concentration than the bulk, which indicates the important role oxygen plays in α-case formation. However, the measurements using optical microscopy undervalued the α-case thickness of some samples. In the best cases, all three methods showed similar measurements. In the worst case, the hardness test and EPMA showed an α-case thickness of 159 and 154 μm respectively, while optical microscopy measured 107 μm for the same sample. As this is the typical method for evaluating α-case, it is a disconcerting result.
2.3. Microstructural evolution

The microstructural evolution of Ti-6Al-4V starts with solidification from liquidus which occurs at 1709 °C calculated in Thermo-Calc for the composition studied in this thesis. As the material is cooled, it will pass the \( \beta \)-transus temperature (967 °C, also calculated in Thermo-Calc) and enter the \( \alpha + \beta \) phase region where \( \alpha \) will begin to nucleate. Depending on the cooling rate, the nucleation can take on different forms. Table 2 is a schematic description of the different types of \( \alpha \) nucleation that commonly occurs in titanium alloys due to cooling rate. Additionally, the thickness of \( \alpha \)-laths has a significant impact on tensile properties [5]. Figure 6 shows decreasing yield strength with increasing \( \alpha \)-lath thickness.

![Figure 6: Alpha-lath thickness impact on yield strength [1].](image)

A sufficiently slow cooling rate will yield equilibrium, or near equilibrium, fractions of both \( \alpha \) and \( \beta \). The microstructure will then be defined by large primary \( \alpha \) grains with some retained \( \beta \) along the grain boundaries. This is referred to as an equiaxed microstructure.

For a moderately slow cooling rate, \( \alpha \) will begin to nucleate at \( \beta \) grain boundaries upon entering the dual phase region. Since the nucleation progresses slowly, thick and continuous \( \alpha \)-laths will grow along \( \beta \) planes and ultimately encounter other \( \alpha \) laths. This leads to a Widmanstätten microstructure consisting of thicker \( \alpha \)-laths in a \( \beta \) matrix.

<table>
<thead>
<tr>
<th>Cooling rate</th>
<th>Transformation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slow</td>
<td>( \beta \rightarrow \alpha \text{Glob} + \beta )</td>
<td>Globular/equiaxed ( \alpha )</td>
</tr>
<tr>
<td>Moderately slow</td>
<td>( \beta \rightarrow \alpha \text{GB} + \beta )</td>
<td>Grain boundary precipitation, Widmanstätten with thick ( \alpha ) laths</td>
</tr>
<tr>
<td>Moderately fast</td>
<td>( \beta \rightarrow \alpha \text{BW} + \beta )</td>
<td>Fine Widmanstätten or basket weave with thinner ( \alpha )-laths, precipitation inside grains and in grain boundaries, ( \alpha )-colonies</td>
</tr>
<tr>
<td>Fast (quenching)</td>
<td>( \beta \rightarrow \alpha', \alpha'' + \beta )</td>
<td>Martensitic</td>
</tr>
</tbody>
</table>

Table 2: Schematic description of different \( \alpha \)-nucleation mechanisms.
For a moderately fast cooling rate, $\alpha$ will nucleate at multiple sites: inside the $\beta$ grains and in grain boundaries. The laths formed in the grains and along the boundaries will eventually grow with different orientations into each other in a woven manner, commonly described as a basket weave microstructure. Grain boundary $\alpha$ is highly dependent on cooling rate. As cooling rate increases, grain boundary $\alpha$ will form in a thinner and discontinuous, or not form at all.

In the case of a fast cooling (i.e. quenching), a diffusionless transformation can occur, which can in turn result in two different crystallographic structures, referred to as acicular ($a'$) and orthorhombic ($a''$) martensite. Acicular martensite has a distorted HCP crystal structure and is often found in alloys containing less $\beta$ stabilizing elements [5]. If sufficient $\beta$ stabilizers are present in the alloys, the crystal structure of the martensite will be orthorhombic instead.

It is appropriate to distinguish between the aforementioned terms ‘Widmanstätten’ and ‘basket weave’. In this thesis, the term basket weave refers to the case where $\alpha$-laths have nucleated within grains as well as in grain boundaries; thus, the colliding laths will have different crystallographic orientations relative to each other. Widmanstätten is used to describe a microstructure where $\alpha$-laths have nucleated solely in grain boundaries and will therefore have the same crystallographic orientation. This contrasts to some cases found in the literature where basket weave is simply referred to as a fine Widmanstätten structure with no mention of crystallographic orientation.

Another critical microstructural feature is the formation of $\alpha$-colonies critical microstructural feature. These colonies are regions of $\alpha$ laths that can vary in size and thickness. Yield strength and ultimate tensile strength are affected by the $\alpha$-lath dimensions [5].

General microstructural types

The overall microstructure of titanium alloys has been thoroughly covered in the scientific literature. Common microstructural types include equiaxed (Figure 7), bimodal (Figure 8), Widmanstätten (Figure 9), martensitic (Figure 10) as well as the aforementioned basket-weave structure (Figure 11).
Figure 7-11: Commonly occurring titanium alloy microstructures such as globular/equiaxed (Figure 7), bi-modal (Figure 8), Widmanstätten (Figure 9), martensitic (Figure 10) and basket weave (Figure 11).

Figure 7: Fully globular, or equiaxed, α-microstructure in a Ti-6Al-2Sn-4Zr-2Mo alloy after slow cooling [1].

Figure 8: The bi-modal microstructure is a mixture of globular or primary α in a lamellar α+β matrix. The figure on the right shows an IMI 834 alloy post rapid cooling [1].

Figure 9: Typical example of a Ti-6Al-2Sn-4Zr-2Mo Widmanstätten microstructure with α laths in a β matrix [1].

Figure 10: Picture of Ti-6Al-4V manufactured with AM and quenched in water resulting in a martensitic microstructure [10].

Figure 11: Basket weave microstructure of a Ti-5Al-5Mo-5V-1Cr-1Fe near β titanium alloy [11]. Note how the fine α-laths are woven together with the β matrix and how it differs to the Widmanstätten example above.
3. Additive Manufacturing

Additive manufacturing refers to processes based on computer-aided design where material is deposited directly in single or multiple layers until the final geometry is achieved. This stands in contrast to subtractive manufacturing where a final component is formed through material removal by machining. Dating back to the early 1980’s, the field of additive manufacturing (AM) has grown substantially in the past five years. According to the Wohlers Report of 2016 (an annual report surveying the AM industry), the global market of AM products and services worldwide grew from $4.103 billion to $5.165 billion USD in 2015.

Emmanuel Sachs, together with a team of researchers, pioneered 3D-printing in the 90’s by highlighting the potential and developing several concepts that form the foundation of additive manufacturing today. In the concluding paragraph of their paper, it is stated that the mechanisms which govern AM are different from that of subtractive manufacturing. Every AM process will have unique characteristics that decide the feasibility and competitiveness of each individual process. It also lends flexibility to the effort of finding tailored solutions to specific requirements.

There is a large number of processes that AM can refer to, yet there are certain attributes that they all have in common. All methods include three things: a power source, material addition and some type of protected environment to shield the material from contamination at elevated temperatures. The power source is typically a laser, an electron beam or a TIG-torch (tungsten inert gas). Material addition is done by either feeding powder or wire directly into the power source ray or by depositing a layer of powder and then letting the power source traverse across the powder layer. A protected environment is achieved either locally or in a sealed chamber, by either lowering the pressure to near vacuum or utilizing a protective gas, such as argon. AM is typically divided by material addition into two major categories: PBF (powder bed fusion) and DED (direct energy deposition). Figure 12 shows an overview of different commercially available methods of additive manufacturing. This thesis will focus mainly on wire-based laser metal deposition (LMD-w). When AM is mentioned hereafter, it refers to additive manufacturing of metals.

![Figure 12: Power source, material feedstock and chamber environment of different AM processes.](image-url)
3.1. Additive manufacturing in aerospace

All processes offer their own set of advantages and constraints. PBF processes are generally better at building complex geometries, such as electron beam melting (EBM) which allows precise control over energy addition due to the electron beam power source. There is however a maximum constraint on the build dimensions. The build chamber typically only allows for components smaller than 30x30x30 cm. Furthermore, all powder-based processes experience more problems with porosity compared to using wire as a feedstock. On the other hand, DED processes have fewer constraints on the maximum size of the build and have higher deposition rates. To establish a general idea of what additive manufacturing has to offer the aerospace industry, the following section is focused on material properties, the buy-to-fly ratio, component complexity and technology readiness levels.

Material Properties

The main concern of materials engineers and scientists is whether additive manufacturing is able to produce components of equal quality to that of cast or wrought metals. The range of properties that can be produced by AM dictates its applications and is fundamental for its competitiveness. In the last decade, a large number of studies has investigated properties of various AM techniques ranging from powder-bed technologies, such as EBM and SLM, to methods using a feedstock of wire or powder, such as LMD. Table 3 and Figure 13 is a summary of three independent studies of LMD-w sample tensile properties (UTS, YS, elongation and high-cycle fatigue (HCF)) [14] [15] [16].

<table>
<thead>
<tr>
<th>Property</th>
<th>AMS4999A (min)</th>
<th>Wrought (min)</th>
<th>Cast [14] (min)</th>
<th>[15] (min)</th>
<th>[16] (min)</th>
<th>Average of referenced LMD-w samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength, X/Y-directions [MPa]</td>
<td>889</td>
<td>896</td>
<td>861</td>
<td>930-1054</td>
<td>~960</td>
<td>864-987</td>
</tr>
<tr>
<td>Tensile strength, Z-direction [MPa]</td>
<td>855</td>
<td>896</td>
<td>861</td>
<td>930-1054</td>
<td>918</td>
<td>937-1011</td>
</tr>
<tr>
<td>Yield strength, X/Y-directions [MPa]</td>
<td>799</td>
<td>827</td>
<td>772</td>
<td>859-984</td>
<td>~870</td>
<td>~790-880</td>
</tr>
<tr>
<td>Yield strength, Z-direction [MPa]</td>
<td>765</td>
<td>827</td>
<td>772</td>
<td>859-984</td>
<td>803</td>
<td>~780-950</td>
</tr>
<tr>
<td>Elongation, X/Y-directions [%]</td>
<td>6</td>
<td>10</td>
<td>5</td>
<td>4.1-11.4</td>
<td>~2.5-2-12</td>
<td>~2-13.5</td>
</tr>
<tr>
<td>Elongation, Z-direction [%]</td>
<td>5</td>
<td>10</td>
<td>5</td>
<td>4.1-11.4</td>
<td>~12-17</td>
<td>~8-15.5</td>
</tr>
</tbody>
</table>

Table 3: Tensile properties LMD-w samples according to literature with regard to specifications for cast, wrought and directly deposited material.
Table 3 highlights the average tensile properties that LMD-w is capable of producing. The three selected publications aimed to evaluate different heat treatments and process parameters. It is worth mentioning that examples from the literature include both as-deposited and heat-treated specimens, and therefore does not represent the maximum capabilities of the LMD-w process. Most specimen fall within AMS and ASTM requirements on all points although half of the material tested as-deposited tended to fail elongation criteria. Different heat treatment recipes in the range of 600-843 °C for two hours would typically resolve this by slightly lowering the UTS and YS while increasing elongation and remaining within specification.

Figure 13 shows high-cycle fatigue properties of heat treated Ti-6Al-4V in the x- and z-direction. The lower limit was set to 760 MPa (samples would run out at this stress level) and the upper limit was set under the YS as plastic deformation is undesired during testing.

![Figure 13: High-cycle fatigue life of LMD-w samples in x- and z-direction post heat treatment compared to cast, wrought and substrate material [14].](image)

**Buy-to-fly ratio**

The buy-to-fly ratio is a commonly used performance indicator within aerospace. It is defined as the ratio of raw material weight and weight of the final component. In the ideal scenario, a part has a buy-to-fly ratio of 1, which indicates that no material has been wasted. In reality, it is likely impossible to achieve a ratio of exactly 1 but achieving a buy-to-fly ratio between 1 and 2 is indeed possible for additively manufactured components. This is considered a significant improvement from standard production, since for certain aerospace components, material removal can be as high as 95% yielding a buy-to-fly ratio of 20. Lowering the buy-to-fly ratio through additive manufacturing for a product can lead to lowered costs in terms of shorter process times, fewer process steps and less material waste. Thus, components with high buy-to-fly ratios are likely to be targeted first.
Technology Readiness Level

Technology readiness level (TRL) is a method that was developed by NASA (incorporated in 1991) with the aim of evaluating matureness of components, methods, concepts and technology in aerospace programs [17]. Today, TRL is a standard method that all major aerospace companies and organizations follow. It consists of nine levels comprising different stages of technology maturity, ranging from basic research to fully operational (Figure 14). Reaching new readiness levels for novel technologies is a time- and resource-consuming activity. The TRL system not only comprises technical requirements but also sets demands for cost-effectiveness and sustainability. The aerospace industry has historically been a conservative field and introduction of the TRL system has not changed that. It stands to reason that particular caution needs to be paid to avoid catastrophic failures when manufacturing components intended for commercial and military aircraft.

![Figure 14: Stages of TRL [15].](image)

Because of the TRL system, introducing new processes, including AM, to the aerospace industry thus takes time. Every commercially available AM process has to pass through the first six TRL levels before it can be introduced to production and start to generate income. Additive manufacturing also requires heavy investments in adequate machinery and skilled personnel. In light of all this, reaching production status with a process such as LMD-w is no small feat not trivial.

The TRL system also serves as a benchmark for other industries. If a process reaches the upper TRL levels it signals to other industries that the technology is feasible and competitive. Therefore, the TRL system is both a challenge and a facilitator for the applicability of AM processes as it forces new technologies to prove validity and robustness.
In conventional manufacturing, geometrical complexity and production cost have long been closely correlated [18]. More specifically, increasing the complexity of the component traditionally increases subsequent machining steps and requires customized process paths, altogether increasing the cost of assembling a product. A fundamental benefit of AM is the need for little or no subsequent machining for most products, although there are of course exceptions depending on the process and application. The term “complexity for free” implies a dissociation of product complexity and increased production cost, which has been a governing principle of mass component manufacturing. In truth, it goes beyond controlling merely the geometrical complexity of the build. Altering composition layer-by-layer, or even point-by-point, is now a possibility, enabling precise compositional variations in the material that would be impossible to achieve with traditional means.

3.2 Laser metal deposition

Laser metal deposition (LMD) is a process where wire or powder is continuously fed directly into a laser spot while the laser travels across a substrate. A component’s final geometry is produced by depositing layers until the desired shape is formed. Each layer is comprised of single or multiple beads. The focus of this thesis is LMD using a Ti-6Al-4V wire (LMD-w). A schematic of the process can be seen in Figure 15.

![Schematic view of LMD using metal wire as feedstock](image)

Figure 15: Schematic view of LMD using metal wire as feedstock [19].

Compared to wire-based LMD processes, powder-based LMD has been more widely used since it enables more complex geometries to be shaped. However, there are several drawbacks relative to a wire-based process; the main disadvantages include longer process times, powder escaping from the feed and increased susceptibility to contamination due to increased surface area [14]. Therefore, LMD-w is suitable for any component as long as the geometry is not too complex or small.

The wire-based LMD process can be used for a large range of applications where bosses, fan case stiffeners and flanges are some examples. The size ranges from...
single-bead depositions (~1-2 mm in diameter) up to structures as large as 3 m in diameter. Essentially any detail that is typically welded to a larger structure could potentially be produced by LMD-w.

Similar to other methods of additive manufacturing, both powder- and wire-based LMD requires finely tuned parameters and a skilled operator for the process to be stable. In the past decade, serious advances have been made in order to achieve a fully automated process [19, 20].

3.2.1. Process parameters

When constructing a part using LMD, it is critical that the process is stable. Process stability can be defined as smooth material transfer from the solid wire into liquid metal and requires the wire to melt close to the interface of the melt pool. The complexity of LMD-manufacturing lies in finely tuning the many process parameters to achieve this goal. A useful way to outline the process is to define a process window that describes the upper and lower limits of the chosen parameters while sustaining a stable process. An example from Heralić’s thesis [19], can be viewed in Figure 16. The following section will focus on the primary process parameters that affect process stability. Secondary parameters are stated; see [19] for a more thorough description.

![Figure 16: Example of LMD process window. Changes in the wire feed angle affects both the sensitivity to the building direction and the width of the process window [19].](image-url)
Material transfer from the nozzle to the substrate can essentially occur in three different ways: globular, smooth or by plunging. In short, it has to do with achieving the proper timing of the melting of the wire. If it melts too early, droplets will have time to form and the transfer mode will be globular. If melting occurs too late, solid wire will be plunged into the melt pool, Figure 17. Thus, a stable process refers to smooth material transfer found in the middle image of the figure.

In his dissertation, Heralić identifies three primary process parameters and several secondary that has to be carefully fine-tuned in order to achieve a stable process. The three primary parameters are the following:

1. Laser power – the laser power directly decides the maximum energy input and thus the melt pool size as well as the maximum wire feed rate that can be added for a given traverse speed.
2. Wire feed rate – the wire feed rate is principally the amount of material that is added per time unit. Preferably chosen with regard to the laser power and traverse speed.
3. Traverse speed – the traverse speed decides the manufacturing speed and the amount of energy added to the process. Building at a slower traverse speed makes a stable process easier to maintain but will also contribute more energy to the process. This will in turn affect the thermal cycles that multi-layered structures undergo during manufacturing and will make the component susceptible to contamination from the environment as well as affecting the cooling rate and resulting microstructure. Building at a higher traverse speed will decrease the added energy but demands more accuracy from the motion of the robot.

Secondary parameters:

- Laser power distribution
- Laser/wire angle
- Laser/substrate angle
- Laser beam size and shape
- Laser beam focal length
- Laser wavelength
- Wire diameter
- Wire/substrate angle
- Wire diameter
- Wire stick-out
- Feeding direction
- Building direction
Fine-tuning the aforementioned parameters will yield a stable deposition process with few defects. Porosity, one of the most common defects found in powder-based processes, is typically not found on a significant scale when wire is used as a feedstock [14]. The resulting material is typically free from porosity once a stable process is ensured. Another benefit is reduced contamination from the environment due to the reduced surface area compared to powders. However, LMD-w is not free from detects. A commonly occurring defect is lack-of-fusion (LOF). LOF defects can appear for several reasons, common triggers being non-smooth material transfer or incorrect bead step length. Additional defects include cracks and substrate deformation due to thermal stress.

3.2.2. Microstructural evolution in LMD-w processes

The thermal history of LMD-w deposited material generates a complex microstructure. Numerous publications in the past decade have improved our understanding of microstructural evolution [10] [11] [15] [21]. A qualitative study in two parts from 2002 by S. M. Kelly and S.L. Kampe [22] [23], focused on characterizing the microstructure and thermal history of Ti-6Al-4V manufactured by LMD-w. The thermal history can be seen in Figure 18, which schematically describes the temperatures that a single layer experience after subsequent layers are deposited [5].

![Figure 18: Schematic thermal history of a single layer after deposition and 6 subsequent layers [5].](image)

We can see that a small portion of the first layer will be remelted when the second is deposited and that the β-transus temperature will be crossed until three subsequent layers have been deposited. Thus, it can be concluded that each layer undergoes multiple thermal cycles and phase transformations during a manufacturing sequence. Predicting the exact microstructure is therefore a highly complex task, yet Kelly and Kampe managed to conceptually describe the microstructural evolution.
The microstructure was characterized as seen in Figure 19, where we can take special note of what happens when layer $n+3$ is deposited. A small part of the initial layer $n$ will experience a short excursion into the $\beta$-phase field, followed by slow cooling which yields a region with a colony morphology, thus explaining the layered band structure common in LMD-$w$ samples (Figure 20).

Due to the $\beta$-transus line being crossed several times for each individual layer, epitaxial growth of $\beta$ grains across multiple layers is enabled. These grains transform as $\alpha$ nucleates upon cooling and they are commonly referred to as prior $\beta$-grains. Other microstructural features include the heat affected zone in the substrate (HAZ), $\alpha$-case, a mixed microstructure of Widmanstätten and basket-weave as well as grain boundary $\alpha$. Note how the bands are not present in the top part of Figure 20, supporting the fact that it is caused by thermal impact of subsequent deposition.
4. Experimental

Three walls and three bosses (Figure 21) were manufactured at different targeted oxygen ppm levels in the chamber (100, 500 and 850 ppm). All samples were heat treated and evaluated through a microstructural analysis (including $\alpha$-case measurements), hardness and chemical composition. Tensile tests were also performed on the wall samples. Chemical composition of the wire used as feedstock in the process was measured.

![Figure 21: Images showing top side of a boss (left) and side of a wall (right).](image)

4.1. Samples

Double-bead walls and bosses (cylinder-shaped) were deposited using a Ti-6Al-4V wire on a Ti-6Al-4V substrate. Dimensions of wire, bosses, walls and substrates are detailed in Table 4.

<table>
<thead>
<tr>
<th>Wire</th>
<th>Boss substrate</th>
<th>Bosses</th>
<th>Wall substrate</th>
<th>Walls</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter 1.14</td>
<td>Thickness 3.2</td>
<td>Diameter 65</td>
<td>Thickness 3.2</td>
<td>Height 45</td>
</tr>
<tr>
<td>Width 190</td>
<td>Height 20</td>
<td>Width 30</td>
<td>Width 220</td>
<td></td>
</tr>
<tr>
<td>Depth 190</td>
<td></td>
<td>Depth 10</td>
<td>Depth 8</td>
<td></td>
</tr>
</tbody>
</table>

The varied geometries were chosen because of the difference in retained heat. The “colder” build, e.g. the wall, will experience less heat exposure, since the start and end point are located further away from each other compared to the boss. More specifically, during wall deposition, the laser is constantly moving away from newly melted material. In contrast, when depositing bosses, the laser is essentially rotating around the center point which will significantly affect cooling rate and retained heat.

4.2. Chamber

A tent (1.3 m in diameter and 1 m in height) made from transparent plastic was used as protective chamber. It cannot be regarded as completely sealed; the protection comes from the gas pressure inside the tent preventing air from entering. Before
beginning deposition, when the ppm level has been stabilized, argon will accumulate in the bottom of the tent near the build due to the higher density of the argon gas. Thus, as a steady state is reached, the lower part of the chamber will consist of argon while lighter gases are located at the top. Utilizing a tent instead of a container common in other processes lends flexibility of movement for the robot and allows for larger variation in possible build geometries.

The experimental setup included two separate gas inlets placed side-by-side at the bottom of the chamber; one for the primary argon flow (with a maximum flow of 500 l/min), and one for a mixed flow of air and secondary argon (max 50 l/min). Figure 22 shows a schematic view of the experimental setup. The amount of air introduced to the process was varied in order to achieve different ppm intervals of oxygen. The air inlet was controlled through a gas mixer combined with mass flow meters.

Air flows between 0-20 l/min could accurately be used to vary the oxygen content in the environment. Back pressure was monitored in order to detect anomalies in the mixed flow inlet.

The concentration of nitrogen is assumed to be ~3.727 times the concentration of oxygen as air is used to contaminate the chamber [24].

Oxygen was measured with an ORBMAX oximeter from Orbitalum Tools GmbH [25]. The oximeter was placed in two different locations. During the first experiment (wall deposition), the oximeter was placed at the gas inlets. For the second experiment (boss deposition), the oximeter was placed as close to the build as possible. The reason for this was due to fluctuating readings from the oximeter, especially at higher ppm values (800-900). Therefore, the oxygen sensor was attached to the plate that controlled the position of wire nozzle and laser. This provided more stable readings from the oximeter.
The compositions of feedstock, substrate and final component according to the respective AMS specifications are presented in Table 5.

Table 5: AMS specifications for composition of titanium plate substrate, welding wire, and deposited Ti-6Al-4V.

<table>
<thead>
<tr>
<th>Element</th>
<th>Substrate: AMS4911M</th>
<th>Feedstock: AMS4954</th>
<th>Deposited Ti-64: AMS4999A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Min</td>
<td>Max</td>
<td>Min</td>
</tr>
<tr>
<td>Ti</td>
<td>Bal.</td>
<td>Bal.</td>
<td>5.50</td>
</tr>
<tr>
<td>Al</td>
<td>5.50</td>
<td>6.75</td>
<td>5.50</td>
</tr>
<tr>
<td>V</td>
<td>3.50</td>
<td>4.50</td>
<td>3.50</td>
</tr>
<tr>
<td>Fe</td>
<td>-</td>
<td>0.30</td>
<td>-</td>
</tr>
<tr>
<td>O</td>
<td>-</td>
<td>0.20</td>
<td>0.12</td>
</tr>
<tr>
<td>C</td>
<td>-</td>
<td>0.08</td>
<td>-</td>
</tr>
<tr>
<td>N</td>
<td>-</td>
<td>0.05</td>
<td>-</td>
</tr>
<tr>
<td>H</td>
<td>-</td>
<td>0.015 (150 ppm)</td>
<td>-</td>
</tr>
<tr>
<td>Y</td>
<td>-</td>
<td>0.005 (50 ppm)</td>
<td>-</td>
</tr>
<tr>
<td>Other elements, each/total</td>
<td>-</td>
<td>0.10/0.20</td>
<td>-</td>
</tr>
</tbody>
</table>

4.3. Heat treatment and sample preparation

All samples were heat treated in vacuum at 977 °K for 2 hours. Samples were cut out using a water jet to avoid heat contamination. Microstructural samples were thoroughly cleaned with isopropanol. Etching was done with Kroll’s Reagent. An aqueous hydrofluoric acid solution was used after Kroll’s when etching for α-case.
4.4. Methods of evaluation

The specimens were evaluated according to the test matrix seen in Table 6. Each test method is explained and attested in the corresponding results section.

<table>
<thead>
<tr>
<th>Test method</th>
<th>Walls (Yes/No)</th>
<th>Bosses (Yes/No)</th>
<th>Wire (Yes/No)</th>
<th>Expected result</th>
<th>Performed at</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOM</td>
<td>Y</td>
<td>Y</td>
<td>N</td>
<td>Microstructural analysis and α-case depth</td>
<td>GKN Trollhättan</td>
</tr>
<tr>
<td>Hardness</td>
<td>Y</td>
<td>Y</td>
<td>N</td>
<td>-</td>
<td>GKN Trollhättan</td>
</tr>
<tr>
<td>Tensile</td>
<td>Y</td>
<td>N</td>
<td>N</td>
<td>Tensile properties</td>
<td>Metcut Research Inc.</td>
</tr>
<tr>
<td>IGFA</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
<td>Total O and N content in surface samples &amp; O, N and H content in wire</td>
<td>Dirats Laboratories</td>
</tr>
<tr>
<td>ICP-EA</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Content of metallic elements in wire</td>
<td>Dirats Laboratories</td>
</tr>
<tr>
<td>Combustion</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Content of C in wire</td>
<td>Dirats Laboratories</td>
</tr>
</tbody>
</table>

Light optical microscopy (LOM)

A thorough study of the samples was performed using light optical microscopy to yield a comprehensive view of microstructural characteristics and α-case measurements.

Hardness

Hardness testing was done in order to compare with previous studies.

Tensile tests

Tensile properties were analyzed to see if contamination of the chamber environment would lead to degradation of mechanical properties. Four samples from each wall were extracted at different locations in the build (samples 8-11 in Figure 24). Tests were performed according to ASTM E8/E8M-16a by Metcut Research Inc. in Cincinnati, OH, United States [27]. The outer and inner diameter of the tensile samples were ~5 and ~3 mm respectively.

Inert gas fusion analysis

Inert gas fusion analysis is a method of combustion analysis designed for retrieving the composition of materials. Small elements, especially oxygen and nitrogen, are
possible to measure at concentrations as low as 80 ppm for oxygen and 20 ppm for nitrogen [26]. Samples were taken from the surface (max 3 mm) in order to see if composition was within specification limits. The tests were performed according to ASTM E1409-13 by Dirats Laboratories in Westfield, MA, United States [26]. The feedstock composition was evaluated to verify that the wire used during deposition was within specification.

In his licentiate thesis, Magnus Neikter discovered that the microstructural texture of LMD-w samples built with Ti-6Al-4V was more pronounced closer to the substrate [5]. As the degree of texture impacts mechanical properties, one could expect lower tensile strength in samples 10-11 than in 8-9 in Figure 20 as they are closer to the substrate. Cutout locations in the different samples can be seen in Figure 23 and Figure 24.

Figure 23: Picture of boss cutouts. 1-6=LOM/SEM/Hardness, 7-10=IGFA.

Figure 24: Picture of wall cutouts. 1-3=LOM/SEM/Hardness, 4-7=IGFA, 8-11=Tensile.
5. Results

Samples are listed in Table 7 according to their corresponding target oxygen ppm level.

5.1. Chamber environment

Table 7 contains a statistical summary of data gathered from the oximeter log.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Target O ppm</th>
<th>Mean</th>
<th>Min-Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boss 1</td>
<td>100</td>
<td>96 (358)</td>
<td>77-112 (287-417)</td>
</tr>
<tr>
<td>Boss 2</td>
<td>500</td>
<td>478 (1781)</td>
<td>448-506 (1669-1886)</td>
</tr>
<tr>
<td>Boss 3</td>
<td>850</td>
<td>829 (3090)</td>
<td>770-867 (2870-3231)</td>
</tr>
<tr>
<td>Wall 1</td>
<td>100</td>
<td>112 (417)</td>
<td>104-119 (387-444)</td>
</tr>
<tr>
<td>Wall 2</td>
<td>500</td>
<td>474 (1766)</td>
<td>439-527 (1636-1964)</td>
</tr>
<tr>
<td>Wall 3</td>
<td>850</td>
<td>861 (3208)</td>
<td>788-934 (2937-3481)</td>
</tr>
</tbody>
</table>

The chamber environment was controlled through online surveillance of oxygen level and mass flow of injected air. Line plots of oxygen measurements can be seen in Figure 25. Approximately 250 seconds is required to reach a stable ppm level of 100, as can be seen in the left part of Figure 25. Reaching the oxygen ppm level commonly used for manufacturing (50 ppm and below) required approximately another 250 seconds. Due to a technical error, data from the first half of the 100-ppm wall deposition could not be accounted for. The process was still monitored manually by the author and an operator at all times during deposition.

![Figure 25: Oximeter log of data points showing oxygen ppm level in the chamber during deposition.](image-url)
5.2. Compositional analysis

Different wires were used for bosses and walls. Compositional analysis using ICP-EA, IGFA and combustion showed that both wires were within specification. Wire oxygen content was measured at 0.15 wt. % (wall samples) and 0.155 wt. % (boss samples), while wire nitrogen content was measured at 0.006 wt. % (walls) and 0.007 wt. % (bosses). All samples were within specification (0.2 wt. % O and 0.05 wt. % N) even when accounting for the error.

Figure 26 and Figure 27 shows the wire content, average element uptake and maximum reported values for each sample. The maximum reported value was found in the 500-ppm boss (Boss 2, 0.195 wt. % oxygen).

5.2.1. Oxygen

![IGFA Results Oxygen](image)

Figure 26: IGFA results of oxygen showing wire content, average oxygen uptake and maximum reported values for each sample.

5.2.2. Nitrogen

![IGFA Results Nitrogen](image)

Figure 27: IGFA results of nitrogen showing wire content, average oxygen uptake and maximum reported values for each sample.
5.3. Tensile testing

Results from the tensile tests can be seen in Figure 28 through Figure 30 including the minimum requirements according to specification AMS4999A for directly deposited Ti-6Al-4V. Four samples from each wall, two from the bottom layers and two from the top layers (~30 mm and ~15 mm from the substrate), were evaluated making it a total of 12 tensile tests. The comparison of top and bottom samples will be elaborated on in 6.4. All samples were tested post heat treatment. No apparent correlation was found between tensile properties and the tested levels of oxygen in the chamber. Furthermore, the reported tensile values were not significantly different from values reported in the literature, [11] [14] [15] [21] [28] [29]. An important note is that no samples failed specification requirements.

![Figure 28: UTS and elongation of wall samples tested in the x-direction (build direction). Dotted lines show the AMS4999A minimum requirement.](image)

![Figure 29: UTS and elongation of wall samples tested in the x-direction (build direction). Top and bottom samples were located ~30 mm and ~15 mm from the substrate respectively. Dotted lines show the AMS4999A minimum requirement.](image)
Figure 30: YS and RA of wall samples tested in x-direction (build direction). Dotted line shows the AMS4999A minimum requirement (no present requirement on area reduction).
5.4. Evaluation of microstructure

The first section contains results from α-case measurements and the second section is a qualitative comparison regarding microstructural differences between the samples. No specific microstructural property, besides α-case, could be correlated to oxygen concentration in the build chamber during deposition.

5.4.1. A-case measurements

A-case measurements were performed on two samples from each wall and five samples from each boss. All surfaces of substrate and deposited material were examined. Figure 31 displays exemplary images of α-case found in both 100-ppm samples.

![Exemplary α-case images](image)

**Figure 31:** Exemplary α-case images of wall 1 (left) and boss 1 (right) at a magnification of 500.

Figure 32 shows the interval of α-case depth in every sample whereas Table 8 reveals information regarding the location of measurements.

![Intervals of α-case depth by sample](image)

**Figure 32:** Intervals of α-case depth by sample.
Table 8: Summary of α-case measurements. Samples numbered after target oxygen ppm level (wall 1 = 100 ppm, wall 2 = 500 ppm etc.). Values reported in µm.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Sides (walls)</th>
<th>Top</th>
<th>Centre (bosses)</th>
<th>hole</th>
<th>Outer side (bosses)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wall 1</td>
<td>9-10</td>
<td>7-20</td>
<td>7-21</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wall 2</td>
<td>7-19</td>
<td>7-20</td>
<td>10-18</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wall 3</td>
<td>8-11</td>
<td>10-22</td>
<td>9-20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Boss 1</td>
<td>8-41</td>
<td>8-17</td>
<td>40-53</td>
<td>39-45</td>
<td></td>
</tr>
<tr>
<td>Boss 2</td>
<td>7-32</td>
<td>16-20</td>
<td>55</td>
<td>17-34</td>
<td></td>
</tr>
<tr>
<td>Boss 3</td>
<td>7-61</td>
<td>15-26</td>
<td>21-22</td>
<td>15-52</td>
<td></td>
</tr>
</tbody>
</table>

5.4.2. General microstructure

A microstructural comparison between the two different sample geometries is presented in this section. Most of the characteristic microstructural features of a LMD-w sample can be identified in Figure 33, including the layered bands, heat affected zone of the substrate (HAZ), epitaxial prior β grains and the lack of layered bands in the final deposited layers. HAZ refers to the heat affected zone of the substrate located directly beneath the first layer.
Figure 34 displays a boss sample, evidently lacking the layered bands and consisting of a coarser microstructure. Epitaxial prior $\beta$ grains spanning across multiple layers are evident in this sample. The HAZ contains a coarser microstructure closer to the center hole (bottom left in Figure 34) than the outer side (bottom right).

Figure 33: Overall microstructure of wall 1 (100-ppm) showing the bottom and top halves (left and right respectively).

Figure 34: Microstructural overview of a boss sample (100 ppm) with a highlighted prior $\beta$ grain. The arrow indicates increasing coarseness in the HAZ.
Figures 36-43 shows typical examples of microstructural variations found in boss and wall samples. Generally, both bosses and walls contain similar microstructural features including Widmanstätten, basket weave, α-colonies and occurrences of grain boundary α and globular α. The microstructural details however, were generally smaller in walls than in bosses as can be seen in the images above. In bosses, basket-
weave would only appear more frequently towards the top layers. Basket weave in wall samples were a common feature throughout the build. The grain boundary $\alpha$ and $\alpha$-laths were significantly thicker in boss samples. The microstructure found in wall samples was more irregular relative to the bosses.

5.5 Hardness

Hardness was evaluated using a Vickers diamond with a load of 0.3 kg. Each measurement series was done starting from the middle of the sample in the substrate and stepping to the final layer at even distances. Figure 44 shows the resulting values where the first (1) and last (10) measurement points refers to the substrate and top layer respectively.

![Graph showing Hardness values](image)

Figure 44: Hardness values stepping from the substrate (1) to the top layer (10).
6. Discussion

6.1. Chamber environment

As can be seen in the graphs (Figure 25), the oxygen level fluctuation increases as more air is allowed into the chamber making it harder to control. The placement of the oximeter during boss deposition showed more stable readings than when the walls were built.

Controlling the chamber environment exactly on a ppm level is a difficult task considering the extensive gas flow (500+ l/min) in the chamber during deposition and the fluctuating temperatures in the chamber which affects oxygen and nitrogen affinity. The setup adequately simulates a production environment as was the intention.

Approximately 4.5-9 minutes could potentially be saved from filling a chamber of this size. If manufacturing suffers from long lead times due to frequent readjustment of the chamber, then this could provide significant time savings.

6.2. Compositional analysis

The IGFA results display a clear trend with increased uptake of interstitial elements as oxygen and nitrogen level in the chamber was increased. Boss samples showed a larger average uptake than walls indicating that heat input affects interstitial uptake.

An interesting note is that the maximum oxygen and nitrogen content was reported for the Boss 2 sample built at a target of 500 ppm, hence not the highest ppm sample. The maximum reported values for the Boss 2 sample could be dismissed as a local phenomenon yet the fact that the average nitrogen content is higher than for Boss 3 indicates that the former experienced more contamination than the latter.

As mentioned previously, all samples were reportedly within the compositional requirements of AMS4999A even when accounting for the error term. The oxygen content in Boss 2 (0.195 wt. %) was closest to the allowed limit of 0.2 wt. %.

6.3. Tensile testing

The tensile tests show that all samples were above the minimum requirements with no apparent correlation between oxygen content in the chamber and tensile properties.

Samples were taken at 15 mm and 30 mm from the substrate and Figure 29 shows that the bottom samples had a lower UTS. In his licentiate thesis, Magnus Neikter discovered a higher degree of microstructural texture closer to the substrate in Ti-6Al-4V samples created using LMD-w [5]. This means that more grains will be aligned in the same crystallographic direction at lower parts of the build. A higher degree of texture is known to decrease mechanical properties such as UTS which could explain the difference found in the studied samples.
6.4. Evaluation of microstructure

6.4.1. A-case measurements

As expected, boss samples showed the greatest \( \alpha \)-case contamination (2.4-2.7 times the thickness of wall samples) and also the largest variation in thickness. Bosses also showed a slight increase in maximum \( \alpha \)-case depth at higher oxygen concentrations while there was no significant difference between wall samples. All surfaces contained \( \alpha \)-case in at least one examined sample. Two places in the build were contaminated in all samples; namely beneath the substrate directly under the first layer as well as the lowest point on the side surface.

The findings of Gaddam et al [9], who reported of underestimated \( \alpha \)-case thickness using LOM, should be considered when setting a requirement for material removal due to \( \alpha \)-case contamination.

6.4.2. General microstructure

The general microstructural details of the builds were similar to that which has been reported in previous studies [10] [21]. Åkerfeldt et al ([21]) concluded that a faster cooling rate rendered smaller microstructural features (e.g., smaller prior \( \beta \) grains, \( \alpha \) colonies) which would improve yield strength and ultimate tensile strength but lower elongation. Furthermore, the thickness of \( \alpha \)-laths has a significant effect on tensile properties [1]. Although no tensile tests were performed on boss samples, lower YS and UTS and higher elongation should be expected due to the thicker \( \alpha \)-laths and larger microstructural features.

Mainly due to the higher amount of retained heat in the bosses, the HAZ spanned across the entire substrate while also displaying a coarser microstructure closer to the center of the sample. The latter indicates a thermal gradient in the radial direction within the build during deposition. This stands in contrast to wall samples where approximately half the substrate thickness is affected enough by heat to initiate microstructural transformation. Furthermore, there was no change in coarseness throughout the HAZ in wall samples.

6.5. Hardness

The hardness profiles show no significant trend in hardness vs. location in the build. This differs from results found in the literature. Figure 45 shows a typical LMD-w hardness profile with values increasing when stepping to the top. This is not apparent when looking at Figure 44. Although the load was different in Figure 45, the trend would be expected to remain the same. The mean hardness value of all samples was within 328-340 HV and the measurements vary around the mean regardless of position.
6.6. Experimental uncertainties

Chamber environment

The setup resembles a production environment in size and gas flow. Establishing an exact oxygen ppm level with no deviation is not possible, due to the large flow of gas (up to 500 l/min) and the increased oxygen affinity of titanium at higher temperatures.

Compositional analysis

The error of IGFA analysis was ±0.002 wt. %.

Tensile tests

For a full description of uncertainties related to the tensile testing see ASTM E8/E8M – 16a: Standard test methods for tension testing of metallic materials [27].

A-case measurements

A-case measurements using light optical microscopy is in most cases a viable alternative (error in the magnitude of ±2µm). However, there are situations where LOM has underestimated the thickness by as much as 40-50% [9]. When analyzing microstructures of AM material, which hasn’t been as thoroughly studied as conventionally manufactured material, it is worth taking this into consideration.
7. Conclusions

The following can be concluded with regard to the research question. The tested oxygen levels in the chamber did not have a significant impact on mechanical properties or microstructural properties (with the exception of \( \alpha \)-case formation). An increased uptake of oxygen and nitrogen was detected, although not enough to contaminate the specimen out of current aerospace specifications.

Due to the occurrence of significantly thick \( \alpha \)-case layers in all samples, it was also concluded that depositing material at higher oxygen levels it is not suitable for components without subsequent processing such as etching or machining.

The most important findings can be summarized as:

- In terms of chemical composition and tensile properties, all samples were in accordance with requirements set by AMS and ASTM for directly deposited Ti-6Al-4V.
- With the exception of \( \alpha \)-case formation, oxygen concentration in the build chamber was not shown to have an impact on microstructure.
- Maximum \( \alpha \)-case thickness in bosses was measured at 53, 55 and 61 \( \mu \)m following the increased oxygen concentration in the build chamber.
- Maximum \( \alpha \)-case thickness in walls was measured at 21, 21 and 22 \( \mu \)m showing no significant increase with regard to the evaluated interval of oxygen concentration in the build chamber.
- Due to the thickness of measured \( \alpha \)-case layers, subsequent processing is recommended for Ti-6Al-4V components manufactured with LMD-w at chamber oxygen concentration levels of 100 ppm and above.
- The profiles of hardness measurements did not follow the trend common in LMD-w samples found in the literature.
8. Future Work

Nitrogen and its effects on α-case creation is still relatively unexplored. Measurements of surface chemical composition indicate that a significant amount of nitrogen is dissolved in the material during processing. As it is a powerful stabilizer of the α-phase, it is an avenue worthy of exploration in order to understand the full process of α-case formation. Furthermore, EPMA analysis of AM samples could be utilized as a complement to light optical microscopy to verify the thickness of α-case layers and lower measurement uncertainties.
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10. References


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