Microstructure evolution and mechanical properties of selective laser melted Ti-6Al-4V

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Metadata Record: [https://dspace.lboro.ac.uk/2134/15070](https://dspace.lboro.ac.uk/2134/15070)

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“Microstructure Evolution and Mechanical Properties of Selective Laser Melted Ti-6Al-4V”

by

Marco Simonelli

Doctoral Thesis

Submitted in Partial Fulfilment of the Requirements for the Award of Doctor of Philosophy

2014

School of Aeronautical, Automotive, Chemical and Materials Engineering, Department of Materials

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Acknowledgements

First and foremost I would like to express my gratitude to my supervisors Dr. Yau Yau Tse and Dr. Chris Tuck for their patient, support and guidance throughout this study.

Yau Yau has taught me many things in these past three years. Not only she has helped me to understand the difficult principles that underpin this work, but also, she has been the first person to walk down in the labs to help whenever I needed. I feel privileged to have encountered Yau Yau in this time of my life. She has certainly contributed to my growth as an individual and as a researcher providing me with an outstanding example of professionalism and intelligence. For this, I would like to thank you Yau Yau.

I also want to express my gratitude to Chris. Chris has always been there when I need help and reassurance. I feel that I have learned much from his supervision. I still remember his words on research “..a PhD should be exciting [well, hopefully most of the time] but it should also be a bit frustrating..”. After countless hours of study, experiments, and writing there are indeed moments of frustration and awareness that what you have done is not enough. Chris has taught me that I should learn from these hard times. His constructive and insightful comments throughout my research have been priceless, thanks Chris.

I would like to express a sincere thanks to Mark Hardy and Mark East. Without them, the microstructural study in this research may have never started and I might still be trying to “tune” the Renishaw system. Thanks a million guys! I consider the “two Mark” as fellow friends. They have created a pleasure environment where I felt always welcomed. Thanks for the innumerable teas that you guys have prepared for me. Thanks for the laughs and for sharing your stories with me. I look forward to spending other years of work with you!

A sincere thanks goes to Geoff West who has helped me in most of the microscopy work presented in this thesis. Thanks for being so patient with me every time I made mistakes. Thanks for helping me whenever I was stuck in front of the microscope! Thanks to Shaun Fowler. Shaun you have supported me in many occasions. Thanks for sharing your passion for life with me.
Ruth and Borja, I am so glad that I have met you! You are an example of determination. Thanks to you I have understood that I should always try to go after my dreams..you have been source of motivation, thanks!

A special thanks goes to my dearest friends Gari, Zucca, Ale and Davide. They are the most clever people that I know. Even though we now live far apart, I feel that you are always there for me. In these times of uncertainty you guys have always shown to be solid rocks, a handhold for me...you are the best friends that I can possible image.

I am eternally grateful to my mum and dad. Without your continuous support I would have never gotten to where I am today. You have always been there to listen and help me when needed..I feel blessed when I think to have such a wonderful family. Thank you!

Finally, I wish to say thank you to Eli, for her love, patience and support. I am confident that our efforts will soon pay off. For a life full of smiles. You are my motivation, my strength. This work is mainly dedicated to you.
Abstract

Selective laser melting (SLM) has been shown to be an attractive manufacturing route for the production of α/β titanium alloys, and in particular Ti-6Al-4V. A thorough understanding of the relationship between the process, microstructure and mechanical properties of the components produced by this technology is however crucial for the establishment of SLM as an alternative manufacturing route. The purpose of the present study is thus to determine the microstructure evolution, crystallographic texture and the mechanical properties of SLM Ti-6Al-4V.

The effect of several processing parameters on the density and the microstructure of the SLM samples were initially investigated. It was found that different sets of process parameters can be used to fabricate near fully dense components. It was found that the samples built using the optimised process window consist exclusively of α′ martensitic phase precipitated from prior β columnar grains. It was observed that the β grain solidification is influenced by the laser scan strategy and that the β phase has a strong \(<100>\) texture along its grain growth direction. The α′ martensitic laths that originate from the parent β grains precipitate according to the Burgers orientation relationship. It was found that α′ laths clusters from the same β grain have a specific misorientation that minimise the local shape strain. Texture inheritance across successive deposited layers was also observed and discussed in relation to various variant selection mechanisms.

The mechanical properties of as-built and stress relieved SLM Ti-6Al-4V built using the same optimised process parameters were then investigated. It was found that the build orientation affects the tensile properties, and in particular the ductility of the samples. Samples built perpendicularly to the building direction showed higher ductility than those built in the vertical orientation. It was also observed that a stress relief heat treatment was beneficial to the mechanical properties of SLM Ti-6Al-4V. The ductility of the stress relieved samples was indeed higher than those found in the as-built condition. It was found that the predominant fracture mode during tensile testing is inter-granular. In terms of high-cycle fatigue, it was found that SLM Ti-6Al-4V is comparable to HIPed cast Ti-6Al-4V but it has a significantly lower fatigue resistance than that of wrought and annealed alloys. It was observed that porosity and the elongated prior β grain boundaries decrease substantially the...
fatigue life of the components. Cracks propagate either by fatigue striation or ductile tearing mechanisms.

Using alternative laser scan strategies it was possible to control the microstructure of the as-built samples. It was observed that the laser scan vector length influences several microstructural features, such as the width of the prior β grains and the thickness of the α’ laths. It was found that re-melting the same layer has instead little effect on the microstructure. A novel laser scan strategy characterised by much lower laser power and scan speed than those typically used in SLM enabled finally to fabricate SLM Ti-6Al-4V with a microstructure close to that of conventionally manufactured Ti-6Al-4V.

This study investigates for the first time the crystallographic texture evolution in Ti-6Al-4V manufactured by SLM. Further, this research presents for the first time the effect of the characteristic microstructure and crystallographic texture on the mechanical properties and fracture of SLM Ti-6Al-4V. Lastly, for the first time this research shows examples of microstructural control during the SLM fabrication of the same alloy using long laser dwell times.
List of publications


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Titanium alloys, and in particular Ti-6Al-4V alloys, have a wide range of applications including the aerospace, medical, automotive and the chemical industries, thanks to their high specific strength to density ratio, excellent corrosion resistance and excellent mechanical properties at high temperatures. The cost of production of titanium alloys represents however, a limit to a still broader utilisation of these alloys [Banerjee and Williams, 2013].

The cost of titanium alloys derives mainly from two aspects: 1) the cost related to its extraction method (the currently used Kroll’s reduction process is energy-intensive and not yet scalable) and 2) the cost of the current fabrication processing that requires protective environments, high energy consumption and typically involves significant material waste [Imam and Froes, 2010; Banerjee and Williams, 2013]. To decrease the overall cost of production, research has therefore focussed on alternative efficient manufacturing methods [Lütjering and Williams, 2007]. In recent years, additive manufacturing (AM) has certainly created great interest to combat some of these issues. In principle, AM allows one to fabricate a component of almost any desired shape from a digital model in an additive manner where successive layers of material are deposited and fused together.

Besides giving the possibility to create components with new functional geometries that are impossible to achieve with conventional processing, the use of AM is associated to a significant number of other advantages [Gibson et al., 2010]. As an example, AM uses just the raw material that is needed to make a component, irrespectively of its shape, reducing significantly the material waste, with the exception of support structures in some cases. Research has shown that AM is also environmentally sustainable: AM allows a reduction in the energy consumption and CO$_2$ emissions compared to traditional processing of metallic materials [Baumers et al., 2010].

One of the most promising AM technologies for the production of titanium alloys is Selective Laser Melting (SLM). Studies in the literature have shown that highly dense (99.7% or more) SLM titanium alloy can be manufactured after fine tuning of the SLM system. Thus, highly dense SLM titanium alloys possess mechanical properties comparable to that of the alloys manufactured conventionally. The majority of the research on SLM of titanium alloys has however focussed on choice of the process parameters that can increase the density of the samples and the build rate with little regard of the microstructure of the parts. For this reason, part quality, repeatability and
mechanical properties of SLM samples are often inconsistent when different SLM systems are used.

Limited studies have characterised the microstructure of SLM titanium, and in particular SLM Ti-6Al-4V. The literature lacks therefore a systematic study of the origin and the evolution of the microstructure during SLM. It is clear however that such a study has crucial importance, especially for the acceptance of SLM as a viable alternative method for the fabrication of titanium alloys. Similarly, the effect of the microstructure and crystallographic texture on the fracture mechanisms in the SLM parts has not yet been fully understood. In turn, the mechanical performance of the SLM Ti-6Al-4V has not yet been systematically interpreted.

The aim of the study is thus to increase the understanding of these aspects: this research work will primarily discuss the microstructure evolution of SLM Ti-6Al-4V and the major microstructural effects on the tensile and fatigue fracture of the fabricated samples. A preliminary study on how the process parameters affect the densification of SLM Ti-6Al-4V has been carried out. It is, in fact, fundamental to establish a procedure to ensure that the results and the quality in the components is highly repeatability. The objective of this research was also to assess whether the microstructure of SLM Ti-6Al-4V can be controlled during the process whilst maintaining full density. Being able to directly manufacture SLM Ti-6Al-4V with a microstructure similar to that of conventionally made α+β titanium alloys would undoubtedly facilitate the comprehension and predictability of the mechanical properties of the alloy, and in consequence, increase the acceptance of SLM as a manufacturing process.

A review on the physical metallurgy of titanium alloys (e.g. Ti-6Al-4V) obtained using traditional manufacturing and AM methods is reported in Chapter 2. The Chapter also includes a review of the state of the art of AM of titanium alloys and a description of the EBSD technique. EBSD was in fact extensively used in this research work to study the microstructural evolution of the SLM Ti-6Al-4V and the fracture behaviour of the parts. The materials and the experimental procedures of this research work are reported in Chapter 3. Chapter 4 discusses the microstructure of the plasma atomised Ti-6Al-4V, i.e. the starting powder material. Chapter 5 outlines the aspects related to the process parameters selection for the fabrication of near fully dense samples. The microstructure and crystallographic texture evolution of the SLM Ti-6Al-4V is then discussed in Chapter 6. Chapter 7 presents the tensile and fatigue properties of SLM Ti-6Al-4V. The attempts to control the microstructure directly during SLM are reported in Chapter 8. Finally the conclusions and the suggestions for future work are reported in Chapters 9 and 10.
CHAPTER TWO

2 Literature Review

2.1 Physical Metallurgy of Titanium and Titanium alloys

2.1.1 Introduction

The main objective of this study is to understand the evolution of the microstructure and the mechanical properties of Ti-6Al-4V manufactured by Selective Laser Melting (SLM). To explore the real potential of the SLM production of Ti-6Al-4V and to contextualise the results presented in this research work, a review of the physical metallurgy of titanium and titanium alloys processed by traditional manufacturing will be presented in this chapter.

Despite the fact that titanium and titanium alloys are generally appreciated for their high specific strength and corrosion resistance, the physical properties of this material system vary significantly with the phase composition of the alloys, the relative microstructure and crystallographic texture. Thus, the complex relationship between the traditional processing and properties of titanium and in particular Ti-6Al-4V is discussed in the first part of the literature review.

A classification of titanium alloys is provided in Section 2.1.2. This section gives information of where Ti-6Al-4V stands among the numerous Ti alloys and the most common application for these alloys. Section 2.1.3 describes the technological aspects related to the traditional manufacturing routes of Ti alloys. This review will pose particular reflections on the current possibility and limitation of Ti alloys manufacturing. The common nomenclature and the microstructure (and texture) of the conventionally produced titanium alloys and in particular Ti-6Al-4V are then reviewed in Section 2.1.4. It is well known that the microstructural and texture development has a significant impact on the mechanical performance of titanium alloys and Ti-6Al-4V and thus the rules of solidification and solid phase transformations during conventional processing of Ti alloys are reviewed.
2.1.2 Titanium Alloy Classification

Titanium and in particular titanium alloys derive their mechanical properties mainly from their phase composition and the arrangement of the phase constituent at a microscopic scale [Lütjering, 1998]. The classification of Ti alloys is generally based on the volume fraction of the main phase present in the alloy at room temperature that in turn is affected by the elemental composition of the alloy. At room temperature, pure titanium (Ti) possesses a hexagonal close packed structure, that is generally referred to as α phase [Donachie, 2000]. Ti is however an allotropic material and when the temperature reaches ~ 882°C (temperature commonly known as β transition temperature, or β transus), it transforms fully into a body-centered cubic (β) phase [Donachie, 2000]. Alloying pure Ti generally alters significantly the transition temperature of the allotropic phase and the volume fraction of the α and β phase at room temperature [Donachie, 2000]. Alloying elements can either:

- stabilize further the α phase by raising the α/β transition temperature
- stabilize the β phase by lowering the α/β transition temperature
- act only as solid solution straighteners and not affect the transition temperature.

Figure 1 shows the effect of some β stabilizers on the volume fraction of the α and β phase at room temperature. The diagram also indicates the martensite start temperature (Ms) and the lower transformation temperature (Mf). As it will be discussed in Section 2.1.4.1, non-equilibrium martensitic phases can occur during quenching of certain Ti alloys.

Ti-6Al-4V is obtained by alloying pure Ti with 6 wt% Al (α stabiliser) and 4 wt% V (β stabilizer). The addition of V causes the lowering of the α/β transition temperature and therefore at room temperature the microstructure of Ti-6Al-4V consists of a mixture of α+β equilibrium phases. The volume fraction and the microstructure of the α+β equilibrium phases can be controlled with designed heat-treatments and/or thermo-mechanical processing [Donachie, 2000].
The relation between processing, microstructure and the mechanical properties of Ti alloys is reviewed in Section 2.1.3.

2.1.3 Traditional Manufacturing of Titanium Alloys

The production of Ti alloy components from Ti ore can be summarised in four major steps [Pederson, 2002]:

- Ti ore is firstly reduced into a porous form referred to as Ti sponge.
- Ti sponge is then melted and re-melted several times for refining purposes to form ingots of controlled composition and homogeneous microstructure
- ingots are then converted into general mill products
- finally, mill products are fabricated into finished end-use components.

Details of the processing at different stage will be discussed hereafter in this section.
2.1.3.1 Primary Fabrication of Ti Alloys

The sponge form of Ti derives from rutile ore (TiO$_2$) after a process of chlorination and reduction. The current industrial reduction process (Kroll’s reduction) is energy-intensive and therefore it represents an significant element of cost for the production of Ti alloys [Froes et al., 2004]. Research efforts have been made to find suitable alternative reduction methods but, to-date, Kroll’s reduction remains the principal reduction method in use [Banerjee and Williams, 2013].

Ti sponge is then purified from external chemical compounds or segregates, such as hard, brittle and refractory Ti oxides, Ti nitrides or complex oxynitride particles that could become crack initiation sites during the subsequent production steps [Lütjering and Williams, 2007].

Ingots are then formed by melting the sponge Ti typically through vacuum arc melting (VAM) [Lütjering and Williams, 2007]. This is the production step where alloying elements can be added. The chemical and microstructural homogeneity of the ingots is controlled through successive heat treatments conducted below the β transus temperature and controlled cooling rates to room temperature. Ingots are then used to fabricate generic mill products through a variety of processes generally referred to as primary fabrication of Ti alloy.

Typical mill products include billets, bars, plates, sheets, powders and wires [Welsch et al., 1994]. Billets, bars, plates, sheets are typically forged in an open die press and are thermo-mechanically processed to the final ordered dimensions and required microstructural and mechanical properties.

2.1.3.2 Secondary Fabrication of Ti Alloys

The primary method to shape mill products into final titanium components is forging [Lütjering and Williams, 2007]. The advantage offered by forging is that the final shape of the components is given through successive forging steps and heat treatments that allow a close control of the final microstructure of the alloy. Forging is generally followed by extensive machining to provide the component with its final shape and surface finish. Forging requires dedicated dies, a series of dedicated furnaces to control the oxidation of the alloys and extensive machining and hence is a costly manufacturing process [Froes et al., 2007]. Research has estimated that forging alone represent the 70% of the entire Ti production cost [Murr et al., 2009].
In order to reduce both the material waste associated with the machining of the forged components and the difficulty associated to the assembly/join of multiple distinct parts into one components, casting of Ti alloys has received great attention in the recent year [Lütjering and Williams, 2007; Froes et al., 2007; Imam and Froes, 2013]. Contrary to forging, casting of Ti alloys is however subject to quality issues (i.e. porosity and small cracks) especially in the case of cast components with a complex shape [Lütjering and Williams, 2007]. Casting, therefore, is generally followed by hot isostatic pressing (HIP) that has been shown to be greatly beneficial to close internal pores present in the casting of Ti alloys [Murr et al., 2009].

Components with large sections can be produced through traditional sheet forming of Ti alloys [Welsch et al., 1994]. Forming of Ti alloys however has to be conducted at high temperatures and requires relatively long times because of the high specific yield stress typically associated with Ti alloys [Donachie, 2000; Lütjering and Williams, 2007]. Traditional forming of Ti alloys is thus expensive and is progressively being replaced by a more recent forming technology known as superplastic forming [Boyer, 1995; Boyer, 1996]. Superplastic forming of Ti alloys has proved to be a convenient technology to shape sheets of Ti alloys in a near net shape without the use of fasteners or the need of joining of multiple parts or machining [Lütjering and Williams, 2007]. Superplastic forming can be achieved thanks to the superplastic behaviour of Ti alloys, i.e. stress flow and plastic deformation are sensitive to strain rate, temperature and microstructure. For this reason, when Ti alloys are applied a low strain rate, in the α+β temperature phase field, superplastic deformation can occur. Shaping of several sheets of Ti alloys by superplastic forming is usually combined with diffusion bonding. Because of surface oxide layers, Ti alloys sheets are capable of forming mechanical bonds by diffusion bonding. It has been shown that using superplastic combined with diffusion bonding is possible to fabricate honeycomb structures and components with large sections in a cost effective manner and thus this manufacturing technology is gaining more interest in the aerospace industry [Leyens et al., 2003]. However superplastic forming coupled with diffusion bonding is now limited to the production of non structural components, because of the risk associated with lack of bonding between the individual layers that could cause early fatigue and failure [Lütjering and Williams, 2007].

2.1.3.3 Powder Metallurgy of Ti Alloys

In the last 20 years, powder metallurgy, i.e. compaction of Ti powders into final component, has been identified as a cost effective manufacturing methods to produce near net shape Ti
Pre-alloyed or pure Ti powders are mainly prepared by plasma or gas atomization [Froes and Eylon, 1990; Froes et al., 2004]. Plasma atomization (PA) is accomplished in two consequent steps: 1) shearing of a liquid metal stream and 2) freezing of the liquid into separate droplets. During PA, a titanium alloy wire is fed into the apex of three argon (Ar) plasma torches, which melt the wire directly. Droplets are then formed as a result of the aerodynamic drag experience by the liquid. The droplets are then rapidly cooled during their free-fall in an inert Ar atmosphere with a cooling rate in the range of 100°C – 1000°C/s. The particles produced by plasma atomization are therefore spherical and their size distribution ranges between 25–250 μm [Froes et al., 2004; Wang et al., 2010]. Gas atomization (GA) relies on a variety of gravity feed systems that are capable of generating a liquid metal stream from either bars, ingots or other mill products and therefore is more common than PA. Once the starting material is melted an Ar gas at a pressure of 2-3 MPa with a flow rate of 10-15 m³/min is then used to atomize the liquid stream of metal. The resulting Ti powders have are mainly spherical with diameters generally smaller than 180 μm [Wang et al., 2010]. The particle size distribution associated with GA is broader than that of PA, and some satellites (smaller particles that remain attached to larger ones during solidification) or small asymmetrical particle can originate [Wang et al., 2010]. Examples of Ti-6Al-4V powders produced by PA and GA are shown in Figure 2.

Figure 2: Micrographs showing: a) gas and b) plasma atomised Ti-6Al-4V powders [Ahsan et al., 2011].
Two powder metallurgy techniques, hot isostatic pressing of pre-alloyed powders, cold isostatic and sintering of blended elemental powders, have led to promising results of producing parts with good microstructure and mechanical properties [Lütjering and Williams, 2007; Singh et al., 2013].

During hot isostatic pressing of pre-alloyed powder, the powders are generally packed into a capsule/mould of a shape corresponding to the final intended object geometry. Powders are then sintered placing the capsule into a cylindrical hot isostatic press. Argon gas is generally used to achieve high hydrostatic pressure. The consolidation is generally carried out at high temperature (just below the β transus temperature). Once powder has been consolidated the capsule can be removed leaving behind the desired component. Hot isostatic pressing of prealloyed powder has shown the possibility of manufacturing components with homogeneous full density and requiring minimal final machining. Optimal microstructures can be tailored using successive furnace heat treatments. Given the high cost of pre-alloyed powders and the HIPing process itself the future of hot isostatic pressing of pre-alloyed powered remains uncertain [Singh et al., 2013].

Cold isostatic and sintering of blended elemental powders is conducted by cold pressing in a die or cold-isostatic pressing blended elemental Ti powders. The parts are then sintered at high temperature above the β transus temperature. Although cold isostatic pressing and sintering of blended powders has proved to be more economical than hot isostatic pressing of pre-alloyed powder, porosity is often an issue in the final components and secondary post process operations such as HIPing are required to eliminate porous defects [Lütjering and Williams, 2007].

2.1.4 Microstructure and Texture Formation in Ti Alloys

Thermo-mechanical processing of α+β Ti alloys can create three distinct microstructures: the fully lamellar, fully equiaxed and the bimodal (or duplex) microstructure. The relationship between the process and microstructure is reviewed in this section. The crystallographic texture formation in Ti alloys, with particular regard to Ti-6Al-4V is summarised in Section 2.1.4.4. Although it is certain that microtexture plays a significant role in the mechanical properties of Ti alloys, the understanding of the texture evolution during Ti alloy manufacturing remains an open challenge of fundamental importance [Semiatin and Bieler, 2001].
2.1.4.1 The Formation of Fully Lamellar Microstructure in α+β Ti alloys

The fully lamellar microstructure derives its name from the fact the α phase present in this microstructure has a lamellar or plate-like shape as shown in Figure 3.

![Micrographs of fully lamellar microstructures](image)

*Figure 3: Optical micrograph showing examples of fully lamellar microstructures derived from different cooling rates; a) slow and b) intermediate cooling rate, c) quenching [Lütjering, 1998].*

The fully lamellar microstructure can be achieved through a 4-stage processing route as shown schematically in Figure 4.

![Processing route](image)

*Figure 4: Processing route for the formation of fully lamellar microstructures [Lütjering and Williams, 2007]*

The microstructure of the ingot is initially homogenised in the β phase field (stage I), then either rolled or forged at high temperature typically around the β transus temperature of the alloy (stage II). The component is then heated in the β phase field (stage III) and then cooled to room
temperature in a controlled cooling rate. Finally, the parts are typically annealed in the \( \alpha+\beta \) phase field temperature (stage IV) as a stress relieving treatment, to eliminate segregates or to allow solid phase transformations between any martensitic phases towards the equilibrium \( \alpha+\beta \) phase mixture.

The most influential parameters to produce fully lamellar microstructures are stage III and IV [Lütjering and Williams, 2007]. The size of the grains in the \( \beta \) phase field, also known as prior \( \beta \) grain size, is in fact determined by the temperature and the time of the \( \beta \) phase field treatment. Ti-6Al-4V is generally processed within 30 – 50°C above the \( \beta \) transus to avoid an excessive \( \beta \) grain growth. The cooling rate of stage III determines the size of the \( \alpha \) lamellar grains (also known as \( \alpha \) lath size), their arrangement and the amount of retained \( \beta \) phase at room temperature as schematically shown in Figure 3a-c. Despite the composition of the \( \alpha+\beta \) Ti alloys, higher cooling rates produce lamellar microstructure with finer \( \alpha \) grains and limited retained \( \beta \) phase. Furnace cooling of Ti-6Al-4V generally produces lamellar microstructure with packets \( \alpha \) laths arranged in a parallel manner (also known as \( \alpha \) colonies) as shown in Figure 3a. This fully lamellar microstructures generally contain retained lamellar \( \beta \) phase around the \( \alpha \) grain boundaries as illustrated Figure 3a. \( \alpha \) colonies typically originate at the prior \( \beta \) grain boundary and grow towards the interior of the prior \( \beta \) grain [Banerjee and Williams, 2013]. Furnace cooling determines fully lamellar microstructures with largest possible \( \alpha \) colony size.

Air cooling of Ti-6Al-4V generates the so-called basketweave microstructure (also known as Widmanstätten microstructure), where \( \alpha \) laths are arranged similarly to weave pattern of a basket [Pederson, 2002]. Basketweave microstructures contain limited retained \( \beta \) phase, that if present, is located at the \( \alpha \) grain boundaries.

Finally if the cooling rate is increased further (for example in case of oil or water quenching), fine martensitic microstructures are produced with no retained \( \beta \) phase [Pederson, 2002]. This microstructure is shown in Figure 3c. In basketweave and martensitic microstructures, it is more difficult to locate the sites where the \( \beta \) In initiated, and only occasionally \( \alpha \) lath grown at the prior \( \beta \) grain boundary can be found.

The martensitic phase of Ti results when the \( \alpha \) phase is supersaturated in \( \beta \) stabilizing elements. Research has shown that the martensitic transformation occurs by shear transformation, where atoms transform homogeneously in a collaborative ordered movement [Pilchak, 2009].

The martensitic phase in Ti alloys, designated as \( \alpha' \), occurs in two similar morphologies: acicular martensite and massive martensite. Acicular martensitic \( \alpha' \) grains are shaped and pointed like
needles and have a thin lenticular aspect in 3D. Acicular martensitic α’ laths tend to cross each other, and are difficult to discern at the optical level [Banerjee and Williams, 2013]. Each lath has generally a distinct crystal orientation and for this reason the α’ colony size corresponds to the thickness of a single α’ laths. For relative lower cooling rates, massive α’ martensite tends to form in place of acicular α’ martensite. The fundamental difference between the two martensitic phases is that α’ massive martensite consists of irregular zones (size 50-100 nm) of laths that share the same crystallographic orientation [Lütjering and Williams, 2007]. Although the crystal structure of α’ martensitic phase (acicular and massive) is similar to equilibrium α phase, the chemical composition of the martensitic α’ phase differs from the equilibrium α phase. For example, in the case of Ti-6Al-4V, α’ phase presents a higher content of V respect to the equilibrium α phase [Ahmed and Rack, 1998]. Studies on the crystal structure of the α’ phase have shown that α’ plates possess high density of dislocation of crystal defects such as staking faults and twins respect to the equilibrium α phase [Qazi et al., 2001].

During the last annealing stage (stage IV), depending on the choice of the annealing temperature it is possible 1) to eliminate compounds that have micro-segregated (typically Ti₃Al ), 2) stress relieve the microstructure that might result from a non-homogeneous quenching from the β phase field or 3) trigger a solid phase transformation during which the martensitic phase transformed into the equilibrium mixture of the α+β phase.

### 2.1.4.2 Static and Dynamic Mechanical Properties of Fully Lamellar Microstructures

The mechanical properties of α+β Ti alloys vary in relation to the microstructure of the alloy. Table 1 provides an example of such variation in Ti-6Al-4V with fully lamellar microstructure.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Yield Strength [Mpa]</th>
<th>UTS [Mpa]</th>
<th>Elongation [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V&lt;sub&gt;water quench&lt;/sub&gt;</td>
<td>1035</td>
<td>1095</td>
<td>13</td>
</tr>
<tr>
<td>Ti-6Al-4V&lt;sub&gt;air quench&lt;/sub&gt;</td>
<td>970</td>
<td>1040</td>
<td>15</td>
</tr>
<tr>
<td>Ti-6Al-4V&lt;sub&gt;furnace cooling&lt;/sub&gt;</td>
<td>910</td>
<td>980</td>
<td>16</td>
</tr>
<tr>
<td>cast Ti-6Al-4V</td>
<td>900 - 1000</td>
<td>950 - 1050</td>
<td>5 - 7</td>
</tr>
<tr>
<td>cast + HIPing</td>
<td>800 - 900</td>
<td>850 - 950</td>
<td>8 - 10</td>
</tr>
</tbody>
</table>
The size of the $\alpha$ colony is the fundamental parameter that affects the mechanical performance of Ti alloys with a fully lamellar microstructure [Lütjering, 1998]. Studies have confirmed that the size of the colony determines the slip length and therefore determine in practice the onset plastic deformation in $\alpha+\beta$ Ti alloys [Chesnutt et al., 1976; Chesnutt et al., 1978; Lütjering 1998]. As the yield strength depends on the resistance to dislocation motion, increasing the $\alpha$ colony size (and thus the slip length) corresponds to a decrease in the yield strength in the alloy. For this reason, Ti alloys that are furnace cooled during stage III have the lowest yield strength among the alloys with fully lamellar microstructures as listed in Table 1. Similarly, the ductility of the lamellar microstructures is also affected by the size of the $\alpha$ colony. Ductility decreases with a decrease in $\alpha$ colony size consistently with the fact that the slip length in the alloy decreases. Fine lamellar (or martensitic) microstructures, where slip length is essentially the size of a single $\alpha$ lath, present thus the lowest ductility among the fully lamellar microstructures. In these microstructures, fracture is generally dominated by intergranular fracture mechanisms and for this reason the tensile ductility decreases [Lütjering and Williams, 2007]. As the resistance to crack nucleation depends on the resistance to dislocation motion, the fatigue resistance in the high-cycle regime scales directly with the yield strength of a given alloy. The resistance to high-cycle fatigue (HCF) is significantly affected by the $\alpha$ colony size [Chesnutt et al., 1976]. It has been demonstrated that fine lamellar microstructure with small $\alpha$ colonies better retard micro-crack propagation than microstructures with large colonies [Nalla et al., 2002]. On the other hand, it has been shown propagation of large cracks is mainly retarded by the deflection of the crack tip. Large $\alpha$ colonies provide, in this sense, better resistant to the propagation of large cracks [Nalla et al., 2002].

The second most important parameter in terms of effect on mechanical behaviour of fully lamellar is the length of the prior $\beta$ grains [Lütjering and Williams, 2007; Hu et al., 2000]. Grain boundaries are weak points in the microstructure where cracks can propagate. Fully lamellar microstructures with long prior $\beta$ grain boundaries exhibit in general poor ductility and resistance to short crack propagation. Long prior $\beta$ grain boundaries can retard the propagation of large cracks as they induce crack deflection similarly to the crack propagation behaviour for large $\alpha$ colonies. It has been shown that large cracks are deflected along the grain boundaries and thus crack path of microstructure with long grain boundary are generally tortuous and thus crack propagation in these microstructures is associated with more energy expense [Nalla et al., 2002; Birosca et al., 2009].
2.1.4.3 Bimodal Microstructures

The bimodal (or duplex) microstructure consists of α phase in the form of lamellar and equiaxed grains as shown in Figure 5.

Figure 5: Optical micrographs showing examples of bimodal microstructures [Lütjering, 1998]. A difference in the size of the equiaxed grains can be observed in the micrographs.

The bimodal microstructure can be produced using a processing route similar to that shown for the fully lamellar microstructure. In this case however, the recrystallization stage (stage III) is conducted in the α + β phase field. The microstructure of the ingot is initially homogenised in the β phase field (stage I) and then cooled down to room temperature. The cooling rate associated with this processing stage determines the width of the lamellar α grains and the maximum size of the equiaxed grain [Lütjering and Williams, 2007]. Figure 5 show two examples of bimodal microstructure with different equiaxed grain size. During stage II (generally conducted in the α+β phase field), the α lamellar grains are then plastically deformed. At this stage, a preferred crystallographic texture of the can be imparted. Details will be covered in Section 2.1.4.5. Recrystallization (stage III) is then carried out. New undeformed equiaxed α grains (also known as α_{primary}) can therefore grow at the expense of the deformed α lamellar grains. The recrystallization temperature determines the volume fraction of equiaxed grains in the microstructure [Lütjering and Williams, 2007]. Depending on the cooling rate, the retained α lamellar grains (also known as secondary α) will have a typical lath width as described in Section 2.1.4.2. The final annealing stage (stage IV) is conducted to eliminate micro-segregates and residual stresses in the microstructure [Lütjering and Williams, 2007].
2.1.4.4 Static and Dynamic Mechanical Properties of Bimodal Microstructures

Figure 6 and Table 2 show the mechanical properties of several important α+β Ti with bimodal microstructure. In general, α+β Ti alloys with a bimodal microstructure have good ductility and high elongation at break [Lütjering, 1998]. This is explained by the fact that generally bimodal microstructures have a higher volume fraction of β phase compared to the fully lamellar microstructures obtained with a similar cooling rate [Pilchak et al., 2009].

Table 2: Properties of Ti-6Al-4V with bimodal microstructure [data from: Chesnutt et al., 1978; Lee, 2004; Hines and Lütjering, 1999]

<table>
<thead>
<tr>
<th>Alloy (%volume fraction α_p*)</th>
<th>Yield Strength [MPa]</th>
<th>UTS [Mpa]</th>
<th>Elongation [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V (10)</td>
<td>940</td>
<td>990</td>
<td>12.3</td>
</tr>
<tr>
<td>Ti-6Al-4V (30)</td>
<td>972</td>
<td>1069</td>
<td>14</td>
</tr>
<tr>
<td>Ti-6242 (5)</td>
<td>790</td>
<td>953</td>
<td>11</td>
</tr>
<tr>
<td>Ti-6242 (33)</td>
<td>826</td>
<td>956</td>
<td>12</td>
</tr>
<tr>
<td>Ti-6242 (60)</td>
<td>886</td>
<td>982</td>
<td>15</td>
</tr>
<tr>
<td>Ti-6242 (69)</td>
<td>883</td>
<td>972</td>
<td>16</td>
</tr>
</tbody>
</table>

*α_p*: equiaxed primary α

Figure 6: S-N curve comparison for Ti-6Al-4V with bimodal and lamellar microstructure [Nalla et al., 2002]
The mechanical properties of bimodal microstructures are mainly controlled by the annealing at stage III. Stage III determines the size of the β grains in the microstructure that is inversely proportional to the size of primary equiaxed α grains ($\alpha_p$) and their volume fraction. As shown in Table 2, with the increase of the volume fraction of the $\alpha_p$ the strength and the ductility of the alloys increase Lee, 2004. The actual size of the β grains instead, determines the final maximum α colony size, and therefore small β grains are associated with high yield strength, good ductility, and good resistance to high-cycle fatigue.

It has been shown that when alloying element partitioning is eliminated, the bimodal microstructure has however, a lower resistance to high-cycle fatigue compared to fully lamellar microstructures consistent with their typical lower strength (Figure 6) [Nalla et al., 2002]. However, in the low-cycle fatigue regime, this behaviour is reversed as shown in Figure 6. This is consistent with the fact that bimodal microstructure shows in general a higher ductility than fully lamellar microstructures [Nalla et al., 2002].

As a final remark, it should be noted that alloying element partitioning can affect the mechanical performance of bimodal microstructures and thus represent an issue to take into consideration during [Lütjering and Williams, 2007]. It is reported that during the formation of equiaxed primary α grains (stage III) the alloying elements can partition within the α and β phase according to their solubility in the two phases. Micro-segregation and brittle precipitates such as Ti$_3$Al can thus form in the equiaxed α phase causing a general decrease in the yield strength of the alloy [Lee, 2004]. In case of alloying element partitioning lamellar α grains are depleted with alloying elements thus decrease the resistance to high-cycle fatigue of the bimodal microstructure.
2.1.4.5 Fully Equiaxed Microstructure

Fully equiaxed microstructure consists exclusively of α phase in the form of equiaxed grains as shown in Figure 7.

Figure 7: Example of Ti-6Al-4V with a fully equiaxed microstructure [Lütjering, 1998]

The fully equiaxed microstructure can be obtained by modifying the processing route described in Section 2.1.4.3 for the bimodal microstructure. One method to create fully equiaxed microstructures is to impose a slow cooling rate during the stage III [Lütjering and Williams, 2007]. In this way, the volume fraction of primary α (equiaxed grains) will dominate the microstructure at the expense of the lamellar α grains. The second methods to obtain fully equiaxed microstructures are to conduct the recrystallization stage (stage III) at a relatively low temperature so that equiaxed grain can form directly during recrystallization of the deformed grains [Lütjering and Williams, 2007]. In terms of mechanical properties, yield strength, ductility and high-cycle fatigue resistance depend on the slip length, similar to that reported in Section 2.1.3.4. The size of the equiaxed α grains determines the mechanical properties of Ti alloys with fully equiaxed microstructures [Lütjering, 1998].

2.1.4.6 Texture Formation in Titanium Alloys

The crystallographic texture can be defined as the distribution of preferred crystallographic orientations in a polycrystalline material. The analysis of crystallographic texture of Ti alloys is important for a number of reasons. As texture is usually formed through deformation during primary and secondary fabrication, texture analysis can be used to deduce the unknown thermo-
mechanical history of a Ti alloy [Pilchak, 2009]. In addition, as the evolution of crystallographic texture in Ti alloys is strictly related to their solidification process, texture analysis allows a precise interpretation of the microstructure in study [Al-Bermani et al., 2010; Knipling and Fonda, 2011]. Finally, studies have shown that the fracture mechanism of Ti alloys in a number of condition is linked to the local microscopic orientation of the grains, thus texture has a significant effect on tensile and fatigue properties of Ti alloys [Peters et al., 1984; Pilchak and Williams, 2011; Pilchak et al., 2012]. To facilitate the discussion of the results presented in this research, this section reviews the development of texture in traditional processing of Ti alloys and the relationship between plastic deformation and crystallographic texture in Ti alloys. The methods used to quantitatively study and represent texture are discussed later in Section 2.3. The origin of the formation of crystallographic texture in metal systems (and Ti alloys) are well established [Kocks 1998 and 2000]. When polycrystalline metals are strained, grains undergo plastic deformation through specific known slip or twinning systems. As a consequence of the deformation under external loading, the atoms in the crystal lattice assume new equilibrium positions, and cause a lattice rotation with respect to the initial orientation of the undeformed grain. The new grain orientations are thus referred to as equilibrium orientations. Although modelling and prediction of the equilibrium orientations in a polycrystalline material is complex, it has been shown that equilibrium orientations can be deduced from a few established theories [Beausir et al., 2007]. Given a certain initial orientation of the grain, it has been shown that the end equilibrium orientation depends on the symmetry of the crystal phase of the polycrystalline material. In particular, grains deform to maximise the resolved shear stress on the slip system with the lowest critical resolved shear stress [Beausir et al., 2007]. In the case of α+β Ti alloys, the microstructure is generally dominated by the α phase that has three main slip systems as shown in Figure 8.

![Figure 8: Schematic representation of the a) slip systems in the α phase and b) their critical resolved shear stress (CRSS) [Banerjee and Williams, 2013].](image)
The slip system associated with the lowest critical resolved shear stress (CRSS) is the prismatic \( \langle a \rangle \) slip \( \{10\overline{1}0\}[1\overline{1}20] \). As shown in Figure 8, this is followed by the basal \( \langle a \rangle \) slip \( \{0002\}[1\overline{1}20] \), and the pyramidal \( \langle c+a \rangle \) slip \( \{1\overline{1}22\}[1\overline{1}23] \) that is about two to five times more difficult to be activated than the two other slip systems [Lütjering and Williams, 2007]. As only a limited number of slip systems are available to accommodation the deformation, mechanical working of \( \alpha+\beta \) Ti alloys can thus determine a strong texture in the \( \alpha \) phase and thus in the material.

In practise, the \( \alpha \) texture of a Ti alloy component is affected by the following main processing stages:

- solidification stage during ingot/cast processing (solidification texture)
- last deformation stage in the \( \alpha+\beta \) or \( \beta \) phase field experienced by the alloy (stage II) (deformation texture)
- cooling rate after the recrystallization (or annealing) stage (stage III) (transformation texture)

The transformation from the \( \beta \) to the \( \alpha \) phase is based on the Burgers crystallographic orientation relationship (BOR), according to which:

\[
\{0001\}_\alpha // \{110\}_\beta \text{ and } <11\overline{2}0>_\alpha // <111>_\beta
\]

In other words, after phase transformation the \{0001\} basal planes of the \( \alpha \) phase is parallel to the \{110\} of the \( \beta \) phase and the close-packed \( <1\overline{2}0> \) direction is parallel to the \( <111> \) direction [Donachie, 2000]. Because there are six non-parallel \{110\} planes and two non-parallel \( <111> \) directions, a maximum of 12 different crystal orientations (also known as 12 \( \alpha \) variants) can originate from a single \( \beta \) grain. For this reason, each preferred orientation imposed on the \( \beta \) phase during solidification or mechanical deformation limits the number of possible \( \alpha \) variants that can originate.

During primary fabrication, Ti ingots have a solidification microstructure that consists either of columnar \( \beta \) grains that develop from the chill zones adjacent to water-cooled crucibles or equiaxed \( \beta \) grains. In case of columnar \( \beta \) grains, it has been shown that the \( \beta \) phase develops a \( <100> \) fibre texture along the \( <100> \) growth direction of the columnar grains [Banerjee and Williams, 2013]. On the other hand, equiaxed \( \beta \) grains that result in a solidification microstructure have a weak or random \( \beta \) texture [Banerjee and Williams, 2013].
In the case of β n the case of jee and Williams, 2013], microstβ grains, a relative weak α texture will develop. On the other hand, when the β phase exhibits a strong < 100 > fibre texture, areas with α texture of various strength can be formed [Banerjee and Williams, 2013].

In order to achieve Ti alloys with homogeneous and desirable α texture, a large amount of research has been carried out to study the development of deformation texture in Ti alloy [Semiatin et al., 1997; Gey et al., 1996; Gey et al., 1997; Gey et al., 2002; Zaefferer et al., 2003; Lütjering and Williams, 2007; Salem et al., 2008; Obasi et al., 2012]. It has been shown that if mechanical deformation is conducted at low temperatures in the α+β phase field (generally 800 °C), preferred texture is directly imparted in the α phase of the alloy [Lütjering and Williams, 2007]. The imparted texture symmetry depends on the deformation path. In the case of unidirectional rolling, a so-called basal/transverse (B/T) texture would be developed as indicated in Figure 9.

Figure 9: Deformation textures in α+β Ti alloys represented through basal {0001} pole figures [Lütjering and Williams, 2007]

As the deformation process temperature increases (and so the volume fraction of β phase), the (B/T) texture tends to vanish, as the deformation is predominantly in the β phase (Figure 9). Ti alloys with moderate to strong α texture can, however, still be formed. As an example, in the case of unidirectional rolling conducted below the β transus temperature a so-called transverse (T) texture has shown to be developed preferentially (Figure 9).

The correlation of the texture between the deformed β and the subsequent α is not trivial and as yet not completely understood [Sargent et al., 2012]. It is known that at temperature close or higher than the β transus temperature, the mechanical deformation induces a preferred texture in the β phase. In this case, experimental work has shown that the strength of the resulting α texture would depend on the deformation path and the imposed cooling rate. The plausible explanation
for these findings is based on the fact that those β grains that were predominantly mechanically deformed provide favourable nucleation sites for the α phase [Sargent et al., 2012]. Crystal slip activity is associated with high dislocation density and thus high elastic stored energy that can then prompt α nucleation during cooling. According to this study, intense activity on the \{110\} <111> and \{112\} <111> slip systems could favor the growth of plates corresponding to specific variants [Gey and Humbert, 2003]. The cooling rate imposed during the β → α phase transformation would then determine the size of the α colony that grows from the specific α variant and thus the strength of the α texture of the material.

Several studies have focussed on the effect of the cooling rate on the α variant selection during β → α phase transformation (also known as α transformation texture) in absence of prior mechanical deformation [Gey and Humbert, 2003; Karthikeyan et al., 2010; He et al., 2012; Sargent et al., 2012; Semiatin et al., 2013].

Despite the fact that there are 12 possible solutions during the β → α phase transformation, as the cooling rate decreases there is no uniform distribution of 12 variants within the sample. Several theories have been suggested to explain the correlation between the cooling rate of the β → α and preferred variant selection [Banerjee and Williams, 2013].

In the presence of adjacent β grains with a set of parallel \{110\} planes, it is suggested that specific variants with nearly parallel [0001] directions on either side of the grain boundary are promoted when cooling rates are low enough [Bhattacharyya et al., 2003; Stanford and Bate, 2004]. This implies that each variant at a grain boundary should satisfy the Burgers orientation relationship with the prior β grain as well as with the adjacent prior β grains. Interestingly, this criterion has also been found in ferrite-to-austenite transformation in micro-alloyed steels [Lischewski and Gottstein, 2011]. For slow cooling rates the colonies of α laths would keep growing in the same orientation as the preferred selected variant to minimise the energy involved in the nucleation of α phase with different orientation.

Formation of α colonies that have originated from the interior of the prior β grain (and not the grain boundary) are however, common in Ti alloys and thus the proposed mechanism appears to be not exhaustive [Lütjering and Williams, 2007]. Moreover, recent studies have been shown that the secondary α texture that forms after annealing and cooling from the α+β of Ti-6Al-4V is correlated to the primary α texture [Sargent et al., 2012]. For this reason, the mechanisms at the basis of variant selection have been studied in terms of the stresses and strains that are associated with the β → α phase transformation [Sargent et al., 2012]. It is suggested that local stresses can develop during cooling due to the differences in coefficients of thermal expansion of the two
phases. Therefore it was proposed that in order to minimise the elastic strain energy associated with the β phases. Therefore it was proposed that in order to minimise the elastic orientation of the parent β phase [Humbert et al., 2006].

Research has also shown that β grains with the exact same texture would be formed when Ti alloys are β annealed and cooled to room temperature repeatedly [Semiatin, 2012]. This so called memory effect of the β phase would be justified by a minimisation of the strain energy generated during the α → β transformation. Similarly, it has been shown that the α texture that forms after repeated annealing stages is maintained through the heating cycles (phenomenon referred to as texture inheritance), showing that the same principle of energy minimisation applies during repeated α → β → α phase transformations [Wenk et al., 2004; Lonardelli et al., 2007; Obasi et al., 2012].

A recent grain to grain analysis of the orientation relation between the α and β phase has however shown that there are cases where the energy minimisation criterion is not satisfied [Sargent et al., 2012]. It was observed that several individual grains possess “non-favourable” orientations, i.e. individual grains originated from phase transformations that do not generate the minimum possible strains. It was noticed however that the average strain calculated over multiple neighbouring β grains was significantly smaller than the strain generated in the case of total random phase transformation (i.e. no variant selection). The research work suggested that strain energy minimisation should then be studied considering multiple neighbouring grains rather than individual grains.

As the cooling rate associated with the β → α increases, the size of the α colonies decreases and a more uniform distribution of the 12 possible variants is generally observed [He et al., 2012; Banerjee and Williams, 2013].

Triangular arrangements of specific α variants are typically present in the interior of the prior β grains. Although it is difficult to believe that these arrangements occur as a statistical consequence of random combination of the 12 α variants, their origin is not yet completely understood [Banerjee and Williams, 2013]. The most validated explanation for the occurrence of these α variants clusters is based on the so called self-accommodation theory of neighbouring variants [Wang et al., 2003]. Several independent studies have shown mathematically that certain groups of variants are preferred over others to minimise the local strain caused by the phase transformation [Banerjee and Williams, 2013]. Two types of clusters, each with three variants forming a triangular arrangement, were found to be the most energetically favourable
combinations. The two types of clusters consisted of α variants misoriented as indicated in Wang et al., 2003.

The study of the origin of the texture in α+β Ti alloys such as Ti-6Al-4V is generally complicated because limited β phase is retained at room temperature, thus it is not possible to directly measure the texture of the β phase with confidence. Several examples of deduction of the texture of the β phase from the measure of the α phase texture are present in the literature [Glavicic et al., 2003; Gey and Humbert, 2003]. In practice, the β phase texture is generated by reconstruction (i.e. back calculation), where the orientation of the α variants is determined first (typically with EBSD) and then the texture of the β phase is deduced assuming that the two phases are related by the Burgers orientation relationship.

2.2 Additive Manufacturing of Titanium Alloys

2.2.1 Introduction

Although Ti and Ti alloys have excellent high specific strength and resistance to corrosion the cost associated with their production limits the number of their applications [Imam and Froes, 2013]. The main cost associated with the production of Ti and Ti alloys derives from the numerous processing steps during the secondary fabrication of Ti as described in Section 2.1 [Froes, 2010; Imam and Froes, 2010; Imam and Froes, 2013]. End use components are usually obtained from mill products after forging and extensive machining. This approach to fabrication causes the generation of waste material and therefore the expenditure of considerable resources. This becomes particularly evident in the aerospace industry where the components have often intricate complex shapes that allow maximum weight savings. In certain cases, the ratio between the weight of the raw material bought to manufacture the end use component and the final flying component (referred to as “buy-to-fly ratio”) is typically 5-10 [Boyer, 2010]. To reduce the cost brought by the secondary fabrication of Ti alloys, extensive research has been carried out on near net shape additive manufacturing (AM) technologies [Arcella and Froes, 2000; Henriques, 2005; Kobryn and Semiatin, 2001; Kobryn et al., 2006; Dehoff et al., 2013].

As suggested by the name, the base concept of these technologies relies on the principle that the product is made by adding material layer by layer. It is clear that AM has a completely different approach to conventional manufacturing where material is constantly removed when shaping the components. The principles of AM are introduced in Section 2.2.2. The general advantages
associated with these technologies are firstly reviewed. Section 2.2.3 describes the recent efforts to produce large components in various Ti alloys (and in particular Ti-6Al-4V) with the principal laser forming techniques that represent the first AM processes specific for the fabrication of Ti alloys components. Thanks to the success of these applications and a greater understating of the process-material interaction, in recent years research has focussed on the use of smaller AM systems (grouped under the category of powder bed fusion systems). The state of the art of the Ti alloy components produced with the powder bed fusion processes (i.e. Electron Beam Melting and Selective Laser Melting) is presented in Section 2.2.4.

2.2.2 Additive Manufacturing: Shared Approach to Revolutionise Production

The concept of producing prototypes by adding layers of material to build up an object was developed in the late 1980’s. The term “rapid prototyping” was in fact introduced and commercialised in 1986 by Chuck Hull who patented stereolithography as a method to produce three-dimensional polymer objects in a quick way [Hull, 1986]. During the following decade, with the development of new rapid prototyping technologies and the improvement of the existing ones, the concept of “rapid manufacturing” was then introduced to indicate that the technologies had progressed beyond prototype applications [Dickens et al., 1995]. Recently the term “additive manufacturing” (AM) has been chosen as the standard term to indicate the technologies that produce end-use components directly from a digital model adding one layer of material at a time.

Schematically, any AM technology can be broken down into 7 sequel steps [Gibson et al., 2010]:

  1. Creation of a digital model corresponding to the object that is intended to print

The digital model of the product is created in a virtual environment using any design software (typically CAD). Because the model is created in a digital environment, design with optimised topology, extended functionality and radical new shapes can be created.

  2. Conversion of a digital model into an .STL format

The digital model is then converted to the STL (STereoLithography or also know as Standard Tessellation Language) format directly into the design software or through auxiliary software. During this conversion, the surface of the model is triangulated and stored in terms of the unit
normal and vertices of the generated triangles. The STL format enables a series of operations that are crucial for the AM. Firstly, the model is oriented in the AM building platform coordinate system. Any overhangs present the model and typically the base of the STL model are then automatically (or manually) supported by auxiliary secondary structures. The supported STL model is finally sliced in a number of layers whose thickness is decided by the user. The orientation of the part into the building platform and the choice of the layer thickness dictate thus the geometric accuracy of the final part.

3. Loading of the STL model in the AM machine environment

Once the STL model has been oriented, supported and layered, it can be transferred directly into the AM machine environment. At this stage the STL model can be repositioned on to the building platform. Different parts can be added into the AM machine for simultaneous fabrication.

4. Set up of the process parameters for the physical fabrication of the STL model

At this stage, the process parameters can be setup in relation to the material that is going to be processed. The process parameters determine the quality of the built part and therefore represent a crucial aspect for the AM fabrication. It is noteworthy that the process parameters to print quality parts vary depending on the AM system and the starting material being used. Chapter 5 discusses the effect of the process parameters on the melting and solidification of Ti-6Al-4V, the alloy investigated in this research work.

5. Building time

The process is then launched and the part is created one layer at the time depositing powder (or wire) material according to the digital model corresponding to the intended part.

6. Removal and cleanup of the end-use component from the building platform

Once the part is completed, it will be detached from the build platform. If the part was built on top of auxiliary secondary supporting structures, these supports and the surrounding build material must be taken off from the product.

7. Post processing (optional)

This stage, specific for the intended application, refers to the finishing of the part: it might include polishing of the product, accuracy or aesthetic improvement of the component or properties enhancement by thermal or non-thermal treatments. AM presents therefore a radically different approach to forming or subtractive manufacturing, and for this reason AM presents
several advantages. Using AM each component is produced from a digital model and depositing material only where it is needed, there is no need for a production line, to make specific tools, moulds, dies or furnaces. Thus it has been shown that AM is extremely advantageous compared to traditional manufacturing for the realisation of new designs or low volume production of specific parts. As any modification of the digital model does not translate into added cost for its production, AM is giving the possibility to design and manufacture products around the customer needs such as patient specific medical devices and consumer goods. Because parts are created in a digital environment and then fabricated with great accuracy (order of μm), AM increases greatly the design freedom. AM offers therefore exciting possibilities as products with new functional geometries can created without adding any extra cost to the conventional designs. Case studies have shown that AM allowed reduction in cost of production for components that would be extremely costly if manufactured with conventional processes [Hague, 2007; Petrovic et al., 2011; Hopkinson et al., 2006; General Electric 2013; EADS 2013]. Studies related to the environmental sustainability of AM have shown that raw material consumption is significantly reduced as well as the CO$_2$ emissions [Reeves, 2008; Baumers et al., 2010]. Finally AM compresses the entire supply chain as the entire component is produced directly within the AM system without the need of modular parts or external kits. At the moment only a limited number of materials are available in powder or wire form, and thus the majority of the research on AM has focussed on a limited number of metal alloys and plastics (Table 3) [Reeves 2013; Hopkinson et al., 2006]. However the possibility to develop new materials by mixing the initial powders represents another plausible exciting development for AM [Hopkinson et al., 2006]

Table 3: Examples of materials that are currently used in AM.

<table>
<thead>
<tr>
<th>Polymeric Materials</th>
<th>Metallic Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABS</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>PLA plastics</td>
<td>Tool steel</td>
</tr>
<tr>
<td>VisiJet®</td>
<td>Ni-Ti superalloys</td>
</tr>
<tr>
<td>Accura plastics</td>
<td>Ag</td>
</tr>
<tr>
<td>DuraForm®</td>
<td>Au</td>
</tr>
<tr>
<td>PA plastics</td>
<td>Ti- Ti alloys</td>
</tr>
<tr>
<td>PrimeCast®</td>
<td>Co-Cr alloys</td>
</tr>
<tr>
<td>PAEK</td>
<td>Al alloys</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>Bronze alloys</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>Inconel</td>
</tr>
</tbody>
</table>
2.2.3 Laser Beam Deposition Systems: Lasform\textsuperscript{SM}, LENS and Wire-based Systems

The first research on AM systems for Ti alloys (and in particular Ti-6Al-4V) was on laser forming of Ti alloys using laser beam deposition systems [Arcella and Froes, 2000; Lütjering and Williams, 2007; Santos 2006; Gibson \textit{et al.}, 2010; Kobryn and Semiatin, 2001; Brice \textit{et al.}, 1999; Kelly and Kampe, 2004]. Laser beam deposition systems consist generally of a “deposition head” that deposits metal material onto a substrate. The deposition head is equipped with 1) a set of optics to focus to the laser onto the substrate, 2) singular or multiple nozzles that deliver the powder (or wire) materials to where the laser is focussed onto, 3) the gas outlets that inject inert gases (such as Ar) in the building chamber [Gibson \textit{et al.}, 2010]. The substrate generally consists of a flat platform (generally made of the same alloys that is being deposited) or a pre-existing part where new material is added. The deposition is generally carried out by controlling the position of the substrate or relative to the deposition head. The first successful laser beam deposition process for Ti alloys is Lasform\textsuperscript{SM} [Arcella and Froes, 2000]. The schematic of Lasform\textsuperscript{SM} is shown in Figure 10.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{image}
\caption{Figure 10: a) Schematic of the Lasform\textsuperscript{SM} system and b) real AeroMet Corporation/ARL Lasform\textsuperscript{SM} apparatus [Arcella and Froes, 2000].}
\end{figure}
This laser forming process has shown the possibility to make large Ti-6Al-4V aerospace components (door panels, rings, cones and flanges) with good accuracy and reproducibility [Lütjering and Williams, 2007].

With a typical deposition rate that ranges between 0.9 to 4.5 kg per hour Lasform\textsuperscript{SM} proved to be a promising and quick manufacturing technology for repairs and the creation of large components with a complicated design in their near net shape requiring relative little machining compared to the traditional processing for Ti alloys [Arcella and Froes, 2000].

### 2.2.3.1 The Microstructure of Laser Beam Deposited Ti Alloys

The microstructure analysis of the first Lasform\textsuperscript{SM} Ti-6Al-4V parts revealed that the microstructure of laser deposited parts is significantly different from that observed in conventionally manufactured Ti alloys. In particular it has been reported that the microstructure of Lasform\textsuperscript{SM} Ti-6Al-4V consists of columnar prior β grains that are similar to those in thin casting and fine lamellar α arranged in a basketweave microstructure [Arcella and Froes, 2000].

Upon tuning the process parameters, parts with high density have been fabricated. The mechanical properties of machined and heat treated tensile samples have evidenced that static properties comparable to those of wrought Ti-6Al-4V can be obtained [Arcella and Froes, 2000].

The fatigue resistance of the Lasform\textsuperscript{SM} Ti-6Al-4V is comparable to that of cast Ti-6Al-4V, but little research was conducted to clarify the role of the microstructure in the fracture resistance of those parts.

The success of Lasform\textsuperscript{SM} led to the development of another more cost effective laser beam deposition system, known as Laser Engineering Net Shaping (LENS) [Keicher \textit{et al.}, 1998; Atwood \textit{et al.}, 1998; Hofmeister 1999; Griffith \textit{et al.}, 2000]. LENS was developed by Sandia National Laboratories about in the early 2000s and shares a similar working principle of Lasform\textsuperscript{SM}. A schematic of the LENS process is shown in Figure 11.
LENS offers better accuracy and reproducibility during the deposition than Lasform\textsuperscript{SM} thanks to a smaller laser beam diameter and spot size [Kobryn and Semiatin, 2001; Gibson \textit{et al.}, 2010]. Several studies have contributed to clarify the aspects of microstructural evolution of Ti-6Al-4V processed by LENS [Kobryn and Semiatin, 2001; Semiatin \textit{et al.}, 2001; Kobryn and Semiatin, 2003; Klingbeil \textit{et al.}, 2004].

The microstructure of LENS Ti-6Al-4V deposits consists of acicular $\alpha$ grains, similar but finer than those found in the Lasform\textsuperscript{SM} builds. This result indicates that cooling rates experienced by the melt pool during each layer deposition LENS is higher than that of Lasform\textsuperscript{SM} which has been confirmed by heat transfer modelling analysis [Klingbeil \textit{et al.}, 2004; Bontha, 2006; Bontha \textit{et al.}, 2009].

The other microstructural difference that was found in between the two laser beam deposition systems is that the prior $\beta$ grains growth in LENS Ti-6Al-4V is directly related to the laser motion path and the heat loss direction [Kobryn and Semiatin, 2003].

This suggested that the solidification of the prior $\beta$ grains could be predicted and controlled by altering the laser scan speed and power. However, to-date no published research work has reported a set of process parameters that avoids the formation of columnar grains during solidification of LENS Ti alloys.

It has been reported that during the process, the re-heating of the deposited substrate induced by successive layer depositions causes coarsening of the $\alpha$ grains in period thin regions parallel to the build platform [Kobryn and Semiatin, 2003]. It is not clear however if this microstructural
change, also known as macroscopic banding, has any effect on the deformation mechanism of the Ti alloys [Kobryn and Semiatin, 2003; Kelly and Kampe, 2004].

The effect of the anisotropy of the microstructure of LENS Ti-6Al-4V on the static mechanical properties was investigated [Kobryn and Semiatin, 2001]. Stress relieved deposited samples built perpendicularly to the building direction (indicated in the study by the $x$- or $y$-orientation) have shown excellent tensile properties comparable to that of conventionally manufactured Ti-6Al-4V. It was noticed however that porosity entrapped in the components has a great effect on the tensile elongation and tensile strength. As porosity tends to form between the deposited layers, specimens with the loading axis parallel with the building direction (and thus indicated as $z$-oriented specimens) had the smallest elongation and mechanical strength. HIPing of LENS Ti-6Al-4V has proved to be beneficial as it is capable to close the existing residual porosity. The ductility and tensile strength have shown to improve after the HIPing of LENS Ti-6AL-4V, although it is reported that $z$-oriented samples still remain the worst orientation in terms of mechanical properties [Kobryn and Semiatin, 2001]. It has been reported that the difference in the mechanical behaviour of parts built in different orientation is caused from the directionality of prior $\beta$ grain boundaries and crystallographic $\alpha$ texture, however, their contribution to the fracture of LENS Ti-6Al-4V remains unclear [Kobryn and Semiatin, 2001]. Residual porosity poses a severe limitation on the fatigue resistance of LENS Ti-6Al-4V. Although limited studies have been carried out on the fatigue performance of LENS Ti-6Al-4V, a HIPing post treatment appears necessary to meet the fatigue endurance limit of conventional cast Ti alloys.

Although LENS remains the main laser beam deposition system, recent research has shown the advantages of using wire feedstock material in place of the powders [Baufeld et al., 2010; Baufeld et al. 2010; Baufeld et al., 2011; Brandl et al., 2010; Brandl et al., 2011]. Laser beam deposition system that use wire instead of powder have generally a higher deposition rate, and reduce potential hazardous chemical contamination of the build [Brandl et al., 2011]. The deposition of Ti alloys through wire-based laser systems occurs within open chambers that are constantly pumped with Ar gas. Wire-feed laser beams have proved to build nearly fully dense Ti-6Al-4V blocks.

The microstructure of these deposits however is not homogeneous and consists of a mixed basketweave/colony $\alpha$ and martensitic $\alpha'$ microstructure and some retained $\beta$ phase [Baufeld et al., 2011]. Despite the fact the wire-feed laser beam Ti-6Al-4V present fully dense microstructures, it has been demonstrated that the build orientation has a significant effect on the tensile properties of the as-built specimens [Baufeld et al., 2011; Brandl et al., 2011]. It is not clear however how
the microstructure and the orientation of wire-deposited parts affect the deformation mechanisms of the parts. Contrasting results are reported in two independent studies [Baufeld et al., 2011; Brandl et al., 2011]. Limited research on the fatigue behaviour of wire-feed laser beam Ti-6Al-4V has shown that parts have a fatigue resistance similar or superior to that of cast Ti-6Al-4V [Baufeld et al., 2011; Baufeld et al., 2009].

The future of wire-feed laser beam deposition systems is however uncertain because of the poor resolution that these systems can offer and consequently the extent of machining that is necessary to carry out to obtain a good surface finish and good part accuracy.

### 2.2.4 Powder Bed Systems for the Production of Ti Alloy

In response to the need of creating parts with a complex shape with great accuracy (thus requiring minimal final machining) powder bed AM systems for the production of small Ti alloys components are being recently explored [Santos, 2002; Santos et al., 2004; Wehmöller et al., 2005; Hollander et al., 2003; Murr et al., 2009; Heinl et al., 2008; Parthasarathy et al., 2010; Gu et al., 2012; General Electric 2013; EADS, 2013].

Powder bed fusion technologies differ from laser beam deposition in the fact that the laser unit is kept separate from the delivery mechanism of the powder on the substrate [Gibson et al., 2010]. This separation simplifies the delivery of the powder and thus makes it possible to correctly deposit material despite of the shape of the part. The deposition of the parts during powder bed fusion takes place in closed air-tight chambers.

In principle, powders are firstly applied in thin layers (tens of μm) and then a laser (or electron) beam melts selected areas of the powder bed. The subsequent solidification realises the first cross section of the component. The build platform is then lowered for an amount that corresponds to the thickness of one layer and new layer of powder is applied across the platform. The completed part is thus created repeating the steps involved in the creation of the first cross section, layer by layer. The electron or laser heat source used by the powder bed fusion system are generally more focussed than that of the laser beam deposition systems and for this reason even parts of high complexity can be fabricated requiring minimum final machining [Gibson et al., 2010]. The two main powder bed fusion technologies for the production of Ti alloys are Electron Beam Melting (EBM) and Selective Laser Melting (SLM).

A list of the most common EBM and SLM systems available today is shown in Table 4.
Electron Beam Melting (EBM) was commercialised in 2001 by Arcam AB. Electron beam melting uses a high powered electron beam (typically 4 kW) that scans a layer of powder. Because gases in the build chamber would interfere with the electron beam, EBM is carried out in vacuum (typically $10^{-4}$-$10^{-5}$ mbar). This is the first fundamental difference with the laser-based processes described so far that are operated in a open environment that is continuously flushed with inert gas species. Figure 12 shows the schematic of the electron beam melting process and a corresponding commercial example (A2 Arcam system).

EBM is carried out in a hot environment to facilitate the electrical conduction of the powder bed. For this reason the build platform is kept at high temperature (500 or 700°C when Ti-6Al-4V is being processed). In practice during EBM each layer of powder is scanned twice. The first initial electron beam scan carried out by using low electron beam current and high scan rates to heat up the powder bed. The melting of the selective areas of the powders bed to the substrate below is then achieved with a second scan typically conducted by an high powered electron beam that rasters the cross section of the part with scan speeds up to 1000 mm/s.

<table>
<thead>
<tr>
<th>Commercial Name</th>
<th>Build Envelope [mm]</th>
<th>Company</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenix PXL</td>
<td>250x250x300</td>
<td>3D Systems</td>
</tr>
<tr>
<td>Arcam Q10</td>
<td>200x200x180</td>
<td>Arcam AB</td>
</tr>
<tr>
<td>M1 Cusing</td>
<td>250x250x250</td>
<td>Concept Laser</td>
</tr>
<tr>
<td>M2 Cusing</td>
<td>250x250x280</td>
<td>Concept Laser</td>
</tr>
<tr>
<td>EOSINT M 280</td>
<td>250x250x325</td>
<td>EOS</td>
</tr>
<tr>
<td>EOS M400</td>
<td>400x400x400</td>
<td>EOS</td>
</tr>
<tr>
<td>Realizer SLM50</td>
<td>70(Ø)x40</td>
<td>ReaLizer</td>
</tr>
<tr>
<td>Realizer SLM250</td>
<td>250x250x300</td>
<td>ReaLizer</td>
</tr>
<tr>
<td>Realizer SLM300</td>
<td>300x300x300</td>
<td>ReaLizer</td>
</tr>
<tr>
<td>AM250</td>
<td>245x245x300</td>
<td>Renishaw</td>
</tr>
<tr>
<td>SLM250 HL</td>
<td>248x248x250</td>
<td>SLM Solutions GmdH</td>
</tr>
<tr>
<td>SLM280 HL</td>
<td>280x280x350</td>
<td>SLM Solutions GmdH</td>
</tr>
</tbody>
</table>

2.2.4.1 Electron Beam Melting (EBM) of Ti Alloys
Figure 12: a) Schematic of the electron beam process. Key components: 1) electron gun assembly, 2) focussing lenses for the electron beam (EB), 3)x-y deflection coils, 4) powder cassettes, 5)polution layer rake, 6) built specimen, 7) build table [Murr et al., 2009]; b) A2X Arcam electron beam melting machine [Arcam 2014].

2.2.4.2 Microstructure and Mechanical Behaviour of Electron Beam Melted Ti Alloys

As EBM systems process at high temperature and each processed layer is heated before the melting starts, the columnar microstructure of EBM Ti-6Al-4V contains a mixture of α+β equilibrium phases [Al-Bermani et al., 2010; Hrabe and Quinn, 2013]. The α phase is arranged in fine lamellae with a basketweave morphology. Retained β can be found at the α grains boundaries. The origin of this microstructure derives from the fact that although the each deposited layer solidifies in the α’ martensitic phase [Cormier et al., 2004] the thermal mass offered by the powder bed and the built part triggers the phase transformation into the equilibrium phases. Contrary to that observed in laser beam deposited Ti components, EBM is characterised by a relatively high intra-batch variability [Hrabe and Quinn, 2013]. It was observed for example that small components have a microstructure that is significantly different from bulky and bigger samples [Al-Bermani et al., 2010]. This difference stems from the fact that small parts have a
lower thermal mass and therefore are unable to activate the annealing process that is observed in larger parts. Similarly, it has been reported that the microstructure of EBM Ti-6Al-4V can vary when parts are built isolated on the platform or adjacent to large parts that contribute to temperature rises through thermal radiation [Hrabe and Quinn, 2013]. As parts are typically deposited in layer of 70-100 μm and the electron beam creates a large melt pool, the surface finish of the EBM parts is generally poor (typical surface roughness ~ 30 μm). Parts for structural application are therefore built oversized during EBM and then typically machined to the desired surface finish.

Despite these variations, EBM Ti-6Al-4V parts are near fully dense, and therefore the tensile properties of as-built machined components are comparable to that of wrought and annealed Ti-6Al-4V [Al-Bermani et al., 2010]. The good ductility exhibited by EBM parts suggests the importance of the retained β phase which contributes to the plastic deformation at the crystalline level. Similar to that observed on laser beam deposited specimens, the tensile properties are also affected by the build orientation. Vertically oriented parts (z-orientation) have a significant reduction in elongation at fracture (up to 30%) compared to the other build orientations. It is suggested that this difference is due to residual porosity and the columnar nature of the prior β grain boundaries [Al-Bermani et al., 2010].

Studies on the fatigue properties of the as-built and machined EBM Ti-6Al-4V show that fatigue resistance is lower than wrought but comparable to that of as-cast Ti-6Al-4V due to residual pores present in the microstructure. HIPing of EBM components has proved to be extremely beneficial for fatigue resistance and are therefore recommended for structural parts that have to endure fatigue loading [Facchini et al., 2009; Murr et al., 2009].

2.2.4.3 Selective Laser Melting (SLM) of Ti Alloys

Selective laser melting (SLM) was developed by MCP Tooling Technologies in 1995 [Gibson et al., 2010] but research on SLM of Ti alloys started only in late 2000s. The SLM process resembles EBM in many aspects with a few fundamental differences. Figure 13 shows a schematic for the selective laser melting process and a commercial system.
Figure 13: a) Schematic of the selective laser melting process. Key components: 1) laser source, 2) laser optical train, 3) laser beam focus lens, 4) powder feeder system, 5) building platform, 6) wiper for powder deposition, 7) powder recycle system. b) A commercial example of SLM: Renishaw AM250 system [Renishaw, 2014]

Typically, SLM systems operate within a vacuum chamber that is back filled with inert gas species such as Ar or N. This is possible because inert gas species are transparent at the laser wavelengths that are used in SLM of metals [Gibson et al., 2010]. The SLM process starts with a thin layer of metal powder (typically ~50 μm) that is spread on to a build platform that is generally kept at relatively low temperatures (typically less than 200°C). A high powered laser then fully melts selected areas of the powder bed. Once that the laser scan is finished, the build platform is lowered to the thickness of the next layer and the process repeats with the application of a new layer of powder.

The great advantage of SLM is that modern laser beam are can be focussed to a spot size on the powder bed and therefore the level of resolution and accuracy that SLM can achieve is undisputed [Gebhardt et al., 2010; Hötter et al., 2013]. The second advantage of operating with small spot sizes is that the melt pool created by the laser during SLM is much smaller than that of EBM and for this reason the surface finish of the as-built components is generally superior [Gibson et al., 2010; Rafi et al., 2013].

The first studies of SLM on Ti alloys were focussed on the production of cellular scaffolds and small customised medical prosthesis (such as dental roots, teeth models, hard tissue replacements) that would have been extremely costly to make using traditional manufacturing [Tolochko et al.,]
2002; Wehmöller et al., 2005; Hollander et al., 2003; Kruth et al., 2005; Laoui et al., 2004; Vanderbroucke et al., 2007; Gebhardt et al., 2010]. More recent case studies have also shown that SLM could be used to manufacture components for the aerospace industry [Brant et al., 2013; Guo and Leu, 2013; General Electric, 2013; European Space Agency 2013; EADS, 2013].

2.2.4.4 Microstructure and Mechanical Behaviour of Selective Laser Melted Ti Alloys

Despite the success of niche applications that have triggered a wider interest around SLM, early studies of SLM Ti and Ti alloys evidenced systematic porosity in the parts that were therefore not suitable for demanding structural applications [Santos et al., 2004; Yadroitsev et al., 2007]. The interaction between SLM process parameters and the Ti powder bed was then investigated with particular interest in the densification mechanisms that occur during SLM.

It was shown that using a suitable combination of the process parameters (laser power, scan speed and distance between adjacent scan tracks) could lead to a production of relatively dense pure Ti [Santos et al., 2004]. Recent research on α+β Ti alloys has shown promising results and parts with a density up to 99.7% have been reported [Sercombe et al., 2008; Thijs et al., 2010].

Recent research has elucidated the main mechanisms responsible for formation of porosity during SLM. The origin of porosity during SLM is strictly related to the behaviour of the melt pool created at each laser pass and the quality of the initial powders. It has been shown that the laser scan track formed by the laser can become unstable and generate high surface roughness or balling for erroneous choice of laser power and scan speed [Kruth et al., 2007; Zhang et al., 2011]. Similarly, it has been shown that incorrect overlapping of adjacent scan tracks can cause powder denudation in some areas of the powder bed and therefore porosity [Yadroitsev et al., 2007; Yadroitsev et al., 2010; Gusarov et al., 2007]. Although powder technology processes are well established and thus the powders provided are typically homogeneous and fully dense, it has been argued that powders might adsorb gas species (in particular H₂) onto their surface. Because these gas species have low solubility at the high temperature of the melt pool, it has been suggested that gaseous bubbles (and therefore spherical porosity) might originate in the microstructure as the powders are melted [Mohandas et al., 1999].

In terms of microstructure, α+β Ti processed by SLM display similar features to that of EBM components although there are few differences caused by the fact that SLM is conducted at lower temperature. Similarly to EBM, the microstructure of SLM α+β Ti consists of columnar prior β
grains as a result of the thermal history experienced by the layers [Sercombe et al., 2008; Thijs et al., 2010]. However, upon cooling to room temperature the columnar grains transform into a fully martensitic α' phase instead of the equilibrium mixture of α+β found in EBM of Ti-6Al-4V [Facchini et al., 2010; Vilaro et al., 2011]. Irrespective of the size of the build component no annealing of the microstructure occurs during SLM due to the fact the SLM is typically carried out with a build platform kept at relatively low temperatures (generally < 200°C).

One main advantage of building at lower temperatures is that the microstructure of the SLM build is homogeneous throughout the part despite build size, orientation or location on the platform. However the difference of temperature between the deposited part and the top layer that is being processed causes the development of thermal stresses that, as reported in some case studies, can exceed the yield strength and cause plastic deformation of the SLM parts [Santos et al., 2004; Mercelis and Kruth, 2006; Schiomi 2004]. Although the mechanisms at the basis of thermal stresses are well understood, research has shown that it is not trivial to control the thermal gradients and build near fully dense parts simultaneously.

During the fabrication of each layer, the laser scans firstly the contour of the cross section (i.e. the perimeter of the cross section) and then rasters the area of the cross section [Gibson et al., 2010]. Recent studies have explored the use of different scan strategies to reduce the overall thermal stresses that develop at each layer deposition [Gibson et al., 2010; Bo et al., 2012; Mercelis and Kruth, 2006]. It has been shown, for example, that changing the direction of the scan vector at every layer reduces the residual stresses in the build component. Other studies have suggested that residual stresses are proportional to the length of the scan vector and thus it has been suggested to use “checkerboard” scan strategies that divide the entire cross-section into small squares (also known as islands) and then scan them randomly to minimise the temperature inhomogeneity and thus to reduce the development of the internal stresses [Gibson et al., 2010].

As the internal stresses are correlated with the length of the scan track, the choice of the build orientation affects the intensity of the internal stresses. Orientations that have the minimum cross sectional area are indeed less susceptible to warpage. Unfortunately the relation between the laser scan strategy and the microstructure has not yet been studied systematically. It has been shown that changing the scan direction at each layer can have an effect of the prior β grain growth direction but the origin of the relationship is empirical and not well understood [Thijs et al., 2010]. Similarly, SLM Ti-6Al-4V processed with a checkerboard scan strategy exhibits inferior density [Qiu et al., 2013].
The tensile properties of SLM Ti alloy vary greatly depending on the systems in use and the porosity of each built part [Vilaro et al., 2011; Chlebus et al., 2009; Vrancken et al., 2012; Facchini et al., 2010; Rafi et al., 2013; Qiu et al., 2013; Murr et al., 2009]. Near fully dense machined Ti-6Al-4V SLM bars tested perpendicular to the build direction have shown tensile strengths comparable to that of HIPed cast or wrought Ti-6Al-4V. However, the martensitic microstructure and residual porosity limit the ductility of SLM Ti-6Al-4V, which in most cases is lower than that of conventionally made material. It has been suggested that the build orientation on SLM significantly affects the ductility of the parts [Chlebus et al., 2009; Vilaro et al., 2011]. The limited studies available in the literature indicate that build components with a high number of layers are prone to fail due to porosity. Heat treatments tailored for α+β Ti SLM have shown to generally improve the ductility of the parts [Vilaro et al., 2011; Vrancken et al., 2012]. It has been shown that the microstructure of SLM Ti-6Al-4V can evolve towards equiaxed microstructures if as-built deposits are treated with β annealing and controlled cooling [Vilaro et al., 2011].

Studies on the fatigue properties of SLM parts are very limited [Leuders et al., 2012; Rafi et al., 2013; B Van Hooreweder et al., 2012]. Published research has evidenced that the fine martensitic microstructure, internal stresses and porosity limit the fatigue life of the as-built SLM Ti and Ti alloys components. Similarly to that described for EBM of Ti alloys, HIPing is generally recommended for SLM specimens that have to endure fatigue loading [Leuders et al., 2012]. In a recent study, it was found that the fatigue resistance of HIPed Ti-6Al-4V specimens is similar to that of cast Ti-6Al-4V [Leuders et al., 2012]. It is not clear how the crystallographic texture and the columnar microstructure of the SLM parts play in the fatigue resistance of the build components.

2.3 Quantitative Analysis and Representation of Crystallographic Texture

Crystallographic texture in polycrystalline metals can be quantified using several established methods [Bhattacharyya, 2004]. Electron backscatter diffraction (EBDS) has proved to be a powerful technique which allows mapping the orientations of a polycrystalline polished sample, and hence giving microstructural and crystallographic texture information at the same time [Bieler et al., 2002; Bhattacharyya, 2004]. Therefore EBSD is extremely useful to the study of the microstructure, and especially the texture evolution in SLM components. This section reviews
the EBSD working principles and the methods that are used in this study to represent and discuss the texture of SLM Ti-6Al-4V.

2.3.1 Principles of Electron Backscatter Diffraction (EBSD)

EBSD is based on the interaction of a high-energy electron beam with the crystal grains at the surface of a polished crystalline material. The interaction of the e-beam with the material leads to coherent or incoherent and elastic or inelastic electron scattering. If all of the electrons in the scattered wave are in phase coherent scattering is formed. On the other hand, during incoherent scattering the electrons are scattered away from the material in multiple directions and are out of phase. If during the collision and subsequent change in direction the electrons do not lose their energy, elastic scattering occurs. On the other hand, inelastic scattering implies a loss in energy.

The generation of the EBSD signal (or EBSD patterns) that is used to obtain the orientation information of the material in analysis is shown schematically in Figure 14.

![Figure 14: Schematic of the generation of the EBSD signal. The sample coordinate system is also shown in the Figure (ND, RD, TD) [EDAX, 2014]](image)

EBSD patterns form when the incident electrons of the e-beam collide and backscatter elastically from the atomic planes of the crystal grain that satisfy the Bragg’s law [Warren, 1969]:

\[ n\lambda = 2d_{hkl} \sin \theta \]
where \( n \) is an integer number, \( \lambda \) is the wavelength of the incident electrons, \( \theta \) is the Bragg angle for a particular plane \( \{hkl\} \) and \( d_{hkl} \) is the interplanar spacing for that plane. The backscattering of the electrons in the Bragg condition forms Kossel cones with an angle \( \theta \) between them [Schwartz, 2000]. When a sensitive phosphor screen is places in close proximity to the surface of the sample, part of the Kossel cones can be captured. The captured Kossel cones on the phosphor screen is in the form of band patterns, where each band, known as Kikuchi band, has a luminance that depends on the intensity of the diffraction. More specifically, the pattern of Kikuchi bands recorded on the phosphor screen is known as an EBSD pattern [Dingley and Field, 1997]. An example of diffraction pattern is shown in Figure 15.

![Figure 15: Example of EBSD pattern showing zone axis (pointed by the arrows) [EDAX 2014]](image_url)

EBSD patterns carry two types of information. The crystal symmetry of the grain in analysis determines the intersection of the Kikuchi bands in the pattern (zone axes). Thus, from the analysis of the Kikuchi bands intersections, the crystal structure of the grain can be deduced. Also, it has been shown that there is an inverse correlation between the width of the Kikuchi bands and the interplanar spacing of the grain crystal structure. The EBSD pattern contains therefore all of the angular information that are necessary to calculate the orientation of the grain in analysis relative to a fixed coordinate reference system [Schwartz, 2000]. Interpreting the EBSD patterns generated by the grains allows the identification of the phases existing in the material and the orientation of each individual investigated grain.

The interpretation of the EBSD patterns (also known as indexing) can be performed manually or automatically. The automated indexing of the patterns relies on a mathematical algorithm known as Hough transform that allows the detection of the location of the Kikuchi bands in the EBSD pattern [Schwartz, 2000]. Finally, once the crystal structure (or material phase) is deduced from
the analysis of zone axes in the patterns, the interplanar spacing and angles that derive from the
width of the measured bands are compared to theoretical values to identify most probable crystal
orientation.

2.3.2 Representing EBSD Results

The results from EBSD scans can be represented in a number of ways. Among the most intuitive
methods of representation there are inverse pole figure (IPF) maps and discrete pole figures (PF)
[Bhattacharyya, 2004].

2.3.2.1 Inverse Pole Figure Analysis

One of the most intuitive methods to represent the results from EBSD analysis is through the use
of inverse pole figure (IPF) colour coded orientation maps. IPF orientation maps are based on the
idea that each crystal orientation relative to a fixed user defined reference frame is represented by
a specific colour. Each measured crystal orientation is reduced to a point on an inverse pole
figure (known as unit triangle). A unique colour is then assigned to each point in the unit triangle
on the basis of its orientation relative to a user defined sample frame direction. In other words, a
point (or colour) in the unit triangle represents a specific \{hkl\} or \{hkil\} plane whose normal is
parallel to the user defined sample frame direction, generally defined as normal direction ND,
rolling direction RD and transversal direction TD).

Figure 16 illustrates an example of IPF colour coding. In this example, the HCP orientation is
fixed relative to the sample reference frame (ND, RD, TD). If the IPF is plotted along ND
(expressed in a compact notation by [001] IPF) the HCP crystal orientation of Figure 16 will be
assign to colour blue according to colour scheme inset. Grains with this specific orientation have
the \{10\bar{1}0\} planes normal to ND as indicated in the unit triangle. The \{010\} IPF plot of the same
orientation would show as red colour instead (Figure 16). As the \{0001\} planes are normal to the
RD, the orientation would have been expressed with a point in correspondence of the \{0001\}
vertex of the unit triangle (Figure 16).
Figure 16: A HCP crystal orientation can be defined by three Euler angles $\theta_1, \theta_2, \theta_3$. This orientation can be plotted in form of inverse pole figures. The colours in the unit triangles show which crystallographic plane is parallel to the chosen sample direction.

Depending on the selection of the axis for colour coordinating, the same single crystal orientation could be represented in different colours. Through the use of IPF maps, it is possible to resolve the grain structure of the area that is investigated and whether areas of identical crystallographic orientation occur in the sample (such as $\alpha$ colonies in Ti alloys). An example of this analysis is shown in Figure 17.
Another intuitive way to represent the grain orientation is offered by discrete or contour pole figures. Discrete pole figure are stereograms that represent the orientation of individual grains relative to an external sample frame of reference.

In practice the plot is built considering the projection of a specific crystal cell plane normal (pole) onto a unit sphere based on the sample coordinate system (Figure 18). The projected points are then connected to the southern hemisphere of the unit sphere. The intersections with the equatorial plane consist thus of distinct univocal points for a specific crystal cell orientation.

Discrete pole figures provide a comparison between orientations of the crystal grains, and are very useful to study orientation relationship between adjacent grains and different phases.

Contour pole figures are built in a similar way, but contrary to the discrete pole figures carry information relative to the whole set of grains in the dataset and not individual ones. In this case the pole figures show areas of equal pole density (relative to a random background) whose level is defined on a corresponding colour scheme legend. Contour pole figures give a more quantitative analysis of the orientation distribution in a polycrystalline sample and therefore are useful to study the texture of a metallic sample. The orientation density is typically shown in

Figure 17: [001] IPF map of SLM Ti-6Al-4V and corresponding colour scheme unit triangle. In this map, α grains with [0001] plane normal parallel to ND are shown in red [Simonelli et al., 2014]
areas of different colours or through density of contours (i.e. greatly spaced contours indicate low pole density). An example of coloured contour pole figure is shown in Figure 19.

Figure 18: a) Spherical projection of the poles of the crystal cell b) equivalent discrete pole figure representation [EDAX, 2014].

Figure 19: a) \{0001\} and \{11\overline{2}0\} contour pole figures from a small dataset (~ 100 grains)[Simonelli et al., 2014]; b) corresponding colour scheme legend.
2.4 Summary

The production of Ti and Ti alloys to the finished component using traditional forming (or subtractive) manufacturing methods are associated with high costs and the generation of considerable waste material. It is not surprising that much research in recent years has focus on the alternative production routes, such as Selective Laser Melting (SLM), that not only allow material saving, but also give greater level of design freedom. In the last 20 years SLM has evolved from a technology capable of realising prototypes to a manufacturing technique enabling a direct production of finished components.

Recent research on SLM of Ti alloys has thus focussed on the processing-microstructure relationship as the quality of the components and the repeatability of the obtained results are the main requirements for the further establishment of SLM. These efforts have resulted in better understanding of the densification mechanisms during SLM and the literature shows that for optimised processing windows it is possible to produce near fully dense components. Unfortunately despite the fact that the operating principle behind SLM is unique, each commercial SLM systems presents distinct characteristic (laser source, delivery mechanism for the powder bed, temperature of the platform, etc.). Therefore extensive trials are usually required to determine the optimal process windows. The analysis of the literature shows a lack of a systematic methodology to study the laser-metal interaction that would be beneficial to the progress of SLM.

Research conducted on the mechanical properties of near fully dense SLM Ti-6Al-4V has shown that mechanical properties comparable to those of conventional fabricated products can be obtained. Limited work has however been done to show the correlation between the microstructure features and the crystallographic texture of the SLM parts with their mechanical properties. It is however undoubted that several features, such as porosity, prior-β grain boundaries, the existing phases, and crystallographic texture have a great influence on the mechanical behaviour and crack propagation in Ti alloys. In addition, most of the available research has focussed only on samples built in one unique orientation and therefore the effect of the building orientation on the mechanical properties remains unclear.
Finally the literature shows a continuous evolution of the available SLM systems and in particular the laser source that is being used. SLM systems operating in a wide laser power range and with adjustable laser beam diameter are in fact now available. This progress has enabled to improve the resolution achievable by the additive manufactured systems, and in turn, has enabled researchers to realise shapes with extended functionalities. No evidence of microstructural control has however been reported. This research work aims to improve the understanding of these aspects.

In the first place, this research aims at understanding how the laser process parameters affect the melting and solidification of Ti-6Al-4V. It is believed that the understanding of the laser-material interaction would enable the identification of optimised processing windows that, in turn, would allow the fabrication of dense and repeatable samples.

In addition, this research work aims to increase the knowledge regarding the mechanical properties of SLM Ti-6Al-4V. As the mechanical performance of this alloy is affected by the peculiar microstructure that originates during SLM, an extensive study of the microstructure and crystallographic texture evolution of SLM Ti-6Al-4V was carried out.

The final part of this research focussed on attempts to achieve samples with an $\alpha + \beta$ microstructure. It is well accepted that samples with an $\alpha+\beta$ microstructure would offer superior ductility than the martensitic samples typically obtained using SLM.
3 Materials and Methods

This chapter describes the experimental work involved in this research including materials, processing and characterisation technologies for SLM of Ti-6Al-4V. The sections are summarised as follow: Section 3.1 includes the materials that were used for the tuning of the process parameters in the SLM systems. In this section it is also describes the characteristic of the powders that were used for the building of the samples. introduces the SLM machines used in this research. Section 3.2 illustrates the methodology that was used to obtain various processing windows to fabricate near fully dense components. Section 3.3 gives a description of the process parameters that produced the near fully dense components at the highest build rate. The microstructure and the crystallographic texture of the components built using this process window will be discussed in Chapter 6. Tensile bars of different build orientation and fatigue bars were also fabricated using optimised processing windows with the highest build rate as described in Section 3.3. The final part of this section describes the alternative scan strategies that allowed the building of near fully dense components with a lower build rate. As it will be discussed in Chapter 8, these alternative processing windows enabled the production of components with controlled microstructure. Section 3.4 describes the microscopy techniques used to characterise the microstructure and crystallographic texture of the samples. The methodology used for the reconstruction of the parent β phase and for the calculation of the residual stresses in the as-fabricated samples are also reported in this section.
3.1 Materials used in this Research

3.1.1 Microstructure of Wrought Ti-6Al-4V

Rolled Ti-6Al-4V plates with approximate dimensions of 4 cm x 3 cm x 1 cm were used to identify the processing window that could lead to near fully dense SLM samples. The exact thermo-mechanical history of the plates was unknown, however the transverse section of the plates shows a bimodal microstructure containing ~15μm equiaxed α grains with slightly smaller secondary α and laminar β grains as shown in Figure 20a (low magnification) and Figure 20b (high magnification). As discussed in Chapter 2 (Section 2.14), this is the typical microstructure of mill-annealed Ti-6Al-4V that is deformed in the α + β phase and then stress relieved in the α + β phase field.

![Microstructure](image)

Figure 20: Electron backscatter micrographs showing the microstructure of the rolled Ti-6Al-4V. Conventionally made α/β Ti alloys (such as Ti-6Al-4V) are generally associated with this microstructure.

3.1.2 Plasma Atomised Ti-6Al-4V Particles

All the samples that are discussed in the present work were fabricated using plasma atomised Ti-6Al-4V powders provided by LPW Ltd. The chemical composition of the powders is reported in Table 5.
Table 5: Chemical composition of the plasma atomised Ti-6Al-4V grade 5

<table>
<thead>
<tr>
<th>Element</th>
<th>Chemical composition [wt. %]</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>0.01</td>
</tr>
<tr>
<td>C</td>
<td>0.01</td>
</tr>
<tr>
<td>H</td>
<td>0.0051</td>
</tr>
<tr>
<td>Fe</td>
<td>0.07</td>
</tr>
<tr>
<td>Al</td>
<td>6</td>
</tr>
<tr>
<td>V</td>
<td>4.1</td>
</tr>
<tr>
<td>Ti</td>
<td>Balance</td>
</tr>
</tbody>
</table>

*Source: LWP Technologies*

The powder size and distribution were determined using a Malvern MasterSizer machine by a laser diffraction method known as low angle laser light scattering (LALLS). The particle size distribution is shown in Figure 21.

*Figure 21: Powder size distribution calculated from one batch of the plasma atomised powder used in this study*

The particle size distribution was within 15 and 70 μm, but, approximately 75% of the examined particle size ranged between 25 to 50 μm. SEM images of the particles are shown in Figure 22. The particles appear spherical, smooth with few satellites (smaller condensed particles attached to the main particle). The microstructure of the plasma atomised particles is discussed in Chapter 4.
3.2 Aspects of fabrication: SLM systems used in this research

Two SLM systems have been used to fabricate the components that were studied in this research work: a Renishaw AM250 Laser Melting System and a ReaLizer SLM50. The majority of the research work presented in this thesis was conducted on the Renishaw AM250 Laser Melting System. Details on this system are provided in Section 3.2.1. A conspicuous part of the results presented in Chapter 8 were however obtained using the ReaLizer SLM50 system. This desktop laser system features a fibre laser with a smaller beam diameter (and thus spot size) and hence enabled the development of alternative scan strategy to control the microstructure during the selective laser melting process. Details on this system are reported in Section 3.2.2.

3.2.1 AM250 Laser Melting System: characteristic of the system

The Renishaw AM250 laser melting system uses a pulsed ytterbium (Yb) fibre laser with a wavelength of 1070 nm. The maximum nominal laser power is 200W whilst the maximum laser scan speed is up to 1000 mm/s. The laser beam can be collimated to a minimum nominal powder bed spot size of 70 μm. The system allows the user to change the building platform material that can also be heated at a maximum temperature of 150°C. All the samples studied in this research would...
work were however built on a Ti-6Al-4V substrate that was heated to 70°C. The levelling system
used for the powder bed preparation consists of a single silicone bladed arm, that wipes pre-
deposited powder onto the build platform in a single motion. The building process occurs in a
protective Ar atmosphere in order to minimise contamination and oxidation. Other technical
details of the AM250 are listed in Table 6.

Table 6: Specification of the Renishaw AM250 Laser Melting system

<table>
<thead>
<tr>
<th>Renishaw AM250 Laser Melting System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Build Envelope [mm]</td>
</tr>
<tr>
<td>External Dimensions [mm]</td>
</tr>
<tr>
<td>Weight [kg]</td>
</tr>
<tr>
<td>Power Supply</td>
</tr>
<tr>
<td>Layer Thickness</td>
</tr>
</tbody>
</table>

Marcam Autofab software was used to support the overhangs (when needed) and orientate the
parts before the building process. This software also allows the specification of the laser scan
strategy and the process parameters involved during SLM. The process parameters that can be set
in Marcam Autofab are:

- layer thickness, i.e. the building platform downward vertical movement at each layer
  completion;
- laser power (the maximum nominal laser power is 200W);
- building platform temperature (the maximum building platform temperature is 200°C);
- hatch spacing, i.e. the distance between two adjacent laser scan vectors;
- nominal scan speed, i.e. the ratio between point distance and the laser dwell time on each
  point (exposure time); it is noteworthy that the laser off time in between two consecutive
  pulses is not accounted in this formulation.
- laser focus position, i.e. the distance from the laser focus plane and the building platform
  (Figure 23);
- laser scan strategy, i.e. the pattern followed by the laser during each layer processing.
On the basis of the measured particle size distribution it was chosen to fabricate all the components with a layer thickness of 50 μm. The nominal laser power used for the fabrication of the samples was 200W, although it should be noted that the maximum laser power at the workpiece was 157W due to energy losses in the optical train. These values of layer thickness and laser power were adopted as a compromise to achieve high build rates, part resolution and final density of the components.

The scan strategy that was used predominantly in this work is generally referred to as “meander scan strategy” and is shown in Figure 24.
The meander scan strategy can be described as follows. Each layer cross section is initially offset 100 μm from the external edges, forming an internal area known as “area-volume”. The laser scans initially this area with parallel alternating scan vectors. The direction of the scan vectors changes by 67° at each layer, as depicted in Figure 24. By default the laser always begins with the area-volume scan from the back left corner of the cross section. The laser then scans along the offset boarder (i.e. 100 μm from the external edges) and the external border of part in order to improve the component surface finish and reduce the number of defects near the surface of the component.

Extensive research was conducted on the laser/Ti-6Al-4V interaction for a wide range of values of hatch spacing, laser focus position and scan speed in order to establish with confidence a set of laser parameters that combined with the meander scan strategy could lead to the production of dense components. The investigated ranges are summarised in Table 7. These results are reported in Chapter 5.

Table 7: Ranges of process parameters that were investigated on the AM250 Laser Melting system. The chosen scan strategy was the meander scan strategy.

<table>
<thead>
<tr>
<th>Process Variable</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100 - 300</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>190 - 320</td>
</tr>
<tr>
<td>Laser Focus Position [mm]</td>
<td>-9 to +9</td>
</tr>
</tbody>
</table>

In an attempt to control the microstructure of the as-built SLM Ti-6Al-4V two different scan strategies were also used (Figure 25 and Figure 26). The scan strategy shown in Figure 25 is known as the checkerboard scan strategy.
Figure 25: Schematic of the checkerboard scan strategy: the inner volume of the layer (N) is divided in several smaller “islands” that are scanned independently (light grey area in the schematic).

The area-volume is firstly divided into a number of square ‘islands’. The islands are then scanned back to front of the chamber (or in other words, along the y-axis) and left to right (i.e. along the x-axis). The scan direction is rotated by 90° between neighbouring islands. When the scanning of the checkerboard is completed, a contour scan was performed along the offset and the external border in an attempt to improve the surface finish. Different combinations of hatch spacing and laser scan speed were used to evaluate the optimal overlapping between two neighbouring islands and the overlap between the contour scan vector and the individual islands.

The third scan strategy that was used is instead generally referred to as double scan strategy. In this scan strategy the interior part of each layer is scanned twice using the pattern illustrated in Figure 26. The interior part of the cross sectional area is then scanned twice with alternating parallel vectors. The direction of the scan vectors during the second scan is however rotated 90° to that of the first scan. Similarly the offset and external border scan are repeated twice on each layer.
Figure 26: Schematic of the double scan strategy. Each layer (N) is scanned twice. The scan direction in the inner volume is rotated by 90° after the first scan. The offset and external border are then scanned twice.

3.2.2 ReaLizer SLM50: Characteristic of the System

Part of the work on the microstructural control was carried out using a SLM50 ReaLizer desktop machine. Some technical details of the SLM50 are listed in Table 8. SLM50 presents a few significant differences compared to the Renishaw AM250. SLM50 uses a continuous Yb-fibre laser with a maximum nominal power of 100W and a powder bed spot size of 30 μm. The laser has a wavelength of 1070 nm. All the building process took place on a Ti-6Al-4V platform, in a protective Ar atmosphere. The levelling system consists of a double bladed level bar. The level bar wipes the powder bed twice in order to improve the levelling of the layer of powder. The building platform temperature was set to 200°C.

Table 8: Technical specifications of the ReaLizer SLM50

<table>
<thead>
<tr>
<th>ReaLizer SLM50</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Build Envelope [mm]</td>
<td>70x40 (diameter, Z)</td>
</tr>
<tr>
<td>External Dimensions [mm]</td>
<td>800x700x500</td>
</tr>
<tr>
<td>Weight [kg]</td>
<td>80</td>
</tr>
<tr>
<td>Power Supply</td>
<td>230 V, 16 A</td>
</tr>
<tr>
<td>Layer Thickness</td>
<td>20-100 μm</td>
</tr>
</tbody>
</table>
ReaLizer Control Software was used to set the process parameters and the build orientation. Similar to Marcam Autofab (Section 3.2.1), this software allows control of the laser at each process layer through a set of user-defined commands. Each layer can be scanned multiple times. At each laser scan, the user can define the laser powder, the hatch spacing, the laser scan speed and the laser scan strategy.

To control the microstructure of the as-fabricated samples and maintain high density, the interaction of the laser with rolled Ti-6Al-4V (i.e. the reference material) varying several process parameters have been studied.

The scan strategy that was predominantly used with the ReaLizer SLM50 is a double scan strategy described in Section 3.2.1.

### 3.2.3 Process parameters determination for the fabrication of near fully dense components

Extensive research work was carried out to study the effect of the laser parameters on the density of the build components. This section describes the general methodology that was used to establish the optimal processing windows to fabricate near fully dense Ti-6Al-4V using the AM250 Laser Melting System and the ReaLizer SLM50. The results obtained from these experiments are reported in Chapter 5.

In order to study the laser effect on the melting and solidification of Ti-6Al-4V, the reference material (Section 3.1.1) was laser scanned using different sets of process parameters. A reference Ti-6Al-4V plate was initially polished to mirror finish and adhered to a build platform for each of the SLM systems. The build platform was then lowered by an amount corresponding to the thickness of the reference material. In this way, it was possible to bring the rolled plate of Ti-6Al-4V to the laser focus plane level \(z = 0\). In a protective Ar atmosphere, square portions of the reference rolled Ti-6Al-4V were then irradiated by the laser beam. By observing the change in the microstructure of the reference Ti-6Al-4V plate induced by the laser heating and successive rapid cooling, it was possible to study the laser penetration depth and therefore the effect that each laser parameters has on the melting and solidification of Ti-6Al-4V. This methodology is schematically shown in Figure 27a. It was thus also possible to estimate the surface roughness created by the laser or in other words the roughness created upon solidification of the melt pools. The set of parameters that created irregular heat affected zones (as shown in the example of
Figure 27b) were considered not optimal for the SLM processing. It is clear that in order to produce dense components each layer of melting has to be as homogenous as possible. In addition, one prerequisite for fabricating dense components is to process an even flat powder bed, thus sets of laser parameters that created high surface roughness were discarded.

![Figure 27: a) Schematic diagram showing the laser beam that irradiates a plate of bimodal Ti-6Al-4V. b) corresponding micrograph showing the marked change in the microstructure caused by the laser irradiation. As the laser rasters Ti-6Al-4V it creates melt pools of specific depth and width that rapidly solidify as martensitic microstructure.]

The first stage of this methodology consisted of a detailed investigation of the phase transformation that takes place when the bimodal microstructure of the reference material is scanned by the laser. As no prior knowledge was available, the reference material was scanned with a set of process parameters established from previous research on SLM of stainless steel (Table 9).

Table 9: List of process parameters that were used to study the laser scan track formation

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>50</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>200</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>0</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>
As stated in Section 3.2.1, it should be noted that in all the following experiments the laser power at the workpiece was in fact 157 W and not 200 W (the nominal value set in the machine). Experiments to assess whether the laser beam was uniform across different location of the building platform were then carried out. Three plates of reference Ti-6Al-4V were placed in three different locations on the building platform, as indicated in Figure 28. The laser then scanned these distinct regions of the reference material using the same process parameters (listed in Table 10).

Figure 28: Schematic diagram showing three different locations on the platform. The front and the back of the SLM machine and the gas flow are also indicated.

Table 10: Process parameters used to assess the laser stability over time and across the building platform

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>
The effect of the hatch spacing on the laser heat affected zone was studied, scanning the reference Ti-6Al-4V with the parameters listed in Table 11. All the laser parameters were kept fixed but the hatch spacing which was varied between 100 – 250μm.

Table 11: List of parameters used to assess the effect of the laser hatch spacing on the melting of Ti-6Al-4V

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100-300</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

The effect of the nominal laser scan speed (that neglects the laser off time in between two consecutive pulses) on the laser heat affected zone was also studied with the reference Ti-6Al-4V with the parameters listed in Table 12. All the laser parameters were kept fixed but the nominal scan speed which was varied between 180 – 320 mm/s.

Table 12: List of parameters used to assess the effect of the laser scan speed on the melting of Ti-6Al-4V

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>140 - 250</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>190 – 320</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
</tbody>
</table>

The effect of the laser focus position on the laser heat affected zone was then studied investigating the rolled Ti-6Al-4V with the parameters listed in Table 13. All the laser
parameters were kept fixed but the laser focus position which was varied between –9 to +9 mm from the focus position (z = 0).

*Table 13: Process parameters used to investigate the effect of the laser focus position of the heat affected zone*

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>220</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>205</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>-9 to +9</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

Finally, in order to investigate the effect of the laser scan strategy on the laser melting of Ti-6Al-4V, the reference material was laser scanned dividing each individual cross-section in islands of different size (checkerboard scan strategy). In this way, it was possible to study the effect of the laser scan vector length on the melting and solidification of Ti-6Al-4V. The process parameters that were used in this investigation are reported in *Table 14*.

*Table 14: Process parameters used to investigate the effect of the scan strategy on the heat affected zone*

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>220</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
<tr>
<td>Scan Vector Length [mm]</td>
<td>0.2 − 4</td>
</tr>
</tbody>
</table>
In order to validate the results obtained from these experiments, 1cm³ test cubes were built varying one process parameter at a time. The density of these cubes was then compared. To validate the effect of the hatch spacing on the density of the components several cubes were built using the parameters listed in Table 15. As can be noticed, only the laser hatch spacing was varied across the builds.

*Table 15: Process parameters used to validate the experiments on the laser hatch spacing*

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>220</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>225</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100 - 180</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

Similarly, the effect of the laser scan speed and focus position was validated comparing the density of cubic components built using the parameters listed in Table 16 and 17, respectively. Finally, the density of the cubic components built in different locations of the platform was compared. The components were built using the parameters listed in Table 9.

*Table 16: Process parameters used to validate the experiments on the laser scan speed*

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>125 - 200</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>225 - 365</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>
Table 17: Process parameters used to validate the experiments on the laser focus position

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal Laser Power [W]</td>
<td>200</td>
</tr>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Scan Speed [mm/s]</td>
<td>225</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>-2 to +2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

3.3 SLM Sample Types

This section describes the samples that were used for microstructural and mechanical investigation. These samples were fabricated after studying the interaction of each process parameter with Ti-6Al-4V. For this reason all the samples described in this section are near-fully dense.

3.3.1 SLM Samples with High Density and Production Rate

The optimal set of process parameters that allowed the production of near-fully dense samples at the highest measured build rate are listed in Table 18. These samples were built using the Renishaw AM250 Laser Melting system (Section 3.2.1). The laser focus plane was set to approximately +2 mm above the powder bed. Cubic samples of 1cm³ were then built using the meander scan strategy described in Section 3.2.1.
Table 18: List of optimal process parameters for the production of Ti-6Al-4V

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Layer Thickness [μm]</td>
<td>50</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

The microstructure of these components was studied on orthogonal planes following ASTM F2921-11. In particular, the microstructure and crystallographic texture were studied on the frontal yz-plane, the lateral xz-plane and the horizontal xy-plane as shown in Figure 29.

![Figure 29: Cubic samples used for the microstructural evaluation. The building direction is parallel to the z-axis.](image)

**3.3.2 Tensile Samples and Tensile Test Description**

Using the optimised set of process parameters (Section 3.3.1), bars with a gauge thickness, width and length of 3mm, 6mm and 35 mm respectively were fabricated using the AM250 Laser Melting system for the evaluation of the tensile properties of SLM Ti-6Al-4V. Two batches of 12 tensile bars were built in total. In accordance with the ASTM F2921, where the orientation of the built part is described listing the axes of the AM machine that are parallel to the longest and
second longest dimensions on the part, each batch contained four tensile bars of vertical \( zx \)-, edge \( xz \)- and flat \( xy \)- orientations (Figure 30).

![Figure 30: Models of components that were tested to assess the tensile behaviour of SLM Ti-6Al-4V.](image)

A batch of tensile bars was stress relieved at 730°C for 2h in a \( \text{N}_2 \) protective atmosphere. The stress relief temperature and holding time were chosen to relieve the residual stresses that occur in the SLM parts [Vilaro et al., 2011]. Once the stress relieving treatment was completed, the tensile bars were furnace cooled to room temperature at an imposed cooling rate of 10°C/min (ten times faster than typical furnace cooling rate). The tensile bars were then mechanically polished before the tensile test. This batch of tensile bars will be referred to as “stress relieved condition” in the following sections. The tensile bars which were not stress relieved (i.e. the second batch) were also mechanically polished to the same surface finish prior to the tensile test. This second batch of tensile bars will be referred “as-built condition” hereafter. Mechanical polishing (machining) was necessary because the surface roughness measured on the horizontal \( xy \)-planes, i.e. those planes scanned by the laser, differed significantly from the surface roughness measured on the frontal \( xz \)- or lateral \( yz \)- planes where sintered powder particles can remain attached (Table 19). Some of the flat \( xy \)-orientation tensile bars curled during the building process and for this reason machining was carried out on those bars to minimise warpage. After the machining process, the \( xy \)-oriented tensile bars had a smaller gauge thickness (2 mm) and width (3 mm) compared to the bars built in the two other orientations.
Table 19: Surface roughness of the SLM Ti-6Al-4V

<table>
<thead>
<tr>
<th>Surface Roughness Ra [μm]</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>As-built lateral surfaces -yz planes</td>
<td>27.58</td>
</tr>
<tr>
<td>As-built top surface -xy plane</td>
<td>6.83</td>
</tr>
<tr>
<td>After polishing - all surfaces</td>
<td>0.39</td>
</tr>
</tbody>
</table>

The tensile tests were performed on an Instron 3369 tensile system (Instron Ltd, Bucks, UK). The test was conducted at room temperature at a strain rate of 2 mm/min. Young’s modulus, yield stress, ultimate tensile stress and elongation at failure were determined according the ASTM E8/E8M using both an extensometer and the measured cross-head strain.

3.3.3 Fatigue Samples and Fatigue Test Description

Fatigue specimens were made in the xz-orientation (ASTM F2921-11) as shown in Figure 31.

![Figure 31: Models of components that were tested to assess the fatigue behaviour of SLM Ti-6Al-4V](image)

The specimens had a rectangular gauge section, similar to that of the tensile samples. Specimens were built via SLM directly to net dimensions and machined to improve their surface finish. After machining the bars had gauge thickness, width and length of ~3mm, 6mm and 65 mm respectively.
A total of five samples were tested. Each specimen was tested at constant maximum stress level of 500 MPa. Tensile-tensile testing of the samples was carried out at a frequency of 10 Hz and a load ratio of $R = 0.1$. The fatigue test was performed on an Instron 8801 at room temperature.

### 3.3.4 Near-fully Dense Samples obtained with Alternative Processing Windows

Significant changes in the microstructure of the samples were found in cubic samples that were processed with three alternative sets of parameters. In particular, using the set of parameters described in Section 3.3.4.3 it was possible to fabricate components with an equilibrium microstructure directly during production.

#### 3.3.4.1 Checkerboard Scan Strategy on AM250 Laser Melting System

In order to reduce the cooling rates during the solidification of the melt pool created by the laser, several cubic samples were fabricated using the checkerboard scan strategy illustrated in Section 3.2.1. The smallest checkerboard “island” allowed by the control software was used to confine the heat induced by the laser in a narrow zone of the powder bed. The optimal set of process parameters that allowed a production of near-fully dense samples using the checkerboard scan strategy are listed in Table 20.

**Table 20: List of optimal process parameters for the production of Ti-6Al-4V using a checkerboard scan strategy (AM250)**

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Layer Thickness [µm]</td>
<td>50</td>
</tr>
<tr>
<td>Point Distance [µm]</td>
<td>45</td>
</tr>
<tr>
<td>Island Size [mm]</td>
<td>0.5</td>
</tr>
<tr>
<td>Exposure Time [µs]</td>
<td>260</td>
</tr>
<tr>
<td>Hatch Spacing [µm]</td>
<td>50</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
</tbody>
</table>
The laser focus plane was set approx. +2 mm above the powder bed. The microstructure and the crystallographic texture of these components were studied in the orthogonal planes as suggested by the ASTM F2921-11 similarly to that described in Section 3.3.1.

### 3.3.4.2 Double Scan Strategy on AM250 Laser Melting System

To improve the density further, several cubic samples were fabricated using the double scan strategy outlined in Section 3.2.1. The set of process parameters that were used are listed in Table 21. In this case, each layer was processed twice using the same process parameters. These samples were built using the Renishaw AM250 Laser Melting system.

*Table 21: List of optimal process parameters for the production of Ti-6Al-4V using double scan strategy (AM250)*

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured Laser Power [W]</td>
<td>157</td>
</tr>
<tr>
<td>Layer Thickness [μm]</td>
<td>50</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>45</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>200</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>100</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

The microstructure and the crystallographic texture of 1cm$^3$ cubic samples were studied in the orthogonal planes as suggested by the ASTM F2921-11.

### 3.3.4.3 Double Scan Strategy on the SLM50 System

The final batch of samples presented in this thesis was obtained using a ReaLizer SLM50 system. This system is equipped with a fibre laser of lower power that can be focused on to the powder bed to a spot size of 30μm. As the energy is delivered in a much smaller area than in the case of the AM250 Laser Melting system, it was possible to fabricate near-fully dense components using a relatively low laser power (i.e. 42W). The list of parameters that were used in this configuration is given in Table 22.
Table 22: List of optimal process parameters for the production of Ti-6Al-4V using double scan strategy in the SLM50 system

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser Power [W]</td>
<td>42</td>
</tr>
<tr>
<td>Layer Thickness [μm]</td>
<td>50</td>
</tr>
<tr>
<td>Point Distance [μm]</td>
<td>50</td>
</tr>
<tr>
<td>Exposure Time [μs]</td>
<td>320</td>
</tr>
<tr>
<td>Hatch Spacing [μm]</td>
<td>60</td>
</tr>
<tr>
<td>Laser Focus [mm]</td>
<td>+2</td>
</tr>
<tr>
<td>Platform Temperature [K]</td>
<td>342</td>
</tr>
</tbody>
</table>

It is noteworthy that, similar to that observed in the AM250 Laser Melting system, the focus position was kept above the plane layer processing plane. The build platform was however heated to 200°C (i.e. 473 K). These samples were processed with the so called “double scan strategy” described in Section 3.2.1. The microstructure of these samples was studied in the orthogonal planes as suggested by the ASTM F2921-11.

3.4 Sample Preparation for Microstructural Characterisation

The as-fabricated cubic components were initially cross-sectioned and polished to a mirror finish to carry out microstructure and crystallographic texture analysis.

A Struers Accutom 5 machine with a silica carbide cutting blade was used for sectioning of the parts. The cutting speed was kept between 0.2 and 0.5 mm/min and water was used as coolant. Before the grinding and polishing steps, specimens were mounted in conductive Bakelite using the Struers Citopress-1 mounting machine. Grinding and polishing of the specimens was performed in reference to the protocol suggested by Struers for Ti alloys. Details on the grinding and polishing steps can be found in Table 23.
Table 23: Polishing procedure used in this research work

<table>
<thead>
<tr>
<th>Process</th>
<th>Wheel/Cloth</th>
<th>Lubricant</th>
<th>Force</th>
<th>Speed</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plane grinding</td>
<td>600 mesh</td>
<td>Water</td>
<td>25N</td>
<td>300 rpm</td>
<td>5 min</td>
</tr>
<tr>
<td>Fine grinding</td>
<td>1200 mesh</td>
<td>9 μm diamond paste</td>
<td>30N</td>
<td>150 rpm</td>
<td>7 min</td>
</tr>
<tr>
<td>Polishing</td>
<td>MD-Chem</td>
<td>OP-S + 10% H₂O₂ (30%)</td>
<td>35N</td>
<td>150 rpm</td>
<td>as needed</td>
</tr>
</tbody>
</table>

The final step was a chemical-mechanical polishing with a mixture of colloidal silica (OP-S) and hydrogen peroxide (30% diluted) on a MD-chem cloth (Struers). The addition of hydrogen peroxide allows a continuous removal of the reaction product between titanium and the silica suspension leaving the surface free of mechanical deformation. The parts were then cleaned individually under tap water, and then dried with methylated spirits and a strong stream of hot air. The etchant solution used in this study is the Kroll’s reagent; a dilute aqueous solution containing HF and HNO₃, which is the most widely used for commercial titanium alloys [Lütjering and Williams, 2007]. The Kroll’s etchant consists of:

- 5ml of HNO₃;
- 10ml of HF (48% concentration);
- 85ml H₂O.

In order to gain a further understanding of the fine microstructure of the SLM components, TEM samples were analysed. Thin foils with electron transparent regions about 100nm thick were prepared. Slices approximately 1 mm thick were firstly cut out from the as-built component. Using a spark erosion system, 3 mm discs were extracted from the slices. The disks were then thinned to an average thickness of 100 μm using Si-C paper with a grit size of 1200 combined with water. The electropolishing of the disks was conducted in a Struers Tenupol twinjet machine using a solution consisting of 90% methanol and 10% perchloric acid (60% concentration). The Electropolishing was done in a temperature range of -20 to -30°C at 20V.

3.5 Techniques for Microstructural and Crystallographic Texture Characterisation

Various characterisation techniques were used in this research work in order to obtain detailed understanding of the microstructure SLM Ti-6Al-4V. Further details are reported in the following sections.
3.5.1 Microstructure and Density Analysis

Observations on the quality and porosity of the samples were carried out using two optical microscopes:

- Reichert-Jung MEF3 optical microscope
- Nikon Optiphot 100 optical microscope

Quantification of the density values were performed from image analysis using ImageJ, that enables an automatic quantification of the area occupied by the pores in the micrograph. Although this analysis does not provide precise information on the real 3D morphology of the pores and the real density of the sample, it certainly gives an indication on the quality of the parts. X-ray micro computed tomography (X-ray micro CT) might have given clearer insights on these aspects. Due to low penetration of X-rays in metals (and Ti alloys) high acquisition times are often necessary to achieve a reasonable voxel resolution capable of describing the real density of the samples. Given the instability of the AM250 Laser Melting system and the fact X-ray samples might be not representative to describe the porosity of larger fabricated components, X-ray micro CT was thus considered to be not ideal to measure the density of the parts. A detailed study of the microstructural evolution was performed using the following electron microscopes:

- FEI Nova 600 Nanolab dual beam FIB/FEG-SEM
- Carl Zeiss (Leo) 1530 VP FEG-SE
- JEOL 2000FX TEM

The microstructure, grain morphology and size was determined using secondary and backscatter electron imaging. Details on the chosen settings can be found in Table 24. The chemical composition of the grains in the microstructure was studied using Energy Dispersive X-ray Spectroscopy (EDS). This investigation was also used to verify the existence of the secondary phases, as SLM involved the re-heating of the deposited layers that can potentially trigger, when processing Ti-6Al-4V, the formation of titanium aluminides such as Ti₃Al or the aluminium rich Ti₃Al. The crystallographic structure and chemical composition of individual grains in the microstructure of the as-fabricated samples were also studied using a TEM Jeol 2000FX microscope operated at 200kV.
Table 24: Microscopy techniques and settings used to carry out the microstructural characterisation

<table>
<thead>
<tr>
<th>System</th>
<th>Analysis</th>
<th>Electron Voltage [ kV]</th>
<th>Working Distance [mm]</th>
<th>Probe current</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leo</td>
<td>SE-Imaging</td>
<td>20</td>
<td>variable below 10</td>
<td>variable</td>
</tr>
<tr>
<td>Leo</td>
<td>BS-Imaging</td>
<td>20</td>
<td>variable below 10</td>
<td>variable</td>
</tr>
<tr>
<td>Leo</td>
<td>EBSD</td>
<td>20</td>
<td>Variable below 8</td>
<td>26 nA</td>
</tr>
<tr>
<td>Leo</td>
<td>EDS</td>
<td>10</td>
<td>8</td>
<td>variable</td>
</tr>
<tr>
<td>FIB</td>
<td>BS-Imaging</td>
<td>5</td>
<td>6</td>
<td>variable</td>
</tr>
<tr>
<td>FIB</td>
<td>EBSD</td>
<td>20</td>
<td>Variable below 8</td>
<td>26 nA</td>
</tr>
<tr>
<td>FIB</td>
<td>EDS</td>
<td>5</td>
<td>5</td>
<td>5 nA</td>
</tr>
</tbody>
</table>

3.5.2 Crystallographic Texture Analysis of SLM Ti-6Al-4V

The distribution of crystallographic orientations of the sample were studied using electron backscatter diffraction (EBSD) analysis using the FEI Nova 600 Nanolab Dualbeam FIB/FEG-SEM and the Carl Zeiss (Leo) 1530 VP FEG-SEM systems. These systems are equipped with a EDAX EBSD camera and TSL acquisition and data analysis software. The mounted samples was glued onto a 70° pre-tilted aluminium holder (for optimising the EBSD data collection). The mounting resin was ground sideways to carry out EBSD at smaller working distances and thus improve the backscatter diffraction signal.

The back-scattered electrons diffracted from a specimen were captured on the phosphor screen of an EBSD camera. Before the acquisition, the background signal was measured over a relatively large area and subtracted from the backscatter pattern in order to improve the automatic indexing of Kikuchi bands. The scanning step size for the EBSD map was 1 μm for the acquisition of large maps and 0.3 or 0.1μm for high-resolution maps. The obtained data was analysed using TSL OIM software.

3.5.2.1 Reconstruction of the Prior β Phase

Since SLM occurs at elevated temperature, it is of interest to know the crystallographic texture at high temperature (when the solidification process takes place) and how the high temperature
phase texture affects the texture of the low temperature phase. However, because of the $\beta$ inceallotropic phase transformation, the high temperature $\beta$ texture is masked by the transformation $\alpha$ phase product. Using the Burgers orientation relationship however it is possible to calculate, or reconstruct, the parent $\beta$ grain orientations based solely on knowledge of the room temperature $\alpha$ phase orientations. Several methods have been proposed in the literature to accomplish this procedure using the orientations measured from $\alpha$ variants in EBSD scans [Humbert et al., 1995, Glavicic et al., 2003a, Glavicic, 2003b, Cayron et al., 2006; Cayron, 2006; Pilchak et al., 2009, Pilchak and Williams, 2011b].

In this research work, a Matlab script was developed and used to perform the automatic reconstruction of the $\beta$ phase. The script is presented in Appendix A. In practise the reconstruction of the $\beta$ phase was carried out considering every triplet of neighbouring $\alpha$ grains in the EBSD data set as suggested in Humbert, 1995 and 1996. The orientation of each $\alpha$ grain present in the measured dataset was initially imported to Matlab using the Euler angles $\varphi_1$, $\Phi$, $\varphi_2$. Euler angles express the consecutive rotations about the axis of the crystal coordinate system that are needed to bring the crystal coordinate system into coincidence with the sample coordinate system (i.e. passive rotations). Because of the crystal symmetry however, multiple rotations can result in an equivalent orientation of a certain crystal. For this reason, it was also necessary to take into account the rotational symmetry elements of $\alpha$ and $\beta$ phases in order to be able to distinguish independent orientations (Appendix A). In the present approach the rotational symmetry elements of the $\alpha$ and $\beta$ phases (12 and 24 respectively) were described using the equations found in Humbert, 1995. As the $\alpha$ and $\beta$ phase are related through the Burgers orientation relationship $\{0001\}_\alpha // \{110\}_\beta$ and $\langle 11\bar{2}0 \rangle_a // \langle 111 \rangle_b$, which is equivalent to a Bunge’s rotation expressed by the matrix $D$ (135°, 90°, 325°), the solution common to the $\alpha$ triplet (or the solution with the least misorientation) was considered as orientation of the parent $\beta$ phase. Solutions exceeding a misorientation of 8° were discarded from the original dataset to reconstruct the parent $\beta$ phase with the accuracy of 8° orientation spread and thus also enabling the generation of an orientation map from a representative number of data points.

Variant selection and variant frequency distribution were determined confronting the misorientation between the measured $\alpha$ grain orientation and the 12 possible solutions generated by the corresponding parent $\beta$ phase. The variant frequency distribution was calculated from detailed orientation maps (step size of 0.3μm) and only considering those $\alpha$ grains equal or larger than ~1 μm.
3.5.3 3D Reconstruction of Plasma Atomised Powders Microstructure

In order to establish the complete microstructure evolution that occurs during SLM, the microstructure of plasma atomised powders used for processing was initially studied using the FEI Nova 600 Nanolab Dualbeam FIB/FEG-SEM. This system features a FEG-SEM and an ion beam column, a gas injectors (Pt), a macromanipulator, and EBSD camera, solid state backscatter and EDS detector. A random particle was picked up from a powder bed using the macromanipulator featured in the dual beam FIB/FEG-SEM and then welded to a 3mm Cu TEM-grid. This procedure is schematised in the sequence of micrographs shown in Figure 32. Once that the particle was securely attached to the Cu grid, the ion beam was used to mill the material, then a 180° stage rotation was carried out from the milling position to the EBSD data acquiring position (Figure 33). The serial sectioning, stage rotation and EBSD acquisition were performed automatically according to the procedure outlined in West and Thomson, 2009.

Figure 32: Sequence of micrograph that shows a) the powder bed of plasma atomised Ti-6Al-4V particles, b), c)and d) the particle lifting and subsequent attachment to a Cu TEM-grid. The welding of the particle to the grid was performed depositing Pt from a gas injector.
Figure 33: a) The region of interest (ROI) forms an angle of 52º to the x-axis and is perpendicular to the ion beam (milling position); b) the ROI forms an angle of 70º to the x-axis to improve the EBSD signal collection (EBSD position). The milling and EBSD positions are achieved mounting the sample on a pre-tilted sample holder (36º to the x-axis) and tilting the stage of 16º [West and Thomson, 2009].

A total of 200 slices with a uniform thickness of 200 nm were milled and analysed during the serial sectioning.

3.5.4 Residual Stress Analysis

Residual stresses of the as-fabricated samples were measured in the middle of the frontal section (xz-plane) of a 1cm³ cube at about 1mm from the edge using X-ray diffraction. As evidenced in the literature, X-rays penetrate metals only to a depth of about 30μm therefore mirror final SiO₂-H₂O₂ polishing of the surface under investigation was carried out prior to the stress analysis [Mecelis and Kruth, 2006, Leuders et al., 2012]. Due to the low penetration depth of the X-rays in the material, a plane stress state (σ₃₃=0) was assumed. A Bruker X-ray diffractometer (AXS D8 Advance) equipped with a Ka (λ=0.54 nm) X-ray point source and a SOL-XE detector was used for the stress analysis. The shift in the diffraction peak position at 2θ of 142°, i.e. the scattering of the (α₃₃) planes, due to the residual stresses was measured according the side inclination method [Cullity and Stock, 2001]. The peak shifts that correspond to induced lattice strains, were measured at three φ angular rotation (0°, -45° and -90° respectively) and six ψ tilting angles (0°, 9°, 18°, 27°,36° and 45° respectively) as shown schematically in Figure 34. The stress tensor of the irradiated volume was calculated from the observed lattice strains using a Young modulus of E=115 GPa and a Poisson’s ratio ν=0.324. As the peak shift might be
introduced by potential system misalignments during $\psi$ tilting, the experiment was repeated at the same $\psi$ tilting angles and $\varphi$ angular rotations on stress free powder material and the stress tensor was then calibrated [Cullity and Stock, 2001].

\[ \text{Figure 34: Schematic showing the location on the sample where the residual stresses were evaluated and the rotations involved during the stress calculation} \]
The general microstructural features of the plasma atomised Ti-6Al-4V particles were studied on several ion beam cross sections of a randomly selected particle. Figure 35a illustrates the image quality map of one of the such cross-sections. The gray scale of the images reveals the quality of the electron backscattered pattern obtained from the examined cross-section – thus, brighter regions indicate higher quality EBSD patterns have been collected.

Figure 35: EBSD micrographs showing a) the image quality and, b) the phase map of a particle cross-section

The phase map (Figure 35b) indicates that the lamellae are α phase (indicated by the light blue colour of the plates). The β phase indexed in the patterns (less than 4% volume fraction) has a low confidence index and thus it does not represent a reliable dataset. There are no visible prior β grain boundaries indicating that the α laths probably originated from one parent β grain. The fully lamellar microstructure results from the high cooling rate experienced by the powder during plasma atomisation (PA) [Wang et al., 2010]. Similar findings are reported in several independent studies [Murr et al., 2009; Heidloff et al., 2010]. Research on Ti alloy powders
focuses mainly on the morphology of the powders rather than their microstructure [Kobryn and Semiatin, 1996; Froes, 1998; Medina and Kruth, 2013] thus a comparison of these results with the literature is not straightforward. The maximum length α laths was 25μm, i.e. grains span through the entire width of the particle. On average the length and width of the laths was 16.1±0.3μm and 1.9±0.3μm respectively.

The 3D morphology and orientation information of the α phase in the powder was characterised by studying the crystallographic texture of the powder from EBSD datasets obtained using a FIB assisted dual beam system through serial cutting (Section 3.5.3). Figure 36 shows the [001] inverse pole figure maps relative to cross sections of 15 slices distanced 800nm.

![Figure 36: [001] IPF maps: the maps correspond to cross sections distanced 800 nm each other. The colour scheme of the IPF maps is indicated in the unit triangle.](image)

The α laths have a few dominant crystallographic orientations as indicated by the different colours in the Figure 36. It is noteworthy that the same colours represent the same orientations. The orientation of the α laths in terms of Euler angles are reported in Table 25. As shown in the corresponding discrete pole figure plots the particle consists of 5 different α variants that originate from the same parent prior β grain. As the α variants precipitate from one single β grain following the Burgers orientation relationship, each of the (0001) poles of the α grains is parallel to one of the (110) poles of the reconstructed β grain.
Table 25: Euler angles describe the orientation of the α laths that form the examined particle

<table>
<thead>
<tr>
<th>Grain</th>
<th>θ1</th>
<th>θ2</th>
<th>θ3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>103.5</td>
<td>17.1</td>
<td>101.8</td>
</tr>
<tr>
<td></td>
<td>166.8</td>
<td>130</td>
<td>247.5</td>
</tr>
<tr>
<td></td>
<td>329.3</td>
<td>107.7</td>
<td>335.6</td>
</tr>
<tr>
<td></td>
<td>87.3</td>
<td>72.7</td>
<td>231.6</td>
</tr>
<tr>
<td></td>
<td>79.1</td>
<td>136.5</td>
<td>51.6</td>
</tr>
<tr>
<td>Reconstructed β</td>
<td>44.7</td>
<td>51.7</td>
<td>67.8</td>
</tr>
</tbody>
</table>

Similarly, one of three <1\bar{1}20> directions of the α grains is parallel to one of the <111> directions in the reconstructed β grain as shown in Figure 37.

Figure 37: Discrete pole figures corresponding to a) the orientation components of the α grains, b) the corresponding orientation components of the reconstructed parent β phase. As imposed by the Burgers orientation relationship, the {0001}_α plane is parallel to the {110}_β plane and the <1\bar{1}20>_α directions are parallel to the <111>_β directions.

The 5 variants that correspond to phase transformation are reported in in a compact notation in Table 26 (the definition of all the rotation matrices is instead described in Appendix A).
Table 26: The variants transformations associated with the α grains of the particle in study

<table>
<thead>
<tr>
<th>Transformation number</th>
<th>Matricial description</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>D(^{1})C(_{31})_z</td>
</tr>
<tr>
<td>3</td>
<td>D(^{1})C(_{2e})_z</td>
</tr>
<tr>
<td>12</td>
<td>D(^{1})C(_{2x})_z</td>
</tr>
<tr>
<td>2</td>
<td>D(^{1})C(_{32+})_z</td>
</tr>
<tr>
<td>11</td>
<td>D(^{1})C(_{2b})_z</td>
</tr>
</tbody>
</table>

A reconstructed three-dimensional orientation image of the α laths is shown in Figure 38. Each α lath colour represents a particular crystal orientation component.

Figure 38: a), b), c) and d) are snapshots representing the 3D morphology of the α grains

As shown in Figure 38 the α plates grow throughout the particle and rarely cut across each other; moreover, the α laths have a plate-like shape. Most of the α laths have a similar size indicating that during the PA process they have probably nucleated and grown at the same rate. This 3D
study also confirms that the particles are fully dense and do not show any spherical pores. TEM analysis of the particle reveals that the α laths have few dislocations (Figure 39 and 40).

![TEM micrograph showing the substructure of few α grains of the plasma atomised Ti-6Al-4V](image)

**Figure 39: TEM micrograph showing the substructure of few α grains of the plasma atomised Ti-6Al-4V**

![TEM micrograph that shows an example of the EDS analysis point. The red circle on the grain indicates the estimated interaction area where the composition was measured](image)

**Figure 40: TEM micrograph that shows an example of the EDS analysis point. The red circle on the grain indicates the estimated interaction area where the composition was measured**

The EDS analysis of on the α laths of the same foil is summarised in Table 27. The investigated α grains exhibit a slightly higher content in Al than that specified in the supplier’s data sheet.
Table 27: Weight % of the detected elements during the ESD acquisition

<table>
<thead>
<tr>
<th>Element [ wt%]</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>7.16 ± 0.56</td>
</tr>
<tr>
<td>Ti</td>
<td>88.84 ± 0.34</td>
</tr>
<tr>
<td>V</td>
<td>4.37 ± 0.19</td>
</tr>
</tbody>
</table>
The repeatability and reproducibility of SLM production represents a crucial aspect for the future establishment of this processing technology [Mumtaz and Hopkinson, 2010; Ferrar et al., 2012; Leuders et al., 2012]. Indeed, as reported in Chapter 2 several studies on the mechanical performance of SLM of Ti alloys have shown great variability of the performance of the components [Vilaro et al., 2011; Chlebus et al., 2011; Vrancken et al., 2012]. To minimise the inter-batch variability, i.e. variations that manifest among different builds, and the intra-batch variability, i.e. the differences that occur within the same building process, a series of experiments were carried out to characterise the laser / material interaction.

The interaction of the laser beam with Ti-6Al-4V was initially studied by investigating individual scan tracks created on rolled Ti-6Al-4V (reference material) using an arbitrary set of process parameters (Section 5.1). The stability of the laser beam output was then characterised to make sure that the same energy was delivered over time and across different locations of the building platform (Section 5.2). Sections 5.3 and 5.4 describe the effect of the hatch spacing and the scan speed respectively on the scan track formation. The effect of the laser focus position and the use of various scan strategies is discusses in Section 5.5 and 5.6 respectively. The results from these investigations were then validated by building various test parts and comparing their density (Section 5.7). Finally a summary describing the most important consideration for processing Ti-6Al-4V quality parts is provided in Section 5.8.
5.1 Laser Scan Track Formation on Ti-6Al-4V

Laser scan tracks were created by scanning the polished Ti-6Al-4V plate with the process parameters listed in Table 9. The evolution of the microstructure of Ti-6Al-4V plate after the laser scanning is shown in a Figure 41a. The laser creates a melt pool that upon solidification shows a different microstructure than the parent material. A Heat Affected Zone (HAZ) consisting of finer grains with respect to the parent material can also be observed.

The $xy$-plane, i.e. the plane where the laser scan occurs, shows scan tracks created by the laser. The laser scan direction imposes a directionality on the scan tracks is shown in Figure 41b. It can be noticed that a lenticular melt pool is formed in correspondence to the point where the laser dwells. The width of the melt pool, that depends essentially on the laser power and the laser focus position [Vasinonta et al., 2001], is larger than the chosen hatch spacing (180 μm), and thus adjacent scan tracks overlap by around 40%.

The microstructure on the $xz$- and the $yz$-planes revels information regarding the depth of the melt pool and the HAZ. Figure 41c shows that the depth of the melt pool and the HAZ is not constant along the $x$-axis, but varies with a periodicity of ~ 180 μm, i.e. the adopted hatch spacing. Consistent with the studies on the scan track stability [Yadroitsev et al., 2010] the scan track reaches the maximum height in correspondence with the middle part of the melt pool. The scan track depth then diminishes where the scan tracks overlap. This explains why the melt pool and the HAZ depth is wavy and periodic.

The microstructural analysis on the $yz$-plane reveals that the scan track depth along the scan track length (i.e. along the $y$-axis), was approximately constant. This is caused by the fact that as the laser advances, adjacent melt pools overlap for most of their entire length as the laser point distance was set at 50 μm (Figure 41d).
Figure 41: a) 3D representation of the microstructure after laser scanning. The microstructure varies according to the orthogonal planes as shown in the micrographs b), c) and d). The sharp change of the microstructure that allows the study the laser HAZ distinguishes the laser HAZ from the parent reference material.

The phase composition and the microstructural evolution of the solidified melt pool and HAZ was then studied using EBSD. Figure 42 shows a [001] IPF orientation map with an image quality contrast of a small region of the solidified melt pool and HAZ. It can be noted that the microstructure of the melt pool consists of fine acicular grains with a HCP crystal structure, as confirmed by the EBSD analysis, vertical grain boundaries. Given the crystal structure and the morphology of the grains, and considering the typical cooling rates that are involved during laser melting at room temperature (4000 °C/s [Vilaro et al., 2011]), it is believed that the microstructure of the solidified melt pool consists of fully α' martensitic phase. This result is consistent to that reported in other studies on laser cladding of Ti-6Al-4V [Sun et al., 2001;
Cottam, 2011] where the cooling rates are similar to quenching of Ti-6Al-4V [Welsch et al., 1994].

Figure 42: [001] IPF orientation map overlaid on the image quality map showing the microstructure changes of rolled Ti-6Al-4V after the laser scan.

Figure 42 also shows columnar grain boundaries of an average width of 12 ± 4μm. The origin of the columnar grain boundaries is related to the heat dissipation during solidification of the melt pool. As reported in various studies on the heat dissipation in laser melting of metals, during SLM heat is lost predominantly through the metallic component that is being scanned [Badrossamay and Childs, 2007; Yadroitsev et al., 2010]. Thus, heat is predominantly lost through the Ti-6Al-4V plate. During solidification, that for Ti-6Al-4V occurs in the β phase field, the grains would grow according to the principal heat loss direction and hence assume a columnar elongated morphology. Upon cooling to room temperature, the entire β phase would transform into α’ phase leaving the vertical prior β grain boundaries visible in the microstructure as shown in Figure 42.

The section of the scan track shown in Figure 42 reveals the HAZ below the martensitic region. This area interfaces the melt pool created by the laser and the bimodal microstructure of the plate and consists of a refined bimodal microstructure. As the α grains maintain an equiaxed and lamellar morphology (and not lamellar morphology exclusively) this indicates that this area did not reach temperatures above the β transus temperature. It is plausible instead that during the
laser scan this area was heated in the high portion of the $\alpha + \beta$ phase field (~900°C) and allowed partial $\alpha \rightarrow \beta$ solid phase transformation. During the subsequent fast cooling rate to room temperature small $\alpha$ grains would then precipitate resulting in the microstructure shown in Figure 42.

5.2 Stability of the Laser Output over Time and different Locations

Experiments were carried out to assess whether the laser beam was uniform across different locations of the building platform. Three polished Ti-6Al-4V plates were placed in different positions of the building platform, as indicated in Figure 28 (Chapter 3). The laser then scanned the Ti-6Al-4V plates using the same process parameters (Table 10). The depth of the HAZ in the Ti-6Al-4V plates at different locations is shown in Figure 43 whereas the measured penetration depth of the laser is listed in Table 28.

![Figure 43: The HAZ of a) sample in the back-left corner, b) middle and c) front-right corner of the building platform.](image)

The laser output varies slightly across the platform, likely due to the f-theta lens not keeping a spherical beam away from the centre. As a result, it was preferred to build the majority of the
components in the middle of the build platform, where the laser power output was found to reach the highest value as indicated by the higher penetration depth.

Table 28: Average and standard deviation of the penetration depth (average $P$ and SD $P$ respectively) measured in three distinct locations of the building platform

<table>
<thead>
<tr>
<th>Location</th>
<th>Average $P$[μm]</th>
<th>SD $P$[μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>48</td>
<td>3</td>
</tr>
<tr>
<td>B</td>
<td>55</td>
<td>2</td>
</tr>
<tr>
<td>C</td>
<td>46</td>
<td>5</td>
</tr>
</tbody>
</table>

The stability of the laser output over time was then investigated for different laser focus positions. The HAZ zone created by laser scanning the Ti-6Al-4V plate for more than 60 seconds was evaluated. The maximum penetration depth (in correspondence of the point where the laser dwelled) and its minimum depth (in correspondence of the regions where adjacent scan tracks overlapped) was measured every 1.5 seconds.

Irrespective of the chosen focus position, it can be noticed that the laser output displays a transient and a steady-state regime (Figure 44). The transient regime duration that is very short (typically a few seconds). In this regime the laser has the greatest power output, as shown in Figure 44. The laser output however decreases as the optical train warms up, i.e. over time. In the steady-state regime the laser output was approximately constant.

The laser penetration depth as a function of time is described by sinusoidal curves, consistently with the fact that the scan tracks were generated with hatch spacing smaller than the melt pool width.
Figure 44: The penetration depth variation with time: it can be noticed that in the first few seconds the laser output is unstable. Elapsed the transient time (i.e. in the steady-state regime), the laser is characterised by a constant output.

Figure 44 also shows that, in the steady-state regime the laser output is similar irrespective of the laser focus position. These unexpected results demonstrate that the optical train losses significantly affect the laser output, and even with the laser beam in the focus position it could not reach the nominal power of 200W.

In order to overcome the potential problems related to the laser fluctuations in the transient regime, a sacrificial dummy block was processed at the beginning of each layer deposition. In this way, the intended parts were processed in the laser steady state regime.
5.3 Effect of Laser Hatch Spacing

The effect of hatch spacing on the laser HAZ was studied in detail by scanning the Ti-6Al-4V plate with the parameters listed in Table 11. It should be noted a sacrificial block was initially scanned by the laser to dissipate the transient regime that characterised the laser in the AM250. All the results reported were obtained using the laser in its steady-state regime. The martensitic layer formed in relation to the various hatch spacing values is shown in Figure 45. The depth of the HAZ for the various laser hatch-spacing is reported in Table 29.

Figure 45: Effect of hatch spacing on the bimodal microstructure of the reference Ti-6Al-4V: a) 100 μm, b) 120 μm, c) 180 μm, d) 200 μm, e) 250 μm, f) 300 μm. The micrographs are taken from the frontal xz-plane.
As the hatch spacing increases, the average laser penetration depth decreases linearly as shown in Figure 46. The overlapping of the molten area decreases if the hatch spacing increases. For this reason the deviation from the average penetration depth increases with the hatch spacing.

Table 29: Average and standard deviation of the penetration depth (average \( P \) and \( SD \ P \) respectively) created using different laser hatch spacings

<table>
<thead>
<tr>
<th>Hatch [( \mu m )]</th>
<th>100</th>
<th>120</th>
<th>160</th>
<th>180</th>
<th>200</th>
<th>250</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average ( P ) [( \mu m )]</td>
<td>58</td>
<td>55</td>
<td>50</td>
<td>46</td>
<td>42</td>
<td>38</td>
</tr>
<tr>
<td>SD ( P ) [( \mu m )]</td>
<td>5</td>
<td>3</td>
<td>6</td>
<td>8</td>
<td>12</td>
<td>16</td>
</tr>
</tbody>
</table>

When all the other laser parameters were kept fixed, and the hatch spacing increases the overall energy density delivered by the laser beam decreases.

Figure 46: The linear relationship between the hatch spacing and the average penetration depth of the laser. The deviation from the average penetration depth are also indicated in the graph.

It was also noted that the hatch spacing has a great effect on the surface roughness of the melted layer, or in other words the “waviness” seen on the surface layer in Figure 45. Indeed, as the hatch spacing increases the cylindrical profile of each scan track tends to emerge, and thus the HAZ becomes more wavy. The surface roughness generated by the laser melting of Ti-6Al-4V plate was hence studied more in detail. The 3D surface profiles of portion of rolled Ti-6Al-4V scanned with different hatch spacing are shown in Figure 47. Figure 47 shows clearly that the
surface roughness diminishes as the overlap of adjacent scan tracks increases. When the hatch spacing decreases, the peaks of the cylindrical scan tracks are brought closer together combining the melted portion of material to a similar height. The measure of the surface roughness along a line that crosses multiple scan tracks confirms this suggested mechanism (Figure 48).

Figure 47: 3D surface profiles of the scan tracks generated with different laser hatch spacings: a) 250, b) 200, c) 180, d) 160 and e) 100 μm.
Figure 48: Surface roughness measured along a line crossing the surface of the reference material scanned with different hatch spacings: a) 250, b) 200, c) 180, d) 160 and e) 100 μm.

5.4 Effect of Laser Scan Speed

The effect of the nominal laser scan speed on the laser HAZ was studied in detail investigating the Ti-6Al-4V plate with the parameters listed in Table 12. All the results reported here were obtained using the laser in its steady-state regime. The microstructures of the Ti-6Al-4V plate after laser modification with the various laser scan speed values is shown in Figure 49. The average depth and standard deviation of the laser penetration depth are listed in Table 30. It is noteworthy that no apparent change in surface roughness is shown in Figure 49 when the laser scan speed varies.
Figure 49: The effect of laser scan speed on the bimodal microstructure of the rolled Ti-6Al-4V: a) 320, b) 265, c) 225, d) 205, e) 187 mm/s. The micrographs are taken from the frontal xz-plane.

As the experiments were carried out using a hatch spacing of 100 μm and significant overlap of the adjacent scan tracks occurred irrespectively of their depth. When the laser parameters are kept fixed and the scan speed is increased, the overall volumetric energy density applied by the laser beam decreases, hence a minor portion of solid material is melted. The relationship between the laser scan speed and the average penetration depth is again linear (Figure 50).
Table 30: Average depth and standard deviation of the penetration depth (average $P$ and SD $P$ respectively) created by the laser scanning at different speeds

<table>
<thead>
<tr>
<th>Scan Speed [mm/s]</th>
<th>321</th>
<th>265</th>
<th>225</th>
<th>205</th>
<th>187</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average $P$ [$\mu m$]</td>
<td>51</td>
<td>59</td>
<td>63</td>
<td>70</td>
<td>74</td>
</tr>
<tr>
<td>SD $P$ [$\mu m$]</td>
<td>1</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>2</td>
</tr>
</tbody>
</table>

Figure 50: The linear relationship between the laser scan speed and the average penetration depth of the laser. The deviation from the average penetration depth is also shown in the graph.

5.5 Effect of Laser Focus Position

The effect of the laser focus position on the HAZ was studied in detail with the parameters listed in Table 13. Again, all the results reported here were obtained using the laser in its steady-state regime. Figure 51 shows the microstructure of the rolled Ti-6Al-4V after it is has been scanned with the laser at different focus positions.
It is shown that the focus position of the laser beam has a substantial effect on the HAZ of Ti-6Al-4V. When the laser is focussed below the surface (+ve focus), individual scan tracks can be noticed as shown in **Figure 51a-b**. Despite the small hatch spacing (100μm) individual scan tracks can be noticed. The individual scan tracks are even more visible when the laser is focussed above the surface to scan (–ve focus) as shown in **Figure 51h-i**. In this case the deepest melts pools are created. It is clear that the laser penetration when the laser is out of the focus position is not desirable, as a non uniform melting process caused by the inferior penetration depth in between adjacent scan tracks (**Figure 51a-b** and **h-i**) can lead to discontinuities and porosity during SLM. From these results it can be concluded that only the “zero” position (laser focused at the material surface) and the “+2” position (**Figure 51d-e**) are suitable for processing. Indeed these two focus positions can melt Ti-6Al-4V homogenously and effectively, i.e. the thickness of the martensitic layer was relatively constant and thicker than that obtained with the other focus positions capable of creating homogeneous HAZs.

The lack of symmetry of laser output at the same but opposite distances from the focus position was unexpected. In a theoretical condition, the penetration depth of the laser depends on two variables: 1) the laser beam spot size and 2) the laser output. At fixed laser output (i.e. there are no losses in the optical train), the penetration depth is maximum when the laser focus spot is located on the surface of the material. In this condition, the smallest spot size and the maximum energy density is obtained [Olson and Swope, 1991; Bandyopadhyay et al., 2002;
Bandyopadhyay et al., 2005]. On the other hand, if the laser is focussed far from the material’s plane, the energy density decreases and broader but shallower volume interactions (or melt pools) occur [Olson and Swope, 1991]. Figure 51 indicates that the losses in the optical train (and therefore the laser output) depend on the laser focus position: losses increase when the laser is focussed below the surface to scan, and decrease for negative focus positions.

The surface roughness of the scan tracks created by the laser in focus (0 mm) and at +2mm were then compared. The 3D profiles of the scan tracks surfaces are shown in Figure 52.

![Figure 52](image)

*Figure 52: Three dimensional scan track profiles for a) laser focus at the material surface; b) laser focused 2mm below the material surface (+2mm)*

The comparison reveals no discernible differences in the surface roughness of the scan tracks created at these two focus positions. For this reason, all the samples built with the AM250 were processed focusing either at the surface of the material (powder bed) or 2mm below (focus position +2mm).

### 5.6 Effect of Laser Scan Strategy

In order to investigate the effect of the laser scan strategy on the laser melting of Ti-6Al-4V, the material was laser scanned using the checkerboard scan strategy (details in Section 3.2.2), i.e. dividing the cross section in islands of different size. The laser beam was used in the steady-state regime. Figure 53 shows the HAZ in the microstructure of the Ti-6Al-4V plate using the checkerboard scan strategy with different square island size (0.2, 0.5, 1 and 2 mm).

It can be noticed however that a lack of melting occurs in correspondence to the borders of adjacent islands as shown by the arrows in Figure 53. Marcam Autofab software allows the
setting of the length of the square islands and the hatch spacing (in this case 100μm). Each island is then scanned with a number of scan vectors equal to the ratio of the island length to the hatch spacing (e.g. 2 for an island size of 200μm and hatch spacing of 100μm). The laser then starts to scan the island either from the island bottom or its left-hand side and scans the remaining area with the last scan vectors. This is shown in the schematic of Figure 54. As not all the sides are of each island are scanned equally, discontinuities can be formed in the cross section as shown in the micrographs of Figure 54. In practice, an additional border scan would thus be beneficial if and when the checkerboard scan strategy is used.

Figure 53: The HAZ created using different scan vector lengths a) 0.2 mm, b) 0.5 mm, c) 1 mm, d) 2 mm. The discontinuities are at the ends of the islands are marked by red arrows, whereas examples of different island sizes are enclosed by a dotted black lines.

Figure 54: Schematic of the division of the cross section into islands on 0.2mm size
Table 31 shows that the average penetration depth of the laser decreases if the island size decreases (i.e. square islands with a length of 0.2 mm show the smallest penetration depth). This result shows that the melt pools created in islands larger than 1mm are significantly deeper than those created in smaller islands. This result could occur because more scan tracks overlap during the layer processing although further characterisation is required.

Table 31: Average, standard deviation, minimum and maximum of the laser penetration depth (Average p, SD p, Min p, and Max p respectively) corresponding to islands length of 2mm, 1mm, 0.5mm, 0.2mm

<table>
<thead>
<tr>
<th>Island length [mm]</th>
<th>2</th>
<th>1</th>
<th>0.5</th>
<th>0.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average P [μm]</td>
<td>73</td>
<td>68</td>
<td>50</td>
<td>49</td>
</tr>
<tr>
<td>SD P [μm]</td>
<td>2</td>
<td>3</td>
<td>15</td>
<td>13</td>
</tr>
<tr>
<td>Min P [μm]</td>
<td>66</td>
<td>67</td>
<td>24</td>
<td>21</td>
</tr>
<tr>
<td>Max P [μm]</td>
<td>77</td>
<td>71</td>
<td>66</td>
<td>59</td>
</tr>
</tbody>
</table>

No significant differences were observed in the microstructure of the heat affected zone corresponding to the islands of different size. As an example, the microstructure of the HAZ of the island of 0.2mm length is shown in Figure 55. This result shows that although the cooling rate associated with the solidification of the scan tracks decreases with the size of the island, as adjacent tracks are scanned rapidly one after the other leaving little time for the island to cool down, the diffusional β Thiphase transformation is still suppressed.

Figure 55: Detail of the microstructure of a scan track obtained using a scan vector length of 0.2 mm.
5.7 Validation of test cubes

In order to validate the process parameters obtained from scanning the Ti-6Al-4V plate, the density of 1cm³ test cubes built using similar process parameters were compared. Figure 56 and Table 32 summarise the effect of the hatch spacing on the density of the samples. All the other laser processing parameters were kept fixed and are listed in Table 15.

Figure 56: Effect of the hatch spacing on the density of the samples: the hatch spacing used are :a) 180 μm, b) 160 μm, c) 120 μm and d) 100 μm.

Table 32: Area fraction of porosity (average and standard deviation) in the samples built with different hatch spacing

<table>
<thead>
<tr>
<th>Hatch Spacing [μm]</th>
<th>Pore Area Fraction [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.1 ± 0.2</td>
</tr>
<tr>
<td>120</td>
<td>0.4 ± 0.2</td>
</tr>
<tr>
<td>160</td>
<td>0.5 ± 0.3</td>
</tr>
<tr>
<td>180</td>
<td>0.7 ± 0.2</td>
</tr>
</tbody>
</table>
It can be noticed that the density of the parts increases when the hatch spacing decreases. This is consistent with that reported in Section 5.3. It was in fact observed that decreasing the hatch spacing the layer surface roughness decreases. Similarly decreasing the hatch spacing it was noted that the penetration depth is more homogenous. Thus it can be assumed that for processing dense components it is suitable to use a close hatch spacing. The effect of the laser scan speed on the density of cubic components is shown in Figure 57 and summarised in Table 33. Again all the other laser scan parameters are kept fixed and are listed in Table 16. Figure 57 show that when the laser scan speed exceeded a critical value the powder bed is not melted effectively and the volume fraction of porosity in the samples increases. In the components that were scanned using high laser scan speed, it appears that porosity occurs predominantly in regions of the sample at the same distance from the platform (as shown by the red dotted line in Figure 57a). This result could be caused by a lack of efficient layer melting as the laser melts the powders to a penetration not sufficient to guarantee a fully dense weld to the layer below. In other words, the laser does not have sufficient time to homogeneously melt the layer of powder.

![Figure 57: Effect of laser scan speed on the density of cubic components. The laser scan speed is: a) 365 mm/s b) 315 mm/s c)275 mm/s and d) 225 mm/s. The red dotted line shows the horizontal alignment of the pores.](image)

Figure 57: Effect of laser scan speed on the density of cubic components. The laser scan speed is: a) 365 mm/s b) 315 mm/s c) 275 mm/s and d) 225 mm/s. The red dotted line shows the horizontal alignment of the pores.
Table 33: Area fraction of porosity (average and standard deviation) in the samples built with different laser scan speeds

<table>
<thead>
<tr>
<th>Scan Speed [mm/s]</th>
<th>Pore Area Fraction [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>225</td>
<td>0.1 ± 0.3</td>
</tr>
<tr>
<td>275</td>
<td>0.4 ± 0.3</td>
</tr>
<tr>
<td>315</td>
<td>0.5 ± 0.3</td>
</tr>
<tr>
<td>365</td>
<td>0.7 ± 0.2</td>
</tr>
</tbody>
</table>

Cubic components were also built in different locations of the build platform to establish the effect of the variation of the laser output across the platform. The components were built using the same process parameters (Table 9). Figure 58 shows, at least qualitatively, that porosity is present in all the components as the parts were processed with a relatively large hatch spacing (200μm).

Figure 58: Schematic showing the location on the platform of the build components and the corresponding effect on the density of the components.
Despite the difference in the laser output measured in Section 5.1 no visible trend appears in the micrographs. The porosity showed in the micrograph is not surprising as these components were built using a set of parameters previously optimised for the production of SLM stainless steel. Finally, cubic parts were built using different laser focus positions (Table 17). The laser focus position has a significant effect on the density of the samples as shown in Figure 59 and Table 34.

Figure 59: Effect of the laser focus position on the density of cubic components. Laser focus position set at a) -2 mm, b) 0 mm, c) +2 mm.

Table 34: Area fraction of porosity (average and standard deviation) in the samples built with different laser focus positions

<table>
<thead>
<tr>
<th>Focus Position [mm]</th>
<th>Pore Area Fraction [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>-2</td>
<td>1.2 ± 0.3</td>
</tr>
<tr>
<td>0</td>
<td>0.6 ± 0.3</td>
</tr>
<tr>
<td>+2</td>
<td>0.2 ± 0.2</td>
</tr>
</tbody>
</table>

It can be noticed that the sample processed with laser below the powder bed (focus position at +2 mm) showed the highest density. This is consistent with the fact that the penetration depth of the laser focused on the powder bed or slightly below is more homogeneous as reported in Section 5.5. On the other hand, it was shown that using the laser out of focus produces discontinuities between individual scan tracks. These discontinuities represent sites where lack of melting might occur and thus regions where porosity can form.
The objective of this series of experiments was to characterise the laser beam output and its interaction with Ti-6Al-4V for a variety of different processing windows. A series of experiments was carried out to study how the laser parameters affect the melting and solidification processes of rolled Ti-6Al-4V. The useful insights that these experiments provided were then validated by comparing the density of the simple cubic components built under different processing conditions. It was found that:

- The laser output was not constant across the building platform. As a result it was chosen to built in the middle of the platform whenever this was allowed by the geometry of the build;
- The laser output was not constant over time. It displayed a transient and steady-state regime. In order to use the laser in its steady state regime a sacrificial dummy part was scanned at the beginning of each layer of processing;
- The distance between adjacent scan tracks (i.e. laser hatch spacing) affects the depth of the melt pool and surface roughness of the sample. Decreasing the hatch spacing produce larger laser heat affected zones. The relationship was linear. The overlap of adjacent scan tracks reduces the surface roughness and therefore small hatch spacing distances (e.g. 100μm) are preferred to produce dense parts;
- The laser scan speed affects the depth of the laser heat affected zone; similarly to that found for the hatch spacing, the energy input is inversely proportion to the laser scan speed, i.e. lower laser scan speeds produce larger melt pools;
- The depth of the heated affected zone was sensitive to the laser focus position. Despite the laser having no symmetrical behaviour, regular scan tracks are obtained for a laser focussed slightly below or on the surface of the metal (or powder bed);
- Short scan tracks are recommended to reduce the residual stresses in metal processing [Gibson et al., 2011]. However, using a checkerboard scan strategy where only the interior of the islands was scanned can cause discontinuities in the melting of the powder bed;
- The results obtained by studying the melting of the reference Ti-6Al-4V plates provide guidelines to build components with high density.
In this Chapter the microstructure and the crystallographic texture of SLM Ti-6Al-4V built with the optimised parameters is studied. Firstly the microstructure of the components in the as-built condition is described (Section 6.1). Further understanding of the solidification of the Ti-6Al-4V is then obtained through a detail analysis of the crystallographic texture of the components in the as-built condition (Section 6.2).

As during SLM considerable thermal stresses might develop in the parts, a post stress relief heat treatment is often necessary. For this reason, it was of interest to study the effect of a stress relief heat treatment on the microstructure and the crystallographic texture of Ti-6Al-4V. These aspects are discussed in Sections 6.3 and 6.4 respectively.

6.1 Microstructure Evolution of as-built SLM Ti-6Al-4V

Optical microscopy analysis revealed that the microstructure of the components was composed of fine acicular α' grains throughout the sample and prior β grain boundaries can be clearly identified after the solidification (Figure 60) [Sercombe et al., 2008, Facchini et al., 2010, Thijs et al., 2010, Vrancken et al., 2012]. Ti-6Al-4V is an allotropic alloy that transforms fully into the β phase field above the β transus temperature (~ 950°C) and into a α+β phase mixture below this critical temperature. Nevertheless the amount β phase retained at room temperature is governed by the cooling rate experienced from the β phase field [Glavicic et al., 2003]. During SLM, each layer cools down very rapidly (in the orders of thousands degrees per second [Roberts et al., 2009]) thus the microstructure is fully martensitic.
Figure 60: a) 3D optical metallograph (OM image) composite showing the microstructure of SLM Ti-6Al-4V components in the as-built condition. The micrographs show the microstructure in b) the frontal plane, c) the lateral plane and d) the horizontal plane. The vector $\vec{g}$ indicates the prior β grain growth. The samples were built using a meander laser scan strategy.

The frontal plane ($xz$-plane) shows continuous vertical prior β grain boundaries parallel to the build direction (Figure 60b). The presence of prior β columnar grain boundaries is due to the fact that Ti-6Al-4V solidifies in the β phase field and heat is mainly conducted away vertically [Roberts et al., 2009]. Prior β grains have a lenticular morphology with a high aspect-ratio. The average width of the prior β grains was $103\pm32\mu m$ that corresponds to the optimised hatch spacing ($100\mu m$). Prior β grains have a length in the order of millimetres. It is not clear in the micrograph where each layer deposition has occurred, however it can be estimated that β grains have grown through tens of layers as each powder bed layer on the scan platform was approximately $50 \mu m$. Prior β columnar grain boundaries also emerge from the optical microscopy analysis of the lateral $yz$-plane (Figure 60c). Figure 60d shows that the prior β grain growth direction $\vec{g}$ is inclined $\sim 20^\circ$ to the build direction ($z$-axis) as a result of the adopted rotating scan strategy [Thijs et al., 2010]. Although the scan vectors are hatching each layer in alternating directions according to the meander scan strategy (Section 3.2.1), it is reasonable to assume that the $67^\circ$ rotation of the scan direction imposed by the scan strategy causes local heat gradients in the $x$ direction (Figure 24). These thermal gradients would explain the inclination of the β grains shown in Figure 60c. The horizontal plane ($xy$-plane) reveals the cross sections of the prior columnar β grains but the correlation between the scan strategy and the morphology of the prior β grains does not appear as clear as reported in other studies [Thijs et al., 2010] probably due to the complexity of the meander scan strategy. Further heat transfer modelling is required to detail the thermal gradient for this particular scan strategy.
The optical microscopy analysis reveals that the $\alpha'$ grains did not vary in size along the build and scanning directions because the build platform was kept at a temperature much lower than the recrystallization temperature of 800°C for Ti-6Al-4V [Lütjering, 1998]. The build platform that acts as a heat sink and did not affect the as-deposited $\alpha'$ phase size, differently from that reported for electron beam melted and laser metal deposited Ti-6Al-4V where the thermal mass offered by the components built within a heated build platform has an effect on the decomposition of non-equilibrium phases and the grain growth [Facchini et al., 2009; Al-Bermani et al., 2010].

The $\alpha'$ martensitic laths are organised within prior $\beta$ grain boundaries with different inclinations mainly at $\sim \pm 45^\circ$ and $\sim 90^\circ$ to the build direction as shown in Figure 61. This result is consistent with that reported in other studies on SLM Ti-6Al-4V [Sercombe et al., 2008; Vrancken et al., 2012]. The reason behind this preferred arrangement of the $\alpha'$ martensitic laths remains unclear [Banerjee and Williams, 2013].

![Figure 61: a) and b) Backscattered electron micrographs showing the morphology and the arrangement of the $\alpha'$ laths within the prior $\beta$ columnar grains.](image)

The $\alpha'$ grains do not precipitate along the $\beta$ grain boundaries (Figure 61) and this suggests that the $\alpha'$ grains originate simultaneously from different points within the parent $\beta$ grain which is typical of martensitic transformations. This microstructural morphology will be discussed in connection with the crystallographic texture of the $\alpha'$ grains in the following section.

In order to confirm that the mentioned acicular laths are $\alpha'$ martensitic, TEM studies were carried out. A TEM bright field image from the as-built component $xy$-plane clearly shows the typical basketweave arrangement of the martensitic microstructure (Figure 62). The morphological features of individual $\alpha'$ laths are shown in Figure 63. As reported in a related study [Ahmed and Rack, 1998], $\alpha'$ appear as relatively long and narrow laths. Some of these $\alpha'$ laths are arranged...
aligned to one another. The corresponding \textless 1213 \textgreater , \textless 01\bar{1}2 \textgreater selected area diffraction (SAD) patterns and their indexing are shown in Figure 63.

\textbf{Figure 62}: TEM bright-field image showing the arrangement of the $\alpha'$ grains in the microstructure of the as-built components

The martensitic acicular laths possess a HCP structure similar to the $\alpha$ equilibrium phase [Sercombe \textit{et al.}, 2008; Qazi \textit{et al.}, 2001; Qazi 2003; Sato 2010]. SAD patterns confirm therefore that no $\alpha''$ martensitic phase (orthorhombic lattice structure) is present in the area of examination of the as-built components. The substructure of these laths is characterized by high dislocation density and either twins or stacking faults (ribbon-like contrast) (\textbf{Figure 64-65}) [Ahmed and Rack, 1998]. Further work to determine the exact nature of these defects is required.
Figure 63: TEM micrograph from the xz-plane showing a) the morphology of several α' grains, b) indexed SADP of the marked area with a zone axis of $<1213>$, c) $<01\bar{1}2>$

Figure 64: TEM bright field image from the xz-plane on as-built sample. The micrograph reveals presence of crystallographic defects (either twins or stacking faults) in the substructure of the grains.
Figure 65: TEM bright field image of sample showing dislocations in α′ lath.

Figure 66: TEM micrograph showing the area (red box) where several EDS spectra were collected.

The α′ martensitic phase was further confirmed by quantitative TEM-EDS analysis. Figure 66 shows the exact site where EDS analysis was carried out. Quantitative results are summarized in Table 35, which shows the average chemical composition of the area indicated by the red circle in Figure 66. As reported by many authors, this martensitic phase is characterized by a higher content in V than that registered in the equilibrium α. As reported by many authors, this m 1998, Sato 2010]. Figure 67 and Table 36 show similar results obtained from a different grain in the
same TEM foil. The chemical composition of the equilibrium $\alpha$ and $\beta$ phases in the Ti-6Al-6V plate (Table 37) was also measured in order to establish a comparison with the $\alpha'$ martensitic phase found in the SLM components. Comparing Table 35-37 it can be noted that the equilibrium $\alpha$ phase in Ti-6Al-4V is less rich in V when compared to the $\alpha'$ martensitic phase found in the SLM components. As SLM is characterised by rapid cooling rates it is plausible that diffusionless $\beta \rightarrow \alpha'$ transformation occurs during the solidification of the melt pool explaining the different V content measured in the $\alpha'$ phase.

Table 35: Weight percentage of the chemical elements detected in the EDS analysis performed in the area marked in Figure 66

<table>
<thead>
<tr>
<th>Element [wt%]</th>
<th>Average</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>87.3</td>
<td>0.6</td>
</tr>
<tr>
<td>Al</td>
<td>7.4</td>
<td>0.6</td>
</tr>
<tr>
<td>V</td>
<td>5.3</td>
<td>0.3</td>
</tr>
</tbody>
</table>

Figure 67: TEM micrograph showing another area (red box) where seven EDS spectra were collected
Table 36: Weight percentage of the chemical elements detected in the EDS analysis performed in the area marked in Figure 67

<table>
<thead>
<tr>
<th>Element [wt%]</th>
<th>Average</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>86.7</td>
<td>0.5</td>
</tr>
<tr>
<td>Al</td>
<td>7.6</td>
<td>0.5</td>
</tr>
<tr>
<td>V</td>
<td>5.7</td>
<td>0.3</td>
</tr>
</tbody>
</table>

Table 37: Average (± standard deviation) weight percentage of the chemical elements detected in the EDS analysis performed in the Ti-6Al-4V plate (bimodal microstructure)

<table>
<thead>
<tr>
<th>Element [wt%]</th>
<th>α phase</th>
<th>B phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>87.2 ± 0.5</td>
<td>81.5 ± 1.1</td>
</tr>
<tr>
<td>Al</td>
<td>11.0 ± 0.5</td>
<td>6.4 ± 0.3</td>
</tr>
<tr>
<td>V</td>
<td>1.8 ± 0.5</td>
<td>12.1 ± 0.9</td>
</tr>
</tbody>
</table>

6.2 Crystallographic Texture of as-built SLM Ti-6Al-4V

In recent years crystallographic texture has been shown to be extremely useful to understand the relationship between processing and the microstructure of a number of α/β Ti alloys to achieve desirable mechanical properties [Glavicic et al., 2003; Kobryn and Semiatin, 2003; Glavicic et al., 2004]. In order to investigate the texture evolution occurring during SLM, specimens from the lateral yz-, frontal xz- and horizontal xy- planes of the as-deposited material were studied by electron backscatter diffraction (EBSD).

Figure 68a shows the α' orientation map of the lateral plane (yz-plane) of the as-built cubic samples. No packets of α' laths sharing the same crystallographic orientation (also known as α' colonies) appear in the orientation map, contrary to that reported for Ti-6Al-4V processed by shape metal deposition and electron beam melting [Al-Bermani et al., 2010; Baufeld et al., 2010]. It is plausible that this results because of higher cooling rates experienced by the solidifying
powder bed during SLM [Roberts et al., 2009]. The overall α' texture appears random because of the relatively high number of α' variants within each prior β grain (Figure 68b).

Figure 68: a) EBSD [001] IPF α' orientation map and the corresponding colour scheme of a specimen taken from the lateral yz-plane of an as-deposited component; b) corresponding (0001)_α' and (1120)_α' contour pole figures; c) IPF orientation map of the reconstructed β phase. The grain boundaries in the reconstructed map represent β grains misoriented equal or larger than 7°; d) the corresponding (110), (111), (100) contour pole figures.

Figure 68c shows the corresponding reconstructed β orientation map whereas the contour pole figure of the β phase is illustrated in Figure 68d. The orientation map of the reconstructed β phase reveals that each β grain grew epitaxially extending through several deposited layers. The analysis of the corresponding contour pole figures shows a dominant {100} texture component in the direction of grain growth \( \vec{g} \) that tends to develop because during solidification cubic crystals grow faster in the {100} direction [Flemings, 1974; Kobryn and Semiatin, 2000; Al-Bermani et al., 2010]. This result is consistent with that reported form Ti-6Al-4V processed with other AM technologies where columnar solidification occurs [Al-Bermani et al., 2010; Kelly and Kampe, 2004].

The α' texture of the frontal plane (xz-plane) is shown in Figure 69a. Similar to the lateral yz-plane, no α' colonies are present in the microstructure and the α' texture is weak (Figure 69b). The reconstruction of the corresponding high temperature β phase is shown in Figure 69c. Although the orientation components shown in the pole figures (Figure 69b.d) do not appear as
{100} texture, by applying a rotation of ~ 20° around the y-axis (hence aligning the z direction with the inclined grain growth direction \( \vec{g} \)) produces the {100} texture components observed on the lateral plane (yz-plane) confirming that the {100} \( \beta \) grain growth direction is inclined ~ 20° to the build direction.

**Figure 69:** a) EBSD [001] IPF \( \alpha' \) orientation map and the corresponding colour scheme of a specimen taken from the frontal \( xz \)-plane of an as-deposited component; b) corresponding \((0001)_{\alpha'}\) and \((1120)_{\alpha'}\) contour pole figures; c) Orientation map of the reconstructed \( \beta \) phase. The black grain boundaries represent \( \beta \) grains misoriented equal or larger than 7°; d) the corresponding \((110), (111), (100)\) contour pole figures; e) \((0001)_{\alpha'}\) and \((1120)_{\alpha'}\) contour pole figures of the \( \alpha' \) data set following a rotation of ~ 20° around the y-axis; f) \((110), (111), (100)\) contour pole figures of the corresponding \( \beta \) phase from the rotated dataset.

High-resolution EBSD (0.3μm step size) from all the three orthogonal planes (horizontal \( xy \)-plane **Figure 70**, frontal \( xz \)-plane **Figure 71** and lateral \( yz \)-plane **Figure 72**) was also carried out to
study in detail the orientation relationship among the α′ variants, which formed within the columnar β grains and their relationship with the parent β phase.

Figure 70: a) α′ and corresponding reconstructed β [001] IPF orientation maps from the horizontal xy-plane (step size 0.3 μm); b) and c) discrete pole figures of the α′ variants and their parent β grain. The arrow indicates α′ laths that are misoriented type 6. The circles indicate α′ laths that are misoriented type 3 whereas the dashed ovals show clusters of α′ variants misoriented type 2.
Figure 71: a) $\alpha'$ and corresponding reconstructed $\beta$ [001] IPF orientation maps from the frontal xz-plane (step size 0.3 μm). The black arrow shows the build direction; b) and c) discrete pole figures of the $\alpha'$ variants and their parent $\beta$ grain. The dotted ovals show clusters of $\alpha'$ variants misoriented type 4 whereas the dashed ovals indicate $\alpha'$ variants misoriented type 2. The circles indicate $\alpha'$ laths that are misoriented type 3. $\alpha'$ laths forming triangular patterns are marked by a square.

As shown in Figure 70b-c, Figure 71b-c and Figure 72b-c, typically, there are 5 to 6 variants that formed inside every parent $\beta$ grain. The $(0001)_{\alpha'}$ plane of each $\alpha'$ lath was parallel to one of the $(110)_{\beta}$ planes of the reconstructed parent phase and at least one of the $<11\bar{2}0>_{\alpha'}$ directions was parallel to one of the $<111>_{\beta}$. 
Figure 72: a) $\alpha'$ and the corresponding reconstructed $\beta$ [001] IPF orientation maps from the frontal xz-plane (step size 0.3 $\mu$m). The black arrow shows the build direction; b) and c) discrete pole figures of the $\alpha'$ variants and their parent $\beta$ grain. The dotted ovals show clusters of $\alpha'$ variants misoriented type 4 whereas the dashed ovals indicate $\alpha'$ variants misoriented type 2. The circles indicate $\alpha'$ laths that are misoriented type 3.

As indicated in Figure 70-72, clusters of 3 $\alpha'$ variants misoriented 63.26° apart around the $<\overline{1}0553>_{\alpha'}$ axis (type 4 [Wang et al., 2003]) or 60° around $<11\overline{2}0>_{\alpha'}$ (type 2 [Wang et al., 2003]) are predominantly formed, whereas the remaining $\alpha'$ laths tend generally to form smaller clusters misoriented 60.83° around the $<1.377 \overline{1} 2.377 0.359>_{\alpha'}$ axis (type 3 [Wang et al., 2003]). In the example of Figure 70b two $\alpha'$ variants are misoriented type 3 (marked by solid circles) whereas Figure 70c shows clusters of two $\alpha'$ variants misoriented type 4. Figure 71b shows the prior $\beta$ grain has transformed into a cluster of three $\alpha'$ laths misoriented type 4 (marked by dotted oval) and a cluster of two $\alpha'$ laths misoriented type 3 (marked by solid circles). The $\beta$ grain illustrated in Figure 71c was instead made of a cluster of three variants misoriented type 2 (marked by dashed ovals) and two remaining individual laths again misoriented type 3. In Figure 72b-c clusters of two $\alpha'$ variants misoriented type 2 and type 4 are shown. From the analysis of the data sets, it was therefore reasonable to believe that the $\alpha'$ grains precipitate in order to satisfy the self-accommodation of the neighbouring $\alpha$ grains [Wang et al., 2003] according to which small group
α’ variants would prefer to adopt two of the five types of possible grain boundary misorientation angles (i.e. type 2 and type 4) in order to minimize the average shape strain induced by the β → α’ transformation. Occasionally α’ grains share a (0001)α’ pole and have the three (11\overline{2}0)α’ reflections misoriented by 10.53° around the <0001>α’ axes (type 6 [Wang et al., 2003]) as indicated by the arrows in Figure 70 and 72. This particular orientation relationship derives from the 6-fold symmetry of the α’ lattice and by the fact that during the β → α’ transformation the <11\overline{2}0>α’ direction remains parallel to the two possible <111>β close packed directions that lie on the \{110\}β plane [Bhattacharyya et al., 2003].

Triangular geometric patterns occur occasionally from the (11\overline{2}0)α’ pole figures as shown in Figure 71 (marked by a square). These patterns occur when the basal reflections of different α’ grains are not parallel but one or two (11\overline{2}0)α’ reflections are shared, and therefore clusters of three variants misoriented type 4 are formed within the same β grain.

A careful analysis of Figure 71-72 reveals that even though β grains have grown through successive layer depositions, α’ laths with similar orientations have formed dominantly throughout the entire prior β grain. During SLM, the α’ phase around the melt pool is heated into the β phase field and then rapidly cooled down to form the same α’ variants suggesting that texture inheritance / preferred variant selection might have taken place [Lonardelli et al., 2007, Daymond et al., 2010].

SLM involves remelting of the top portion of the deposited part together with a newly deposited layer of powders, thus as continuous α’ → β transformations occur, correspondingly a large number of α’ variant would form if texture inheritance / variant selection did not take place.

As shown in the example of Figure 73, three dominant α’ orientations are observed due to the preferred variant selection within the same prior β grain as indicated in the (0001)α and (11\overline{2}0)α pole figures of the corresponding grains. More specifically, it was observed that the α’ laths indicated with the same colour in Figure 73a, represent the same variant and were misoriented less than 5° despite having formed during successive layer depositions.
Figure 73: Detail from the frontal xz-plane orientation map showing that the prior β columnar grain is composed of three α′ variants that repeat during successive depositions; b) corresponding (0001)_α′ and (1120)_α′ discrete pole figures.

Although texture inheritance has never been reported for Ti-6Al-4V made by AM technologies, it is clear that β grains grow epitaxially and maintain a strong {100} texture during the α′ ↔ β phase transformation associated with the re-melting of successive layers. As a result, just one preferred variant among the 6 possible solutions for the α′ ↔ β transformation has occurred.

During the cooling of the layers (i.e. the β → α′ transformation) variant selection at prior β grain boundaries has been demonstrated [Bhattacharyya et al., 2003; Stanford and Bate, 2004]. Therefore the orientation relationship of the α′ grains across prior β grain boundaries was investigated. Typical examples are shown in Figure 74-75.
Figure 74: a) EBSD orientation map of two reconstructed adjacent β grains and their α' variants; b) superimposition of the (110)_β and (111)_β pole figures of the prior β grains (orange and light-blue reflections corresponding to the prior β grains A and B respectively). The arrows indicate pole reflections that are almost parallel to each other; c) (0001)_α' and d) (1120)_α' pole figures of the α' variants deriving from the two grains.

Figure 74a shows two prior β grains A and B which are 8° misoriented and therefore meet the criteria for variant selection at the grain boundary [Stanford and Bate, 2004]. It was observed that the α' laths formed in the corresponding β grains shown no orientation relationship and thus preferred variant selection across the boundary. In particular, the example of Figure 74b shows that one (110)_β and one (111)_β plane in those two β grains are almost parallel (indicated by the arrows in Figure 74b) but the α' variants that have formed in each β grain have none of the (0001)_α' planes that remained parallel to that shared (110)_β.

Figure 75 shows another example for which it is evident that preferred variant selection across the prior β grain boundary is unlikely for SLM. It was observed that the reconstructed β grains have high angle grain boundaries misorientation (51° in this particular example) and therefore it is reasonable to confirm that selection of α' variants of the same orientation on both sides of the
neighbouring β grains would not be beneficial to lower the α′ nucleation energy during the β ucα′ transformation as reported in [Bhattacharyya et al., 2003; Stanford et al., 2004].

Figure 75: a) EBSD orientation map of two reconstructed adjacent β grains and their α′ variants; b) superimposition of the (110)\textsubscript{β} and (111)\textsubscript{β} pole figures of the prior β grains (red and green reflections corresponding to the prior β grains A and B respectively); c) (0001)\textsubscript{α′} and d) (1120)\textsubscript{α′} pole figures of the α′ variants deriving from the two grains that do not show any orientation relationship because of the high angle misorientation of the prior β grains (51°).

It was observed however that α′ variants of similar inclination to the growth direction have the same crystal orientation shown by the same colour (Figure 73). This suggests that during the β → α′ transformation certain variants are chosen preferably to minimise the work associated with the stresses and strains involved in the phase transformation [Daymond 2010]. Similar mechanisms of local variant selection might take place during SLM.

To verify whether certain α′ variants occurred more frequently, statistics regarding the variant frequency was determined for the data sets acquired on the three orthogonal planes. It was observed that during the β → α′ all the 12 variants occurred but 6 of the 12 solutions constitute about 70% of the data sets (Figure 76).
Figure 76: Variant frequency distribution from the data sets analysed in Figure 70-72

Among the six variants that occur more frequently, it was observed that variants that are misoriented type 6 (for example solutions D$^1$C$_{31}$ and D$^1$C$_{4X}$) [Wang et al., 2003], type 2 (e.g. solutions D$^1$C$_{33+}$ and D$^1$C$_{4X+}$) [Wang et al., 2003], or type 4 (e.g. solutions D$^1$C$_{33+}$ and D$^1$E) [Wang et al., 2003], dominate for all the three orthogonal planes consistently with that observed on the discrete pole figures of Figure 70-72.

It is noteworthy that the variant frequency distribution was determined for all the $\alpha'$ grains equal or larger than about 1μm. It is possible therefore that the small $\alpha'$ grains (~1μm in size) had precipitated within the columnar β grain following no preferred variant selection, explaining the fact that all the 12 solutions are observed in Figure 76.
6.3 Microstructure of Stress Relieved SLM Ti-6Al-4V

The microstructure of the samples after the stress relief heat treatment is shown in Figure 77. As expected, the stress relief process did not alter the morphology or the inclination of the prior β grains as the heat treatment temperature of 730°C was much lower than the β transus temperature of Ti-6Al-4V [Lütjering and Williams, 2007].

![Figure 77: a) 3D optical metallograph (OM image) composite showing the microstructure of SLM Ti-6Al-4V components in the stress relieved condition. The micrograph shows the microstructure in a) the frontal plane, b) the lateral plane and c) the horizontal plane.](image)

The stress relief heat treatment and the following furnace cooling rate have however a significant effect on the phase composition of the specimens under investigation. A comparison of the grain and phase composition of the as-built and the stress relieved components is presented in Figure 78.
Figure 78: Backscatter electron images showing the microstructure of a) as-built SLM Ti-6Al-4V, b) the same component after the stress relief heat treatment.

The α’ grains of the as-built samples span through the width of the prior β grains with a plate-like morphology, with an average length of 8±3μm and width of 0.570±0.130μm, as shown in Figure 78a. The microstructure of the stress relieved samples consists instead of a mixture of α+β phase, where the β phase stands out as the bright contrast phase located at the α laths boundaries of Figure 78b. It is possible that the stress relief temperature of 730°C, being close to the recrystallization temperature of Ti-6Al-4V, allowed the α’ → α + β phase transformation to take place as suggested in [Vilaro et al., 2011]. It was observed that the α grain size increased significantly in the stress relieved samples (average length and width were 8.7±0.4μm and 1.2±0.3μm respectively) confirming that recovery of the α grains had occurred. Stress relieved SLM Ti-6Al-4V consists therefore of a fully lamellar α+β microstructure. The only difference with the lamellar microstructure typical of conventionally made Ti alloys (section 2.1.4) is that stress relieved SLM Ti-6Al-4V features distinctive elongated prior β grain boundaries due the columnar solidification intrinsic to the SLM process.
6.4 Crystallographic Texture of Stress Relieved SLM Ti-6Al-4V

The crystallographic texture of the as-built specimens has been discussed in detail in Section 6.2. It was concluded that the α′ phase of the as-built components has a weak texture because of the relatively high number of variants that precipitate within each columnar β grain. On the other hand, the reconstruction of the β phase texture shows that the columnar grains possess a strong (100) texture in the grain growth direction $\vec{g}$ during solidification.

In order to compare the crystallographic texture of the stress relieved samples to that of the as-built components, EBSD on the frontal $xz$- and lateral $yz$-planes of the stress relieved parts was carried out. The α orientation maps from the frontal $xz$- and lateral $yz$-planes (Figure 79-81a) indicate that even after the stress relieving process no α colonies are present in the microstructure similar to that observed for SLM Ti-6Al-4V in the as-built condition.

![Figure 79](image)

**Figure 79:** a) EBSD [001] IPF α orientation map and the corresponding colour scheme of a specimen taken from the lateral $xz$-plane of a stress relieved component; b) corresponding $(0001)_\alpha$ and $(11\bar{2}0)_\alpha$ contour pole figures; c) Orientation map of the reconstructed β phase. The black grain boundaries represent β grains misoriented equal or larger than 7°; d) the corresponding $(110)$, $(111)$, $(100)$ contour pole figures.
Figure 80: a) EBSD [001] IPF α orientation map and the corresponding colour scheme of a specimen taken from the frontal yz-plane of a stress relieved component; b) corresponding (0001)\textsubscript{α} and (11\overline{2}0)\textsubscript{α} contour pole figures; c) IPF orientation map of the reconstructed β phase. The grain boundaries in the reconstructed map represent β grains misoriented equal or larger than 7º; d) (110), (111), (100) contour pole figures of the reconstructed β phase along the main grain growth direction \( \hat{g} \).

The corresponding α contour pole figures show that the α texture was weak as a result of the multiple variants that have formed from the β columnar grains (Figure 79-81b). As the α phase has a predominant volume fraction in the microstructure and given the low resolution at which the EBDS analysis was carried out in this sample, the indexed β phase was discarded. The indexed β phase was considered insufficient to gain some understanding regarding the solidification texture that take place during the SLM process. For this reason, similar to that seen in the as-built components, the β texture was reconstructed using the method described in Section 3.5.2 and Appendix A.

From the reconstruction of the crystallographic texture of the corresponding β phase (Figure 79-80c), it is evident that during SLM the β grains grow epitaxially through successive deposited layers. It is clear that the prior β phase has a dominant (100) solidification texture along the grain growth direction (Figure 79-80d) consistent to that reported for SLM Ti-6Al-4V in the as-built condition.
Figure 81: a) EBSD [001] IPF α orientation map and the corresponding colour scheme of a specimen taken from the frontal xy-plane of a stress relieved component; b) IPF orientation map of the reconstructed β phase. The grain boundaries in the reconstructed map represent β grains misoriented equal or larger than 7°.

6.5 Summary

The microstructure of the as-fabricated components fabricated with the optimised set of parameters consists entirely of α’ martensitic phase that has formed from prior columnar β grains. No products of segregation, such as titanium aluminides Ti₃Al were found in the microstructure. The nature of the α’ martensitic phase in the as-built components was confirmed by TEM analysis. The α’ grains have a HCP crystal structure similar to the equilibrium α phase. The α’ grains are however richer in V content and possess a substructure characterised by high dislocation density and several crystallographic defects.

The width of the prior β columnar grains was correlated to the chosen hatch spacing and that the formation of the columnar grains was due to the epitaxial solidification across the deposited layers. This was demonstrated by studying the crystallographic texture of the as-built components. In particular, the crystallographic texture of the samples was studied on three orthogonal planes. It was found that prior β grains have a preferred {100} texture along the main grain growth direction which is inclined ~ 20° to the build direction (z-axis). To date, this is first time that epitaxial growth of the prior β grains during SLM of Ti alloys has been demonstrated.
The orientation relationship of the α' grains precipitated within the columnar grains and their parent β phase was then examined. It was found that the β phase′ transformation is governed by the Burgers orientation relationship: most of the α' laths belonging to the same parent β grain have the (0001)α' reflection 60° misoriented around the <0110>α' axes. The (1120)α' pole figures reveal that the α' variants precipitate in clusters forming boundary misorientations that minimise the strain energy associated with martensitic transformation.

During the deposition of successive layers, texture inheritance seems to occur within prior β grains. It was evidenced by the fact that α' grain of similar inclination are characterised by the same crystal orientation. Although variant selection at prior β grain boundaries is unlikely due the fast cooling that occurs during SLM, it is possible that certain variants might be chosen among others to minimise the phase transformation energy. The overall α' texture, however, appears random because of the high number of μm sized α' grains that precipitate within the columnar β grains that grow in multiple directions and random orientation as indicated by the variant frequency distribution. It was found that the rotating scan strategy has a strong influence on the solidification texture and, in consequence, on the development of the final microstructure. In particular the growth of the β grains was slightly inclined to the building direction. Although qualitatively it was easy to see how the correlation between the laser scan strategy and the prior β might arise, further modelling work is required to understand the exact direction of heat loss in the meander scan strategy.

It is well known that thermal stresses can develop during SLM of metals and Ti alloys. It is of great interest therefore to study how the microstructure of the as-built components changes after the stress relief heat treatments. The SLM Ti-6Al-4V microstructure after the application of the stress relief heat treatment was a mixture of the α+β phases. The two phases have a lamellar morphology that closely resembles the fully lamellar α+β microstructure of the conventionally made Ti-6Al-4V. Similar to that found in the as-built components, no other phases such as titanium aluminides were found in the microstructure. The stress relieved components still features elongated prior β grain boundaries and the peculiar acicular porosity that is present in the as-built components. These microstructural features might have a significant impact in the mechanical properties of SLM Ti-6Al-4V as discussed in Chapter 7. The stress relieving treatment did not affect the crystallographic texture of the SLM samples as the heat treatment was conducted below the β transus temperature.
CHAPTER SEVEN

7 Mechanical Properties of SLM Ti-6Al-4V: Tensile and Fatigue Behaviour

In this Chapter the mechanical properties of SLM Ti-6Al-4V built with a set of optimised parameters are studied. The tensile properties of the as-built and stress relieved samples are discussed in Section 7.1. The effect of the building direction on the tensile properties is also investigated in this section. The mode of fracture is discussed through a detailed analysis of the fracture surfaces and profiles of the samples built in the different conditions.

The high-cycle fatigue behaviour of SLM Ti-6Al-4V is discussed in Section 7.2. This section provides insights regarding the fatigue life of the components with cyclic stress at about half of their yield strength (500 MPa). The fatigue performance of the components is discussed in relation to the microstructure of the sample. A detail analysis of the fracture surfaces is also given in this section.

7.1 Tensile Properties of As-Built and Stress Relieved samples

All the tested samples failed after a considerable amount of necking in the gauge length of the specimens. The tensile stress-strain curves are shown in Figure 82a-b and Table 38 summarises the results obtained. Section 7.1.1 compares the stress relieved samples to the as-built parts. The comparison is made for tensile bars built in identical orientations. Section 7.1.2 describes the effect of the build orientation on the tensile behaviour and fracture mechanism of the specimens.
Figure 82: Stress strain curves of the a) as-built and b) stress relieved components. The curves with different colours indicate the build orientation of the tensile bars, namely the edge xz-, vertical zx- and flat xy-orientation. The orientation is expressed following the ASTM F2921.

Table 38: Summary of the tensile properties of SLM Ti-6Al-4V in the as-built and stress relieved condition

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Tensile properties of SLM Ti-6Al-4V in the as-built condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>XZ</td>
<td>115± 6</td>
</tr>
<tr>
<td>ZX</td>
<td>119 ± 7</td>
</tr>
<tr>
<td>XY*</td>
<td>113 ± 5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Tensile properties of SLM Ti-6Al-4V in the stress relieved condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>XZ</td>
<td>113 ± 9</td>
</tr>
<tr>
<td>ZX</td>
<td>117 ± 6</td>
</tr>
<tr>
<td>XY*</td>
<td>112 ± 6</td>
</tr>
</tbody>
</table>

*XY bars has a smaller gauge cross sectional area compared to XZ and ZX bars
7.1.1 Comparison of the Tensile Behaviour of the Stress Relieved and As-Built condition

The Young’s modulus of the stress relieved and as-built samples is consistent for all the tested samples and similar to that reported elsewhere for Ti alloys components made by SLM or other AM technologies [Facchini et al., 2009; Facchini et al., 2010; Vrancken et al., 2012]. It can be observed that the elastic modulus measured on the as-built and stress relieved bars is similar (Table 38).

Table 38 shows that the yield and ultimate tensile strength (UTS) are higher than Ti-6Al-4V produced by conventional processing [Welsch et al., 1994; Murr et al., 2009]. This result can be explained by the fact that the onset of plastic deformation is dependent on the α colony size, i.e. small colony size delays the onset of the plastic deformation. Both the stress relieved and as-built samples have α (or α’) colony sizes equal to the width of a single α (or α’) laths and therefore display high yield stress. It can be observed that the yield stress and UTS measured on the stress relieved samples was lower than on the as-built samples. This was probably due to the α’ → α phase transformation and the fact that the α laths size has increased after the relatively slow cooling rate from the stress relief temperature as discussed in Section 6.3. It should be noted that after the stress relief heat treatment the tensile bars possess yield stress and UTS similar to wrought and annealed Ti-6Al-4V and exceed those of cast Ti-6Al-4V [Welsch et al., 1994; Murr et al., 2009].

Table 38 shows that the stress relieved samples have larger elongation at failure (irrespective of their orientation) and hence improved ductility compared to the as-built bars. The large scatter from the average elongation at failure indicates however, an improvement not as significant as that reported in other related works [Vilaro et al., 2011; Vrancken et al., 2012]. It is well known that poor ductility is the major limitation for AM α+β Ti alloys and thus stress relief has proved to be beneficial for SLM Ti-6Al-4V. The difference in ductility between the stress relieved and as-built parts can be explained considering the plasticity of the α+β phases [Banerjee and Williams, 2013]. The as-built samples consist entirely of α’ phase and hence the plastic deformation is mainly restricted to the basal and prismatic planes of the hexagonal lattice, namely (0002)(11\(\bar{2}0\)) and (10\(\bar{1}\)0)(11\(\bar{2}0\)) [Banerjee and Williams, 2013]. As the α’ grains do not form colonies of laths sharing the same orientation, the effective slip length is confined to single grains. For this reason, the as-built SLM Ti-6Al-4V tensile bars show a poor elongation at failure. Stress
relieved samples, on the other hand, have retained β phase at the grain boundaries of the α laths (Section 6.3). Although the volume fraction of β phase is limited, the existence of β phase improves the ductility of the stress relieved samples due to slip transfer at the interface of the two phases can take place. Slip transfer occurs because of the α/β Burgers orientation relationship between the α and β phase of Ti-6Al-4V [Pilchak and Williams, 2011]. The two primary slip systems of the α phase are, in fact, precisely aligned with the {110}{111} and {112}{111} systems of the β phase, with a third set misoriented only ~ 10.5° and thus slip transfer across the two phases can occur [Pilchak et al., 2011; Bhattacharyya et al., 2003].

The residual stresses in the as-built samples were measured by X-ray diffraction. As explained in Section 3.5.4, the residual stresses were evaluated from side inclination interplanar strain measurements at 2θ of 142° assuming a biaxial stress condition. Generally, when reporting the residual stresses in SLM metallic parts, the normal stress components on the sample coordinate system axis are discussed [Mercelis and Kruth, 2006; Leuders et al., 2012; Shiomi et al., 2004].

The results obtained in the present work are shown in Table 39. In the present work, the normal stress components acting on the x- and z- axis are 96 MPa and 239 MPa respectively. These values correspond well to that reported in the literature for SLM parts built with an alternating scan strategy [Mercelis and Kruth, 2006; Leuders et al., 2012].

**Table 39: Point stress tensor on the middle of the frontal plane of an as-built component**

| Stress tensor T due to the residual stresses on an as-built sample |
|------------------|----------------|----------------|
| 96               | 182            | -188           |
| 182              | -239           | 243            |
| -188             | 243            | 0              |

More interestingly from a design point of view is the principal stress tensor derived from Table 39 and summarised in Table 40. The principal stress tensor shows in fact the largest stress components that can be found from any rotation of the sample coordinate system.

It is evident that these internal stresses, adding up to the axial loading components resolved on the principal axis, affect the mechanical performance of the as-fabricated SLM parts and contribute to explain the difference in elongation at break presented in Table 38. Further analysis of the residual stress distribution in the samples would however be useful to gain a more accurate understanding of internal stresses that develop during SLM.
Table 40: Principal stress components and directions relative to the sample reference frame

<table>
<thead>
<tr>
<th>Principal Stress Component</th>
<th>Principal Stress [Mpa]</th>
<th>Angles between Principal Axis and Sample Coordinate System (X,Y,Z) [degree]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-499</td>
<td>x: 121  y: 54  z: 51</td>
</tr>
<tr>
<td>2</td>
<td>114</td>
<td>x: 40   y: 49  z: 88</td>
</tr>
<tr>
<td>3</td>
<td>242</td>
<td>x: 113  y: 60  z: 141</td>
</tr>
</tbody>
</table>

7.1.2 Anisotropy of SLM Ti-6Al-4V: Tensile Properties comparison for three different Build Orientation

Table 38 shows that the elastic modulus does not vary with the build orientation or in other words that the orientation of the prior β grains do not influence the elastic moduli of the specimens. It has been reported that the α crystal anisotropy has a marked effect of the elastic modulus of Ti-6Al-4V with strong crystallographic texture [Banerjee and Williams, 2013]. However, as discussed in Sections 6.2 and 6.4, all SLM Ti-6Al-4V samples have a weak α’ (or α) texture, thus the elastic moduli do not vary when the build orientation is changed.

The bars built perpendicular to the building direction (xz-orientation) display a yield stress and UTS that is comparable to the vertical oriented bars suggesting that all the orientation have a similar strength (Table 38).

Table 38 also shows that the elongation at failure of the flat oriented tensile bars is the lowest among the tested samples. It is noteworthy that the flat parts curled during the process due to thermal stresses that originate in SLM (Section 3.3.2). Thus it is likely that the samples built in the flat orientation had lower density than the samples built in the other orientations because of uneven powder deposition. In addition, in order to test not-curlcd samples, the flat tensile bars were machined to smaller straight specimens (Section 3.3.2). It is possible that the lower ductility of the flat oriented samples was also caused by a geometrical effect. Indeed the elongation at failure is inversely proportional to $\frac{L}{\sqrt{A}}$, where L and A are the initial gauge length and cross-
sectional area respectively (ASTM E8/E8M), and the flat bars had the smallest cross-sectional area among the tested orientations. In order to establish the real effect of the microstructure on the fracture mechanism of tensile bars built in the different orientation and hence the difference in ductility, fractography for the tested samples was carried out.

**Figures 83** show low magnification SEM images of the fracture surfaces of stress relieved tensile bars built in different orientation. The fracture surfaces are generally rough and dimpled. The fracture surface profiles corresponding to the fracture of tensile bars built in different orientation are shown in **Figure 84**.

![Figures 83](image)

**Figure 83**: Examples of fracture surfaces after tensile test of the tensile bars built in the: a) vertical zx-orientation, b) flat xy-orientation and c) edge xz-orientation.

It was observed that for all the build orientations the fracture surface profiles manifest the typical features of overload failures [Brooks and Choudhury, 2002]. The fracture surface profiles consisted generally of a central portion that is relatively flat and perpendicular to the axial loading and external portions (shear lips) highly inclined (~ 45°) to the loading direction (**Figure 84**). In addition, the fracture surface profiles reveal that the predominant fracture is inter granular where cracks have propagated mainly along the grain boundaries present in the microstructure as shown in the insets of the same figure. This result is consistent with research on fatigue crack propagation of Ti alloys with fine microstructure, where it has been reported that crack
propagation is highly depended on the crystallographic orientation of the α grains encountered by the crack tip [Pilchak and Williams, 2011, Nalla et al., 2002].

Figure 84: Examples of fracture profiles of tensile bars built in different orientations: a) vertical zx-orientation, b) edge xz-orientation and c) flat xy-orientation. The insets show the predominant intergranular fracture along the both the α and prior β grain boundaries (marked by the black dot lines).
It has been shown that the crack tip deflects at the grain boundaries generating inter granular fracture when the neighbouring α grains (or colonies) have multiple distinct crystallographic orientations as typical of fine microstructures [Lütjering, 1998, Chesnutt et al., 1976]. As SLM Ti-6Al-4V has a weak texture, i.e. no α colonies (neighbouring α laths with identical orientation), but α laths that interface with high angle boundaries, inter granular fracture is therefore the most plausible predominant fracture mode.

From the analysis of Figure 84a-c, it was observed that the surface roughness of the central portion of the fracture profiles varies according to the build direction and it is generally higher in tensile bars built in the vertical orientation. These results derive from the fact that the vertical tensile bars fractured predominantly either along the α grain boundaries that can span through the width of an entire prior β grain and are generally inclined at ~±45° to the build direction (z-axis) or along prior β grain boundaries. The inset in Figure 84a shows a typical fracture along the α grain boundary encountered in the vertical oriented tensile bars.

Because the vertical built tensile bars have prior β grain boundaries nearly parallel to the building and loading direction, it is conceivable that fracture along these grain boundaries have generated tortuous crack paths and rougher fracture surface profiles compared to that observed on the other build orientations. The edge and flat bars, on the other hand, have prior β grain boundaries that are nearly perpendicular to axial loading, therefore the inter granular fracture along these boundaries implies profiles with lower roughness (insets in Figure 84). These results indicate that higher crack deflection might increase the strength of the vertical oriented bars.

The fracture surfaces were investigated at higher magnification to study the nature of the fracture in detail (Figure 85). The fracture surface in regions of porosity was smooth and with concentric features as shown in Figure 85 and as reported in the literature [Hrabe and Quinn, 2013]. As the fracture surface is generally rough it is difficult to establish whether or not fracture had initiated and propagated predominantly in regions of porosity.
Figure 85: Higher magnification micrographs of the fracture surfaces of the tensile bars: a) shows an example of extended region where terraces are dominant, b) shows the clear distinction between the topography of the terraces and regions of porosity (circular smooth areas indicated by the arrows). Micrograph c) shows several examples of step marks on the terraces. The step marks are indicated by black arrows.
It was observed that terrace-like features are present in all the fracture surfaces despite the build orientation of the tensile bars. An example of terrace-like features (also referred to as layered fracture [Qiu et al., 2013]) is shown in Figure 85a. Although these peculiar features are generally attributed to fracture in regions of porosity [Vilaro et al., 2011], Figure 85b shows clearly that the terraces have a richer topography than the areas where the powder bed was partially melted and porosity occurred. Indeed, it was observed that equiaxed shallow dimples appear on the surface of the terraces similar to the fracture surface morphology of other high strength metal alloys under tensile loading [Brooks and Choudhury, 2002]. Thus, it seems unlikely that the terraces indicated in Figure 85a are generated from the opening of existing porosity as reported in the literature [Vilaro et al., 2011]. Fracture of laser melted Ti-6Al-4V has been recently discussed and it has been speculated that features similar to that observed in this study might have originated from α cleavage [Qiu et al., 2013; Rafi et al., 2013]. These studies however do not take into account that the terrace size and aspect ratio are more consistent with the prior β grains of the SLM microstructure rather than the α grains that are an order of magnitude smaller (Section 6.1). In addition, cleavage fractures occur on defined crystallographic planes with no associated plastic deformation [Chesnutt et al., 1976]. It is clear instead that the terrace-like features of Figure 85b-c show dimpled fracture surfaces generated by local plastic deformation. The morphology of the terraces reveals no clear directional marks; therefore it is difficult to establish with certainty the crack propagation direction. It was observed however that, occasionally, steps can be found on the terrace fracture surfaces (Figure 85c). It is possible that the steps result as the fracture propagates around α laths of similar orientations that have generated from prior β grains with low angle misorientation [Brooks and Choudhury, 2002]. Indeed from the analysis of the α and β orientation maps in Section 6.2, there is evidence that low angle grain boundaries occur during the solidification of SLM Ti-6Al-4V and therefore it is possible that the crack tip deflected slightly, creating the observed steps, when fracture had occurred around similarly oriented α laths. To gain a better understanding on the nature of the terrace-like features of Figure 85a-c and verifying the intergranular propagation mechanism, EBSD was carried out on a plane parallel close to the fracture surface of one of the terrace features (Figure 86a-b). Figure 86c shows the IPF α orientation map corresponding to the selected feature and the corresponding reconstructed parent β phase. It was observed that the terrace is made of several α laths whose crystal orientation repeat is typical for the variants found within one prior β grain (Section 6.2). The analysis of the orientation of the α laths (Figure 86d) indicates the fracture has occurred in none of the basal or prismatic planes that are instead generally associated with α cleavage fracture [Pilchak et al., 2009; Pilchak and Williams, 2011]. In addition, the reconstruction of the parent β phase shows that all these α laths have originated...
from one unique grain. As evidenced in the fracture surface profiles, the predominant fracture in SLM Ti-6Al-4V is intergranular fracture thus it is believed that terrace-like features that appear on the fracture surfaces might have originated upon crack propagation through prior β grains predominantly along α grain boundaries. Finally, it is believed that if the predominant fracture mechanism was cleavage (i.e. fracture along either the basal or prismatic planes) the fracture surfaces should show extensive formations of elongated cylindrical dimples, generally referred to as flutes. However, studies on the tensile behaviour of SLM Ti-6Al-4V have never reported the existence of flutes of the fracture surfaces of the samples. Flutes form in order to join independent cracks that advance by α cleavage that cannot propagate either on the basal or the prismatic plane of two misoriented α grains [Chesnutt et al., 1977; Meyn and Bayles, 1987; Brooks and Choudhury, 2002; Pilchak, 2009]. In absence of α colonies that share the same orientation (as in the case of SLM Ti-6Al-4V), the propagation of cracks by cleavage would have to be sustained by void coalescence whenever cracks encounter grains whose orientation is not suitable for cleavage.

Figure 86: Micrograph a) shows the terrace that was investigated using dual beam FIB/FESEM lift out. A wedge portion of the terrace was lifted from the fracture surface and prepared for EBDS. Micrographs c) and d) show the corresponding [001] IPF orientation map of the α phase and the reconstructed parent β phase. Micrograph d) is a discrete IPF that shows the plane of fracture of the α laths in the terrace. The orientation of the α laths is reported in terms of Euler angles as described in Appendix A.

The early crack propagation during tensile overload was also investigated in one vertical zx-oriented tensile bar that was pulled until the onset of necking (the test was interrupted at a stress
level of about 1100 MPa and corresponding elongation of ~8%). Figure 87 shows several micro-voids observed in the microstructure of the plastically deformed sample. These voids might represent crack initiation points as they tend to coalesce and have asymmetrical morphology. Although it is not possible to establish with accuracy whether the micro-void had originated from a pre-existing pores or in correspondence of α lath fracture, Figure 87b suggest that the micro-void tend to propagate along the α grain boundaries since the early stage of fracture as observed in α+β Ti alloys with fine microstructure [Banerjee and Williams, 2013].

![Figure 87: a) and b) Optical micrographs showing voids in the microstructure of a plastically deformed sample. It can be noticed the rupture occurs fracturing the α grains and propagating along the α grains boundaries (black arrows).](image)

### 7.2 Fatigue Properties of Stress Relieved SLM Ti-6Al-4V

In order to gain some understanding of the high-cycle fatigue life of SLM Ti-6Al-4V five stress relieved bars built in the xz-orientation (i.e. edge orientation) were tested with a load ratio R=0.1 and maximum stress of 500 MPa. The fatigue life of the samples is listed in Table 41. The results are consistent for all the bars in the analysis and show a relatively low scatter indicating low variability in the microstructure of the samples. As the components were built using the optimised processing window reported in Section 3.3.1, reproducibility of the samples is expected. It is noteworthy that all the samples fracture at the end of the necking region as indicated in Figure 88. The design of the test bars used is indicated in Figure 31 (section 3.3.3). Considering the curvature of the neck and that no apparent macroscopic surface discontinuities...
were visible, it is reasonable to assume that stress concentration may have occurred in the neck region of the bars. The higher resolved stress in the neck region of the sample might have therefore decreased intrinsically the fatigue life of the sample tested in this study.

![Image](image.png)

*Figure 88: The macrograph indicates the fracture location on the bars failed after high-cycle fatigue*

Ignoring the stress concentration that might have concurred to a premature failure of the samples, *Figure 89* compares the fatigue resistance of SLM and traditionally manufactured Ti-6Al-4V. It can be noted that the SLM Ti-6Al-4V fatigue performance is comparable to that of HIPed cast Ti-6Al-4V but it is significantly lower than that of wrought and annealed alloys. The comparison of the present results with Ti-6Al-4V manufactured using similar additive processes is not trivial, as the mechanical performance of SLM Ti-6Al-4V varies significantly with the AM system, the build orientation and the applied post process treatments.

*Table 41: High cycle fatigue life of stress relieved SLM Ti-6Al-4V*

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Cycles to Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18832</td>
</tr>
<tr>
<td>2</td>
<td>31065</td>
</tr>
<tr>
<td>3</td>
<td>23234</td>
</tr>
<tr>
<td>4</td>
<td>22210</td>
</tr>
<tr>
<td>5</td>
<td>28534</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>24775</strong></td>
</tr>
<tr>
<td><strong>STD</strong></td>
<td><strong>4019.6</strong></td>
</tr>
</tbody>
</table>
Figure 89: Graphical comparison between the fatigue life of Ti-6Al-4V processed in a number of different routes and SLM (red dotted cross). It can be noticed that the fatigue resistance of the SLM Ti-6Al-4V is comparable to that of cast plus HIP alloys [Welsch et al., 1994].

Two independent works have reported a fatigue resistance of stress relieved and polished SLM Ti-6Al-4V at 500 MPa superior of $10^5$ cycles [Thöne et al. 2012; Gong et al., 2012]. The fatigue resistance of SLM Ti-6Al-4V reported in these studies is thus significantly higher than that observed in the present study. Unfortunately the microstructure of the samples is not provided in the studies and thus it is difficult to discuss the marked difference in fatigue performance. As mentioned above, the bars in this study failed at the end of their necking region thus it is possible that the resolved stress where the fracture commenced was higher than 500 MPa. This would then account for the reported difference in the fatigue life. Nevertheless, other studies report instead a poor fatigue resistance of the additively made Ti-6Al-4V samples [Edwards and Ramulu, 2013; Knowles et al., 2012; Van Hooreweder et al., 2012] were the fatigue limit at 500 MPa does not exceed $2 \times 10^4$ cycles. In order to gain more understanding on the fracture mechanism at the basis of the fatigue performance of SLM Ti-6Al-4V the fracture surfaces of the samples failed were investigated (Figure 90-92). The crack initiation point and the crack propagation area were studied carefully to establish the effect of the SLM Ti-6Al-4V microstructure in the resistance to high-cycle fatigue.
Figure 90: Micrographs that show examples of the typical features that occur on the fracture surface of high-cycle fatigue SLM Ti-6Al-4V samples. Micrograph a) shows the entire fracture surface. The black arrow indicates the fracture initiation point. The red dotted line shows the direction of crack propagation front. The topography of the fracture surface near the b) initiation point and c) propagating region are shown. The fracture propagates by d) striation growth (arrows) and e) cyclic ductile tearing.
Figure 91: Micrographs that show examples of the typical features that occur on the fracture surface high-cycle fatigue SLM Ti-6Al-4V. Micrograph a) shows the entire fracture surface. The black arrows indicate multiple crack initiation points. The red dotted lines show the directions of crack propagation. The topography of the fracture surface in the propagation region and final fracture are instead shown in micrographs b) and c) respectively.

Most of the crack initiation points in the bars are situated near the external surfaces of the test bars as shown in the Figures 90-92. This may be due to that fact that the laser beam was not characterised prior the building process, and thus the bars might have been processed at a non-ideal laser focus position. Improper melting of the offset region during SLM might have thus occurred causing the formation of porosity near the external surface of the samples. Similar results occurred during the validation of the process parameters optimisation method, as shown in Figure 56-57, Section 5.7. As materials with good high-cycle fatigue performance have good resistance to crack propagation [Pilchak, 2009; Chesnutt et al., 1976], it is evident that porosity has a significant detrimental effect on the fatigue performance of the SLM samples. In particular, it has been reported that the typical acicular porosity of SLM samples can cause stress concentration factors in the range of 5-12 [Santos et al., 2004; Van Hooreweder et al., 2012; Thöne, 2012].
Figure 92: Micrograph a) shows the entire fracture surface of another representative sample. The black arrow indicates the crack initiation point. The red dotted line shows the directions of crack propagation. The topography of the fracture surface near the initiation point and in the crack propagating region are instead shown in micrographs b) and c) respectively.

Given the fact that as-built SLM samples always possess residual pores, components in the as-built condition are not recommended for structural applications that are required to withstand fatigue loads. Therefore a post process HIPing treatment of the samples capable of closing the residual porosity would improve drastically the fatigue life of the SLM samples. The benefits associated with HIPing SLM Ti-6Al-4V are indeed reported in several independent studies [Edwards and Ramulu, 2013; Thöne et al., 2012; Facchini et al., 2012].

The crack propagation region presents a complex topography dominated by tear ridges, features resembling dimples, striations and planar terraces similar to those observed in the mechanically overloaded fracture surfaces. The lack of beachmarks, chevrons, river patterns indicates that the samples have generally fractured rapidly. When the crack propagation area of the fracture surfaces is examined in more detail, two areas with distinct topography are clearly displayed. This is illustrated in Figure 90c: the area near the origin of the crack initiation point displays fatigue striations and poor topography (Figure 90d). As the crack advances however, the fracture surface was dominated by tear ridges and features resembling a ductile fracture (Figure 90e). Although there is a lack of understanding on the exact role of the microstructure of AM Ti alloys during fatigue fracture and the associated fracture mechanisms, the fatigue behaviour of conventionally made Ti alloys has been extensively studied [Williams et al., 1968; Chesnutt et al.,
It is well understood for example that striations such as those shown in Figure 90d occur as a result in blunting of the crack tip during the loading cycle [Laird and Smith, 1966]. When the load is released, the elastic material around the crack tip plastic zone relaxes and results in a local compressive force on the crack tip and reversed plasticity in the plastic zone. This reversed plasticity is responsible for change of direction of the crack tip and thus the generation of the fatigue striations. In a recent study on the fatigue resistance characterisation of SLM Ti-6Al-4V, features with a similar topography were described as transgranular cleavage [Gong et al., 2012].

Ignoring the fact it has been demonstrated that the term “cleavage” is not correct to describe the facet formation in Ti alloys [Pilchak et al., 2009, Pilchak and Williams, 2011a], faceted growth is however generally associated with the formation of smooth features with no visible topography. It is evident that the features present in Figure 90d and also discussed in Gong, 2012, indicate that the fracture has occurred with a substantial amount of plasticity as shown by the rich topography displayed by the fracture surface. In addition, faceted growth seems unlikely to occur in a microstructure with a weak texture and high angle grain boundaries such as that of SLM Ti-6Al-4V.

Figure 90e shows that as the crack advances, fatigue striations diminish and areas with richer topography, extensive ductile tearing and features resembling micro dimples become dominant. It is believed that these features originate from cyclic ductile tearing [Williams et al., 1968; Chessnut 1976; Chessnut 1978]. Ductile tearing is a crack propagation mechanism characterised by tear ridges at the $\alpha$ grain boundaries and $\alpha/\beta$ interfaces and micro cavities that resemble shallow dimples [Chesnutt et al., 1976; Pilchak et al., 2008]. Ductile tearing has been reported in the fatigue fracture of welds of Ti alloys [Baeslack and Becker, 1979; Pilchak et al., 2008] and typically occurs when the grains in the microstructure are smaller than the crack tip plastic zone ahead of the crack tip [Williams et al., 1968]. The SLM components that were tested in this study possess a fine microstructure and thus it is plausible that after an initial crack propagation by fatigue striation, ductile tearing crack growth becomes the dominant crack propagation mechanism. Other examples of what it is believed to be a result of cyclic ductile tearing are shown in Figures 91b and 92c.

Despite the dominant crack propagation mechanism (fatigue striation or ductile tearing) vertical faces and planar features similar to those described in section 7.1 appear occasionally in the fracture surfaces (bottom right of Figure 90c and 91c.). It is believed that these features originate
from intergranular fracture along the prior \( \beta \) grain boundaries. It is generally accepted that the presence of the elongated grain boundaries contributes significantly to the premature failure of the Ti alloys in particular during high-cycle fatigue [Nalla et al., 2002]. As the samples in this research were built in the edge orientation the columnar grain boundaries are perpendicular to the external applied load, and thus represent weak points in the microstructure where crack can propagate with little resistance [Sercombe et al., 2008; Van Hooreweder et al., 2012]. A possible explanation for the fact that cracks do not propagate predominantly along the prior \( \beta \) grain boundaries but by striation or cycling ductile tearing might be that cracks tend arrest when deflected at grain boundaries meeting at triple points [Nalla et al., 2002]. Crack deflection retards the crack propagation in coarse microstructures especially in the latter stages of crack propagation [Lütjering and Williams, 2007].

Finally, the remaining portion of the fracture surface (referred to as final fracture area in the micrographs) is characterised by unstable crack growth and thus closely resembles the monotonic fracture process described in Section 7.1. Figure 91c shows that this area is characterised by extensive microvoid coalescence and the fracture topography varies between dimpled and planar depending on the spatial orientation of the \( \alpha \) grain boundary and probably the crystallographic orientation of the grains. As observed in Section 7.1, it is likely that the predominant fracture in this area is intergranular, either along the prior \( \beta \) grain boundaries or the \( \alpha \) grain boundaries.

### 7.3 Summary

In this Chapter the tensile properties and the high-cycle fatigue resistance of SLM Ti-6Al-4V built with a set of optimised process parameters are examined. Regardless of the build orientation, stress relieving alters the size and the phase composition of the parts and thus has a significant effect on the tensile properties of the samples. The stress relieved components exhibit superior ductility compared to the parts in the as-built condition. For this reason, a stress relief heat treatment is necessary for SLM components intended for structural applications.

It was found that the tensile properties of SLM Ti-6Al-4V are strongly dependent on the build orientation of the parts. In particular, the edge \( xz \)-oriented bars showed the best tensile properties and the greatest elongation at fracture. The results presented in this study indicate that the direction of the prior \( \beta \) grain boundaries affects the fracture mechanisms and the crack propagation in the parts.
The fracture surface profiles of the mechanically overloaded samples differ with the build orientation as well because of the orientation of prior β grain boundaries to the external axial loading direction. The lack of microtexture in SLM Ti-6Al-4V and the absence of α colonies explain why the dominant fracture mode is inter granular α or along the prior β grain boundaries.

Despite the fact that SLM Ti-6Al-4V presents a fine microstructure that could retard the propagation of the small cracks, and thus offer great resistance to high-cycle fatigue, porosity and the elongated prior β grain boundaries decrease substantially the fatigue life of the components. Results show that porosity is the most critical microstructural defect that decreases the fatigue life of the components. Pores act as small cracks and are thus detrimental to the fatigue life of the materials. It is believed that cracks propagate either by fatigue striation or ductile tearing mechanisms, while facet growth is practically absent due to the lack of a dominant texture in the microstructure. Cracks seem also to propagate occasionally on the prior β grain boundaries that therefore represent a weakness points in the microstructure.

It has been shown in the literature that HIPing the microstructure could close all the porosity in the sample and thus increase drastically the high-cycle fatigue resistance of the samples. It can also be speculated that heat treating the as-built samples above the β phase field followed by air or water quenching would break the columnar morphology of the prior β grains and thus create a microstructure more suitable to fatigue resistance.
8 Microstructural Control

This Chapter discusses the microstructure of SLM Ti-6Al-4V built with alternative scan strategies to produce tailorable microstructure. The microstructure of the components built with the alternative scan strategies is described and compared to that of the samples fabricated with the optimised processing window.

Section 8.1 reports the microstructure of samples built using the AM250 Laser Melting system and a checkerboard scan strategy (Section 3.3.4.1). The microstructure of samples built using a double scan strategy (Section 3.3.4.2) is then studied in Section 8.2. Section 8.3 discusses the microstructure of the specimens built using a double scan strategy in the SLM50 system. All the processing parameters that were used to fabricate these components are found in Section 3.3.4.3. A summary and the discussion of the found results are given in Section 8.4.

8.1 Microstructure of Samples obtained with Optimised Checkerboard Scan Strategy (AM250)

The microstructure of the samples fabricated with an optimised checkerboard scan strategy is shown in Figure 93. The optical micrographs show that the components were nearly fully dense with occasional spherical pores. Similar to that described in Section 6.1, porosity does not form any pattern. It is therefore believed that pores arise in the specimens as a result of either pre-existing defects in the plasma atomised powders or uneven powder deposition during the layer preparation rather than a wrong choice of process parameters. The microstructure consists of acicular grains that have formed within elongated prior β grain boundaries. Given the high cooling rates that characterised SLM and the morphology of these grains, it is believed that the microstructure consists of α' martensitic phase [Facchini et al., 2009; Qiu et al., 2013, Thijs et al., 2012].
Figure 93: a) 3D stack of optical micrographs showing the microstructure of SLM Ti-6Al-4V obtained using an optimised checkerboard scan strategy. The microstructure on the frontal, lateral and horizontal planes is shown in the micrograph b), c) and d) respectively.

The general features of this microstructure thus closely resemble that of the as-built components processed described in section 6.1. A comparison between the two microstructures is given in Figure 94 and Table 42.

Figure 94 shows clearly that the average width of the prior β grains in the samples built with the checkerboard scan strategy is significantly larger than those fabricated using the meander scan strategy. The samples processed with the checkerboard scan strategy experienced an overall higher laser energy density input. In the first instance, the former samples were process with a lower nominal laser scan speed and hatch spacing (Sections 3.3.1 and 3.3.4.1). Moreover during the processing of the individual islands, adjacent tracks are scanned rapidly one after the other, as the island size was significantly smaller that than entire section of the cubes (0.5 mm and 10 mm respectively). It is plausible therefore that when using a checkerboard scan strategy, the melt pools remain above the β transus for a longer time than when the meander scan strategy is used. For this reason the prior β grain size in the samples processed with the checkerboard scan strategy increases [Donachie, 2000; Lütjering and Williams, 2007; Welsch et al., 1994].
Figure 94: Comparison of the optical microstructures obtained using a) checkerboard and b) meander scan strategy

Table 42: Comparison of the main microstructure features of the samples built with two different scan strategies

<table>
<thead>
<tr>
<th></th>
<th>Meander</th>
<th>Checkerboard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prior β grain width</td>
<td>103 ± 32 μm</td>
<td>383 ± 32 μm</td>
</tr>
<tr>
<td>Prior β grain length</td>
<td>hundreds of μm</td>
<td>order of mm</td>
</tr>
<tr>
<td>α lath thickness</td>
<td>0.5 ± 0.2 μm</td>
<td>0.9 ± 0.5 μm</td>
</tr>
</tbody>
</table>

The average thickness of the acicular α′ laths that precipitates from the prior β grains was also significantly different as shown in Figure 95a-b. The samples processed with the checkerboard scan strategy consist of thicker α′ laths (Table 42). It is well understood that the increment of the α′ laths size is related to the cooling rate that governs the solidification of Ti alloys [Donachie, 2000; Lütjering and Williams, 2007]. As the scan vector length is substantially decreased (and so the time that the laser needs to start the melting process of an adjacent track) each individual island was processed at a higher average temperature. Because of this, the cooling rate during solidification of the melt pools would be lower, producing therefore a coarser microstructure. No similar results are yet reported in the literature for Ti-6Al-4V made using laser powder-bed systems. The average size of the α′ laths is however similar to that reported in EBM of Ti-6Al-4V [Al-Bermani et al., 2010; Brandl et al., 2012; Hrabe and Quinn, 2013; Antonymsamy et al., 2013].

The distribution of the orientations in the samples is shown in Figure 95c-d. No α′ colonies are present in the microstructure, indicating that cooling rates similar to water quenching have occurred [Welsch et al., 1994]. Figure 96 shows the corresponding backscatter micrographs. It is clear that no β phase was present in the microstructure.
Figure 95: Image quality and IPF orientation maps of the samples built with the a) and c) checkerboard and b) and d) meander scan strategy.

Figure 96: Backscatter micrographs of the as-built samples built with a) checkerboard and b) meander scan strategies.
8.2 Microstructure of Samples obtained with Optimised Double Scan Strategy (AM250)

The microstructure of the samples fabricated with an optimised double scan strategy is shown in Figure 97. The optical micrographs show that the components were near fully dense. Only occasional spherical pores appear in the microstructure. The pores formed without a particular pattern or periodicity. Although the effect of the double scan strategy of SLM Ti-6Al-4V has not been reported in the literature, several studies on the SLM of different steel grades have shown that this strategy can improve the densification of the samples [Yasa et al., 2009; Li et al., 2010; Kruth et al., 2010]. The re-melting of the same layer can improve the surface finish of the layers and thus enable an even distribution of the powder layers as new material is deposited [Yasa, 2011].

Figure 97: a) 3D stack of optical micrographs showing the microstructure of SLM Ti-6Al-4V obtained using an optimised double scan strategy. The microstructure on the frontal, lateral and horizontal planes is shown in the micrograph b), c) and d) respectively.
The microstructure of the double scanned samples consists of acicular grains that have formed within elongated prior β grain boundaries. Considering the high cooling rates involved in SLM it is believed that the acicular grains are indeed α′ martensitic phase. As can be seen in Figure 97 the prior β grain boundaries are vertical in both the frontal and lateral planes, which is different from that reported in Section 6.1. Although a thermal modelling of the heat dissipation has not been carried out in this study, as discussed in related studies of SLM Ti-6Al-4V [Thijs et al., 2010] vertical β grains would originate when using a scan strategy where the laser changes direction by 90°. The microstructure obtained using the double and the meander scan strategy is shown in Figure 98. It can be noticed that the size of the prior β grains was similar in the two microstructures. This indicates that the size of the melt pool and the thermal history experienced by Ti-6Al-4V do not change significantly for both the meander or the double scan strategies.

![Figure 98: Comparison of the optical microstructures obtained using a) double and b) meander scan strategy](image)

On the other hand, the thickness of the α′ laths in the samples fabricated with the double scan strategy is larger than that measured in the samples built with the meander scan strategy (Figure 99a-b and Table 43). This indicates that when using the double scan strategy there was a decrease in the cooling rate during the β → α′ phase transformation. This might be associated with the re-melting of the layer. Lower cooling rates during the re-melting of the layers might derive from the fact that the temperature of the layer at the beginning of re-melting was higher than that of the first scan (~70°C). Given the geometry of the sample and the process parameters described in Section 3.3.4.2 and considering that no re-coating takes place between the first melting and the re-melting, it can be calculated that the time elapsed between the two scans was approximately 4s. It could be that this time was not long enough for the material to cool down to...
the building platform temperature (70°C). Further studies to determine the cooling rates of SLM Ti-6Al-4V in relation to the laser process parameters are required. Similar considerations have recently been made in studies on the heat transfer models associated with a number of AM technologies [Roberts et al., 2009; Al-Bermani et al., 2010; Costa et al., 2011] where it is shown that the temperature in the deposited build rise as new layers are added.

Figure 99: Image quality and IPF orientation maps of the samples built with the a) and c) double and b) and d) meander scan strategy.

Table 43: Comparison of the α' grain thickness in the samples built with the two different scan strategies

<table>
<thead>
<tr>
<th></th>
<th>Meander</th>
<th>Double Scan</th>
</tr>
</thead>
<tbody>
<tr>
<td>α lath thickness</td>
<td>0.5 ± 0.2 μm</td>
<td>0.8 ± 0.3 μm</td>
</tr>
</tbody>
</table>
The cooling rate during the re-melting remains however significantly higher as shown by the microstructure which still exhibits fine acicular grains and no colonies of α’ grains sharing identical orientations (Figure 99c-d).

The backscatter analysis also reveals that no β phase is retained during the solidification (Figure 100) using the double scan strategy.

![Image](image.png)

*Figure 100: Backscatter micrographs describing the phase composition of the as-built samples built with a) double and b) meander scan strategies.*

### 8.3 Microstructure of Samples obtained with Optimised Double Scan Strategy (SLM50)

The microstructure of the samples fabricated with a radical new set of parameters (as described in Section 3.3.4.3) in the three orthogonal planes is shown in Figure 101. The general features of this microstructure resemble closely to that described in Section 6.1. The optical micrographs reveal that the microstructure was predominantly formed by prior β columnar grain boundaries where α phase has precipitated. The prior β grain boundaries are thus mainly vertical in the frontal and lateral plane (Figure 101a-b) while they form irregular shapes in the horizontal plane. However, the α microstructure appears more complex and not easy to distinguish at this level of magnification. The montage in Figure 102 shows the several pores are present in the microstructure. Although the density measured in this samples is greater than 99%, it is evident...
that the samples fabricated with this scan strategy display a lower quality than that reported for the other optimised conditions.

![Image](image_url)

**Figure 101**: a) 3D stack of optical micrographs showing the microstructure of SLM Ti-6Al-4V obtained using an optimised double scan strategy. Micrographs b), c) and d) show the microstructure on the frontal, lateral and horizontal planes respectively.

The origin of porosity during SLM has been extensively discussed in the literature [Kobryn et al., 2000; Das et al., 2003; Vilaro et al., 2011; Thijs et al., 2010; Gu et al., 2012; Qiu et al., 2013; Attar et al., 2013]. As discussed in the literature, it is believed that flat pores originate from an incomplete melting of the layers [Kobryn et al., 2000; Das et al., 2003]. The chosen laser processing parameters allow the melting of solid Ti-6Al-4V for several tens of μm, i.e. provide enough energy for the melting of the individual layers of powder, however, it can be noticed that the layers display a high surface roughness (Figure 103). It has been demonstrated that gradients of the surface tension in the molten material can cause metal to flow and potentially agglomerate when excessive energy is applied to the powder bed [Gu and Shen, 2009; Yadroitsev et al., 2010]. Given the rippled surface of the substrate material, it is thus plausible that uneven layers of powder were in turn deposited. As a result, inhomogeneous melting might have occurred creating the elongated interlayer porosity observed in the microstructure. On the other hand, the spherical porosity (also present in the microstructure of Figure 102) is caused by either defects in the fresh
powder particles or by the evolution of gaseous species adsorbed in the starting material [Qiu et al., 2013; Thijs et al., 2010]. The optical analysis of the samples also shows macroscopic “banding” in the frontal and lateral planes (Figure 101-102).

Figure 102: Microstructure of the xz-plane (frontal plane). Interlayer porosity is evident in the microstructure. Macroscopic banding, indicated as sharp contrast change in the image, is also present in the microstructure.

Figure 103: Microstructure of the xz-plane (frontal plane) indicating the surface roughness of the samples. The micrograph shows also extensive macroscopic banding as indicated by the arrows.

The microstructure was studied in more detail at higher magnification using backscatter imaging. The backscatter analysis on the frontal plane of the samples shows that the average grain size and morphology vary along the building direction (Figure 104a). The microstructure of the first few deposited layers consisted of relatively coarse fully lamellar α+β microstructure (Figure 104b). The average length and width of the α laths was 9.6 ± 3.1μm and 2.3±0.5μm respectively. A slight increase in the α lath size was observed along the y-axis, where regions that were scanned last. The fully lamellar microstructure of these layers originates from columnar β grains whose vertical grain boundaries are visible in the microstructure.
As more layers are deposited however, it was observed that several regions of the sample showed a near fully equiaxed microstructure with some retained β phase at the α grain boundaries (Figure 104c). The α grain size was about 3.0±0.6μm. It was also noted that the size of the equiaxed α grains coarsen in correspondence to the regions close to the last scan along the y-axis, similarly to that observed in the fully lamellar region. The orientation measurement shows that there was preferential solidification grain growth [001] direction in the β phase indicating that the orientation of the columnar grains that have formed during the initial solidification has been altered during the SLM process.

As the height along the building direction increases, it was observed that the volume fraction of equiaxed α grains diminishes at the expense of α grain with the lamellar morphology (Figure 104d). The microstructure of these regions resembles the standard bimodal microstructure of Ti alloy, where recrystallised grains constitute the primary α (α_p) while the remaining α laths can be considered secondary α (α_S). The change of the dominant α grain morphology was accompanied by a change in the average α grain size that, as new layers were added, becomes finer (Figure 104e).

Eventually, martensitic acicular α’ microstructure was observed in the regions corresponding to the last processed layer (Figure 104f). It was noted that the bimodal microstructure (as well as the martensitic microstructure of the last layer) originated from columnar prior β grains that have grown predominantly aligned to building direction. The fully martensitic microstructure of the last layer indicates that after each layer deposition the material cools down to room temperature at such a high rate that the diffusion control β → α is suppressed [Welsch et al., 1994; Donachie, 2000; Lütjering and Williams, 2007]. This indicates that despite the relatively low scan speed and the heating of the build platform to 200°C, cooling rates higher than 410°C s⁻¹ have occurred during solidification of the melt pool, as proved by the resultant α’ martensitic microstructure typical microstructure of SLM Ti-6Al-4V deposits (Figure 104f) [Facchini et al., 2009; Thijs et al., 2010; Vilaro et al., 2011].
Figure 104: a) Backscatter micrograph showing the evolution of the microstructure along the building direction; starting from the bottom of the sample, the microstructure is composed of α+β phases arranged with b) fully lamellar, c) fully equiaxed, d) and e) bimodal morphologies; f) the last deposited layer is instead fully martensitic.

The microstructure analysis presented in this study suggests that as new layers are added, the underlying Ti-6Al-4V workpiece experienced high temperatures that have annealed the microstructure. Finite element models of the heat transfer during metal AM have shown that the thermal cycles caused by the addition of successive layers has a significant effect on the microstructure of Ti-6Al-4V [Roberts et al., 2009; Crespo and Vilar, 2010; Fachinotti et al., 2012]. At first, the thermal cycles experienced by the layers induce epitaxial columnar growth of the prior β grains [Al-Bermani et al., 2010; Simonelli et al., 2014]. In addition, it has been shown that reheating of previously deposited layers can cause decomposition of the α’ into α+β phases if the high annealing temperature is held sufficiently long [Crespo and Vilar, 2010]. The low scan speed caused gradual heating of the workpiece that allowed diffusional solid phase transformation to take place. At the bottom of the workpiece, cyclic heating has caused the martensitic α’ phase to transform into lamellar α+β microstructure (Figure 104b). Similar results have been presented for powder-blown laser beam deposited Ti-6Al-4V, where the laser scan
speed has been shown to influence the average temperature distribution in the workpiece and the cooling rates associated with the melt pool solidification [Crespo and Vilar, 2010]. As the lasers advanced during the scan of the single layer it is possible that the whole workpiece temperature had increased. Thus it is likely that regions that were scanned last experienced (along the x- and y-direction) lower cooling rates than those scanned at the beginning of each layer as indicated by the corresponding coarsening of the microstructure described above. This is shown schematically in Figure 105.

![Figure 105](image)

*Figure 105: (a) and (b) are the schematic diagrams that show the gradual temperature increase as the laser scans the same layer with the two different scan vector directions.*

As more layers were added, areas with fully equiaxed microstructure were observed (Figure 104c). This microstructure suggests that recrystallization of α laths has taken place in these areas. The origin of the driving force that has led to α grain recrystallization might be related to the thermal stresses developed during SLM due to the typical high cooling rates involved in the layer solidification [Santos et al., 2004; Shiomi et al., 2004; Mercelis and Kruth, 2006]. As the first few layers are in contact with lattice supporting structures that have minimal cross sectional area, it is reasonable to surmise that thermal stresses at the bottom of the build did not reach high values to cause crystal deformation. Hence, the microstructure at the bottom of the build is fully lamellar (Figure 104b). With the addition of more layers however, it is likely that residual stresses increased considerably and caused crystal deformation [Gibson et al., 2011]. During reheating cycles, recrystallization of the lamellar α+β microstructure might have occurred in these areas, generating the equiaxed microstructure (Figure 104c). Indeed there is some evidence in the literature that the reheating cycles during other beam deposition systems can cause the formation of α equiaxed grains in Ti-6Al-4V substrate [Kobryn and Semiatin, 2001; Kobryn and Semiatin, 2004]. In the present study, apart from the first few layers, α equiaxed grains have
occurred throughout the majority of the sample microstructure and not just appear in periodic locations as reported in the literature [Kobryn and Semiatin, 2001; Kobryn and Semiatin, 2004]. The volume fraction of α laths that underwent recrystallization decreases as the build height increases probably because far from the building platform the workpiece temperature is generally lower, thus leading to higher cooling rates during the cyclic heating [Crespo and Vilar, 2010].

In order to validate this proposed mechanism, microhardness and EDS analysis were also carried out. The hardness measured in correspondence to the observed microstructural changes is reported in Figure 106. Table 41 lists the average and standard deviation of the measured hardness values. The microhardness measured on the reference rolled material is also included (Section 3.1.1).

![Figure 106: Variation of the microhardness with the distance from the building platform. The highest value of the microhardness was measured in the fully equiaxed region of the specimens.](image)

The microhardness of the lamellar α+β microstructure is the lowest in sample (Table 44). This microhardness is higher than that measured on the reference material but it is comparable to that of solution treated and aged Ti-6Al-4V [Welsch et al., 1994]. Studying the measured composition of the α and the β phases of the lamellar microstructure it is noted that the β phase is rich in V as typically occurs in α+β Ti alloys (Table 45).
Table 44: Microhardness variation along the building direction and microhardness of the reference material

<table>
<thead>
<tr>
<th>Distance from the platform [mm]</th>
<th>Microhardness (Hv)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>508±15</td>
</tr>
<tr>
<td>4.5</td>
<td>525±17</td>
</tr>
<tr>
<td>3.5</td>
<td>534±14</td>
</tr>
<tr>
<td>3</td>
<td>555±21</td>
</tr>
<tr>
<td>2.5</td>
<td>600±23</td>
</tr>
<tr>
<td>1.5</td>
<td>615±25</td>
</tr>
<tr>
<td>1</td>
<td>635±32</td>
</tr>
<tr>
<td>0.5</td>
<td>410±15</td>
</tr>
</tbody>
</table>

*Reference material (Section 3.1.1)* 349±15

Table 45: Area and chemical grain composition of the lamellar α+β microstructure (EDS data)

<table>
<thead>
<tr>
<th>Area</th>
<th>α phase</th>
<th>β phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>90.7 ± 0.1</td>
<td>91.7 ± 0.1</td>
</tr>
<tr>
<td>Al</td>
<td>5.3 ± 0.1</td>
<td>5.8 ± 0.1</td>
</tr>
<tr>
<td>V</td>
<td>4.0 ± 0.1</td>
<td>2.5 ± 0.4</td>
</tr>
</tbody>
</table>

The graph in Figure 106 shows that the hardness reaches the maximum value in correspondence to the fully equiaxed microstructure (Figure 104c) and decreases with the build height to an average value of 508 in the martensitic region.

The hardness value of the martensitic microstructure was high compared to reference Ti-6Al-4V. It is well known that the martensitic phase is harder than that of the equilibrium phases of Ti-6Al-4V [Welsch et al., 1994; Donachie, 2000]. Moreover the average hardness value reported here is in line with that reported in other studies on SLM Ti-6Al-4V [Thijs et al., 2010; Murr et al., 2009]. The EDS analysis shown in Table 46 reveals that the α′ phase is rich in V [Ahmed and Rack, 1998; Donachie, 2000; Qazi et al., 2001; Lütjering and Williams, 2007].

The cyclic re-heating experienced by the layer below the martensitic region that causes the change in microstructure depicted in Figure 107 was also responsible for a progressive hardening
of the microstructure. It is believed that this microhardness change can be explained in terms of the solid-state diffusion of Al and V that takes place during the cycling re-heating/annealing.

The EDS data from the martensitic and the fine bimodal region are compared in Table 46. It can be noticed that during $\alpha' \rightarrow \alpha + \beta$ decomposition, significant solid-state diffusion of Al and V takes place. In particular, it can be noticed that the $\alpha$ phase possess less Al and V than the parent $\alpha'$ martensitic phase whereas the $\beta$ phase enriches in both the elements (Table 46).

![Figure 107: Backscatter micrograph that shows the change in microstructure and microhardness from a) the top deposited martensitic layer to b) fine $\alpha + \beta$ microstructure a few layers below.](image)

**Table 46: Area and grain chemical composition of the microstructures shown in Figure 107 (EDS data)**

<table>
<thead>
<tr>
<th>Area</th>
<th>$\alpha'$ phase</th>
<th>Area</th>
<th>$\alpha$ phase</th>
<th>$\beta$ phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>87.4 ± 0.4</td>
<td>87.3 ± 0.7</td>
<td>90.0 ± 0.1</td>
<td>92.0 ± 0.7</td>
</tr>
<tr>
<td>Al</td>
<td>8.8 ± 0.2</td>
<td>8.7 ± 0.1</td>
<td>5.4 ± 0.1</td>
<td>6.0 ± 0.4</td>
</tr>
<tr>
<td>V</td>
<td>3.8 ± 0.3</td>
<td>4.0 ± 0.7</td>
<td>4.6 ± 0.1</td>
<td>2.0 ± 0.2</td>
</tr>
</tbody>
</table>

A similar trend appears during the change from coarse bimodal to the fully equiaxed microstructure region shown in Figure 108. Table 46 and 47 report the composition of the phases that appear in the microstructure. It can be noticed that the equiaxed $\alpha$ phase is characterised by a lower content of Al and V than the $\alpha$ phase in the lamellar morphology that is, in turn, lower than that measured in the $\alpha'$ martensitic phase.

From Table 46 and 47 it can also be seen that during cyclic reheating the $\beta$ phase progressively enriches in V and Al. This result is not surprising considering the fact the BCC systems can accommodate more solute than the HCP systems [Callister and Rethwisch, 2007].
**Figure 108:** Backscatter micrograph that shows the change in microstructure and microhardness from a) the coarse bimodal to b) fully α+β equiaxed microstructure.

**Table 47:** Area and grain chemical composition of the microstructures shown in Figure 108 (EDS data)

<table>
<thead>
<tr>
<th></th>
<th>Area</th>
<th>α phase</th>
<th>β phase</th>
<th>α phase</th>
<th>α phase</th>
<th>β phase</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>equiaxed</td>
<td>equiaxed</td>
<td>equiaxed</td>
</tr>
<tr>
<td>Ti</td>
<td>90.0</td>
<td>92.0±0.7</td>
<td>87.0±0.9</td>
<td>93.5±0.7</td>
<td>Ti</td>
<td>93.5±0.5</td>
</tr>
<tr>
<td></td>
<td>92.0</td>
<td>92.0±0.7</td>
<td>87.0±0.9</td>
<td>93.5±0.7</td>
<td>(b)</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>5.4</td>
<td>6.0±0.4</td>
<td>5.1±0.5</td>
<td>4.4±0.4</td>
<td>Al</td>
<td>4.5±0.4</td>
</tr>
<tr>
<td></td>
<td>5.4</td>
<td>5.4±0.4</td>
<td>5.1±0.5</td>
<td>4.4±0.4</td>
<td></td>
<td>7.4±0.2</td>
</tr>
<tr>
<td>V</td>
<td>4.6</td>
<td>2.0±0.2</td>
<td>7.9±0.2</td>
<td>2.1±0.2</td>
<td>V</td>
<td>2.0±0.3</td>
</tr>
<tr>
<td></td>
<td>4.6</td>
<td>2.0±0.2</td>
<td>7.9±0.2</td>
<td>2.1±0.2</td>
<td></td>
<td>17.1±0.5</td>
</tr>
</tbody>
</table>

As mentioned above, the samples also show macroscopic “banding” in the frontal and lateral planes of microstructure (Figure 102-103). The dark bands appear in the microstructure with layer periodicity and possess a wavy shape almost parallel to the x-axis (or y-axis if observed in the lateral plane). Although it is generally accepted that banding is caused by the cyclic reheating of previously deposited material during each subsequent deposition, some doubts remain regarding to the exact formation mechanisms of the banding [Kobryn and Semiatin, 2000; Kobryn and Semiatin, 2001; Kelly and Kampe, 2004; Thijs et al., 2010; Baufeld et al., 2011]. **Figure 109** shows the microstructure change in correspondence to one of the observed bands. Two main differences have been observed between the band and the lower portion of material (i.e. outside the band). There is sudden change of predominant grain alignment as indicated by the red marks on the micrograph. In addition, it can be observed that the microstructure in the band present an higher volume fraction of dark grains than the lower portion of the micrograph. It is not trivial to determine whether the average size of the α phase is altered in band region.
Although the reason behind these microstructural changes is not clear, it is noteworthy that macroscopic banding is typically correlated with the use of high energy densities [Kobryn et al., 2000; Kobryn and Semiatin, 2001; Kelly and Kampe, 2004; Thijs et al., 2010]. Under the processing used in this experiment it is possible that reheating cycles cause the formation of heat affected zone that experience temperatures above the β transus. Upon cooling from the β phase, different α variants from those precipitated in the first deposition might form explaining the microstructural change observed in Figure 109b. As new layers are added, the substrate farther away from the melt pool, would instead experience temperatures in the α+β phase field. As the EDS analysis proves that solid-state diffusion of the alloying elements occurs among α phases, it is believed that the dark grains imaged in the bands are, in fact, those grains depleted in Al and V.

![Figure 109: Backscatter micrograph showing the change of the sudden change in orientation of the α grain in correspondence of the observed “banding”](image)

EBSD was finally conducted on the same locations indicated in Figure 104. Figure 110 shows the corresponding phase maps and inverse pole figures (IPFs) orientation maps. Figure 110a shows the phase map relative to the coarse lamellar α+ β microstructure of the component near the bottom region. The microstructure comprises about 5% of β phase. The remaining α phase is arranged in α variants related to the retained β phase through the Burgers orientation relationship [Simonelli et al., 2014]. The phase map of the fully α equiaxed microstructure is shown in Figure 110b. The β phase constitutes about 5% of the microstructure, similarly to that observed at the bottom of the component. The corresponding IPF orientation map shows that several α equiaxed grains have similar crystallographic orientations. This suggests that these grains might have recrystallised from small α colonies. As the sample height increases, a gradual refinement of the
microstructure was noted as shown in Figure 110c-e. The analysis of the phase maps indicate a decrease of the volume fraction of the β phase, e.g. only about the 4% of β phase was observed in Figure 110d. Smaller α colonies are present in the microstructure as a result of higher cooling rates. Similarly to that observed in a related study [Simonelli et al., 2014], the top martensitic layer shows a more equal distribution of the 12 variants, as indicated in IPF orientation map of Figure 110e.

![Figure 110: Phase maps, IPF orientation maps and corresponding colour schemes showing the microstructure evolution along the building direction.](image)

### 8.4 Summary

In this Chapter the microstructure of SLM Ti-6Al-4V built with alternative scan strategies was investigated. The evidence presented in this Chapter suggests that there are various processing windows under which it is possible to fabricate near fully dense SLM Ti-6Al-4V.

The checkerboard scan strategy used in this study, with a island size of 0.5 mm, produces a microstructure with bigger prior β grains and slightly coarser α′ martensitic phase when compared to that obtained using the optimised parameters. It is believed that because of the reduced scan vector length each islands solidifies less rapidly than in the case of the samples processed with the meander scan strategy. The cooling rates are however still high enough to suppress the diffusional $\beta \rightarrow \alpha + \beta$ solid-state transformation and for this reason the samples are entirely
martensitic with no retained β phase. Interestingly, this scan strategy coupled with a heated building platform could produce components with equilibrium microstructures. As thermal stresses during SLM are correlated to the cooling rates of the melt pool, it is reasonable to think that this scan strategy could be beneficial to minimise the thermal stresses in the parts.

A second batch of samples fabricated with a double scan strategy was then examined. It was found that this scan strategy produces samples with a microstructure that resemble closely that of the samples fabricated with the optimised meander scan strategy. The only difference between the two microstructures is in the thickness of the α’ laths. Because of the very little time lapse between the recoating of the layers, the last layer prior the re-melting scan is at a higher temperature than that of the deposited powder bed. For this reason, the solidification of the layer after the re-melting would occur under lower cooling rates and would produce thicker α’ laths than that measured in the samples made with the meander scan strategy. It was also observed that the direction of growth of the columnar prior β grains is vertical, i.e. parallel to the building direction, due to the 90° alternating laser scan direction.

Finally samples built with a new radical process parameters were characterised. These samples were produced using a double scan strategy similar to that described above. However the laser power and scan speed were much lower than those typically used in SLM Ti-6Al-4V. Although the density of the samples is not as high as that measured in the other investigated conditions, the microstructure of the samples is closer to that of conventionally made Ti-6Al-4V. Further work is required to optimise the density of the samples.

Apart from the last layer, it was found that the components produced under this processing condition have a predominant α+β bimodal microstructure. It is believed that the cyclic heating of the layers and the intrinsic deformation caused by the thermal stresses can cause solid-state diffusion of the alloying elements and recrystallization. Future work will be required to investigate the effect of combined long and short laser/material interaction times in order to maintain α+β equilibrium microstructure and improve the density of the as-fabricated samples.
9 Conclusions

In this study the microstructure evolution and the mechanical properties of SLM Ti-6Al-4V have been discussed and compared with the literature. In order to comprehend the evolution of the microstructure, from powder to final products, the starting microstructure of the plasma atomised particles was initially studied. It was found that the particles have a lamellar microstructure that consists exclusively of α phase. The examined particles showed to be fully dense with considerable uniform particle size distribution and thus ideal for the process.

As the objective of the work was to produce quality and repeatable components, a novel methodology to identify the processing windows to fabricate near fully dense components was developed. In particular, the melting and the solidification of the solid Ti-6Al-4V upon laser scan (an approximation of the SLM of powder bed) was studied in detail. It was found that several laser parameters concur to the formation of stable melt pools, scan tracks and homogeneous melt areas and in turn to the densification mechanism during SLM. Four different processing windows capable of producing near fully dense Ti-6Al-4V have been identified. It is evident that studying the melting and solidification on a single layer of bulk material to identify the best production parameters (instead of building parts using a trial and error approach) presents significant advantages both in time and cost and material consumption.

The microstructure of SLM Ti-6Al-4V built using the found four sets of process parameters was then investigated. Extensive EBSD analysis was carried out on the samples that were built using the set of processing parameters that could give the best density and the highest production rate. It was found that the layers solidify in the β phase field and precipitate as α′ martensitic phase. It was observed that what appears to be a random microstructure is, in fact, a microstructure that forms following precise crystallographic rules. The results of this research support the idea that variant selection was occurs during the cyclic heating of the layers. Although the exact principle that drives these phenomena remains unclear, the theories advanced in the literature were compared and discussed. The findings of this study suggest that EBSD can provide useful insights on microstructural formation of metal AM. To date, limited research has however focussed on these aspects. As it is well established that Ti alloys retain the features of the original
microstructure even after several heat treatments, the detailed comprehension of the as-built components appears fundamental to take SLM forward.

The tensile and fatigue properties of samples built using the same set of parameters were then evaluated. In terms of tensile properties, it was found that the tensile properties are sensitive to the build orientation. It was found that dominant fracture mode is inter granular. In turn, the orientation of the prior β columnar grain boundaries relative to the external axial loading has a significant influence in the tensile properties of the SLM components. It was shown that stress relieved samples had improved ductility. The reason for this improvement is twofold. In the first place the recrystallization of the microstructure during the stress relieved allows to the elimination of the residual stresses that form during SLM. Secondly, the heat treatment of the alloys in the α + β phase field (but near to the β transus temperature) causes an transformation of the martensitic microstructure into the equilibrium phases. It is well documented that this microstructural change contributes to the ductility of the alloys. In terms of fatigue properties, it was found that the residual porosity in the as-built samples limits the high-cycle fatigue performance of SLM Ti-6Al-4V considerably. It is likely therefore that SLM Ti-6Al-4V parts intended for structural application require post-treatment capable of closing porosity (such as HIPing).

Finally the microstructure of sample built using alternative scan strategy was evaluated. It was found that the scan vector length (i.e. using a checkerboard scan strategy) has a significant effect on the microstructural formation during SLM. Applying the checkerboard with a reduced island size can reduce the cooling rates and thus presumably produce parts with lower residual stresses. These findings represent a promising result as it can be envisaged that SLM Ti-6Al-4V with equilibrium microstructure could be produced combining this scan strategy with a heated platform. On the other hand, it was found that the re-melting of each deposited layer (double scan strategy) has a minor influence on the microstructure of the sample (i.e. it produces a slight coarsening of the grains). It is evident however that re-melting each layer decreases the build rate considerably and causes a significant increase in energy consumption. As no significant improvement in density was observed, it is believed that the double scan strategy is not beneficial to SLM of Ti alloys. Finally, the microstructure of the samples produced with low laser powder and scan speed was investigated. These samples have shown significant differences in the microstructure that is typically associated with SLM of Ti-6Al-4V. In particular it was found that the as-built components consist of a mixture of α+β phase with varying morphology as the build height increases. Although the density of these samples is not as high as that measured in the
samples scanned with other scan strategies, the findings of this research work indicate first the first time that it is possible to control the microstructure during SLM.

The research work presented in this manuscript has some limitations that are worth to be reported. In the first place it was demonstrated that the laser used in the AM250 Laser Melting system was not stable. In other words, the laser output varies in time and as well as in the focus position. In order to overcome this problem the laser beam was characterised periodically and adjusted accordingly. However, due to the unpractical and time consuming nature of this job, it was not possible to characterise the laser beam prior to each building. Therefore, although the methodology gives an adequate description of the processing conditions, the inhomogeneity of the quality found in the parts might have been caused by the variations of the laser beam output at the time of the build. A tangible example of this occurrence is represented by the porosity observed near the external surface of the fatigue samples.

Only arbitrary samples of powders were investigate in this research work. Similarly the powders used to build the samples were simply sieved through successive finer meshes to ensure that powders were smaller than 70 μm. However no systematic routine was established to make sure that the powders used were adequate in terms of shape, composition, gaseous species adsorbed onto their surfaces.

In this research work porosity was evaluated from optical micrographs of part cross sections. Although this analysis gives a good idea of the quality of parts, no quantitative information are provided regarding the whole volume of the components.

The microstructural study shown in this thesis is based on the analysis of relative small samples of cubic geometry. In reality, the main advantage of producing parts with AM derives from the fact that these processes enable design freedom and thus the fabrication of complex shape with added functionality. Although the results obtained in this research are valid for any SLM Ti-6Al-4V part, the effect of the geometry on the microstructure evolution of the samples was not studied. In addition, it was shown that the laser scan vector length has a significant effect on the melting and solidification of Ti-6Al-4V. In order to process parts with maximum density it might thus be beneficial to use process parameters that change slightly in relation to the size of the cross section to be scanned.

The effect of the gas flow, laser spatter and material evaporation was not included in the discussion of the microstructural formation during SLM. It is however believed that gas flow is an important process variable to consistent repeatable result. Similarly, using alternative
processing windows can lead to the formation of laser spatter and preferential material evaporation, that is turn are responsible for porosity and chemical changes in the deposits.

A thermal model to study the heat loss during SLM would have been useful to gain further insights in the microstructure evolution of SLM Ti-6Al-4V. The experiments on the melting and solidification of solid Ti-6Al-4V carried out in this study represent an approximation of the melting of layer of powder.
10 Future work

The present research has discussed the evolution of the microstructure of SLM Ti-6Al-4V and its mechanical properties. Due to the complex nature of the problem and the limited time, not all the issues that are related to the quality and the reproducibility of the SLM samples have been fully addressed. The main suggestions for future work are here reported.

Research has suggested that defects present in the initial powders and poor flow behaviour of the powders can affect the quality of the SLM samples [Engel and Bourell, 2000; Marcu et al., 2012]. In order to improve reproducibility, the adoption of a standard routine for powder treatment and recycling would be beneficial. For example, powders could be heat treated prior to SLM in order to improve the flowability and favour the degassing of contaminants potentially adsorbed on the powder surface.

A new methodology to fabricate low-cost titanium powders for AM has recently been developed [The Manufacturer, 2013]. Further experimental investigations to assess whether these powders can be used during SLM would be very interesting.

This research work has investigated the evolution of the microstructure of SLM Ti-6Al-4V. It was shown that variant selection and texture inheritance occur during SLM. Further research is however needed to clarify the reasons why specific variants formed predominantly within the prior β grains. These aspects could be clarified using numerical simulations that describe the elastic strain energy associated with the β → α′ phase transformation.

This research qualitatively investigated the relationship between the microstructure development and the SLM samples’ thermal history. A more quantitative strategy to investigate this relationship would be using a numerical model capable of predicting the temperature experienced by the layers in relation to all the laser process parameters, in particular the laser scan strategy and energy density. Firstly, being able to predict the direction of growth of the prior β grains under different processing conditions would allow further control on the microstructure and, to some extent, the mechanical behaviour of the SLM samples. A numerical thermal model of SLM
would also allow the estimation of the cooling rates associated with the solidification of each deposited layer for various processing conditions. Finding a processing window that can decrease the typical cooling rates of SLM would lead to a number of advantages. For example it would reduce the thermal stresses that occur during SLM and enable to manufacture samples with a more desirable microstructure.

In order to improve density and obtain an equilibrium microstructure future work to investigate the effects of combining long and short laser/material interaction times could be performed.

Further research on the effect of the size of the components on the microstructure development would be useful to verify whether the considerations made in this research are applicable to larger components.

As residual porosity was always found in the as-built components, future research on non-destructive evaluation of the quality of the SLM samples is therefore fundamental for the future of technology. In particular, it would be interesting to evaluate the probability of finding defects that could lead to premature failure. Thus using X-ray micro CT to detect pores of critical size as a tool for life prediction could be of great interest. Serial mechanical sectioning could also be considered as an alternative investigation tool to determine the real porosity in the samples.

This research work has investigated in detail the tensile properties of SLM Ti-6Al-4V. Further work to establish the fatigue life and crack growth resistance is however still needed. Studying how the non-conventional microstructure of SLM Ti-6Al-4V influences the initiation and growth of fatigue cracks would be extremely interesting.

Studies on the laser spatter and the potential preferential evaporation that might occur during SLM are practically absent in the literature. The laser spatter that forms from the melt pool can obstruct the optical path of the laser and thus cause degradation of the energy delivered to the powder bed and a general deterioration of the processing conditions. Preferential evaporation of the alloying elements could instead alter the composition of the final deposits.

As a final remark, it is worth mentioning that SLM systems have rapidly evolved in the last few years and now desktop machines featuring small lasers are available on the market. The advent of small lasers is actually redefining the limits of SLM. Due to the fact that small lasers can be collimated to smaller spot sizes these systems offer the possibility to manufacture parts with improved resolution. In addition these systems enable processing of materials with radically different process parameters and opening up the possibility of producing parts with more controlled microstructure. Little attention has been given to this aspect in the literature. It is thus
believed that this could potentially become a primary thrust of exploration for future research on SLM.
11References


Humbert, M., Wagner, F., Moustahfid, H. and Esling, C. “Determination of the Orientation of a Parent Grain from the Orientations of the Inherited Plates in the Phase Transformation from


Reeves, P. “Private communication” University of Nottingham, 2011-2013.


Wenk, H., Lonardelli, I. and Williams, D. “Texture changes in the $\alpha \rightarrow \beta \rightarrow \alpha$ transformation of zirconium studied in situ by neutron diffraction”. *Acta Materialia*, 2004, 52.7: 1899-1907.


One method to express the 3D orientation of a crystal is to define the rotation of the crystal coordinate system relative to the sample coordinate system and consider the symmetry of the crystal itself. The rotation of the crystal is generally described by a triplet of angles, known as Euler angles, \((\phi_1, \Phi, \phi_2)\), that express the consecutive rotations about the axis of the crystal coordinate system that are needed to bring the crystal coordinate system into coincidence with the sample coordinate system (i.e. passive rotations). In the present study a crystal coordinate system for the \(\alpha\) and \(\beta\) phases was chosen as defined in the EDAX Orientation Imaging Microscopy (OIM\textsuperscript{TM}) data analysis software. The 3D rotations were then expressed according to the Bunge’s convention as follow:

\[
R(\phi_1, \Phi, \phi_2) = \begin{bmatrix}
\cos \phi_1 \cos \phi_2 & -\sin \phi_1 \sin \phi_2 \cos \Phi & \sin \phi_1 \cos \phi_2 \\
\cos \phi_1 \sin \phi_2 & \sin \phi_1 \cos \phi_2 \cos \Phi & \sin \phi_1 \sin \phi_2 \\
\sin \phi_1 & -\sin \phi_2 \cos \phi_1 & \cos \phi_1 \cos \phi_2 \\
\end{bmatrix}
\]

Because of the crystal symmetry however, multiple rotations can result in an equivalent 3D orientation of a certain crystal. For this reason, it was also necessary to take into account the rotational symmetry elements of \(\alpha\) and \(\beta\) phases in order to be able to distinguish independent orientations. The rotational symmetry elements of the \(\alpha\) and \(\beta\) phases (12 and 24 respectively) used in this study are listed in the form of rotational matrices (Bunge’s convention) in Table 1 and 2. As the \(\alpha\) and \(\beta\) phase are related through the Burgers orientation relationship which is equivalent to a Bunge’s rotation expressed by the matrix \(D\) (135°, 90°, 325°), it has been shown that starting from three \(\alpha\) grain orientations it is possible to determine the parent \(\beta\) grain orientation with accuracy and conversely each \(\beta\) grain can generate 12 distinct \(\alpha\) grain orientations (\(\alpha\) variants). In the present study, the 12 \(\alpha\) variants generated from one single \(\beta\) grain correspond to the set of rotations listed in Table 3.
### Table 1

<table>
<thead>
<tr>
<th>Rotational elements of the cubic symmetry</th>
</tr>
</thead>
<tbody>
<tr>
<td>E(0,0,0)</td>
</tr>
<tr>
<td>C(_{2x})(0,(\pi),0)</td>
</tr>
<tr>
<td>C(_{2y})((\pi),(\pi),0)</td>
</tr>
<tr>
<td>C(_{2z})((\pi),0,0)</td>
</tr>
<tr>
<td>C(_{31+})((\pi/2),(\pi/2),0)</td>
</tr>
<tr>
<td>C(_{32+})(3(\pi/2),(\pi/2),(\pi))</td>
</tr>
<tr>
<td>C(_{33+})(3(\pi/2),(\pi/2),(\pi/2))</td>
</tr>
<tr>
<td>C(_{31})((\pi),(\pi),2(\pi/3))</td>
</tr>
<tr>
<td>C(_{32})(0,(\pi/2),3(\pi/2))</td>
</tr>
<tr>
<td>C(_{33})((\pi),(\pi/2),(\pi/2))</td>
</tr>
<tr>
<td>C(_{34})(0,(\pi/2),(\pi/2))</td>
</tr>
<tr>
<td>C(_{34+})((\pi/2),(\pi/2),(\pi))</td>
</tr>
<tr>
<td>C(_{4x+})(0,(\pi/2),0)</td>
</tr>
<tr>
<td>C(_{4y+})((\pi/2),(\pi/2),3(\pi/2))</td>
</tr>
<tr>
<td>C(_{4z+})((\pi/2),0,0)</td>
</tr>
<tr>
<td>C(_{4x})((\pi),(\pi/2),(\pi/2))</td>
</tr>
<tr>
<td>C(_{4y})(3(\pi/2),(\pi/2),(\pi/2))</td>
</tr>
<tr>
<td>C(_{4z})((\pi),(\pi/2),3(\pi/2))</td>
</tr>
</tbody>
</table>

### Table 2

<table>
<thead>
<tr>
<th>Rotational elements of the hexagonal symmetry</th>
</tr>
</thead>
<tbody>
<tr>
<td>E(0,0,0)</td>
</tr>
<tr>
<td>H(<em>{C</em>{6z}})((\pi/3),0,0)</td>
</tr>
<tr>
<td>H(<em>{C</em>{3z}})(2(\pi/3),0,0)</td>
</tr>
<tr>
<td>H(<em>{C</em>{2z}})(0,0,(\pi))</td>
</tr>
<tr>
<td>H(<em>{C</em>{3z}})(4(\pi/3),0,0)</td>
</tr>
<tr>
<td>H(<em>{C</em>{6z}})(-(\pi/3),0,0)</td>
</tr>
<tr>
<td>H(<em>{C'</em>{21}})((\pi),(\pi),0)</td>
</tr>
<tr>
<td>H(<em>{C''</em>{21}})((\pi),(\pi),(\pi/3))</td>
</tr>
<tr>
<td>H(<em>{C'</em>{23}})((\pi),(\pi),2(\pi/3))</td>
</tr>
<tr>
<td>H(<em>{C''</em>{31}})((\pi),(\pi),(\pi))</td>
</tr>
<tr>
<td>H(<em>{C'</em>{22}})((\pi),(\pi),4(\pi/3))</td>
</tr>
<tr>
<td>H(<em>{C''</em>{23}})((\pi),(\pi),5(\pi/3))</td>
</tr>
</tbody>
</table>
Table 3

<table>
<thead>
<tr>
<th>α grain rotations deriving from a single parent β grain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. D⁻¹C₄Y.</td>
</tr>
<tr>
<td>2. D⁻¹C₃₂⁺</td>
</tr>
<tr>
<td>3. D⁻¹C₂e</td>
</tr>
<tr>
<td>4. D⁻¹C₃₃⁺</td>
</tr>
<tr>
<td>5. D⁻¹C₃₁⁻</td>
</tr>
<tr>
<td>6. D⁻¹C₄X.</td>
</tr>
<tr>
<td>7. D⁻¹C₃₃⁻</td>
</tr>
<tr>
<td>8. D⁻¹C₂d</td>
</tr>
<tr>
<td>9. D⁻¹E</td>
</tr>
<tr>
<td>10. D⁻¹C₂a</td>
</tr>
<tr>
<td>11. D⁻¹C₂b</td>
</tr>
<tr>
<td>12. D⁻¹C₂X</td>
</tr>
</tbody>
</table>

The back calculation of the parent β phase from the measured α texture is computed numerically in Matlab using the angular information measured during the EBSD analysis. The reconstruction of the β phase from the acquired EBSD dataset use three input files exported from the EDAX Orientation Imaging Microscopy (OIM™) data analysis software. The name and the content of these three exported files is specified as follow:

- **neighbours.txt**. Each row contains ID_grain_n (identification of α grain_n), number of adjacent grains to grain_n, identification of adjacent grains.
- **average.txt** Each row contains an identification of grain_n, three euler angles that define the average orientation of ID (Bunge/TSL convention).
- **pixels.txt** File that contains the spatial information (“x” and “y” coordinates) of each α grain n and the corresponding identification.

### 10.1.1 Launch of “main_reconstruction.m”: main function to reconstruct the parent β phase

The three input files are pasted into a common directory with MTex. MTex is then initialized, typing >>startup in the Matlab workspace. Once MTex is initialized, the reconstruction of the β phase is launched typing >>ebsd=main_reconstruction(threshold). Example:

```matlab
ebsd=main_reconstruction(30);  % launches the script with a threshold of 30°. See Section 1.3 for more info on threshold.
```
The script automatically creates two txt files named *info_beta* and *ebsd_file*. *ebsd_file* contains all the necessary information to calculate the texture of the parent $\beta$ phase and to generate IPF maps. Example:

```plaintext
>> plot(ebsd) % generated an IPF map point to point
```

```plaintext
>> orientation_map(ebsd,8,20) % generates an IPF grouping under an unique $\beta$ grain solutions with a misorientation less than 8°. Grains composed with at least 20 points are considered.
```

```plaintext
>> odf = calcODF(ebsd); % generates an estimated odf from the ebsd dataset in analysis
```

```plaintext
>> plotpdf(odf,[Miller(1,0,0),Miller(1,1,0),Miller(1,1,1)],'antipodal','silent','position',[10 10 600 200]) % generates the [100], [110], [111] contour pole figures corresponding to odf.
```

### 10.1.2 General Comments on “main_reconstruction.m”

Each row of neighbours.txt has a variable length. The default import option inserts “-” (hyphens) in correspondence of short rows. The command *isnan* allows to substitute “-” with “0” (zeros).

*scriptmark* and *storer* are dummy variables that are used later in the loops.

*symmetry* is a built-in function of Mtex to define the symmetry of the crystal cell (variable *cs*) and the sample symmetry (*ss*).

*beta* defines an initial arbitrary grain orientation.

ID_grain_n and the number of its adjacent grains are initially evaluated (*grain1* and *neigh*). If ID_grain_n has a number of adjacent grains inferior of 2 the script evaluates another grain (grain n+1). If ID_grain_n has more than 2 adjacent grains the script attempts to reconstruct the common parent $\beta$ phase using the “*calc_beta*” subfunction. If a solution to the first triplet of $\alpha$ grains is found (see *calc_beta*) the orientation of the $\beta$ phase is stored into *solutionbeta*. The variable *storer* stores the ID of the triplet of $\alpha$ grains that were reconstructed. One new adjacent grain of ID_grain_n is considered at the time. If they can be also be reconstructed with the same calculated $\beta$ orientation (*solutionbeta*) they are added to *storer* otherwise they are discarded. *Info_beta* is then created. *Info_beta* contains the three euler angles that define
the parent β phase and the ID of the α grains that derive from it. These calculations are repeated for all the grains in the dataset and new rows are appended to Info_beta.

Once the entire dataset is examined the spatial information of the α grains is taken into account. Pixels stores the x and y coordinates of each α grain in the dataset. The spatial information of all the α grains that derive from the same parent β phase is retrieved. One α grain is examined at the time. The β solution corresponding to each α of Info_beta is retrieved and stored in phi(ind), theta(ind), psi(ind). The average orientation of the grain is then calculated phi_medio, theta_medio, psi_medio. The x and y coordinate of each α grain is retrieved and stored into posx and posy. A new viable EBSD_file is then created with the solution of the β solution and the x and y corresponding information. A MTex ebsd file called ebsd is then created from EBSD_file. ebsd is a dataset that contain all the information necessary for the analysis and plotting of the reconstructed β phase.

10.1.3 General Comments on “calc_beta.m”

“calc_beta.m” attempts the reconstruction of the parent β phase from a triplet of adjacent α grains. Six β solutions are created from each α of the examined triplet (α1, α2, α3). The six solution of α1 are compared to the six solution of α2. If a common solution can be found (within the misorientation threshold imposed by threshold), the solution is considered as potential solution β_check of the triplet. β_check is then compared to the the six solutions that derive from α3. If a β_check is also a solution for α3 (within the misorientation threshold imposed by threshold) the orientation of the β phase from which the triplet derive is found (α1, α2, α3).

10.2 Scripts used for Representation and Orientation Relationship Analysis

10.2.1 General Comments on “orientation_map.m”

orientation_map(ebsd,threshold_mis,threshold_size) generates an IPF map of the reconstructed β phase. The IPF is made of β grains that consist of all the solutions that are
misoriented less than \textit{threshold\_mis} (typically 8°). Grains that consist of more than \textit{threshold\_size} number of points are considered.

\subsection*{10.2.2 General Comments on “variant\_distribution.m”}

\texttt{variant\_distribution.m} is used to calculate the frequency of the variant distribution in the dataset in analysis. This script uses \texttt{info\_beta.txt} to retrieve information on the orientation of the parent $\beta$ phase and the identification of the $\alpha$ grains that derive from it. The 12 possible $\alpha$ variants from the $\beta$ phase (\textit{ort(1-12)}) are then generated. The script then compares the misorientation between the $\alpha$ grains and the 12 possible $\alpha$ variants that derive from the corresponding parent $\beta$ phase. The solution with the least misorientation is stored as well as the corresponding variant transformation ($x$). The analysis continues until all the $\alpha$ grains in the dataset are examined.

\subsection*{10.2.3 General Comments on “discretepolefigure.m”}

d\texttt{is}e\texttt{tepolefigure.m} generates discrete pole figures of individual (or multiple) $\alpha$ or $\beta$ grains. This function defines the symmetry of the crystal cell ($cs$) and the sample ($ss$). The orientation of the grain is then specified using the Bunge convention, that is three successive rotations around the axis $Z$, $X$ and $Z$ of the crystal coordinate system.

\subsection*{10.2.4 Matlab and OIM\textsuperscript{TM} Data Analysis Compatibility}

Both Matlab and OIM\textsuperscript{TM} data analysis software use the Bunge’s convention to define a crystal rotation, i.e. three consecutive rotation around the $z$-, $x$- and $z$-axis of the crystal coordinate system. For a correct comparison of the results obtaining using OIM\textsuperscript{TM} data analysis software and Matlab, the definition for the orientation of a cubic and hexagonal crystals in both softwares should however be considered. There are two fundamental differences. The first difference is that the $x$-axis in the OIM\textsuperscript{TM} data analysis software and Matlab have opposite verses (Figure 111). It is clear that this affects the representation of the orientation for both the cubic and hexagonal crystal coordinate systems: when the orientation is described using the Euler angles, identical rotations are obtained for opposite angle values.
The second difference emerges when coordinate system definition for the hexagonal crystal is compared in the two systems.

Figure 111: Definition of the crystal coordinate system in a) OIM™ data analysis software and b) Matlab. It can be noticed that the x-axis is defined with opposite verses in the softwares. The y-axis instead are shifted by 30°.

The two crystal coordinate systems are in fact brought into coincidence only by rotating one of the two around the z-axis for 30° (Figure 111). In OIM™ data analysis software the y-axis cuts a vertex of the hexagon. In Matlab, the y-axis passes instead through the middle of one of the hexagon sides. In practise, an HCP orientation described by a triplet of Euler angles \((\phi_1, \Phi, \phi_2)\) in OIM™ data analysis software can be expressed in Matlab by \((-\phi_1, \Phi, \phi_2+30°)\).
function [ebsd] = main_reconstruction (threshold)
startup % start MTex Matlab toolbox, available to download at http://code.google.com/p/mtex/
m=dlmread('neighbors.txt'); % ID grain, number of neighbours, ID neighbours
angles=dlmread('average.txt'); % ID grain , three average euler degree
m(isnan(m))=0;
read1=m;
[totgrains, ncolumns]=size(read1);
scriptmark=1;
storer=1;
cs=symmetry('cubic');
ss=symmetry('triclinic');
beta=orientation('Euler',0,0,0,cs,ss);
for row_n=1:totgrains
    grain1=read1(row_n,1);
nneigh=read1(row_n,2);
    counter=0;
    if (nneigh>=2)% reconstruction if at least 3 adjacent alpha
        numerocolumns=nneigh+2;
cycle_num=numerocolumns-3;% no triplets
        for add=1:cycle_num
            grain2=read1(row_n,2+add);
grain3=read1(row_n,3+add);
            [solutionbeta,min] = calc_beta(grain1, grain2, grain3,angles,threshold);
            if(min<threshold)
                counter=counter+1;
                storer(row_n,1)=grain1;
                if (counter==1)
                    storer(row_n,2)=grain2;
                    storer(row_n,3)=grain3;
                else
                    storer(row_n,counter+2)=grain3;
                end
            end
        end
        [phib,thetab,psib]=Euler(solutionbeta,'Bunge');
        phibeta=rad2deg(phib);
        thetaneta=rad2deg(thetab);
        psibeta=rad2deg(psib);
        angles=[phibeta,thetaneta,psibeta,storer(row_n,:)];
end

[phib,thetab,psib]=Euler(solutionbeta,'Bunge');
phibeta=rad2deg(phib);
thetaneta=rad2deg(thetab);
psibeta=rad2deg(psib);
angles=[phibeta,thetaneta,psibeta,storer(row_n,:)];
```matlab
   dlmwrite('info_beta.txt',angles,'-append','delimiter','
   solutionbeta=0;
   phibeta=0;
   thetaneta=0;
   psibeta=0;
   else
   bananas=999999;
   end
   end
   else
   end
end

m=dlmread('info_beta.txt');
pixels=dlmread('pixels.txt'); % x and y and ID
m(isnan(m))=0;
fileEBSD=m;
[totgrains, width]=size(fileEBSD);
IDDISTINCT=fileEBSD(:,4);
IDAD=pixels(:,3);
matrix=zeros(totgrains, 5);
for row_n=1: totgrains
   ID=IDDISTINCT(row_n);
   [row_ID, columns_ID]=find(fileEBSD==ID);
   molt=size(row_ID);
   multiplicity=molt(1,1);
   for ind=1: multiplicity
       phi(ind)=fileEBSD(row_ID(ind,1),1);
       theta(ind)=fileEBSD(row_ID(ind,1),2);
       psi(ind)=fileEBSD(row_ID(ind,1),3);
   end
   phi_medio=sum(phi)/multiplicity;
   theta_medio=sum(theta)/multiplicity;
   psi_medio=sum(psi)/multiplicity;
   phi=0;
   theta=0;
   psi=0;
   [index_x y]=find(IDpixels==ID);
   molt2=size(index_x);
   multiplicity2=molt2(1,1);
end
```

for roll=1:multicity\n    posx(roll)=pixels(index_x(roll,1),1);
posy(roll)=pixels(index_x(roll,1),2);
    phi_(roll)= phi_medio;
    theta_(roll)= theta_medio;
    psi_(roll)=psi_medio;
end
matrix=[phi_;theta_;psi_;posx;posy]';
dlmwrite('ebsd_file.txt', matrix,'-append','delimiter','	');
matrix=0;
posx=0;
posy=0;
phi_=0;
theta_=0;
psi_=0;
end
cs=symmetry('cubic');
ss=symmetry('triclinic');
ebsd=loadEBSD_generic('ebsd_file.txt', 'CS', cs, 'SS', ss, 'ColumnNames', {'Euler1', 'Euler2', 'Euler3', 'y','x'},
'Bunge');
function [solution,minimo] = calc_beta (grain1, grain2, grain3,angles,threshold)

read2=angles;
ind1=find(read2(:,1)==grain1);
phi1=read2(ind1,2);
theta1=read2(ind1,3);
psi1=read2(ind1,4);
ind2=find(read2(:,1)==grain2);
phi2=read2(ind2,2);
theta2=read2(ind2,3);
psi2=read2(ind2,4);
ind3=find(read2(:,1)==grain3);
phi3=read2(ind3,2);
theta3=read2(ind3,3);
psi3=read2(ind3,4);
var=rotation('Euler',phi1*degree, theta1*degree, psi1*degree, 'Bunge');
var2=rotation('Euler',phi2*degree, theta2*degree, psi2*degree, 'Bunge');
var3=rotation('Euler',phi3*degree, theta3*degree, psi3*degree, 'Bunge');
cs=symmetry('hexagonal');
ss=symmetry('triclinic');
ori_1=orientation(var, cs,ss);
ori_2=orientation(var2, cs,ss);
ori_3=orientation(var3, cs,ss);
s=symmetrise(ori_1);
s2=symmetrise(ori_2);
s3=symmetrise(ori_3);
d=rotation('Euler', 135*degree, 90*degree, 325*degree,'Bunge');
for i=1:12
    beta(i)=s(i)*inverse(d);
    beta2(i)=s2(i)*inverse(d);
    beta3(i)=s3(i)*inverse(d);
end
cs=symmetry('cubic');
ss=symmetry('triclinic');
for j=1:3
    ori(j)=orientation(beta(j), cs,ss);
    ori2(j)=orientation(beta2(j), cs,ss);
    ori3(j)=orientation(beta3(j), cs,ss);
end
for j=4:6
ori(j) = orientation(beta(j+3), cs, ss);
ori2(j) = orientation(beta2(j+3), cs, ss);
ori3(j) = orientation(beta3(j+3), cs, ss);
end
beta_1 = ori;
beta_2 = ori2;
beta_3 = ori3;
for i = 1:6
    for j = 1:6
        mis12(i,j) = angle(beta_1(i), beta_2(j))/degree;
        mis13(i,j) = angle(beta_1(i), beta_3(j))/degree;
        mis32(i,j) = angle(beta_3(i), beta_2(j))/degree;
    end
end
[x, y] = find(mis12 == min(min(mis12)));
value = mis12(x(1), y(1));
if (value < threshold)
    beta1 = beta_1(x(1));
beta2 = beta_2(y(1));
    [xx, yy] = find(mis13 == min(mis13(x(1), :)));
    value2 = mis13(xx(1), yy(1));
    if (value2 < threshold)
        beta3 = beta_3(yy(1));
        value3 = mis32(yy(1), y(1));
        if (value3 < threshold)
            solution = beta1;
            minimo = threshold - 1;
        else
            cancel = 0;
            minimo = threshold + 1;
            solution = 0;
        end
    else
        cancel = 0;
        minimo = threshold + 1;
        solution = 0;
    end
else
    cancel = 0;
    minimo = threshold + 1;
else
    cancel = 0;
    minimo = threshold + 1;
solution=0; end

function [] = discretepolefigure ()

% plots 6 alpha grains 0001 and 11-20 pole figures

cs=symmetry('hexagonal');
ss=symmetry('triclinic');
rotf=rotation('axis',Miller(0,0,1),'angle',89.8*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',125.3*degree);
rott=rotation('axis',Miller(0,0,1),'angle',268*degree);
grain1=rotf*rots*rott;

rotf=rotation('axis',Miller(0,0,1),'angle',325.6*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',85.1*degree);
rott=rotation('axis',Miller(0,0,1),'angle',93.1*degree);
grain2=rotf*rots*rott;

rotf=rotation('axis',Miller(0,0,1),'angle',203.3*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',121.1*degree);
rott=rotation('axis',Miller(0,0,1),'angle',226.6*degree);
grain3=rotf*rots*rott;

rotf=rotation('axis',Miller(0,0,1),'angle',278.1*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',175.9*degree);
rott=rotation('axis',Miller(0,0,1),'angle',108.3*degree);
grain4=rotf*rots*rott;

rotf=rotation('axis',Miller(0,0,1),'angle',104.4*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',133.0*degree);
rott=rotation('axis',Miller(0,0,1),'angle',194.8*degree);
grain5=rotf*rots*rott;

rotf=rotation('axis',Miller(0,0,1),'angle',156.5*degree);
rots=rotation('axis',Miller(-1,0,0),'angle',95.1*degree);
rott=rotation('axis',Miller(0,0,1),'angle',359.1*degree);
grain6=rotf*rots*rott;

or1=orientation(grain1,cs,ss);
or2=orientation(grain2,cs,ss);
or3=orientation(grain3,cs,ss);
or4=orientation(grain4,cs,ss);
or5=orientation(grain5,cs,ss);
or6=orientation(grain5,cs,ss);

plotpdf(or1,Miller(0,0,0,1),'antipodal')
hold on
plotpdf(or2,Miller(0,0,0,1),'antipodal')
hold on
plotpdf(or3,Miller(0,0,0,1),'antipodal')
hold on
plotpdf(or4,Miller(0,0,0,1),'antipodal')
hold on
plotpdf(or5,Miller(0,0,0,1),'antipodal')
hold on
plotpdf(or6,Miller(0,0,0,1),'antipodal')

figure
plotpdf(or1,Miller(1,1,-2,0),'antipodal')
hold on
plotpdf(or2,Miller(1,1,-2,0),'antipodal')
hold on
plotpdf(or3,Miller(1,1,-2,0),'antipodal')
hold on
plotpdf(or4,Miller(1,1,-2,0),'antipodal')
hold on
plotpdf(or5,Miller(1,1,-2,0),'antipodal')
hold on
plotpdf(or6,Miller(1,1,-2,0),'antipodal')
function [] = variant_distribution ()

startup

infobeta=dlmread('info_beta.txt');
average=dlmread('average.txt');
infobeta(isnan(infobeta))=0;
betas=infobeta;

[no_grains, no_columns]=size(betas);

for n=1:1:no_grains
    phi=betas(n,1);
    theta=betas(n,2);
    psi=betas(n,3);
    rot=rotation('Euler', phi*degree, theta*degree, psi*degree, 'Bunge');
    ori_beta=orientation(rot,cs,ss); % orientation reconstructed beta
    s=symmetrise(ori_beta);
    for i=1:8
        var(i)=s(12+i)*d;
    end
    for j=9:12
        var(j)=s(j-4)*d;
    end

end

for n=1:1:no_grains
    phi=betas(n,1);
    theta=betas(n,2);
    psi=betas(n,3);
    rot=rotation('Euler', phi*degree, theta*degree, psi*degree, 'Bunge');
    ori_beta=orientation(rot,cs,ss); % orientation reconstructed beta
    s=symmetrise(ori_beta);
    for i=1:8
        var(i)=s(12+i)*d;
    end
    for j=9:12
        var(j)=s(j-4)*d;
    end

end

% 12 theoretical variants from reconstructed beta
or(1)= orientation(var(1), cs,ss);
or(2)= orientation(var(2), cs,ss);
or(3)= orientation(var(3), cs,ss);
or(4)= orientation(var(4), cs,ss);
or(5)= orientation(var(5), cs,ss);
or(6)= orientation(var(6), cs,ss);
or(7)= orientation(var(7), cs,ss);
or(8)= orientation(var(8), cs, ss);
or(9)= orientation(var(9), cs, ss);
or(10)= orientation(var(10), cs, ss);
or(11)= orientation(var(11), cs, ss);
or(12)= orientation(var(12), cs, ss);
[dummy2, no_columns]=size(betas(n,:));

for i=4:no_columns
    ID1=betas(n,i);
    if ID1==0 | find(storer(:,1)==ID1)
        dummy2=1;
    else
        [pos,dummy2]=find(average==ID1);
    end
    alpha1=average(pos,2:4);
    grain1=rotation('Euler',alpha1(1,1)*degree, alpha1(1,2)*degree, alpha1(1,3)*degree, 'Bunge');
    ori_grain1=orientation(grain1, cs, ss);
    storer(n+i,1)=ID1;
    mis0rientat(1)=angle(or(1),ori_grain1)/degree;
    mis0rientat(2)=angle(or(2),ori_grain1)/degree;
    mis0rientat(3)=angle(or(3),ori_grain1)/degree;
    mis0rientat(4)=angle(or(4),ori_grain1)/degree;
    mis0rientat(5)=angle(or(5),ori_grain1)/degree;
    mis0rientat(6)=angle(or(6),ori_grain1)/degree;
    mis0rientat(7)=angle(or(7),ori_grain1)/degree;
    mis0rientat(8)=angle(or(8),ori_grain1)/degree;
    mis0rientat(9)=angle(or(9),ori_grain1)/degree;
    mis0rientat(10)=angle(or(10),ori_grain1)/degree;
    mis0rientat(11)=angle(or(11),ori_grain1)/degree;
    mis0rientat(12)=angle(or(12),ori_grain1)/degree;
    x=find(mis0rientat==min(min(mis0rientat)));
    dlmwrite('numero_varianti_hori.txt',x,'-append','delimiter','\t');
end
end
end
infovariant=dlmread('numero_varianti_hori.txt');