



High-temperature compressive deformation of additive manufactured Ti6Al4V

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Declaration

I Guyle le Roux hereby declare that this is my own unaided work. I am aware of and have adhered to North-West University's policy on Academic Integrity. This work is being submitted to the degree of Master of Engineering in Mechanical Engineering to North-West University, Potchefstroom, and has not been submitted before for any other degree or for examination in any other university.

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Abstract

Machining titanium alloys such as Ti6Al4V to near-net-shapes is difficult and costly because of their low thermal conductivity, high strength and hardness at elevated temperatures, high chemical reactivity, and relatively low elastic modulus that hinder their usage. Additive manufacturing of Ti6Al4V provides the opportunity to produce complex geometries with minimum machining and short production times, thereby reducing manufacturing costs. However, there is limited research available on the high-temperature compressive deformation of additive manufacturing Ti6Al4V to fully replace conventionally manufactured Ti6Al4V.

In this study, as-built Ti6Al4V additive manufactured (AM) using selective laser melting (SLM) and laser engineered net shaping (LENS) techniques were subjected to hot compression tests at temperatures ranging from 750 to 900°C and strain rates of 0.1 – 10 s⁻¹ using the Gleeble 3500® thermo-mechanical simulator and power dissipation maps to characterise deformation behaviour. Microstructural analyses on the additively manufactured Ti6Al4V before and after hot compression tests were performed using optical microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). An Arrhenius-type equation with a hyperbolic sine function was utilised to compare the experimental flow stress obtained by the Gleeble 3500® thermo-mechanical simulator to the calculated flow stress, with good correlation. In all cases a wrought Ti6Al4V was used as a control.

The results obtained by SEM and XRD showed that additive manufactured Ti6Al4V exhibited larger β -grain boundaries with finer α -phase in between compared to the wrought Ti6Al4V displaying a network of β -phase with equiaxed and elongated α -phase. The α - and β -phase composition showed a notable dependence on manufacturing techniques, with no significant changes in the chemical composition after deformation, but only in the percentages of phases present.

Flow stress curves obtained at 10 s⁻¹ showed that the peak strength of wrought Ti6Al4V decreased by 54.7% when the deformation temperature was increased from 750 to 900°C but decreased by a maximum of 70.6% for the AM Ti6Al4V, with the most significant decrease observed on the samples produced by LENS. At higher strain rates, the flow stress of all the samples, regardless of manufacturing technique, exhibited flow softening after achieving the peak stress value, with stress decreasing by at least 298 MPa at 0.1 s⁻¹, compared to a maximum decrease of 400 MPa at 10 s⁻¹.

Decreasing the strain rate from 10 to 0.1 s⁻¹ resulted in serrated flow stress curves, consistent with the Portevin-Le Chatelier effect, which became more pronounced with an increase in

temperature. In addition, the Portevin-Le Chatelier effect was observed most frequently on the additive manufactured Ti6Al4V, suggesting that the wrought microstructure with coarser elongated and equiaxed α -grains hinders dislocation movement and results in a lower flow stress. Power dissipation maps showed that the predominant mechanisms in the high-power efficiency percentages of deformation were dynamic recrystallisation (DRX) and globularisation for wrought Ti6Al4V; dynamic globularisation, recovery (DRV), and DRX for the samples prepared by SLM; and dynamic globularisation and DRX for the samples produced using LENS. These results imply that additive manufactured Ti6Al4V would be more prone to failure by creep or stress rupture compared to wrought Ti6Al4V.

Keywords: (Titanium alloys, Ti6Al4V, additive manufacturing, high-temperature, compression, power dissipation maps)

Abstrak

Die bewerking van titaanlegerings soos Ti6Al4V na naby-net-vorme is moeilik en duur as gevolg van die legering se lae termiese geleidingsvermoë, hoë sterkte en hardheid by verhoogde temperature, hoë chemiese reaktiwiteit, en lae elastiese modulus, wat die gebruik daarvan belemmer. Additiewe vervaardiging van Ti6Al4V bied die geleentheid om komplekse geometrieë met minimale bewerking en kort produksietye te vervaardig, wat die vervaardigingskoste verlaag. Daar is egter beperkte navorsing beskikbaar oor hoë-temperatuur drukvervorming van additief vervaardigde Ti6Al4V om dit volledig te vervang met konvensioneel vervaardigde Ti6Al4V.

In hierdie studie is as-vervaardigde Ti6Al4V wat deur selektiewe lasersmelting (SLM) en laser-ingenieurs netvorming (LENS) vervaardig is, onderwerp aan warmdruktoetse by temperature wat wissel van 750 tot 900°C en vervormingstempo's van $0.1 - 10 \text{ s}^{-1}$ deur die Gleeble 3500® termomeganiese simulator te gebruik, en krag-dissipasie kaarte wat gebruik is om die vervormingsgedrag te karakteriseer. Mikrostruktuur- en fase-ontledings op die additief vervaardigde Ti6Al4V voor en na warmdruktoetse is uitgevoer met behulp van optiese mikroskopie, skande-elektronmikroskopie (SEM) en X-straal diffraksie (XRD). 'n Arrhenius-tipe vergelyking met 'n hiperboliese sinusfunksie is gebruik om die eksperimentele vloei-spanning wat deur die Gleeble 3500® termomeganiese simulator verkry is te vergelyk met die berekende vloei-spanning, met goeie korrelasie. In alle gevalle is 'n gesmede Ti6Al4V as 'n beheer gebruik.

Die resultate wat deur SEM en XRD verkry is, het getoon dat additief vervaardigde Ti6Al4V groter β -korrelgrense met fyner α -fase tussenin vertoon het in vergelyking met die gesmede Ti6Al4V wat 'n netwerk van β -fase met eweveel en verlengde α -fase vertoon. Die α - en β -fase samestelling het 'n noemenswaardige afhanklikheid van vervaardigingstegnieke getoon, met geen beduidende veranderinge in die chemiese samestelling na vervorming nie, maar net in die persentasies van fases wat teenwoordig is.

Vloei-spanningskurwes wat by 10 s^{-1} verkry is, het getoon dat die pieksterkte van gesmede Ti6Al4V met 54.7% verminder het wanneer die vervormingstemperatuur verhoog is van 750 tot 900°C, maar met 'n maksimum van 70.6% vir die additief vervaardigde Ti6Al4V, met die grootste daling waargeneem op die proefstukke wat deur LENS geproduseer is. By hoër vervormingstempo's het die vloei-spanning van al die proefstukke, ongeag die vervaardigingstegniek, vloei-versagting getoon na die maksimum spanning bereik is, met 'n daling van minstens 298 MPa by 0.1 s^{-1} , in vergelyking met 'n maksimum daling van 400 MPa by 10 s^{-1} .

Die verlaging van die vervormingstempo van 10 tot 0.1 s^{-1} het gesaagde vloeispanningskurwes tot gevolg gehad, in ooreenstemming met die Portevin-Le Chatelier-effek, wat meer uitgesproke geword het met 'n toename in temperatuur. Daarbenewens is die Portevin-Le Chatelier-effek die meeste op die additief vervaardigde Ti6Al4V waargeneem, wat aandui dat die gesmede mikrostruktuur met growwer verlengde en eweveel α -korrels dislokasie-beweging verhinder en 'n laer vloeispanning tot gevolg het. Krag-dissipasierkaart het getoon dat die oorheersende meganismes in die hoë-krag doeltreffendheids persentasies van vervorming dinamiese herkristallasie (DRX) en globularisasie vir gesmede Ti6Al4V was, dinamiese globularisasie, herstel (DRV), en DRX vir die proefstukke voorberei deur SLM, en dinamiese globularisasie en DRX vir die proefstukke wat met LENS geproduseer is. Hierdie resultate impliseer dat additief vervaardigde Ti6Al4V meer geneig sou wees tot mislukking deur kruip of spanningsbreuk in vergelyking met gesmede Ti6Al4V.

Sleutelwoorde: (Titaanlegerings, Ti6Al4V, additief vervaardiging, hoë-temperatuur, kompressie, krag-dissipasierkaart)

Dedications

This work is dedicated to my family that always believed in me, and to myself to show that I can strive to do embrace every opportunity that comes my way. I also dedicate and hope this research work will help the titanium industry, additive manufacturing industry, and everyone who has contributed knowledge to the improvement of titanium alloys for industrial and commercial use.

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List of symbols

α	alpha
$\alpha+\beta$	alpha and beta
β	beta
D_R	ratio of height and diameter
h_o	height of sample
d_o	diameter of sample
$\dot{\varepsilon}$	strain rate
ε	strain
σ	flow stress
T	absolute temperature
Z	Zener-Hollomon parameter
$\dot{n}$	material constant for Arrhenius-type equation
α_1	material constant for Arrhenius-type equation
β_1	material constant for Arrhenius-type equation
A	material constant for Arrhenius-type equation
R	universal gas constant
Q	activation energy
M	measured flow stress
$\bar{M}$	mean measured flow stress
P	predicted flow stress
$\bar{P}$	mean predicted flow stress
m	sensitivity parameter
N	total number of measurements
η	efficiency of power dissipation
R^2	correlation coefficient

List of acronyms

AARE	Average Absolute Relative Error
AM	Additive manufacturing
BCC	Body Centred Cubic
CAD	Computer Aided Design
CSIR	Council for Scientific and Industrial Research
DED	Direct energy deposition
DM	Direct manufacturing
DMD	Direct metal deposition
DMLS	Direct metal laser sintering
DRV	Dynamic recovery
DRX	Dynamic recrystallisation
EBM	Electron beam melting
EDS	Energy Dispersive X-Ray Spectroscopy
HCP	hexagonal close-packed
LENS	Laser engineered net shaping
LM	Laser melting
LMD	Laser melting deposition
PBF	Powder bed fusion
RMSE	Root Mean Square Error
SEM	Scanning electron melting
SLM	Selective laser melting
SLS	Selective laser sintering
WAAM	Wire and arc additive manufacturing
XRD	X-ray diffraction

Chapter 1: Introduction

1.1 Background

Ti6Al4V, also known as Ti64 or TC4, is a grade 5 alloy comprising approximately 6% aluminium to stabilise the α -phase and 4% vanadium to stabilise the β -phase. This dual-phase alloy is the most widely employed titanium alloy for applications such as aerospace, aircraft, chemical, and biomedical industries, particularly those requiring high-temperature strength (Xiao et al., 2019). Favourable properties include: high strength at elevated temperatures, a high strength-to-weight ratio, good creep resistance, and excellent corrosion resistance (Sui et al., 2019; Pushp et al., 2022).

Titanium alloys are important for high-temperature structural applications in the aerospace and automotive industries. Ti6Al4V, for instance, is used in the aerospace industry for compressor blades (Artetxe et al., 2016), which may be exposed to temperatures as high as 1500°C. Ti6Al4V is also used in rods to connect the crankshaft and piston in motorcycles, typically exposed to temperatures up to 300°C, and in intake valves operating within a temperature range of 300 to 400°C (Takahashi et al., 2020), resulting in enhanced driving responsiveness. The melting point of Ti6Al4V is $\approx 1660^\circ\text{C}$ (Romero et al., 2015), rendering it suitable for high-temperature applications. However, manufacturing Ti6Al4V remains a significant challenge (Gupta & Laubscher, 2016; Liu et al., 2019; Bandyopadhyay et al., 2020).

Ti6Al4V presents significant machining challenges owing to its low thermal conductivity of approximately 6.7 W/mK, compared to the thermal conductivity of ≈ 33.6 W/mK for steel alloys (Peet et al., 2011; Vidaller et al., 2014). Thermal conductivity is essential for controlling the temperature gradient during machining. A low thermal conductivity indicates that the alloy would absorb little heat during machining, leading to a low material removal rate and an increased amount of tool heating, thereby diminishing the cutting tool's lifespan. In addition, Ti6Al4V has a low modulus of elasticity of about 110 GPa, compared to ≈ 200 GPa for steel alloys (Bahl et al., 2021). This makes Ti6Al4V prone to deflection during machining, requiring high cutting forces and low cutting speeds.

Additive manufacturing (AM) can eliminate or minimise the need for excessive machining of Ti6Al4V, thereby reducing manufacturing costs and time. Scrap rates during the machining of titanium alloys are expected to range from 80–90%, compared to $<10\%$ when using AM (Rawal, 2018). Significant research has focused on AM in titanium alloys, revealing that the mechanical properties of AM Ti6Al4V are comparable and, in certain instances, up to 25%

superior to those of conventionally produced Ti6Al4V (Bian et al., 2015; Bertolini et al., 2019; Beyl et al., 2019; Nguyen et al., 2022). In addition, the mechanical properties of Ti6Al4V are dependent on factors including thermal history and cooling rates, resulting in varying phases within the alloy's microstructures (Liu & Shin, 2019; Nguyen et al., 2022).

1.2 Problem statement

The deformation of AM Ti6Al4V at ambient temperatures is well studied (Beyl et al., 2019; Xiao et al., 2019; Nguyen et al., 2022). However, there is limited data available on the deformation behaviour of AM Ti6Al4V at elevated temperatures (Ivanov et al., 2021), especially for hot compression deformation. The high cooling rates and layer-by-layer manufacturing produce fine heterogeneous microstructures in AM-produced Ti6Al4V (Elkhateeb & Shin, 2021). This negatively affects the deformation behaviour as inconsistent mechanical properties may be experienced. Combined with the dual-phase microstructure and the β -transus temperature of Ti6Al4V above 950°C, predicting deformation behaviour at high temperatures is difficult (Hémery et al., 2020; Mai et al., 2020).

1.3 Aim and Objectives

This study aims to investigate the deformation behaviour of AM Ti6Al4V at elevated temperatures of 750 to 900°C using hot compression tests. The deformation behaviour of wrought Ti6Al4V will be compared against AM Ti6Al4V.

The objectives of this research work were:

1. To deform AM Ti6Al4V at high temperatures using a thermo-mechanical simulator to determine its plasticity.
2. To compare the deformation of AM Ti6Al4V to wrought Ti6Al4V at high temperatures using flow stress curves and power dissipation maps.
3. To perform microstructural analyses on AM and wrought Ti6Al4V before and after hot compression tests.

1.4 Research Questions

This study seeks to answer the following research questions:

1. What is the flow stress behaviour of AM Ti6Al4V under compressive deformation within the temperature range of 750 to 900°C?
2. What effect does an increase in the strain rate have on the flow stress behaviour of AM Ti6Al4V under compressive deformation?
3. What effect does the microstructure of AM Ti6Al4V have on the high-temperature compressive strength of the material?
4. How does the high-temperature deformation behaviour of AM Ti6Al4V compare with that of wrought Ti6Al4V?

1.5 Hypotheses

The following hypotheses were addressed:

1. The flow stress of AM Ti6Al4V decreases with increasing temperature in the range of 750 to 900°C.
2. Increasing the strain rate from 0.1 to 10 s⁻¹ leads to higher flow stresses with reduced flow softening in AM Ti6Al4V.
3. AM Ti6Al4V will demonstrate similar or superior mechanical properties to wrought Ti6Al4V.

1.6 Delineations

The following delineations apply to this study:

1. The study was confined to a temperature range of 750 to 900°C and strain rates of 0.1, 1, and 10 s⁻¹.
2. Only the as-built microstructures were considered in this study. No heat treatment was applied.
3. Instead of conducting additional compression tests, constitutive models were employed to verify the flow stress findings.

Chapter 2: Literature review

This chapter provides a comprehensive review of the findings provided in books, journal articles, conference proceedings, and technical reports. A broad review of wrought and AM Ti6Al4V is given, with the characteristics of these alloys highlighted. Previous research on the high-temperature deformation of AM Ti6Al4V is also analysed.

2.1 Ti6Al4V alloys

Ti6Al4V, known as the titanium workhorse, accounts for about 50% of the titanium market (Bodunrin et al., 2021; Pimenov et al., 2021). Its significance can be attributed to its outstanding properties, such as high strength-to-weight ratio, good creep resistance, and high-temperature strength (Mroz et al., 2016; Sui et al., 2019). Low-density Ti6Al4V is also used in structural parts for the aerospace and aircraft industries. For example, Ti6Al4V has been used for approximately 14% of the total airframe structures, which is about 50% that of nickel alloys and stainless-steel alloys (Zhu et al., 2018; Hebzda, 2020). With this combination of excellent properties, it provides high service performance in the aerospace, chemical, biomedical, and aircraft industries.

2.1.1 Metallography of Ti6Al4V

Table 2.1 shows the typical composition of Ti6Al4V. Aluminium stabilises the α -phase and increases the strength and creep resistance of Ti6Al4V. In addition, adding vanadium stabilises the β -phase and increases its ductility and corrosion resistance (Sarao et al., 2018).

Table 2.1: Typical chemical composition of Ti6Al4V (Mohammadhosseini et al., 2013).

Element (wt.%)	Al	V	Fe	O	N	C	H	Ti
Min.	5.50	3.50	0	0	0	0	0	balance
Max.	6.75	4.50	0.30	0.20	0.05	0.1	0.01	balance

The α -phase in Ti6Al4V has a hexagonal close-packed (HCP) crystal structure, and the β -phase is body-centred cubic (BCC). At room temperature, the alloy has $\approx 90\%$ α -phase by volume; hence, its mechanical properties are primarily influenced by the α -phase. An increase in the α -phase will increase the strength, while an increase in the β -phase will increase the formability of the alloy (Séchépée et al., 2024). The amount of β -phase present in the alloy

can be manipulated through heat treatment or high-temperature deformation (Becker et al., 2015). The microstructure of the alloy has a significant impact on the mechanical properties and the overall performance in service. For example, at 850°C, the β -phase can increase to 34% and to 72% at higher temperatures of 950°C (Li et al., 2023). The distribution and morphology of the α - and β -phases give rise to different microstructures of Ti6Al4V. Figure 2.1 shows the three microstructures typical in Ti6Al4V.

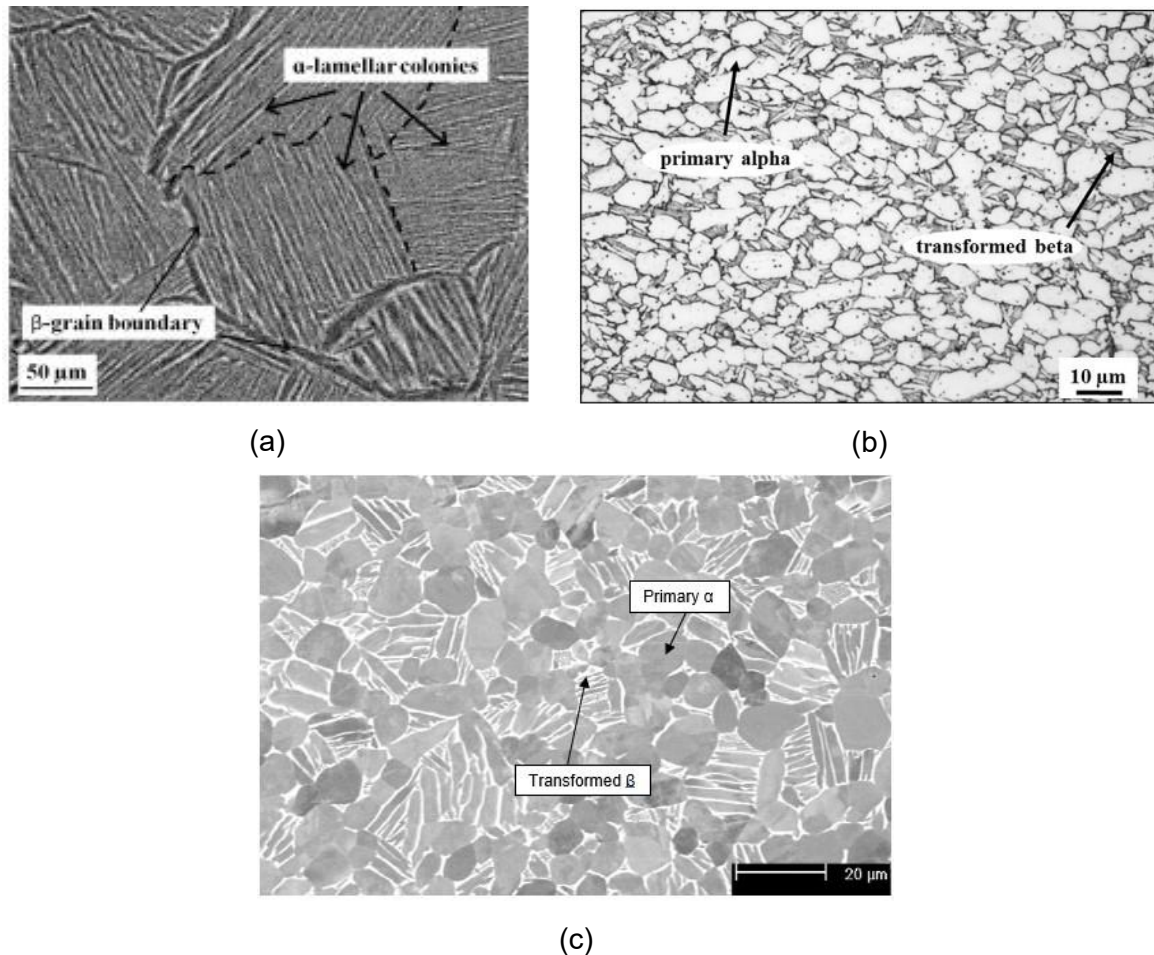


Figure 2.1: Typical microstructures found in Ti6Al4V: (a) lamellar, (b) equiaxed, and (c) bimodal (Spanrad & Tong, 2010; Jopek et al., 2012; Sefer et al., 2016).

A fully lamellar microstructure (Figure 2.1(a)) contains colonies of α -lamellae, with the β -phase at the boundaries (Jopek et al., 2012). Fully lamellar microstructures provide high strength and high fatigue resistance, which is attributed to the coupling effect of crack deflection and twin boundary barrier (Wang et al., 2021). Micro-cracks propagate faster in coarse lamellar microstructures compared to fine lamellar microstructures. Therefore, rapid cooling from the β -phase field creates fine α -lamellae and small α -colonies, which in turn leads to higher strength and fatigue resistance, as small α -colonies are more effective obstacles to dislocation

motion and crack propagation (Bodunrin, 2018). Additionally, the lamellar microstructure is characterised by its high creep resistance.

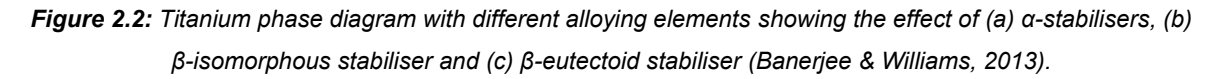
An equiaxed microstructure (Figure 2.1 (b)) is composed of primary α -grains and elongated α needles in a transformed β matrix, where the β -phase is dispersed between the primary α grains (Sefer et al., 2016). The α -grain size in the equiaxed microstructure is as important as the α colony size in fully lamellar microstructures, as it determines the effective slip length. Hence, smaller α -grain sizes favour higher strength, higher ductility, and better resistance to cracks than larger α -grains which, however, have lower creep resistance (Chong et al., 2017). Notably, both equiaxed and bimodal (Figure 2.1(a)) microstructures offer a good balance of strength and ductility at room temperature, making them more widely used in industrial applications (Sieniawski et al., 2013; Chong et al., 2017).

The bimodal microstructure, shown in Figure 2.1(c), consists of equiaxed primary α -phases and transformed β -phases containing lamellar secondary α -phases (Zhao et al., 2023). α -grain size and the alloying element partitioning effect have the greatest effect on the mechanical properties of Ti6Al4V with the bimodal microstructure. With the decrease in the grain size in the bimodal microstructure, more interfaces between primary α -grains and transformed β areas are introduced, and therefore more fractions of pyramidal slip systems could be activated (Chong et al., 2021). Chong et al. (2017) found that the bimodal microstructure exhibited slightly lower strength than the fully lamellar and equiaxed microstructures but a much larger work-hardening ability.

2.1.2 Effect of alloying elements

Titanium is allotropic and will transform from HCP α -phase to BCC β -phase at 885°C. Alloying elements in titanium alloys are classified as α -stabilisers or β -stabilisers, depending on whether the addition of the alloying element raises or lowers the transition temperature from approximately 885°C (Sieniawski et al., 2013; Yadav & Saxena, 2020), as can be seen in Figure 2.2 α -stabilisers tend to stabilise the α -phase and increase the β -transus temperature, while β -stabilisers lower the β -transus temperature (Figures 2.2(b) & (c)). Hence, with the selective addition of alloying elements, it is possible to control the phase composition of titanium alloys and obtain a wide range of material properties. Table 2.2 lists the α - and β -stabilisers typically used in titanium alloys.

The temperature at which the β -transus temperature begins and ends varies depending on the contents of the β -stabilising elements (Sieniawski et al., 2013). This indicates that selectively adding alloying elements to titanium can control its phase composition, resulting in



Effect	Alloying elements
α -stabiliser	Al, N, O, and C, and Ga
β -isomorphous stabiliser	V, Mo, W, and Ta
β -eutectoid stabiliser	Fe, Cu, Mn, Cr, Ni, Co, Si, and H
Neutral elements	Sn, Hf, and Zr

temperature, as shown in Figure 2.2(b). Vanadium is an important addition in Ti6Al4V by increasing the ductility and malleability of the alloy. The other group of β -stabilisers is called β -eutectoid, of which iron is one. These induce a eutectoid reaction, as illustrated in Figure 2.2(c), to produce a two-phase microstructure at room temperature while lowering the temperature at which the α to β transformation takes place (Palka & Pokrowiecki, 2018). Incorporating β -eutectoid elements into a titanium alloy will delay the transformation of β to α through the eutectoid reaction, unlike β -isomorphous elements that completely prevent this transformation (Lin et al., 2016), as can be seen by comparing Figures 2.2(b) and (c).

2.1.3 Phase evolution in Ti6Al4V

Thermo-mechanical processing in the dual-phase region can lead to phase evolution in Ti6Al4V. The phase transformation in Ti6Al4V is strongly influenced by temperature history and cooling rates, which directly govern the resulting microstructure. Rapid cooling, as observed in AM techniques, leads to the formation of α' -martensite through a diffusionless transformation (Krakhmalev et al., 2016). In addition, slower cooling rates, typical of conventional manufacturing methods, facilitate the formation of $\alpha+\beta$ phases through a diffusional process (Krakhmalev et al., 2016).

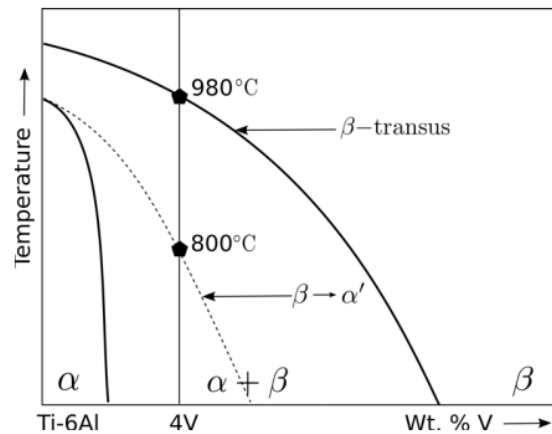


Figure 2.3: Ternary phase diagram of Ti6Al4V with increasing vanadium and temperature (Babu, 2008).

The phase diagram in Figure 2.3 highlights the phase composition of Ti6Al4V as a function of temperature. The transformation of the β -phase to α -phase begins between 960 and 990°C, while the primary α forms through nucleation and growth, and its morphology varies from equiaxed globular to elongated plates (Vilaro et al., 2011; Mai et al., 2020; Prasad et al., 2015). A further decrease in temperature to 800°C, the martensitic transformation temperature (M_s), will cause the transformation of β to α' , which presents as serrated, acicular, and plate-like α -Widmanstätten, or α' -martensite. α' -martensite is formed under a rapid cooling rate, while α -

Widmanstätten is formed under a slower cooling rate. (Liu et al., 2019; Motyka, 2021). The presence of vanadium stabilises the β -phase such that Ti6Al4V contains retained β -phase at room temperature.

2.1.4 High-temperature applications of Ti6Al4V

Ti6Al4V finds widespread use in high-temperature applications such as jet engines, aero engines, and gas turbines due to its high strength at elevated temperatures, low density, high melting point of $\approx 1660^\circ\text{C}$, and good creep resistance (Romero et al., 2015; Liu & Shin, 2019). Replacing nickel-based superalloys with TiAl alloys in gas turbine engines could increase fuel efficiency by 20-30% (Kothari et al. 2012), making titanium alloys highly attractive.

Figure 2.4(a) shows the applications of titanium alloys in aero engines. Ti6Al4V is the most used titanium alloy in the fan, low-pressure compressor, and in some stages of the high-pressure compressor components (Artetxe et al., 2016). These components, some of which are shown in Figure 2.4(b), are typically blisks, disks, and blades that operate at temperatures up to 400°C , typically manufactured of forged Ti6Al4V (Klocke et al., 2014).

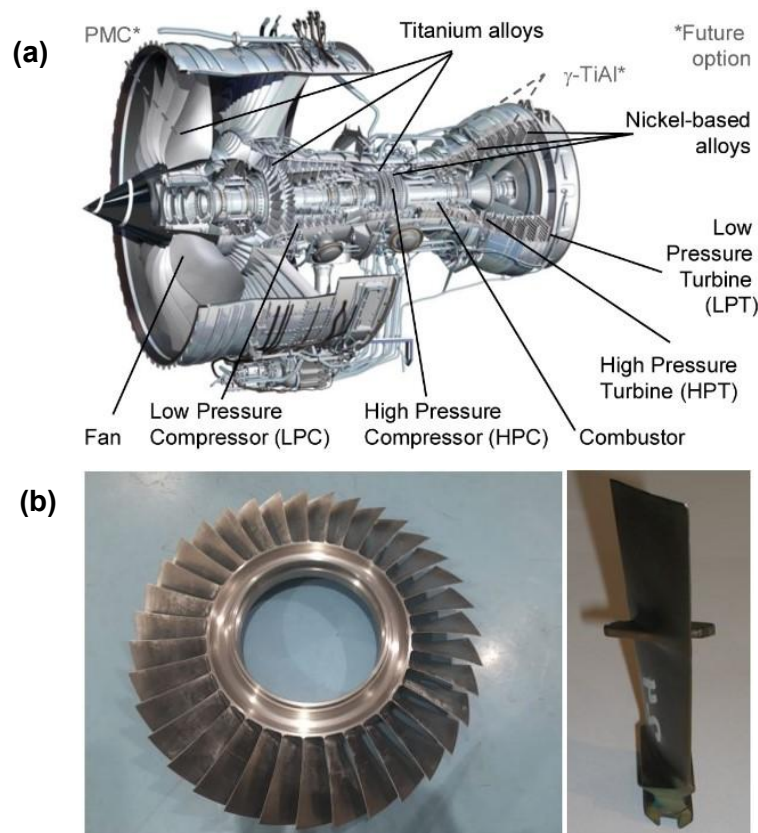


Figure 2.4: Temperature specific applications of Ti6Al4V in aero engines, including a blisk and compressor blade (Kermanpur et al., 2008; Klocke et al., 2014; Swaminathan, 2021).

The demand for advanced aircraft is promoting the development of high-temperature titanium alloys. Advanced aeroplanes are mostly made of titanium alloys, which make up 30%-50% of the total structure weight. For example, in F-22 fighters, titanium alloys make up to 41% of the total weight (Lianggang et al., 2015). In advanced aero-engine and rocket propulsion, high-temperature titanium alloys such as Ti6Al4V improve the thrust-to-weight ratio of the engine and the mobility of the aircraft due to their high specific strength and low density. Low density means that the thrust is higher, and the aircraft can accelerate quicker. In addition, the reduced overall weight of an aircraft decreases fuel consumption and operational expenses.

Ti6Al4V has gained significant interest from the automobile industry due to its lower density compared to steel and its superior high-temperature strength compared to aluminium (Duraiselvam et al., 2014). For example, Ti6Al4V is commonly used in engine valves, connecting rods, and rocker arms, shown in Figure 2.5. Ti6Al4V intake valves typically operate between temperatures of 300 to 400°C (Takahashi et al., 2020).

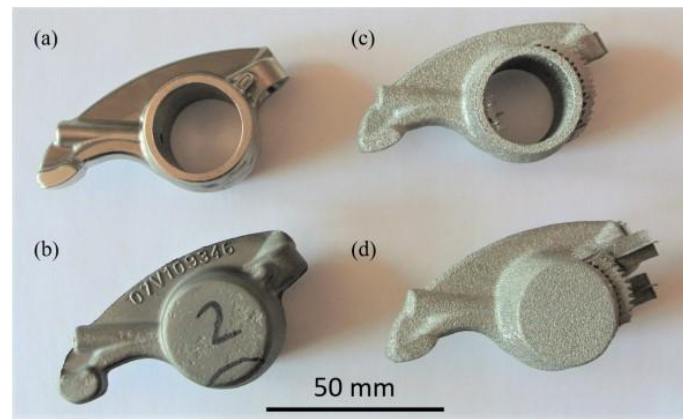


Figure 2.5: Ti6Al4V train rocker arms manufactured through (a & b) FAST-forging and (c & d) SEBM metal additive (Childerhouse & Jackson, 2019).

2.2 Processing Ti6Al4V alloys

The processing of Ti6Al4V in both wrought and AM forms is pivotal in tailoring the properties of Ti6Al4V for specific applications. In the wrought form, Ti6Al4V is typically processed through hot working methods. In addition, AM, particularly SLM and LENS, offers a layer-by-layer approach to building Ti6Al4V components, which allows for the creation of complex geometries that are difficult to achieve through traditional methods. Therefore, the correct manufacturing technique must be used to acquire the desired final part.

2.2.1 Conventional methods for processing Ti6Al4V

Conventional manufacturing processes of Ti6Al4V include casting, forging, and rolling, followed by machining for dimensional accuracy and surface finish. The stock used for these processes typically consists of wrought Ti6Al4V billets, rods, ingots, and slabs that are in the mill-annealed condition and exhibit a variety of microstructures (Bodunrin, 2018), including lamellar $\alpha+\beta$, α -Widmanstätten, α' -martensite, and equiaxed α with a network of intergranular β . The major problems facing conventionally manufactured techniques are high material wastage and low production rate, leading to new methods, such as AM, to replace them.

The as-built microstructure of the wrought Ti6Al4V is defined as a complex or non-uniform microstructure obtained in semi-finished products, comprising equiaxed α grains, elongated α -grains, and equiaxed β -grains (Bodunrin et al., 2019). When wrought Ti6Al4V is heat-treated, such as hot rolling in the $\alpha+\beta$ temperature range and subsequently cooled, an equiaxed microstructure is formed (see Figure 2.6), although complex microstructures have been observed in mill-annealed Ti6Al4V (Becker et al., 2015; Shunmugavel et al., 2015; Bodunrin et al., 2019). Additionally, an acicular microstructure can be formed when cooling from above the β -transus temperature.

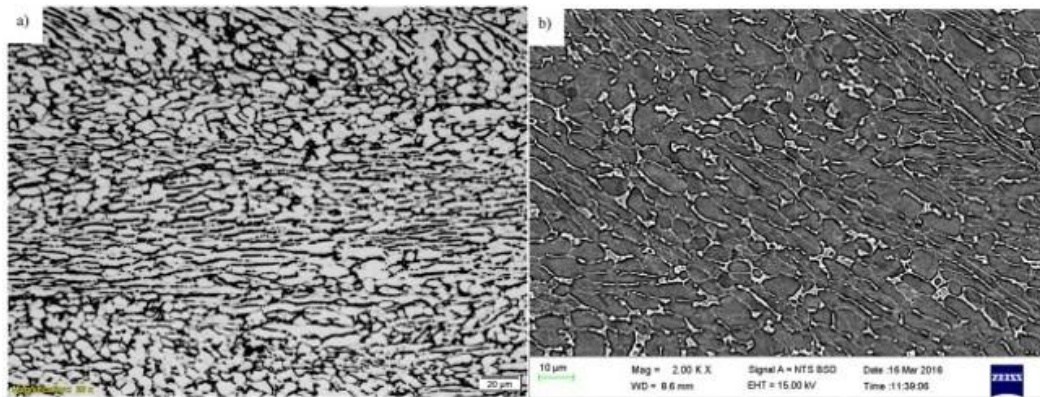


Figure 2.6: Microstructure of the as-received Ti-6Al-4V bar in the mill-annealed condition (a) optical micrograph (b) SEM BSE image (Bodunrin et al., 2019).

2.2.2 Machining of Ti6Al4V

Machining involves creating components to the desired shape and size with accurate tolerances by removing excessive material through a cutting technique. Machining techniques used for Ti6Al4V include drilling, turning, and milling. However, Ti6Al4V has poor machinability because of its low thermal conductivity, high strength at elevated temperatures, high chemical reactivity, and low elastic modulus (Saini et al., 2016).

Among low-density metals, aluminium and magnesium alloys have a much higher thermal conductivity than Ti6Al4V, with 130 W/mK for AA-7075-T6 and 96 W/mK for AZ31 (Vidaller et al., 2014). A low thermal conductivity of 6.7 W/mK means the Ti6Al4V alloy would absorb little heat during machining, leading to a low material removal rate, high cutting temperatures, and an increased amount of heat transfer to the tool rather than the workpiece or chips (Veiga et al., 2013). This can significantly shorten the lifespan of the cutting tool, which may be exacerbated when machining at high speeds. Conventional cutting speeds of Ti6Al4V range from 30 to 60 m/min, during which temperatures can reach more than about 726°C, with higher cutting speeds at 200 m/min increasing cutting temperatures to as high as approximately 1076°C (Pramanik & Littlefair, 2015).

The high strength of Ti6Al4V requires high cutting forces that can be as high as 1000 N (Nouari & Makich, 2013) and can decrease tool life by generating high stresses at the cutting edge and contributing to wear of the cutting tool (Figure 2.7). Tool wear can cause poor surface finish, thus increasing manufacturing costs (Saini et al., 2016; Liang & Liu, 2018), and diminishing product quality. Additionally, the high dynamic shear strength during cutting causes tool notching from the abrasive saw-tooth edges, leading to higher cutting forces and tool wear (Veiga et al., 2013; Saini et al., 2016).

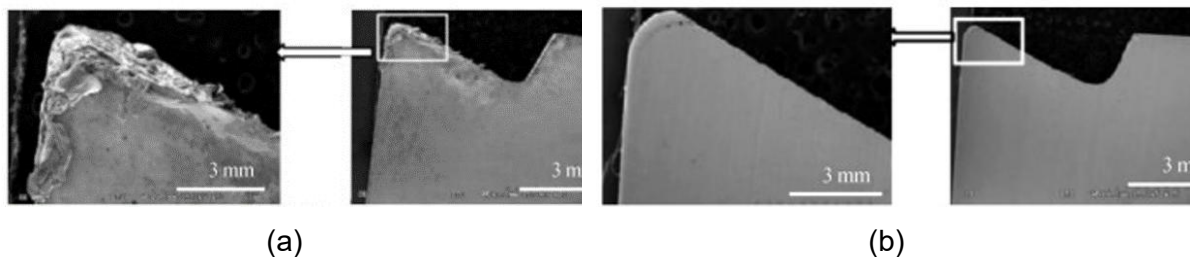


Figure 2.7: Wear during machining of Ti6Al4V (Khettabi et al., 2013).

Titanium alloys are extremely reactive and will react with oxygen, hydrogen, and nitrogen to form oxides, hydrides, and nitrides. This chemical reactivity of titanium causes embrittlement and a decrease in fatigue strength (Veiga et al., 2013), therefore leading to a rough surface finish when machined. Due to the workpiece's high reactivity with the cutting tool, chipping of the surface leads to increased tool wear and poor surface finish (Saini et al., 2016; Liang & Liu, 2018).

The low elastic modulus of Ti6Al4V, about 110 GPa, causes the alloy to deflect and exhibit a spring-back phenomenon during machining (He et al., 2020). This process can result in significant friction and chatter between the workpiece and the tool, leading to poor surface finish (Geetha et al., 2009; Patil et al., 2015). Numerous machining techniques, such as dry

machining, cryogenic cooling, and flood lubrication, have been implemented to machine Ti6Al4V (Gaurav et al., 2021); however, these are expensive and often impractical.

2.2.3 Additive Manufacturing of Ti6Al4V

AM is based on the idea of using computer-aided design (CAD) data, uploading this data onto the machine, and constructing the component layer by layer while adhering to the sliced model data utilising a heat source. Heat sources used in AM include laser, electron beam, or electric arc, and the feedstock can be metal powder or wire (Rawal et al., 2013). Recently, AM has been used to manufacture Ti6Al4V components with a near-net-shaped finish (Li et al., 2022). Figures 2.4, 2.5, and 2.8 show Ti6Al4V parts manufactured by AM. The attractiveness of AM Ti6Al4V lies in its ability to create complex, high-strength components with precision and low material waste.

A combined centrifugal impeller was created, integrating a wrought base plate with an EBM-built component (Figure 2.8). This approach suggested that the integration of wrought and AM parts may reduce overall manufacturing costs and lead times compared to using one method independently (Wang et al., 2017). In addition, this technique can facilitate repairs by enabling the design and addition of new features to existing components.

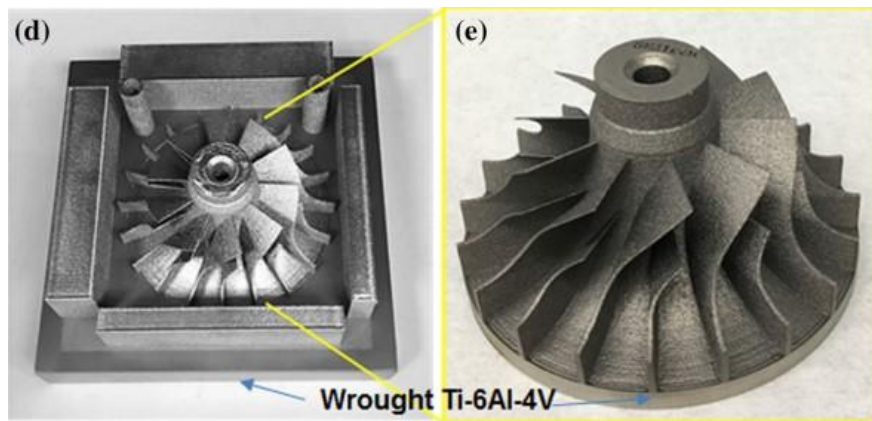


Figure 2.8: (d) Picture of the as-built sample prepared by an Arcam EBM machine and (e) impeller after machining from the wrought Ti-6Al-4V plate (Wang et al., 2017).

AM techniques typically used to manufacture Ti6Al4V are Selective Laser Melting (SLM) and Laser Engineered Net Shaping (LENS) (Zhai et al., 2016; Sola & Nouri, 2019; Xiao et al., 2019). These AM techniques are preferred because they can produce high-density metal parts with superior mechanical properties, leading to near-net-shape components with minimal post-processing. Table 2.3 compares some properties of AM Ti6Al4V to the wrought alloy. These

properties can vary, depending on the processing and deformation history. The AM techniques displayed similar properties with increased strength, hardness, and modulus of elasticity compared to the wrought method. Several authors have found that AM Ti6Al4V exhibits mechanical properties similar to, or even superior to, those of wrought Ti6Al4V alloys (Rawal et al., 2013; Bian et al., 2015; Spigarelli et al., 2022).

Table 2.3: Some properties of Ti6Al4V (Simonelli et al., 2014; Praneeth et al., 2018; Szafrńska et al., 2019; Tao et al., 2019; Pasang et al., 2021; Sarzyńska & paszkowski, 2021; Willemse et al., 2023; Praneeth et al., 2023; Antolak-Dudka et al., 2024).

Property	Manufacturing method		
	Wrought	LENS	SLM
Ultimate tensile strength (MPa)	895	1139	1117
Yield strength (MPa)	825	1080	967
Elastic modulus (GPa)	110	118	117
Density (g/cm ³)	4,43	4,43	4,41
Hardness (HV)	342	380	377

AM is associated with very high cooling rates, about 10^6 K/s, that cause non-equilibrium solidification and promote the formation of fine acicular and lamellar α' -martensite and α -Widmanstätten (Krakhmalev et al., 2016; Arthur, 2019:49; Moridi et al., 2019) as shown in Figure 2.9. These microstructures provide high resistance to dislocation movement such that Ti6Al4V produced by SLM and LENS would exhibit higher strength and hardness (Table 2.3) but lower ductility than wrought Ti6Al4V (Beyl et al., 2019; Nguyen et al., 2022). Therefore, the high strength of AM Ti6Al4V indicates that this material is likely to exhibit high flow stresses when deformed, making it more difficult to deform plastically.

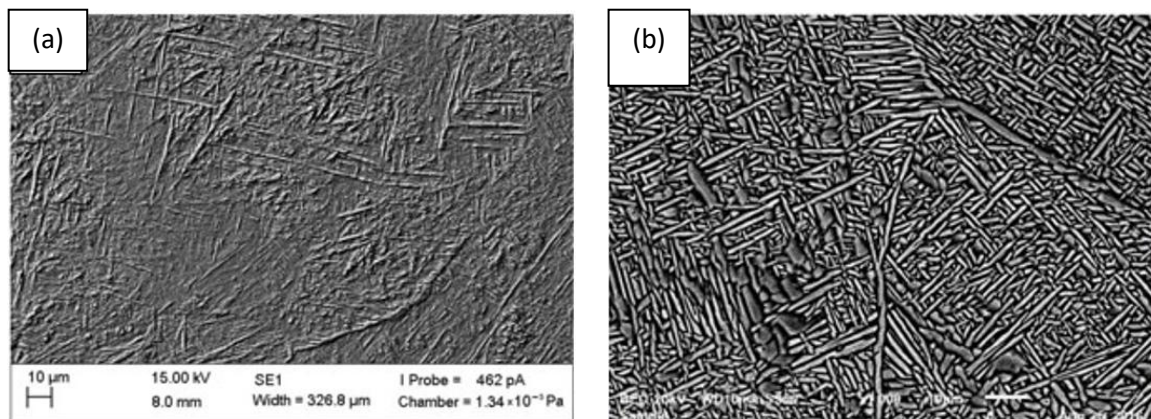


Figure 2.9: Microstructure of Ti6Al4V manufactured by: (a) SLM and (b) LENS (Arthur, 2019; Zochowski et al., 2021).

2.3 Additive manufacturing technologies

AM technologies used for processing titanium and its alloys are divided into direct energy deposition (DED) and powder bed fusion (PBF) methods. These are shown in Table 2.4, with a brief description of each technology given. Complex features, hollow cooling passages, and high-precision parts are best built using PBF technologies; however, these capabilities are limited by the build envelope, limiting the use of only one material per build (Nouri et al., 2021). The DED technologies offer a larger build envelope and higher deposition rate than PBF, but their capacity to create hollow cooling passages and finer geometry is limited (Froes & Dutta, 2014).

Table 2.4: AM technologies for processing titanium and its alloys (Froes & Dutta, 2014).

AM Category	Technology	Description
Direct Energy Deposition (DED)	Direct Metal Deposition (DMD)	Uses laser and metal powder for melting and depositing using a patented close loop process.
	Laser Engineered Net Shaping (LENS)	Uses laser and metal powder for melting and depositing.
	Direct Manufacturing (DM)	Uses an electron beam and metal wire for melting and depositing.
	Shaped Metal Deposition or Wire and Arc Additive Manufacturing (WAAM)	Uses an electric arc and metal wire for melting and depositing.
Powder Bed Fusion (PBF)	Selective Laser Sintering (SLS)	Uses laser and metal powder for sintering and bonding.
	Direct Metal Laser Sintering (DMLS)	Uses laser and metal powder for sintering, melting, and bonding.
	Laser Melting (LM)	Uses laser and metal powder for melting and bonding.
	Selective Laser Melting (SLM)	Uses laser and metal powder for melting and bonding.
	Laser Cusing	Uses laser and metal powder for melting and bonding.
	Electron Beam Melting (EBM)	Uses an electron beam and metal powder for melting and bonding.

2.3.1 Selective laser melting

The SLM technique, shown in Figure 2.10, is based on the placement of a thin layer of metal powder on the build platform, followed by selectively fusing the powder using a high-power laser that either completely or partially melts the powder in its path before re-solidifying and binding together as it cools (Moridi et al., 2019). The CAD data of the part being manufactured controls the layer-by-layer tool path tracing, and the process is repeated until the desired part is produced.

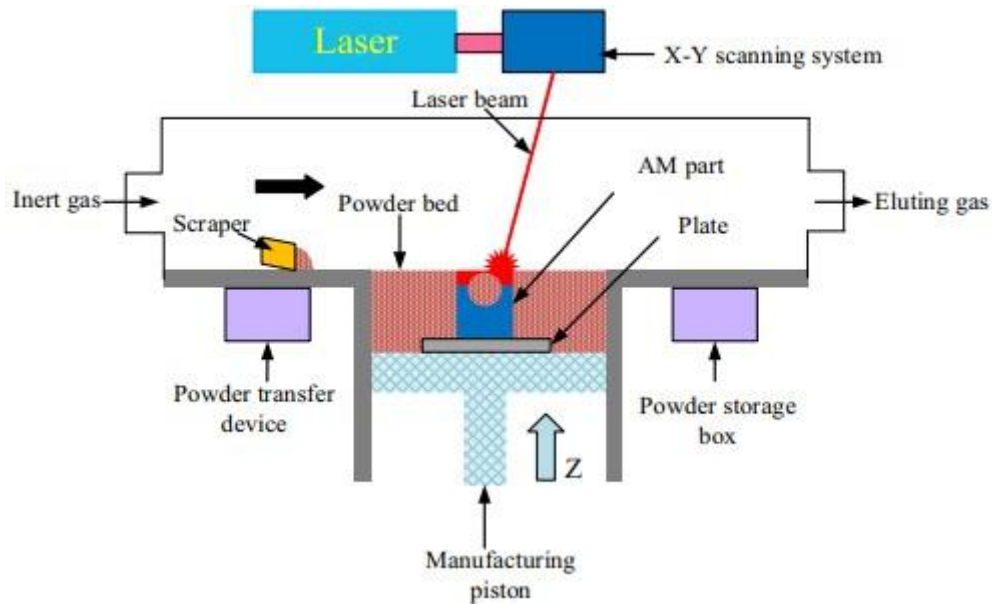


Figure 2.10: Schematic of SLM system (Wang et al., 2021).

SLM is the leading AM technique known to produce high-accuracy and high-density parts with complex geometries, aided using supports during printing, without significantly increasing costs (Harun et al., 2018; Wang et al., 2018). However, SLM has certain limitations, including restricted build sizes due to the powder coating process with rapid heating and cooling rates, which may lead to warping and cracking due to residual stresses (Gao et al., 2023). In addition, the thermal impact of the high-energy laser affects the surrounding powder, altering its physical properties and limiting its potential for reuse (Liu & Shin, 2019).

The typical parameters for processing Ti6Al4V include a layer thickness of 20-100 μm , hatch spacing between 90 and 110 μm , a scan speed of 1-1.4 m/s, and laser power ranging between 250 and 350 W (Praneeth et al., 2023). Argon is typically employed as the shielding gas while the platform may be heated to 200°C, after which the substrate plate is removed, and the samples are sliced using a cutting tool.

2.3.2 Laser engineered net shaping

The LENS manufacturing technique (Figure 2.11) involves injecting powder into the melt pool. A substrate or existing part is placed on the worktable, and a layer of metal is formed as the melt pool solidifies, and the nozzle or tooltip recedes. Successive layers build up the part layer by layer until completion.

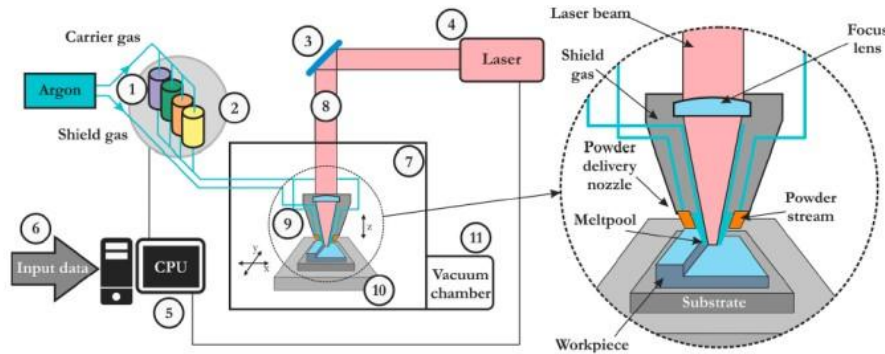


Figure 2.11: Schematic of LENS system (Szafrńska et al., 2019).

The LENS technique is one of the widely used AM techniques, due to its ability to manufacture larger parts with a high density and strength and the ability to modify surfaces or repair components (Harun et al., 2018; Chaudhary et al., 2020). The deposition process benefits from the use of powder-feeding gas coolant, which controls the cooling rate, leading to greater design freedom and quality control (Harun et al., 2018). Consequently, the LENS process is less effective at producing complex components and limited in creating overhanging structures due to the lack of support structures, while exhibiting increased surface roughness compared to SLM techniques (Gao et al., 2023).

The typical parameters for processing SLM Ti6Al4V include a layer thickness of 200 to 1000 μm , hatch spacing between 660 and 900 μm , a scan speed of 0.001-0.04 m/s, and a maximum laser power of 500 W (Liu & Shin, 2019). The LENS process is housed in a chamber purged with argon to limit contamination during deposition. Once the process is complete, the samples are removed using cutting tools, such as wire cutting.

2.4 High-temperature deformation

In high-temperature applications such as in the aerospace industry, Ti6Al4V is preferred because of its high strength and good creep resistance (Sui et al., 2019; Pushp et al., 2022). Ti6Al4V alloys are used to produce many critical components, as explained in Section 2.1.4, such as for disks and blades for high- and low-pressure compressors. During service, these components are always exposed to creep loading due to the sustained exposure to high temperatures (Williams & Boyer, 2020).

Depending on the temperature and stress, different deformation mechanisms are activated, with one mechanism that is dominant, accounting for almost all deformation (Fu et al., 2024). For example, as temperatures rise, many materials show a tendency towards decreased strength and decreased ductility due to mechanisms that occur, such as dynamic recrystallisation (DRX) and dynamic recovery (DRV), in which dislocations move more readily and new, smaller grains develop, resulting in flow softening (Sakai et al., 2014). Flow softening is typically associated with DRX at higher strain rates and lower temperatures, while grain growth is linked to work hardening at higher temperatures and lower strain rates (Fu et al., 2021).

2.4.1 High-temperature deformation of Ti6Al4V

The deformation of Ti6Al4V is sensitive to both temperature and strain rate. Figure 2.12 shows typical stress-strain curves for uniaxial compression testing of wrought Ti6Al4V at strain rates ranging from 0.01 to 10 s⁻¹ between 750 and 950°C. An increase in temperature will decrease the flow stress, decreasing the yield strength and work-hardening capacity (Jiang et al., 2023). This is because elevated temperatures activate dislocation movement by climb and decrease dislocation density as well as the multiplication rate of dislocations, leading to rapid softening and increased ductility (Chen et al., 2015).

With an increase in strain rate, there is an increase in the flow stress of the alloy, implying an increase in the strength. The increase in flow stress with higher strain rates results in faster deformation and generates more dislocations in the material's crystal structure. These dislocations interact and become entangled, making further movement more difficult, which leads to strain hardening and a rise in flow stress (Zhang et al., 2022).

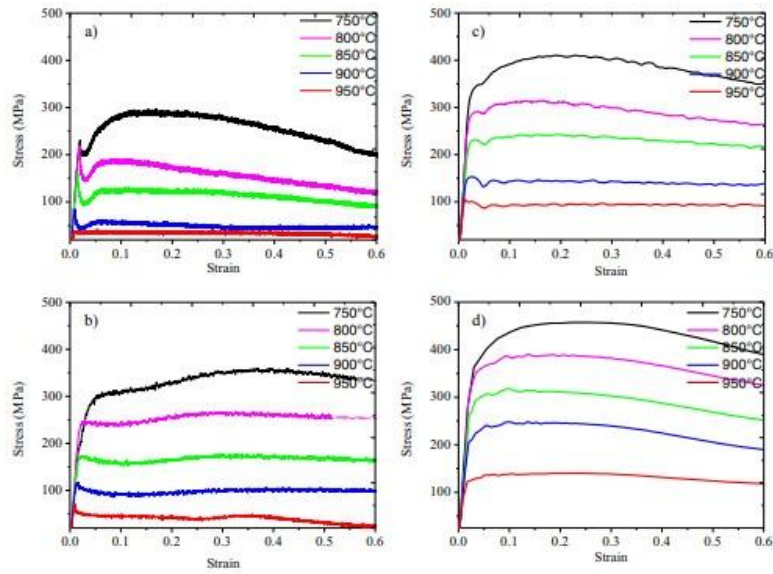


Figure 2.12: True stress-strain curves obtained by uniaxial compression testing of Ti6Al4V in the temperature range 750-950°C and at strain rates of: a) 0.01 s⁻¹, b) 0.1 s⁻¹, c) 1 s⁻¹, and d) 10 s⁻¹ (Bodunrin et al., 2019).

The relative composition of α - and β -phases in Ti6Al4V as a function of temperature is shown in Figure 2.13. The proportion of α -phase decreases with increasing temperature, whereas the proportion of β -phase increases, eventually reaching 100% at the β -transus temperature, which ranges from approximately 960 to 990°C, depending on the elemental weight percentage of each element in Ti6Al4V (Vilaro et al., 2011; Mai et al., 2020; Prasad et al., 2015). This means that the decrease in flow stress with an increase in deformation temperature of Ti6Al4V presented in Figure 2.12 may also be related to the proportion of β -phase at elevated temperatures, since the β -phase has a higher ductility than the α -phase at higher temperatures (Kaschel et al., 2021). Despax et al. (2020) have reported that the volume fraction of β -phase in Ti6Al4V was 12% at 750°C and 52% at 920°C. The BCC β -phase possesses a greater number of active slip systems in operation than the HCP α -phase; although not all slip systems operate concurrently, this increases ductility and work-hardening and can accommodate higher dislocation movement (Séchépée et al., 2024).

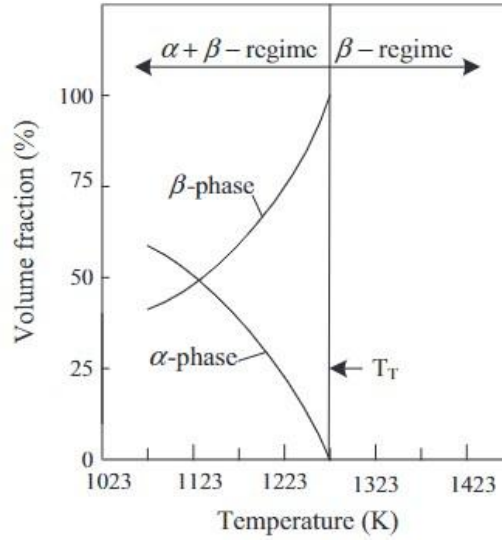


Figure 2.13: The variation in percentages of α -phase and β -phase with temperature for Ti6Al4V (Quan et al., 2015).

The presence of a percolating β -phase during the decomposition of martensite, starting from temperatures $> 400^{\circ}\text{C}$, may be the reason for the reduced flow stress of AM Ti6Al4V at higher temperatures, while the nucleation of the β -phase starts not only at interfaces within individual α' -lamellae boundaries but also at lattice defects within lamellae (Boccardo et al., 2024). Additionally, at higher temperatures, mechanisms such as DRX, diffusion, and grain boundary sliding can become more active, potentially reducing the impact of grain size on the flow stress, making grain boundaries less effective at preventing dislocation movement (Harikrishna et al., 2024; Kareem et al., 2024). In T6Al4V, the mechanism of DRX, which forms globular α grains from the elongated and partially equiaxed α -grains, has been attributed to localised, high-energy shear bands generated within the α -lamellae, which allow the penetration of β -phase at the α/α interfaces and eventually separate the lamellar α grains into smaller segments (Zherebtsov et al., 2011; Zhang et al., 2021). In addition, titanium alloys with thin α lamellae are more easily converted into an equiaxed morphology than those with thick lamellae (Zheng et al., 2022).

Wrought Ti6Al4V generally has a larger α grain size compared to the grain size of AM Ti6Al4V at room temperature; therefore, the AM Ti6Al4V with finer grain sizes will have more grain boundaries and greater resistance to dislocation movement (Beyl et al., 2019). In addition, a-Widmanstätten microstructure typically exhibits larger grains and thicker lamellae compared to microstructures with α' -martensite (Fan et al., 2016). At lower temperatures, such as at room temperature, it is expected that smaller grain sizes indicate an increase in strength, whereas at higher temperatures a finer grain size may make the alloy more ductile and decrease its strength.

At high temperatures, the wrought, LENS, and SLM processes create an equiaxed-type microstructure in Ti6Al4V; however, in AM, the Ti6Al4V microstructures often result in finer sub-microstructures within these equiaxed grains, which differ from the coarser equiaxed grains observed in wrought Ti6Al4V (Motyka, 2021). Therefore, it is expected that the AM Ti6Al4V samples would deform faster at higher temperatures than the wrought Ti6Al4V samples with a decrease in strength, whereas the wrought alloy should have a higher modulus of elasticity and increase in strength as the strain increases but is dependent on the mechanisms active when deformation occurs.

2.4.2 Creep behaviour of Ti6Al4V

Creep is the time-dependent plastic deformation of a material under applied load at high temperatures. Creep typically occurs through dislocation climb, which is aided by diffusion and the accumulation of vacancies, as well as through cross slip and by grain boundary sliding (Abd-Elaziem et al., 2023). Creep may culminate in a fracture, brought about by the formation of cavities at grain boundaries caused by vacancy clustering, with cavity sizes and quantities increasing with an increase in temperature and a decrease in stress (Badea et al., 2014). Creep occurs in three stages: primary creep, characterised by a decreasing creep rate; secondary creep, distinguished by steady-state creep; and tertiary creep, where the material experiences an increasing creep rate that may lead to fracture.

Creep in Ti6Al4V is dependent on the microstructure of the alloy, including the size of both prior β -grain and α -colony, the size of the α lamellae, and the proportion of the α -phase (Sahoo et al., 2018). Since creep occurs predominately by grain boundary sliding, it follows that the fewer the grain boundaries, the higher the resistance to creep (Liu et al., 2023). Hence, coarser prior β -grain, α -colonies, and α lamellae should offer higher resistance to creep as they decrease diffusion rates along grain boundaries (Liu et al., 2023).

Creep in Ti6Al4V is assumed to be regulated by the deformation of the HCP α -phase, which forms more than 90% of the total microstructure at room temperature (Spigarelli et al., 2022), however, its effect depends on the microstructure of Ti6Al4V (Sahoo et al., 2018). For lamellar with an increase in the size of β -grains and α -colonies or a decrease in the thickness of the α -lamellae, while for the bimodal and equiaxed microstructures, steady state creep rate will decrease with decrease in the volume fraction of the α -phase (Sahoo et al., 2018).

2.5 Analysis techniques

High-temperature applications of conventional manufactured and AM alloys, such as in the aerospace, automotive, and energy sectors, demand an understanding of how they deform at elevated temperatures. Techniques used to study the thermomechanical response of materials include high-temperature tensile and compression testing and constitutive modelling techniques such as the Arrhenius-type equation with hyperbolic sine function and power dissipation maps.

2.5.1 Tensile and compression testing

Figure 2.14 shows typical stress-strain curves obtained using high-temperature compression and tensile tests. The force required for compression tests is higher than in tension in the plastic region because of the increase in area under compression, and hence the high flow stress observed in Figure 2.14 (Kubásek et al., 2019). In addition, Figure 2.14 demonstrates that, whether through compression or tensile testing, the results consistently show that the flow stress of the zinc alloy is superior to that of pure zinc metal, with comparable margins of difference between the two (Kubásek et al., 2019). Ayllón et al. (2021) obtained similar results by comparing pure titanium 2 and Ti6Al4V at high temperatures.

The samples loaded in compression do not experience necking when deformed. Therefore, the stress-strain curve of the same material loaded in compression will not exhibit the stress dip typical during non-uniform deformation under tensile loading. In addition, compressive stresses can generate tensile and shear stresses, the latter of which are responsible for dislocation movement and therefore deformation (Yu et al., 2018).

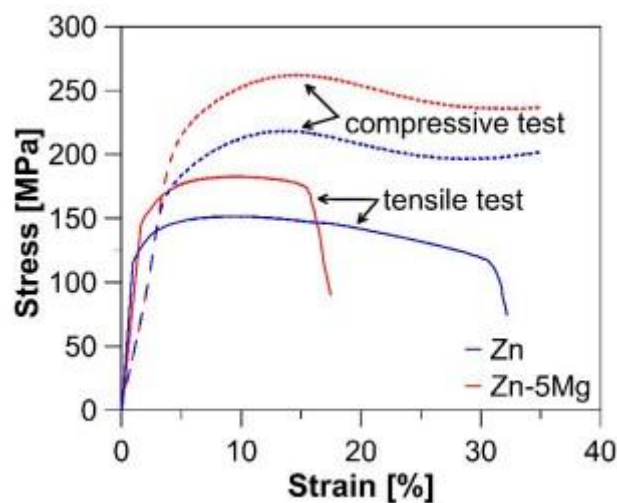


Figure 2.14: Compressive versus tensile stress-strain curves (Kubásek et al., 2019).

Thermo-mechanical deformation testing is a technique designed to understand the mechanical properties of metals by controlling hot deformation parameters. High-temperature deformation of AM Ti6Al4V has been investigated by thermo-mechanical simulators using both tensile tests (Gupta et al., 2021; Ivanov et al., 2021) and compression tests (Saboori et al., 2020; Qi et al., 2023). Gleeble thermo-mechanical simulators, such as the Gleeble 3500[®] thermo-mechanical simulator, have been one of the leading pieces of equipment used for hot compression tests of titanium alloys, such as with Ti6Al4V (Anand et al., 2016; Li et al., 2018; El-Shenawy, 2020). Figure 2.15 shows a typical titanium alloy sample in the Gleeble 3500[®] thermo-mechanical simulator during a compression test at elevated temperature.

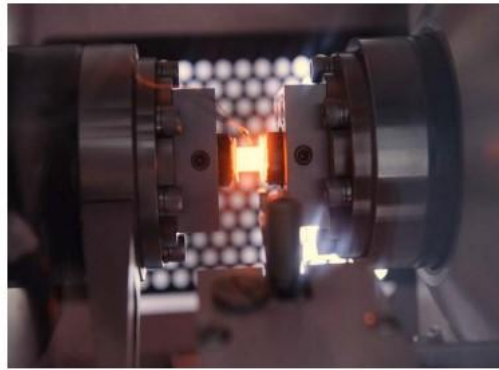


Figure 2.15: Hot compression testing of titanium alloys in the Gleeble 3500[®] thermo-mechanical simulator (Bodunrin, 2018).

Research has demonstrated that understanding material behaviour requires knowledge of the complete stress state, including tensile and compression (Mulay et al., 2016). The advantages of compression testing over tensile testing include (1) simpler sample setup, (2) uniform stress distribution, (3) higher load capacity, and (4) the suitability for brittle materials (Affolter et al., 2015; Baumann et al., 2021). Additionally, where obtaining large tensile creep specimens is difficult, perhaps due to cost, as is the case with Ti6Al4V, small, simple shaped samples can be studied using compression tests (Kim et al., 2018). In compression tests, the aspect ratio (D_R) of a sample is the ratio of the height (h_0) and the diameter (d_0) of the undeformed sample, and it was determined using Equation 2.1.

$$D_R = h_0/d_0 \quad (2.1)$$

According to Roebuck et al. (2006), any D_R value between 1 and 2 may be used, but a D_R value of 1.5 is often preferred. Typical Ti6Al4V compression samples are tested with a height of 12 or 15 mm and a diameter of 8 or 10 mm (Dizlek et al., 2009; Bodunrin et al., 2018; Li et al., 2018), resulting in a D_R value of 1.5. Ultimately, the results from compression tests are just as valuable in understanding high-temperature deformation as those from tensile tests.

2.5.2 Constitutive modelling

Analysis of high-temperature deformation using constitutive modelling techniques involves studying and predicting the mechanical behaviour of materials by employing constitutive models. Constitutive models are, therefore, mathematical formulations between material parameters such as flow stress, strain, and temperature that provide a framework to describe materials' response to different deformation processes.

Arrhenius-type equation with a hyperbolic sine function

The Arrhenius model is typically used to describe the relationship between deformation temperature, flow stress, and strain rate. Saboori et al. (2020) used the Arrhenius model to describe the deformation behaviour of EBM and wrought Ti6Al4V at deformation temperatures ranging from 1000 to 1200°C and strain rates between 0.001 and 1 s⁻¹. In this approach, a hyperbolic sine constitutive equation based on an Arrhenius-type relationship with a temperature-compensated Zener-Hollomon parameter, Z , is used to describe the kinetics of the deformation process and predict the flow stress (Lin et al., 2008; Xiao et al., 2014; Liu et al., 2021).

The effect of strain rate and temperature on the flow characteristics can be expressed by the Zener-Hollomon parameter, also considered as the temperature-compensated strain rate, in the exponential Equation 2.9 (Xiao et al., 2014; Saboori et al., 2020; Liu et al., 2021). The Arrhenius-type equation is expressed in Equation 2.1, where the stress function, $f(\sigma)$, has three main conditions under different stress states, shown in Equation 2.3.

$$\dot{\varepsilon} = A \times f(\sigma) \times \exp\left(\frac{-Q}{RT}\right) \quad (2.2)$$

$$f(\sigma) = \begin{cases} \sigma^{n_1}, & [\alpha_1]\sigma < 0.8 \\ \exp(\beta\sigma), & [\alpha_1]\sigma > 1.2 \\ [\sinh([\alpha_1]\sigma)]^n & \text{for all } \sigma \end{cases} \quad (2.3)$$

In Equations 2.2 and 2.3, $\dot{\varepsilon}$ is the strain rate, σ is the flow stress, n is the stress exponent, Q is the deformation activation energy, R is the universal gas constant (8.314 J/mol), T is the deformation temperature (in K), and α_1 , β_2 , n , n_1 , and A are material constants. To obtain the material constants, Equation 2.3 was substituted into Equation 2.2. The Arrhenius-type equation can then be expressed in three equations following the three different stress states.

$$\dot{\varepsilon} = A1 \times \sigma^{n1} \times \exp\left(\frac{-Q}{RT}\right) \quad (2.4)$$

$$\dot{\varepsilon} = A2 \times \exp([\beta1]\sigma) \times \exp\left(\frac{-Q}{RT}\right) \quad (2.5)$$

$$\dot{\varepsilon} = A \times [\sinh([\alpha1]\sigma)]^n \times \exp\left(\frac{-Q}{RT}\right) \quad (2.6)$$

Based on Equations 2.4 and 2.5, the relationship of $\ln\dot{\varepsilon}$ against σ and $\ln\sigma$ was plotted. The average slope of each curve at the different testing temperatures corresponds to the values of material constant β_1 and $\dot{n}$, which were then used to determine α_1 using Equation 2.7.

$$\alpha1 = \frac{\beta1}{\dot{n}} \quad (2.7)$$

The hyperbolic-sine type can be applied to various temperatures and strain rates. The Zener-Hollomon parameter, Z , was used to describe the kinetics of the deformation process and predict the flow stress. The effect of strain rate and temperature on the flow characteristics was expressed by Z , also considered as the temperature-compensated strain rate, in the exponential Equation 2.8.

$$Z = \dot{\varepsilon} \times \exp\left(\frac{Q}{RT}\right) = A \times f(\sigma) = A \times [\sinh([\alpha1]\sigma)]^n \quad (2.8)$$

The material needs to overcome a specific potential barrier to complete the thermal deformation process, where the potential barrier is the activation energy (Q) during the hot deformation process, and its magnitude reflects the difficulty of dislocation initiation, DRV, and DRX during the hot deformation (Yang et al., 2021). The stress components, n and Q were obtained using Equation 2.9. The value of n was determined as the average slopes of $\ln\dot{\varepsilon}$ against $\ln[\sinh(\alpha\sigma)]$ at the different test temperatures, and the slope average of $\ln[\sinh(\alpha\sigma)]$ against $1000/T$ gave the value of K .

$$Q = RnK = R \left\{ \frac{\delta \ln \dot{\varepsilon}}{\delta \ln [\sinh(\alpha1\sigma)]} \right\}_T \left\{ \frac{\delta \ln [\sinh(\alpha1\sigma)]}{\delta (1/T)} \right\}_\varepsilon \quad (2.9)$$

$$\ln Z = \ln A + n \times \ln [\sinh(\alpha1\sigma)] \quad (2.10)$$

To account for the combined effect of temperature and strain rate, Z was obtained from Equation 2.8 to formulate Equation 2.10. The intercept of the plot of $\ln Z$ against $\ln[\sinh(\alpha\sigma)]$ was used to determine $\ln A$ using the intercept of the line. Using the definition of the hyperbolic sine function to solve Equations 2.8, the predicted flow stress can be written as a function shown in Equation 2.11 of the Z parameter.

$$\sigma = 1/\alpha_1 \left\{ \left(\frac{Z}{A} \right)^{\frac{1}{n}} + \left[\left(\frac{Z}{A} \right) + 1 \right]^{\frac{1}{2}} \right\} \quad (2.11)$$

The statistical root mean square error (RMSE), the percentage of the average absolute relative error (AARE), and the correlation coefficient (R^2) were used to evaluate the predictability of the constitutive equation (Li et al., 2019). These variables are expressed as:

$$AARE (\%) = 1/N \times \sum_{i=1}^N \left| \frac{M_i - P_i}{M_i} \right| \times 100 \quad (2.12)$$

$$RMSE (MPa) = \sqrt{1/N \times \sum_{i=1}^N (M_i - P_i)^2} \quad (2.13)$$

$$R^2 = \frac{\sum_{i=1}^N (M_i - \bar{M})(P_i - \bar{P})}{\sqrt{\sum_{i=1}^N (M_i - \bar{M})^2 \times \sum_{i=1}^N (P_i - \bar{P})^2}} \quad (2.14)$$

where M and P are the measured and the predicted flow stresses, $\bar{M}$ and $\bar{P}$ are the mean measured and predicted flow stress values, and N is the total number of measurements. After comparing the calculated flow stress and experimental flow stress, statistical parameters such as the root mean square error (RMSE), the percentage of the average absolute relative error (AARE) and correlation coefficient (R^2) values are calculated to measure the predictability of the model. RMSE is the prediction error representing the standard deviation of the residuals, while AARE is the magnitude of the difference between the calculated and experimental flow stresses.

Low values of RMSE and AARE indicate that the calculated flow stress correlates with the measured flow stress. The smaller the AARE and RMSE values, the stronger the reliability of the prediction is. Values for R^2 are measured between 0 and 1, with values closer to 1 indicating that the regression predictions perfectly fit the data. The value of R^2 is commonly used to provide information regarding the linear relationship between the predicted and calculated flow stresses (Liu et al., 2021).

Power dissipation maps

The basis for modelling dynamic material behaviour is viewing physical systems as energy manipulators, where they transfer power obtained from the source to the material. In this research study, the dies of the Gleeble 3500® thermo-mechanical simulator were used to transfer power to the material. The absorbed energy is primarily dissipated through two mechanisms: first, the generation of viscoplastic heat due to high temperatures and strain rates, which is released as thermal energy, and second, the microstructural evolution, which dissipates energy through processes such as DRV, DRX, void formation, and phase transformation (Jiang et al., 2023).

The material in this case functions as the workpiece undergoing deformation and is regarded as a system that reacts to the power applied to it during deformation. Power dissipation maps can be represented by a contour plot that depicts the efficiency values of power dissipation achieved under various strain rates and temperature conditions. The power dissipation values indicate the rate of entropy production because of microstructural changes at a given strain under the applied strain rate and temperature (Bodunrin, 2018).

Different iso-efficiency values are dissipated by the various microscopic phenomena that occur during hot deformation, with certain microscopic phenomena advantageous during hot deformation, whereas others are detrimental. Therefore, the power dissipation values can be correlated to the microstructural evolution in the deformed samples. The strain rate sensitivity parameter (m), defined as the increase in stress needed to cause a specific increase in plastic strain rate at a given temperature, was calculated as follows:

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \quad (2.15)$$

The slope of the $\ln \sigma$ versus $\ln \dot{\epsilon}$ plot was used to determine m by Equation 2.15 (Prasad et al., 2015). As suggested by Bodunrin (2018) a cubic spline fit was used to determine the intermediate data point for m as a function of strain rate in the test temperature range 750 – 900°C (Table 3.2). This was done using a SRS1 cubic spline function in Excel. The efficiency of power dissipation (η), in terms of m , was calculated using Equation 2.16 (Prasad et al., 2015).

$$\eta = \frac{2m}{m + 1} \quad (2.16)$$

When compared to microscopic phenomena such as flow localisation, cracking, kink bands, adiabatic shear bands, and other detrimental microscopic phenomena, DRV, DRX, and superplasticity dissipate power with unique efficiencies and are advantageous microscopic occurrences (Prasad et al., 2015). The following was reported by Prasad (2003) and Prasad et al. (2015) between the power dissipation values and the corresponding mechanisms in titanium alloys:

- Power dissipation efficiency below 20% indicates possible flow instabilities.
- DRV correlates to power dissipation efficiency values between 20 and 30%.
- Power dissipation values between 35 and 50% are typical of DRX or globularisation.
- Superplasticity and wedge cracking domain correspond to power efficiency values above 60% although DRX has been reported to dissipate power efficiency above 50%.

Prasad et al. (2015) created an instability criterion based on the extremum principle of irreversible thermodynamics to distinguish between advantageous and detrimental microscopic occurrences during material processing. A processing map is a result of superimposing two maps: one of power dissipation efficiency and the other of instability. An example of a processing map of Ti6Al4V is shown in Figure 2.16, where the red rectangles illustrate the domains, and the shaded area shows the instability regions. In this research work, only the power dissipation maps were used to determine the deformation mechanisms. This enabled the identification of mechanisms responsible for microstructural changes in wrought and AM Ti6Al4V at elevated temperatures and was used to discuss the flow behaviour of the stress-strain curves obtained via hot compression tests.

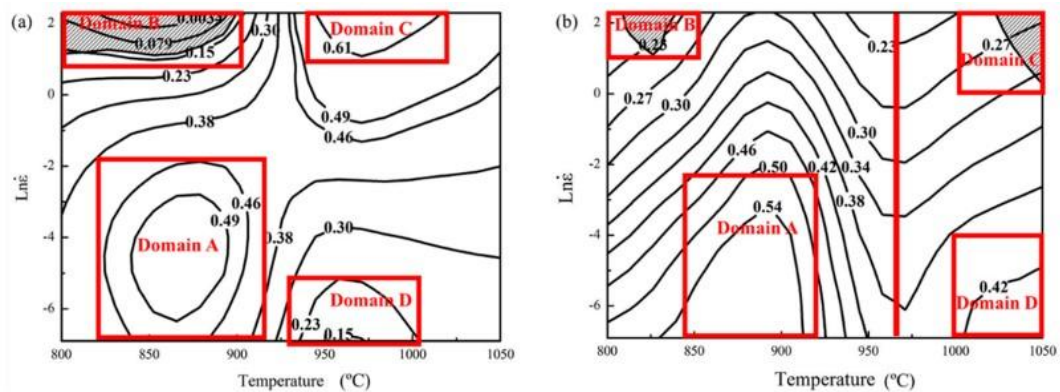


Figure 2.16: Processing maps for Ti6Al4V alloy derived based on (a) power law constitutive equation and (b) Arrhenius-type constitutive equation (Hu et al., 2018).

2.6 Gaps in literature

Several studies have reported on the high-temperature tensile test behaviour of AM-produced Ti6Al4V (Zhao et al., 2019; Ladani et al., 2023; Shah et al., 2024). However, the compressive response of the AM-produced material has not been adequately investigated. Temperatures commonly studied range from 600 to 750°C (Kim et al., 2018; Tao et al., 2019), which is the temperature range used for stress relief. Thus, this study selected a temperature range above that of the stress relief range, but still fell below the β -transus, such that the formation of more β -phase is avoided. Tensile and compression tests are commonly performed at room temperature using conventional machines. However, a Gleeble thermo-mechanical simulation machine can mimic the required high-temperature range and service environment (El-Shenawy, 2020; Chen et al., 2022). Thus, a Gleeble thermo-mechanical simulation machine was selected for experimental testing.

2.7 Summary

This review of literature found that high-temperature deformation of Ti6Al4V is highly dependent on the amount of the α -phase present, while the volume fraction of β -phase increases with the increase in temperature. AM Ti6Al4V exhibit similar or even superior mechanical properties than conventionally manufactured Ti6Al4V. In addition, compressive tests have been used widely to try and understand high-temperature deformation of alloys, while the compressive deformation of AM Ti6Al4V at elevated temperatures is limited.

Chapter 3: Materials and experimental procedure

This chapter gives details on the materials, facilities, experimental procedures, and characterisation techniques used to achieve the objectives of this study, as well as a detailed description of the hot compression testing done using the Gleeble 3500® thermo-mechanical simulator. The methods used to process and analyse the stress-strain data obtained from the hot compression tests are also described.

3.1 Overview of experimental procedures

The objectives of this study were achieved through a series of experimental procedures summarised in Figure 3.1. The sample preparation included cutting, mounting, grinding, polishing, and etching of the samples. In addition, the constitutive modelling techniques included an Arrhenius-type equation with a hyperbolic sine function and power dissipation maps.

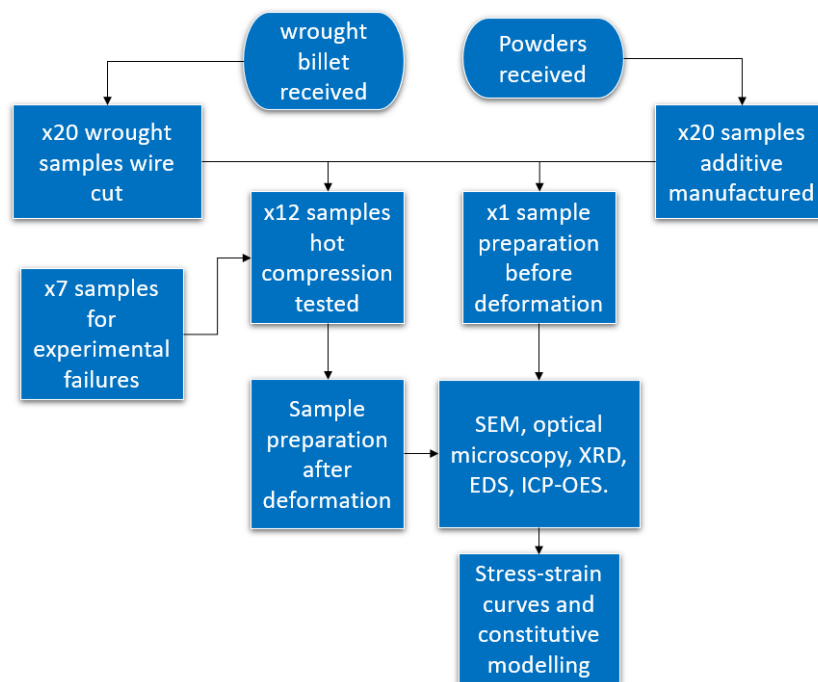


Figure 3.1: Overview of experimental procedure.

3.2 Materials

The AM Ti6Al4V samples used in this research work were prepared using the SLM and LENS techniques described in detail in Sections 2.3.1 and 2.3.2. The starting powders were supplied pre-alloyed and were used as received. Their chemical composition was given as approximately 6.34% aluminium, 3.94% vanadium, and 89.38% titanium with small percentages of iron, oxygen, carbon, nitrogen, and helium, and was confirmed using ICP-OES. The wrought Ti6Al4V used was supplied as a billet and machined with an electric discharge wire cutting machine. It was used as received, and its chemical composition was also confirmed by EDS. Phase analysis of the LENS and SLM powders, as well as the AM and wrought samples, was done using XRD.

3.2.1 Additive manufacturing Ti6Al4V

AM of the Ti6Al4V samples was done at the CSIR Photonics Centre in Pretoria, South Africa, using an Optomec LENS® 850-R system and an in-house developed SLM machine through PBF technology. Argon was employed as the carrier gas for the LENS technique, with a gas flow rate of 4 L/min. The carrier gas for the SLM was argon, and the process started when the oxygen content in the chamber dropped below 1000 ppm. Both techniques were printed on a wrought grade 5 Ti6Al4V baseplate, and no preheating was done in either case. The parameters used to produce the LENS and SLM Ti6Al4V samples are given in Table 3.1. These parameters were optimised through experimental trials to produce Ti6Al4V samples with the highest relative density.

Table 3.1: Parameters used to manufacture the SLM and LENS Ti6Al4V samples.

Parameters	SLM Ti6Al4V	LENS Ti6Al4V
Laser power (W)	1300	240
Scan speed (m/s)	3	0.01016
Layer thickness (µm)	50	200
Hatch distance (µm)	100	550
Scan strategy	No contour, bi-directional	1 contour, bi-directional

Twenty samples were manufactured with each method, and 12 of those were used for hot compression testing. The remaining samples were characterised for chemical composition, hardness testing, and microstructural analysis. The samples were manufactured using the D_R of 1.5, calculated using Equation 2.1. The AM samples used in this study were cylindrical with a height and diameter of 12 and 8 mm, respectively, while wrought samples had a height and

diameter of 15 and 10 mm. The test samples, apparatus, and procedure for axial-load compression testing at room and elevated temperatures were designed in accordance with ASTM E209 and ASTM E9.

3.2.2 Sample preparations

The Ti6Al4V samples were sectioned horizontally and vertically and then prepared for metallographic analysis and hardness testing. A Struers alumina cut-off wheel fitted on a Struers Labotom-S cutting machine was used to cut the samples at a speed of 2845 rpm, and water was used as the coolant. The cut samples were mounted in ClaroFast resin using an AMP 50 automatic mounting press. This process was done at 180°C with a maximum pressure of 2 MPa for 10 minutes, with a cooling temperature of 40°C.

Sample grinding and polishing was done using the Struers TegraPol-25 and TegraPol-31 machines, both with a Struers TegraForce-5. Grinding was performed using abrasive silicon carbide (SiC) paper of progressively decreasing grit sizes from P120 to P4000, with water as the coolant. For polishing, MD Chem (magnetic Chem) polishing cloths were used together with a 0.25 µm oxide polishing suspension, OP-S Suspension.

Kroll's reagent, consisting of 3 ml of HF, 6 ml of HNO₃, and 100 ml of distilled water, prepared in accordance with ASTM E407-187/192. After etching, the samples were cleaned with ethanol and water and finally dried with air. This etching process was done in a fume hood and with adequate protective equipment such as goggles, closed shoes, long pants, nitrile (22 ml) gloves, and acid-resistant aprons. Upon completion of the etching process, the used Kroll's reagent was poured into a chemically compatible sealed container, with proper labelling, and kept in a dedicated space for waste chemicals. The metallographic preparation detailed here was done on both the wrought and AM Ti6Al4V samples before and after deformation in the Gleeble 3500® thermo-mechanical simulator.

3.3 Compression testing

The Gleeble 3500® thermo-mechanical simulator at the University of the Witwatersrand, School of Chemical and Metallurgical Engineering, was used for compression tests. Figure 3.2 shows the Gleeble 3500® thermo-mechanical simulator and the position of the samples during the tests. Before the tests, a type K chromel-alumel thermocouple was spot welded to the mid-span of the samples to be placed in the Hydrawedge autoloader to sense the actual temperature during the deformation process.

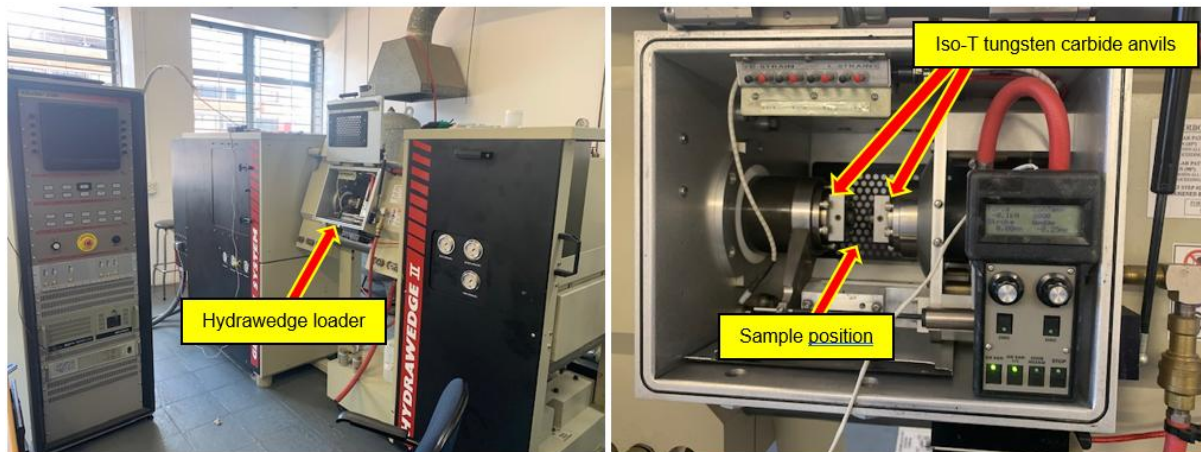


Figure 3.2: Gleeble 3500® thermo-mechanical simulator of the University of the Witwatersrand.

The Ti6Al4V samples were then mounted in the Hydrawedge autoloader as shown in Figure 3.2. Graphite foil and nickel paste were placed between the Iso-T tungsten carbide anvils and the samples to reduce friction during the hot compression deformation. For each compression test, the samples were heated directly to the deformation temperature at a rate of 5°C/s and held for 180 seconds to homogenise them. Following this, the samples were deformed at a predetermined strain rate and then cooled with compressed air. The isothermal compression schedule for hot compression testing used for the experiments is shown in Figure 3.3.

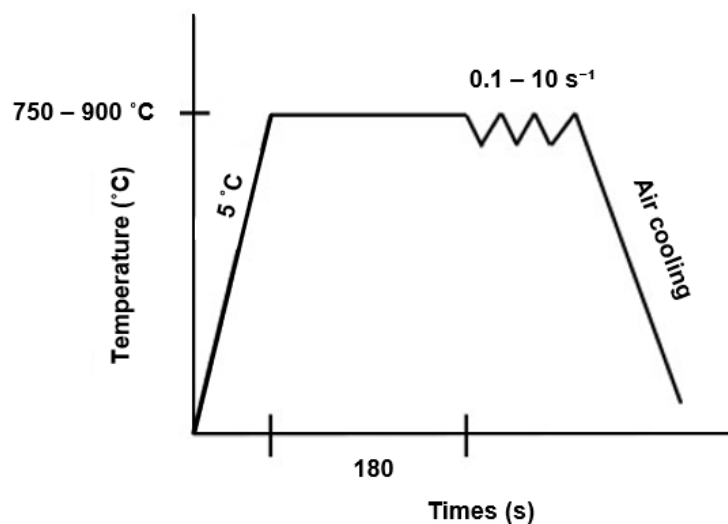


Figure 3.3: Schematic diagram of the isothermal compression testing schedule.

The compression tests were done at temperatures ranging from 750 to 900°C, with increments of 50°C. In this temperature range, Ti6Al4V has a good combination of strength, hardness, creep resistance, and toughness due to the $\alpha+\beta$ phase (Sabban et al., 2019; Pushp et al., 2022). It is typical to correct the flow stress obtained from the experiments for adiabatic heating

to obtain a more accurate prediction of deformation behaviour from processing maps. However, correction for adiabatic heating was not required in this study because the temperature deviation from the preset temperatures did not exceed 30°C. Deformation was done at strain rates of 0.1, 1, and 10 s⁻¹.

Table 3.2 shows the deformation condition for the 12 samples tested per Ti6Al4V manufacturing method (Section 3.2.1). Figure 2.15 shows a typical titanium alloy sample in the Gleeble 3500® thermo-mechanical simulator during a compression test at elevated temperature, while Figure 3.4 shows the setup of the Ti6Al4V samples before hot compression testing. No repetitions of the tests were conducted, as the results were validated using constitutive laws.

Table 3.2: Hot compression testing parameters for the commercial and experimental alloys.

Deformation parameters	Temperature (°C)			
	750	800	850	900
Strain rate (s ⁻¹)				
0.1	Sample 1	Sample 2	Sample 3	Sample 4
1	Sample 5	Sample 6	Sample 7	Sample 8
10	Sample 9	Sample 10	Sample 11	Sample 12

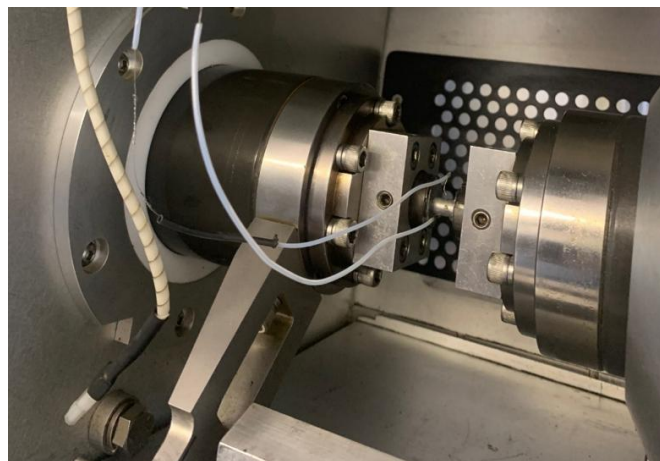


Figure 3.4: Setup of Ti6Al4V sample in the Gleeble 3500® thermo-mechanical simulator.

The results obtained from the Gleeble 3500® thermo-mechanical simulator were converted to true stress-strain curves and power dissipation maps for comprehensive analysis. The stress-strain curves were drawn using Excel, while the power dissipation maps were developed using Origin 2024 software. Additionally, the Arrhenius-type equation with a hyperbolic sine function was calculated using Excel.

3.4 Sample characterisation

The density of the Ti6Al4V samples was measured using an Ohaus explorer[®]. Five AM samples and one wrought Ti6Al4V were tested. The weight of the samples was measured in air and then ethanol, and the density was calculated using Archimedes' principle in Equation 3.1. The density of ethanol is 0.79 g/cm³ and the test temperature was measured as 16.8°C.

$$\frac{\text{Density of sample}}{\text{Density of fluid}} = \frac{\text{Weight in air}}{\text{Weight in air} - \text{Weight in fluid}} \quad (3.1)$$

Porosity of the Ti6Al4V samples was analysed by optical microscopy. An Olympus BX51M optical microscope was used, and the total percentage of porosity was determined using ImageJ software. A stream report was obtained to establish two phases, one phase showing the porosity fraction percentage and the other the material fraction percentage.

The microstructural analysis of both the wrought and AM Ti6Al4V was conducted before and after the hot compression tests. An Olympus BX51M optical microscope and an Olympus GX-51 optical microscope were used at magnifications ranging from 5x to 100x. Additional analysis was done using the FEI Quanta 250 FEG SEM and JSM-IT200 SEM, both equipped with EDS. An accelerating voltage of 20 kV was used for both imaging and EDS.

XRD measurements were done using a PANalytical X'pert Pro instrument with an iron-filtered cobalt K_α radiation source at the North-West University Laboratory for Analytical Services. The measurements were carried out within the range of 2θ = 10-110°, and the results obtained were analysed using X'Pert Highscore software. XRD analysis was done on the LENS and SLM powders as well as on the AM and wrought samples. All XRD analyses were done on the samples before hot compression tests.

The hardness of the wrought and AM Ti6Al4V samples before and after the deformation test was measured using a Matsuzawa Vickers micro-hardness tester fitted with a diamond indenter. The measurements were taken using a force of 300 gf and a dwell time of 10 s, consistent with ASTM E92-17. At least six indentations, each in different positions, were taken on the Ti6Al4V samples, and the average HV_{0.3} value was used to characterise hardness. Care was taken to space out the indents to minimise the effects of strain hardening.

3.5 Analysis techniques

Typical true stress-strain curves developed using data from the Gleeble 3500® thermo-mechanical simulator were used to analyse the hot deformation behaviour of the wrought and AM Ti6Al4V samples at different temperatures and strain rates. The true stress-strain curves were used to develop constitutive equations, and this analysis was complemented by the Arrhenius-type equation with a hyperbolic sine function and processing maps.

3.5.1 Arrhenius-type equation with a hyperbolic sine function

The Arrhenius-type equation with a temperature-compensated strain rate Zener-Hollomon parameter (Z) was used to demonstrate the correlation between deformation temperature, strain rate, and flow stress. In addition, the average activation energy was calculated to determine the energy required to facilitate microstructural changes. The calculations followed, and the recommended values for the statistical tools are shown in Section 2.5.2.

3.5.2 Power dissipation maps

Power dissipation maps were used to describe dynamic material behaviours during high-temperature deformation. Hot compression tests were performed to determine the deformation patterns. The values obtained in Equations 2.15 and 2.16 in Section 2.5.2 were used to plot power dissipation maps for the hot deformation of Ti6Al4V using Origin 2024 software.

Chapter 4: Results and discussions

This chapter presents results obtained from isothermal compression tests using the Gleeble 3500[®] thermo-mechanical simulator and the material characterisation of the AM and wrought Ti6Al4V done using, among other techniques, XRD, SEM and optical microscopy. The deformation behaviour of Ti6Al4V at temperatures ranging from 750 to 900°C was evaluated using stress-strain curves and constitutive modelling techniques, namely the Arrhenius-type equations and processing maps. Section 4.4 of the chapter presents the discussion and interpretation of the results and their possible implications for the deformation of AM Ti6Al4V at elevated temperatures.

4.1 Pre-deformation characterisation

This section presents the results of the chemical composition, density, porosity, hardness, microstructural and phase analyses of the wrought and AM Ti6Al4V samples obtained before hot compression testing. In addition, the results were compared to literature to validate the results.

4.1.1 Compositional analyses

Table 4.1 shows the chemical composition of the LENS and SLM powders, as well as the wrought and AM Ti6Al4V samples before deformation, as determined using ICP-OES for the powders and EDS for the samples. For the most part, the aluminium and vanadium in all the tested samples were within specifications, as given in Table 2.1. Vanadium was between 3.50 and 4.50 wt%, while aluminium was found to be between 5.50 and 6.75 wt%. Titanium was taken as the balance, which was mostly less than 87.20 wt%.

Table 4.1: Composition of the Ti6Al4V alloys before deformation.

Ti6Al4V	Composition (wt %)					
	O	Al	Ti	V	Fe	C
Wrought	3.85	5.87	84.33	4.15	-	2.22
SLM powder	-	5.40	82.60	4.20	-	-
LENS powder	-	5.60	80.00	4.00	-	-
SLM	3.68	5.65	86.59	3.82	0.26	-
LENS	2.90	5.58	87.20	4.02	0.29	-

As can be seen in Table 4.1, all the Ti6Al4V samples tested showed notably high amounts of oxygen, which were more than the maximum of 0.2 wt% specified in Table 2.1. The high oxygen content in the Ti6Al4V samples reported in Table 4.1 was probably due to titanium having a high affinity for oxygen (Kim et al., 2018). Only titanium, aluminium, and vanadium were measured for the powders using ICP-OES, which had a profound effect on the microstructure. Oxygen and iron were not measured and were taken as supplied, as it was not expected that they would have a major impact on the mechanical properties of the samples, and ICP-OES could not reliably measure the oxygen composition. The wt% of the oxygen and iron for the SLM powder was 0.18 and 0.21%, while the LENS powder was 0.08 and 0.25%, respectively. However, the carrier gas used when manufacturing and testing likely limited this oxygen to the Ti6Al4V samples' surface.

The wrought Ti6Al4V sample had about 2.22 wt% carbon content (Table 4.1), the source of which was not immediately obvious. Table 2.1 specifies a maximum carbon content of 0.1 wt%. Small amounts of iron were observed in the AM Ti6Al4V samples, but these were less than the maximum of 0.30 wt% specified in Table 2.1. While iron is a known β -eutectoid stabiliser (Table 2.2), amounts greater than or equal to 2.5% (Bak et al., 2019) would be required to induce eutectoid reactions and lower the α to β transformation temperature.

4.1.2 Density and porosity

The densities of wrought, SLM, and LENS Ti6Al4V samples were determined using Archimedes' principle in Equation 3.1 and were 4.431, 4.411 ± 0.04 , and 4.412 ± 0.03 g/cm³, respectively. These values were in good agreement with those found in literature (Table 2.3) with percent errors < 1%. Densities of the LENS and SLM samples were similar, indicating that the manufacturing methods produced similarly dense materials. Although AM alloys typically exhibit higher porosity percentages compared to conventionally manufactured parts, the densities of the AM Ti6Al4V samples were lower than that of wrought Ti6Al4V, consistent with some typical AM parts (Al-Rubaie et al., 2020). Figure 4.1 shows the percent porosity in the AM Ti6Al4V samples.

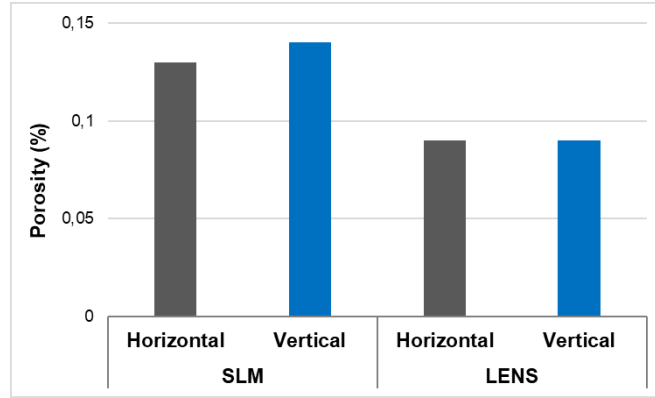


Figure 4.1: Percentage porosity of the Ti6Al4V SLM and LENS samples before deformation.

Figure 4.1 reveals that the percent porosity for the SLM and LENS Ti6Al4V samples was less than 0.15%. This significantly small percentage of porosity suggested that any pores present in the Ti6Al4V samples were unlikely to impact their behaviour during high-temperature deformation. Further analysis of the results presented in Figure 4.1 indicates that the relative densities of AM Ti6Al4V samples were quite high, ranging between 99.86 and 99.91%. These results aligned well with those of previous studies (Wang et al., 2018; Mahmud et al., 2021; Singla et al., 2021), which achieved relative densities greater than 99% for AM Ti6Al4V.

The micrographs in Figure 4.2 show the pores typically observed on the SLM and LENS Ti6Al4V samples. Most of the observed pores were due to lack of fusion, a condition prevalent in AM components (Ghods et al., 2021), which may have been caused by operating at high energy densities and attributed to unforeseen thermal distortion of the part (Collins et al., 2016). Pores due to trapped gases were also noted. In AM of titanium alloys, these types of pores are typically attributed to gas trapped in the raw metal powder particles or inert gas, such as argon, used as the shielding gas. The pores in the SLM and LENS Ti6Al4V samples produced in this study were observed to range between 44 and 50 μm in size.

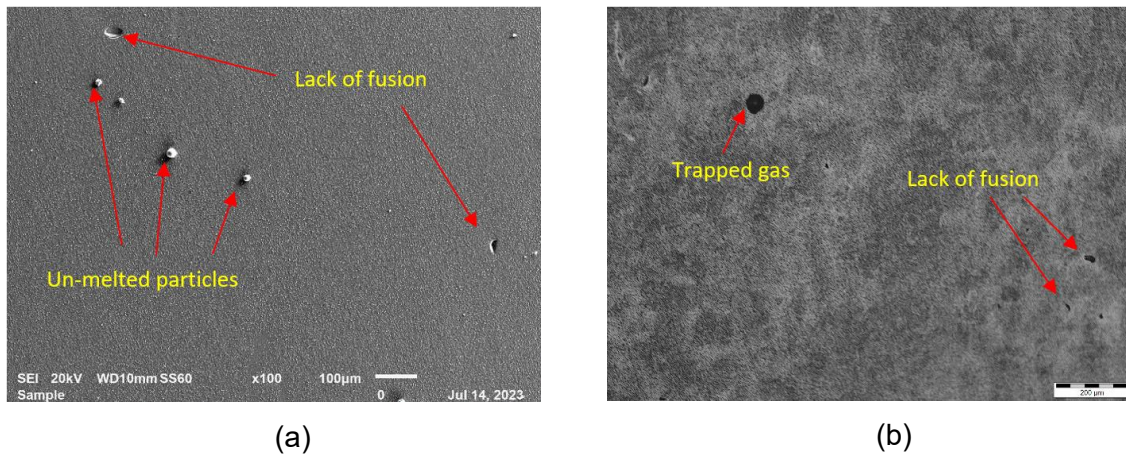


Figure 4.2: Typical defects found in the a) SLM and b) LENS Ti6Al4V samples.

4.1.3 XRD Analyses

Figure 4.3 depicts the XRD diffractograms of the wrought, SLM, and LENS Ti6Al4V samples. For all the samples, four major peaks were detected at around 41, 45, 48 and 63° 2 θ , and they were matched to (100), (002), (101) and (102) reflections of the hexagonal α -phase of Ti6Al4V (PDF files 04-004-8487, 04-002-5207, and 04-015-8644). Only one peak, a (110) reflection detected at 2 θ $\approx$ 75°, was attributed to the cubic β -phase of Ti6Al4V. This was consistent with expectation since Ti6Al4V characteristically contains α as the predominant phase (Section 2.1.1).

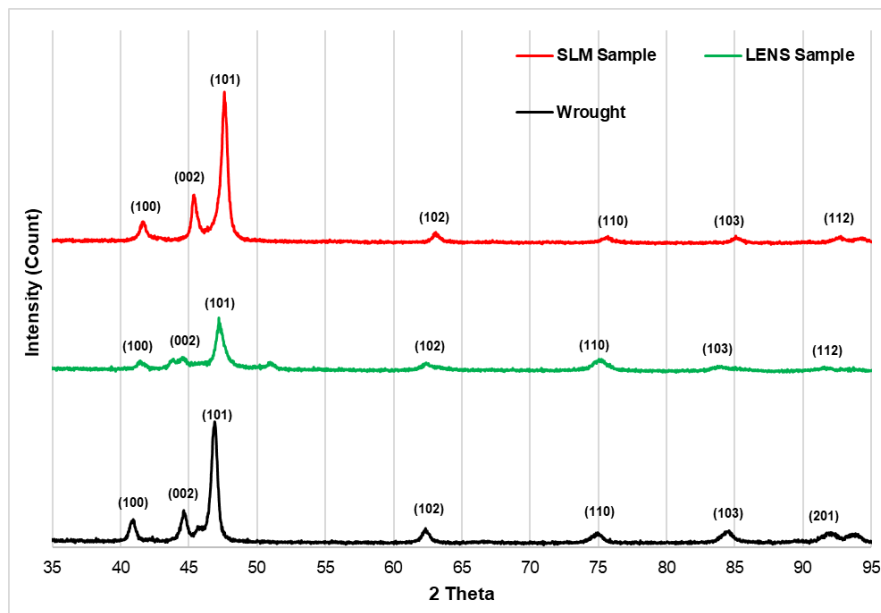


Figure 4.3: XRD diffractogram of the wrought, SLM, and LENS Ti6Al4V samples with the reference codes 04-004-8487, 04-002-5207, and 04-015-8644 respectively.

Figure 4.4 shows the effect of the manufacturing method on the Bragg angle of the (100), (002), and (101) peaks. These peaks are consistent with those of hexagonal α -Ti. The diffractograms in Figure 4.3 show that the α -phase exhibited a preferred crystal orientation along the (101) plane. Teng et al. (2024) noted similar observations. The peaks of the wrought, SLM, and LENS samples shifted more to the left of the reference peaks, which indicated that the wrought and AM Ti6Al4V samples induced more compressive stresses when manufactured.

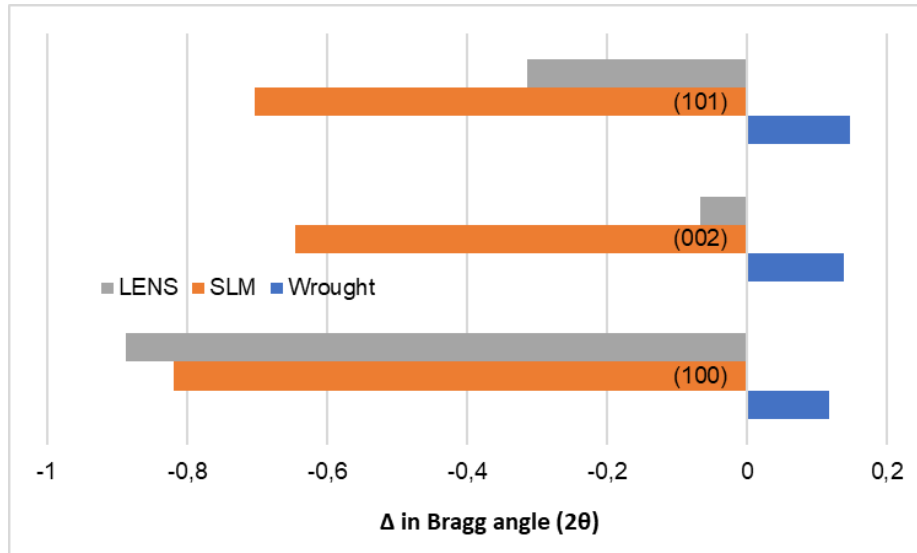


Figure 4.4: Change in Bragg angle (2θ) for the SLM, LENS and wrought Ti6Al4V samples before deformation.

4.1.4 Metallographic analysis

Metallographic analysis was done using optical microscopy and SEM, and the results obtained are presented in Figure 4.5. The microstructure of Ti6Al4V was strongly dependent on the manufacturing method. The wrought Ti6Al4V microstructure exhibited a complex microstructure with equiaxed α -grains and elongated α -grains with a network of intergranular β . Similar results were seen by Bodunrin et al. (2019). As described in Section 2.1.1, such a microstructure would provide the samples with a good balance of strength and ductility.

In contrast, the SLM Ti6Al4V had a fully acicular martensite microstructure characterised by acicular α' -martensite and columnar β -grains, while the LENS sample presented a microstructure consisting of intersecting lamellae of α with prominent β -grain boundaries indicative of a Widmanstätten microstructure. These results were consistent with the high cooling rates associated with AM and agreed well with findings by other researchers (Krakhmalev et al., 2016; Arthur, 2019; Bodunrin et al., 2019; Moridi et al., 2019).

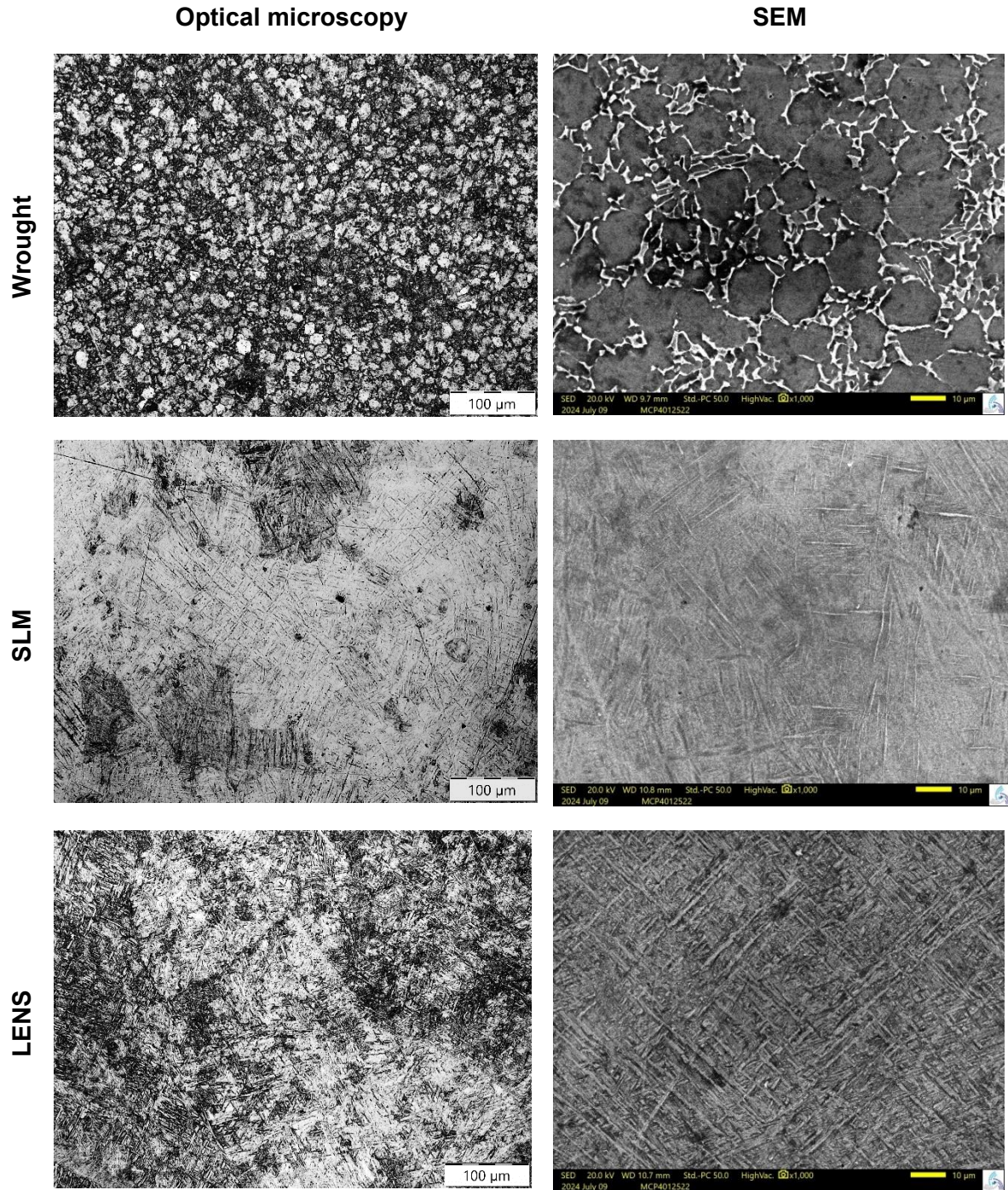


Figure 4.5: Initial microstructure of the wrought, SLM, and LENS Ti6Al4V samples.

Figure 4.6 provides a further comparison of the microstructures of the AM Ti6Al4V samples. The β -grain boundaries for the initial AM Ti6Al4V samples were examined in the vertical cross-sectional area. These columnar β -grain boundaries were observed to be larger compared to the initial wrought sample. The α -phase was established to exist between these β -grain boundaries. It was also observed that the columnar β -grain boundaries of the initial LENS samples were larger than the SLM samples.

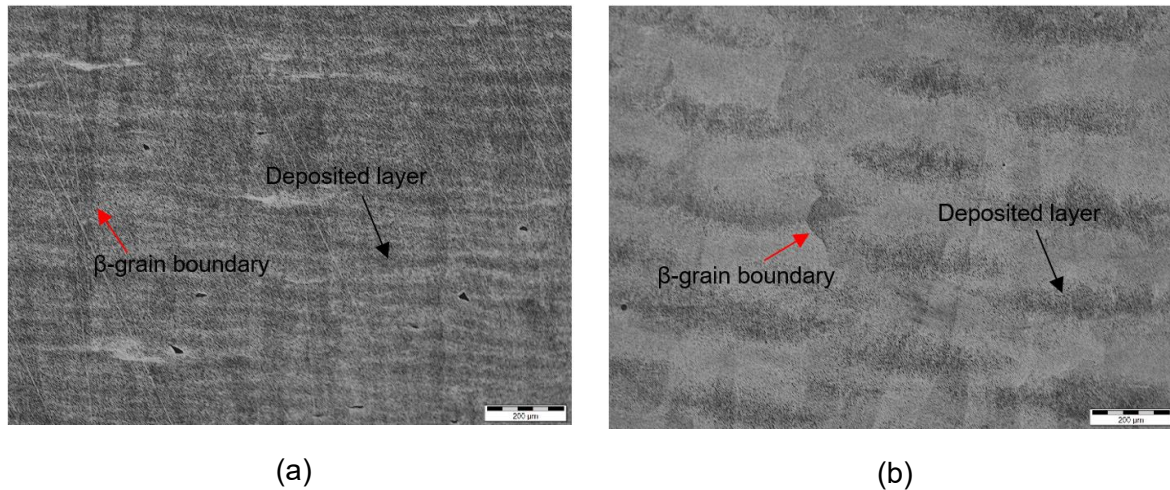
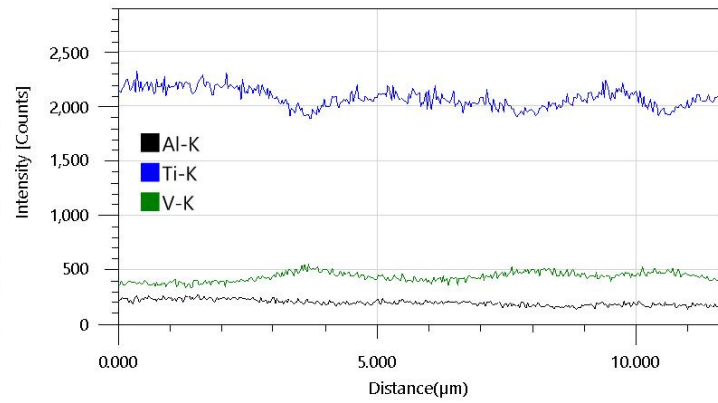
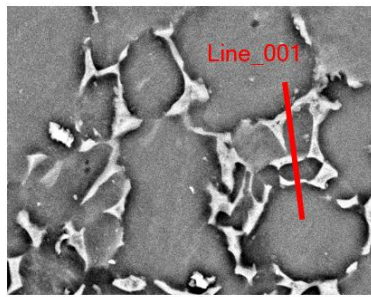


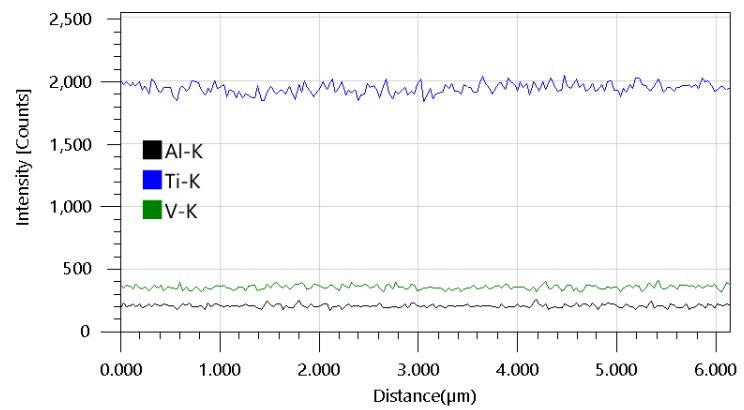
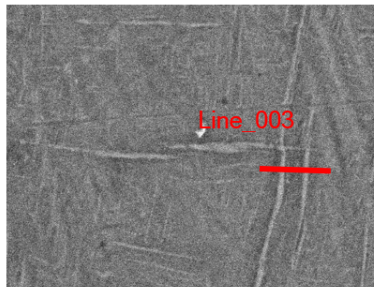
Figure 4.6: Optical micrographs displaying the columnar β -grain boundaries of the initial microstructure of the a) SLM and b) LENS Ti6Al4V samples.

Compositional profiling of the grains of the Ti6Al4V samples was done using EDS, and the results are presented in Figure 4.7. Only the primary alloying elements, titanium, aluminium and vanadium, were considered. Figure 4.7(a) shows an uneven distribution of titanium and aluminium across a typical grain boundary. There was a distinct depletion of titanium and enrichment of vanadium at the grain boundary, consistent with the expectation of the β -phase at the grain boundaries, as vanadium is a known β -stabiliser.

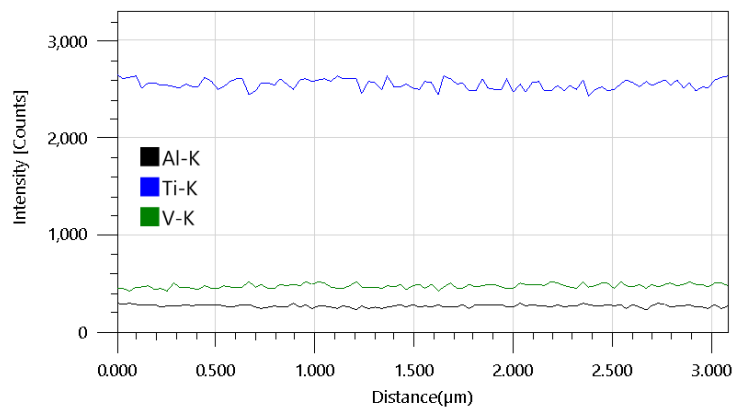
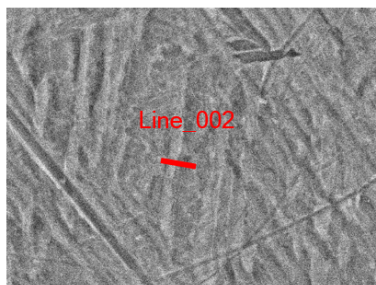
Notably, aluminium did not exhibit preferential partitioning in the sample. This is likely since aluminium has good solubility in both the α - and β -phase as discussed in Section 2.1.2, and will be distributed uniformly in the grains and across the grain boundaries. The EDS line scans for the AM Ti6Al4V given in Figure 4.7(b) and (c) show a uniform distribution of the three elements, titanium, vanadium, and aluminium. Similar elemental distribution in AM alloys was reported by Ran et al. (2020) and was attributed to no chemical composition segregation in AM Ti6Al4V samples.



(a)



(b)



(c)

Figure 4.7: Intensity versus distance using line analyses to distinguish the element differences between the α and β -phases on the initial microstructure of the a) wrought, b) SLM, LENS Ti6Al4V samples.

4.1.5 Hardness

The hardness of each Ti6Al4V sample before deformation is presented in Figure 4.8. Prior to deformation, the average hardness of the wrought, SLM, and LENS Ti6Al4V samples was measured as 340, 375, and 380 HV, respectively. These values were in agreement with those reported in previous studies, as shown in Table 2.3, and were consistent with the expectation that AM alloys inherently have fine grains and therefore would exhibit high hardness.

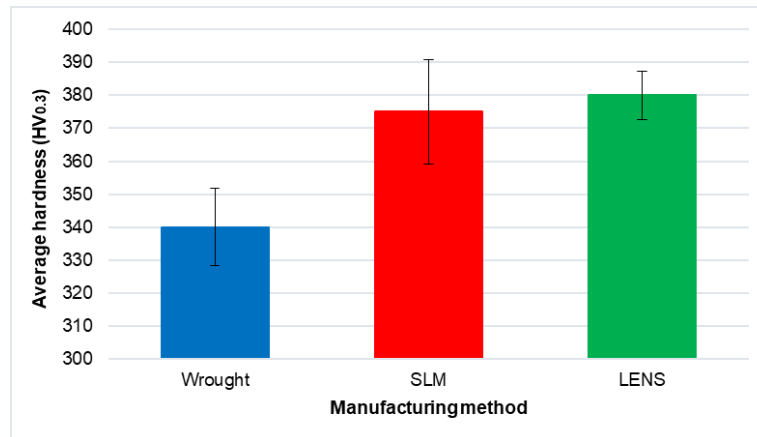


Figure 4.8: Average hardness ($VH_{0.3}$) before deformation of the wrought, SLM, and LENS Ti6Al4V samples.

4.2 High-temperature deformation tests

Hot compression testing was done on the wrought, SLM, and LENS Ti6Al4V samples using the Gleeble 3500® thermo-mechanical simulator at strain rates of 0.1, 1, and 10 s⁻¹ and temperatures ranging from 750 to 900°C. This section of the chapter presents the results obtained from the tests. Stress-strain curves were drawn to evaluate the flow behaviour of the Ti6Al4V samples.

4.2.1 Stress-strain curves

Figures 4.9 to 4.11 show the true stress-strain curves for the wrought, SLM, and LENS Ti6Al4V samples obtained at different temperatures and strain rates. The true stress-strain flow stress values are shown in Appendix A. It is immediately clear that for all the tested samples, flow stress decreased with the decrease in strain rate and increase in temperature. This observation aligns with findings from similar studies in which both commercially manufactured and AM Ti6Al4V were subjected to hot compression testing at various strain rates (Bodunrin et al., 2019; Tao et al., 2019; Jo et al., 2022).

The flow stress curves in Figures 4.9 to 4.11 exhibited an initial sharp peak followed by either a decrease or increase in the flow stress consistent with flow softening or strain hardening. Flow softening is typically related to thermal softening and microstructural softening, caused by mechanisms such as dynamic recrystallisation (DRX) and dynamic recovery (DRV), and is often observed at higher strain rates (Yang et al., 2019). On the other hand, strain hardening may be caused by interaction and multiplication of dislocations during plastic deformation, hindering dislocation movement (Yang & Jiang, 2024).

At lower strain rates, the extent of work hardening increases, as greater stress is required to deform the material. At the strain rate of 10 s^{-1} , the true stress-strain was characterised by a single broad peak suggesting deformation associated with DRX (Xiao et al., 2014; Huang et al., 2023). Notably, at lower strain rates, increasing the deformation temperature resulted in serrated stress-strain curves, consistent with the Portevin-Le Chatelier effect, a phenomenon observed when solute atoms act as obstacles to dislocation movement, temporarily obstructing dislocations and causing repeated propagation of localised stress concentrations during plastic deformation (Tamimi et al., 2015). Similar behaviour was observed by Bodunrin et al. (2018) for wrought Ti6Al4V, which was hot compression tested between 750 and 950°C and strain rates from 0.01 to 10 s^{-1} .

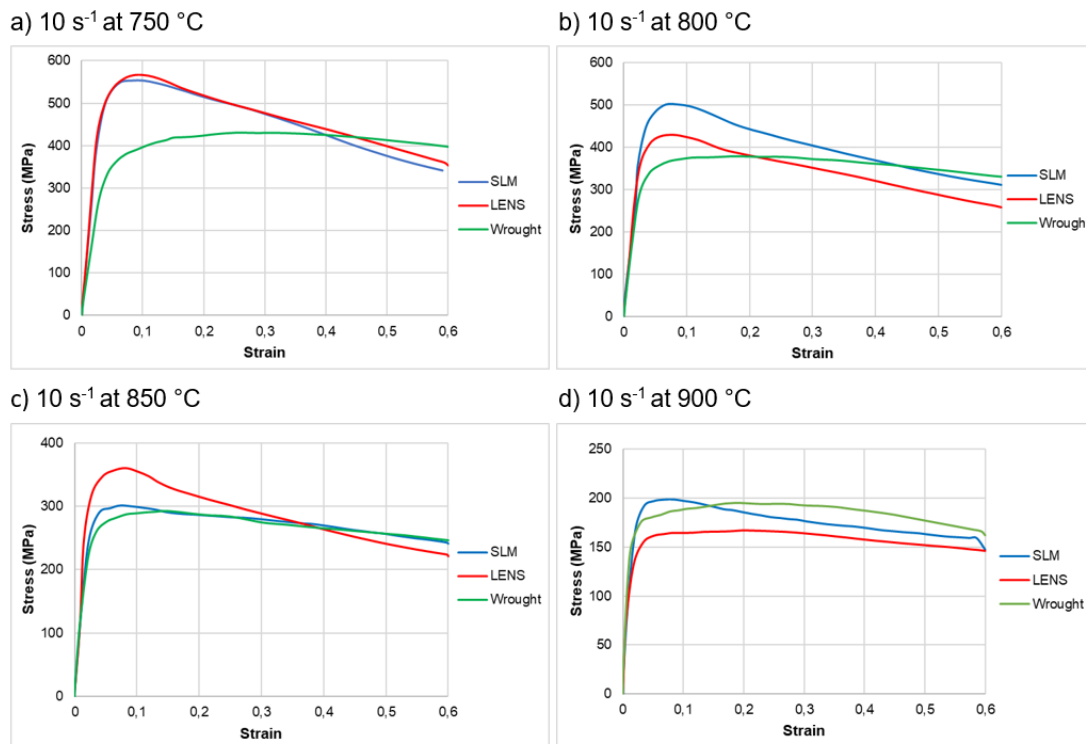


Figure 4.9: True stress-strain curves by hot compression testing of the Ti6Al4V alloys in the temperature range of 750-900°C at a strain rate of 10 s^{-1} .

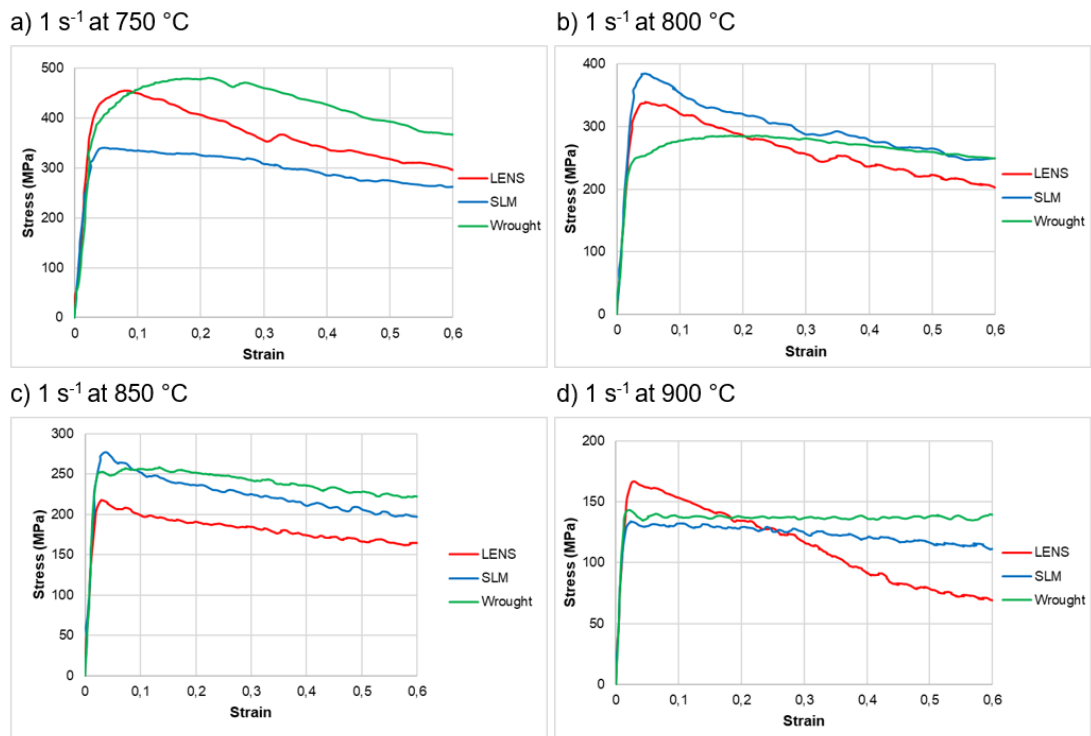


Figure 4.10: True stress-strain curves by hot compression testing of the Ti6Al4V alloys in the temperature range of $750\text{-}900^{\circ}\text{C}$ at a strain rate of 1 s^{-1} .

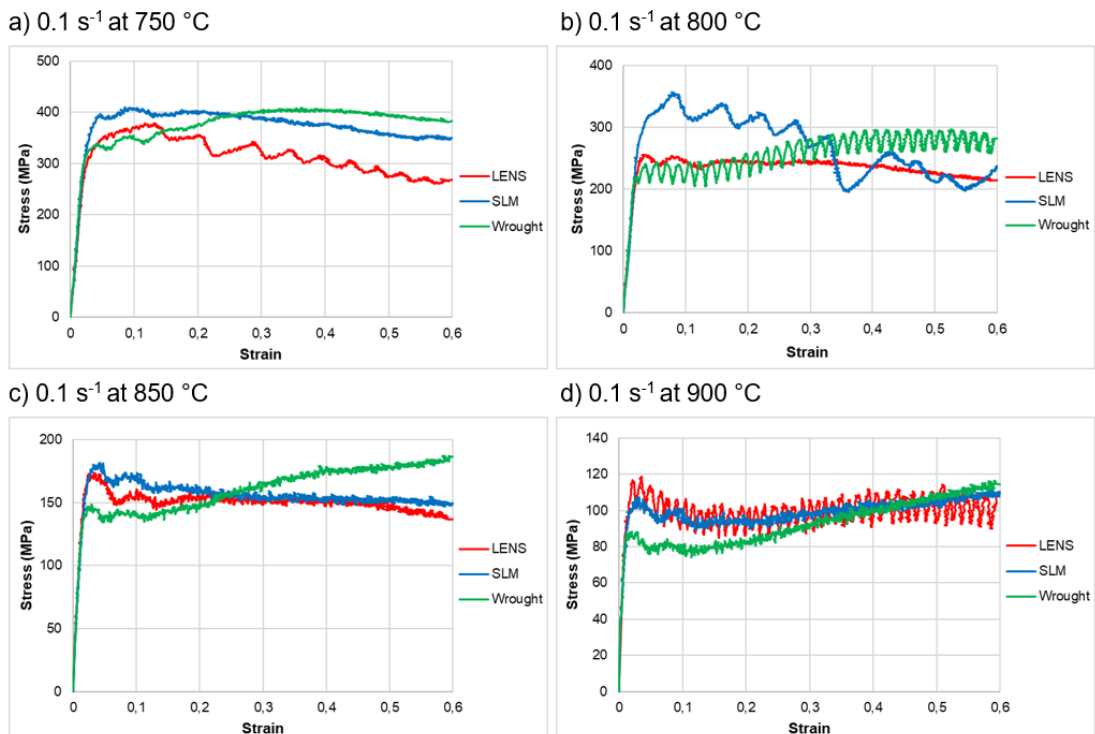


Figure 4.11: True stress-strain curves by hot compression testing of the Ti6Al4V alloys in the temperature range of $750\text{-}900^{\circ}\text{C}$ at a strain rate of 0.1 s^{-1} .

Therefore, it was expected that the AM Ti6Al4V samples would deform faster at higher temperatures compared to the wrought Ti6Al4V samples, which would experience a decrease in strength. In contrast, the wrought alloy was anticipated to have a higher modulus of elasticity and an increase in strength as strain increased, but this outcome was dependent on the mechanisms active during deformation.

In the main, the AM Ti6Al4V samples showed a greater decrease in flow stress compared to the wrought samples after the initial peak. For example, in Figures 4.9(b) and 4.10(b), the decrease was between 104 and 186 MPa for SLM and 117 and 166 MPa for LENS, compared to 34–48 MPa for the wrought under the same deformation conditions. This suggests that the wrought Ti6Al4V was most prone to flow softening than the AM Ti6Al4V. It is possible that the intergranular β -phase observed in Figure 4.5 increased resistance to dislocation movement in the wrought samples and hence resistance to flow softening. In fact, in most cases, flow stress in the wrought Ti6Al4V either increased or remained nearly constant, with some flow softening after the initial peak. The decrease in flow stress was most notable in the LENS Ti6Al4V samples compared to the SLM samples. The primary difference in these AM samples was their microstructure, as shown in Figures 4.5 and 4.6. It is likely that this contributed to the differences in rate of flow softening.

Both the initial AM Ti6Al4V microstructures are characterised by columnar β -grains and α -lamellae, but the LENS samples exhibit larger grains compared to the SLM samples (see Figures 4.6 and 4.7). It was expected that the coarser β -grains and thicker α -lamellae in LENS-produced Ti6Al4V may have promoted dislocation accumulation while facilitating dislocation motion, thereby reducing the availability of sites for mechanisms such as DRX and grain boundary sliding (Fan et al., 2016; Chen et al., 2025). However, the needle-like α -phase in SLM Ti6Al4V further restricted dislocation movement and facilitated resistance to softening, resulting in the flow softening being more gradual (Beyl et al., 2019; Chen et al., 2025). At increasing temperatures, both microstructures underwent globularisation, where the elongated α transformed into rounded grains, as shown in Figures 4.17 and 4.18.

Figure 4.12 presents the influence of the test parameters, strain rate and deformation temperature, on the peak flow stress of the wrought, SLM, and LENS Ti6Al4V samples. Peak flow stress decreased with an increase in temperature and a decrease in strain rate. For example, the peak stress of the wrought sample decreased from 430 to 409 MPa when the strain rate was increased from 10 to 1 s⁻¹ at 750°C and decreased by at least 55% and 72% when the temperature was increased from 750 to 900°C at 10 and 0.1 s⁻¹, respectively. Similar observations were noted by Bodunrin et al. (2019) on Ti6Al4V with a complex microstructure tested at strain rates of 0.01 to 10 s⁻¹ and 750 - 950°C, where there was a decrease of 47%

and 62% for the same test conditions. In most cases, the AM Ti6Al4V samples showed higher peak stresses than the wrought Ti6Al4V samples, and the difference was most pronounced at low temperatures and high strain rates.

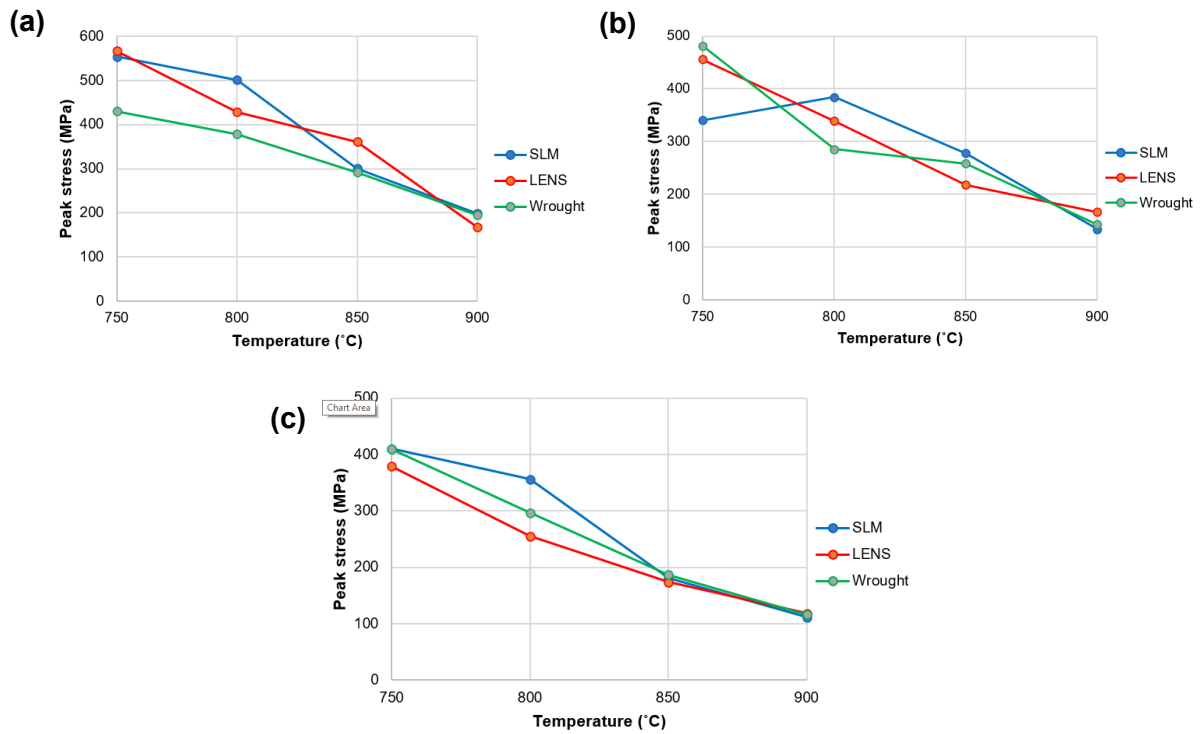


Figure 4.12: Peak flow stresses for the Ti6Al4V samples at different deformation temperatures at strain rates of a) 10 s⁻¹, b) 1 s⁻¹, and c) 0.1 s⁻¹.

4.2.2 Analysis of flow stress using the Arrhenius-type equation

Peak flow stresses calculated using Equation 2.11 were compared to the experimental peak flow stress measured in the hot compression test done using the Gleeble 3500[®] thermo-mechanical simulator. The findings are presented in Figure 4.13, where the AARE, RMSE, and R² values were determined using Equations 2.12 to 2.14. The peak flow stresses were examined to evaluate the influence of undesirable variations such as strain rate fluctuations, temperature deviations, and microstructural changes on the flow stresses of the wrought, SLM, and LENS Ti6Al4V samples.

Using the peak flow stresses between strains of 0.1 to 0.6, the wrought, SLM, and LENS Ti6Al4V showed average activation energies of 683, 776, and 660 KJmol⁻¹. These activation energies are consistent with previous literature with activation energies between 527 and 752 KJmol⁻¹ (Luo et al., 2015). As the strain increased, the activation energy increased. The higher activation energy of the LENS Ti6Al4V may have resulted in a higher accumulation of dislocations required to commence dynamic recrystallisation and dynamic spheroidisation (Lv et al. 2022).

In general, the flow stress comparison of the LENS Ti6Al4V samples at lower strains, illustrated in Figure 4.13, demonstrated the best correlation between the calculated flow stress and the experimental flow stress compared to the wrought and SLM samples, whereas at a strain of 0.6, the wrought samples showed a more consistent comparison. The SLM flow stress correlation was revealed to be the least consistent.

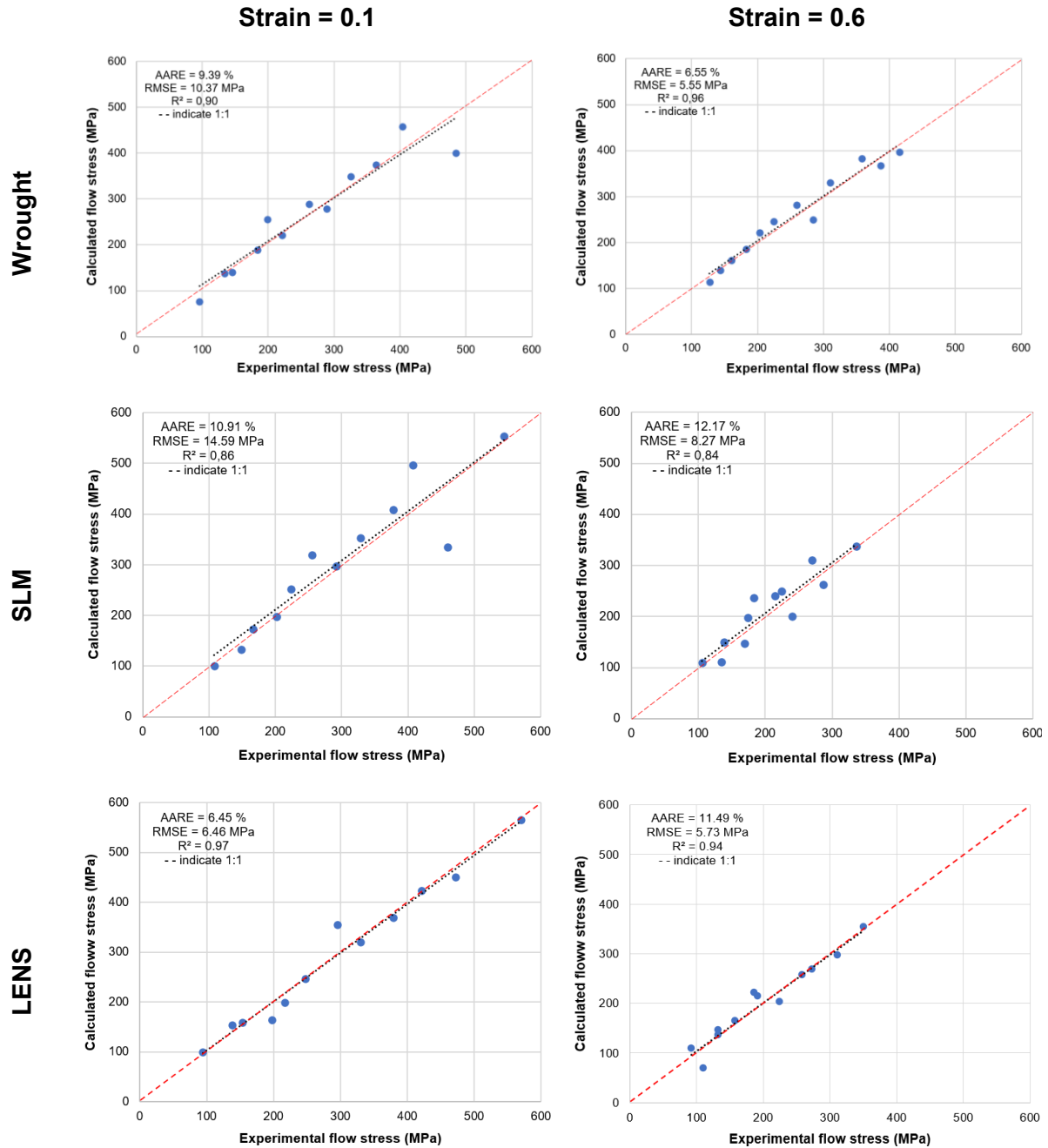


Figure 4.13: Scatter plots of the experimental flow stress and calculated flow stress regarding the statistical parameters of the wrought, SLM, and LENS Ti6Al4V at a strain of 0.1 and 0.6.

The LENS Ti6Al4V samples showed the best linear relationship between the calculated and experimental flow stresses, ranging from 0.94 to 0.97. The value for the SLM samples was calculated between 0.84 and 0.86, which demonstrated the least linear relationship compared to the wrought and LENS samples. The SLM showed the highest prediction error with an RMSE value of 14.59 MPa. The wrought and LENS samples demonstrated much lower RMSE values, which indicated that no abnormal variation was seen between the flow stresses. Niu et al. (2020) observed similar RMSE results for AM Ti6Al4V alloys, ranging from 30.17 to 18.24 MPa.

The highest AARE of the wrought Ti6Al4V samples was taken as 9.39%. This was less than the AARE value of 11.8% observed by Bodunrin et al. (2019). The AARE values for the SLM and LENS at a strain of 0.6 were 12.17% and 11.49%, respectively. These results indicated that the SLM Ti6Al4V samples experienced a greater magnitude of differences between the calculated and experimental flow stresses. The Arrhenius model assumes a consistent relationship between temperature, strain rate, and flow stress, which breaks down when microstructural response varies significantly across the sample (Shakib et al., 2016). Therefore, SLM Ti6Al4V may have showed less consistent flow stress correlation with the Arrhenius-type equation due to its metastable α' -martensitic microstructure, high residual stresses, and anisotropy from rapid solidification (Ricci & Iannitti, 2024). However, LENS Ti6Al4V may have provided a more consistent flow stress correlation due to its more stable Widmanstätten microstructure with a slower cooling rate (Fan et al., 2016; Liu et al., 2019; Motyka, 2021). This resulted in better alignment with the model's assumptions.

4.2.3 Power dissipation maps of Ti6Al4V alloys

During the high-temperature compressive deformation in the Gleeble 3500® thermo-mechanical simulator, the wrought, SLM, and LENS Ti6Al4V samples responded to the imposed testing parameters of temperature, strain rate, and strain by dissipating power in the form of heat and microstructural evolution. The efficiency of power dissipation, η , was obtained from the strain rate sensitivity parameter, m , as described in Equations 2.15 and 2.16, and was used to characterise the ease of the Ti6Al4V samples to undergo plastic deformation as well as predict the deformation mechanisms likely in these samples at different temperatures and strain rates. This was expressed using power dissipation efficiency maps at true strains of 0.1, 0.3, 0.4, and 0.6 and shown in Figure 4.14.

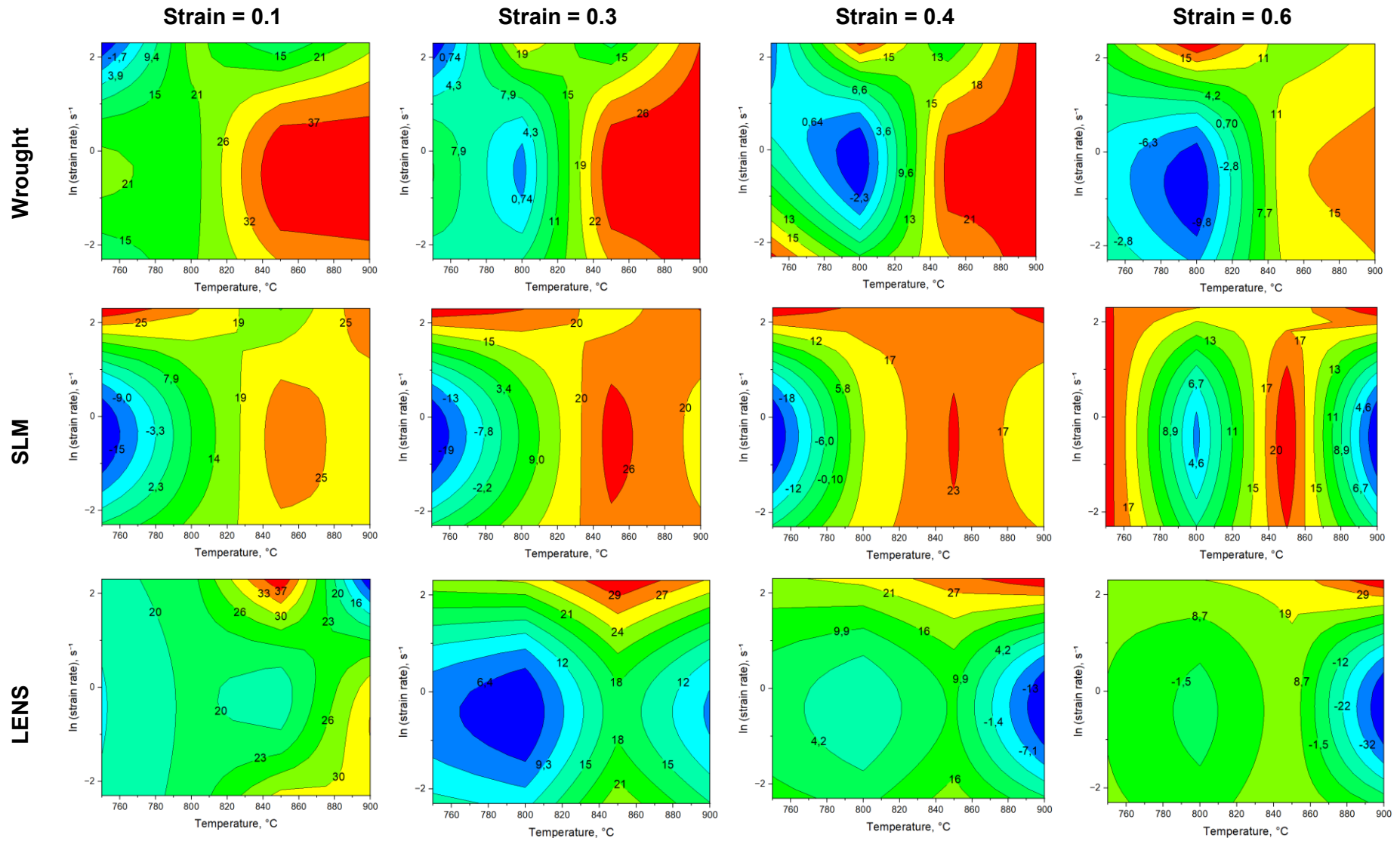


Figure 4.14: Power dissipation efficiency maps for the wrought, SLM, and LENS Ti6Al4V at a strain of 0.1, 0.3, 0.4, and 0.6.

The power dissipation maps in Figure 4.14 were divided into different deformation domains using differently coloured iso-efficiency contours. Red and orange domains represent regions with the highest η , while the blue-coloured domains indicate regions with the lowest η . Generally, values of η between 30 and 50% are associated with DRX, and with DRV between 20 and 30% (Prasad, 2003; Prasad et al., 2015). Low values of η , typically less than 20%, correspond to regions with poor hot deformation.

The highest power dissipation efficiency values for the wrought, SLM, and LENS Ti6Al4V samples ranged approximately between 21-37%, 20-26%, and 20-37%, respectively. These values suggested that mechanisms such as DRV, dynamic globularisation, and DRX were possible mechanisms within these power dissipation ranges. These efficiency values were primarily observed at temperatures exceeding 820°C, with variations in strain rates.

Notably, the LENS Ti6Al4V samples exhibited higher power dissipation efficiency at higher strain rates of approximately 10 s^{-1} , while the SLM samples demonstrated these values at around 1 s^{-1} . In addition, the wrought samples showed consistently high-power dissipation efficiency for all strain rates. However, as the strain increased, the regions of high-power dissipation efficiency started to decrease, indicating that mechanisms such as DRX and DRV became less pronounced. Instead, flow instabilities, including wedge cracking, void formation, intercrystalline cracking, prior particle boundary cracking, and other forms of unstable flow, became more prevalent (Prasad 2003; Prasad et al., 2015).

4.3 Post-deformation characterisation

This section presents the results of the chemical composition, hardness, and microstructural analysis of the wrought and AM Ti6Al4V samples obtained after hot compression testing. The results obtained from the post-deformation characterisation were compared with the pre-deformation results.

4.3.1 Chemical analysis

Figure 4.15 shows the typical change in chemical composition of the wrought and AM Ti6Al4V samples after deformation. Only aluminium, vanadium, carbon, oxygen, and iron were analysed. Positive values display an increase in the change in chemical composition, while a negative value shows a decrease.

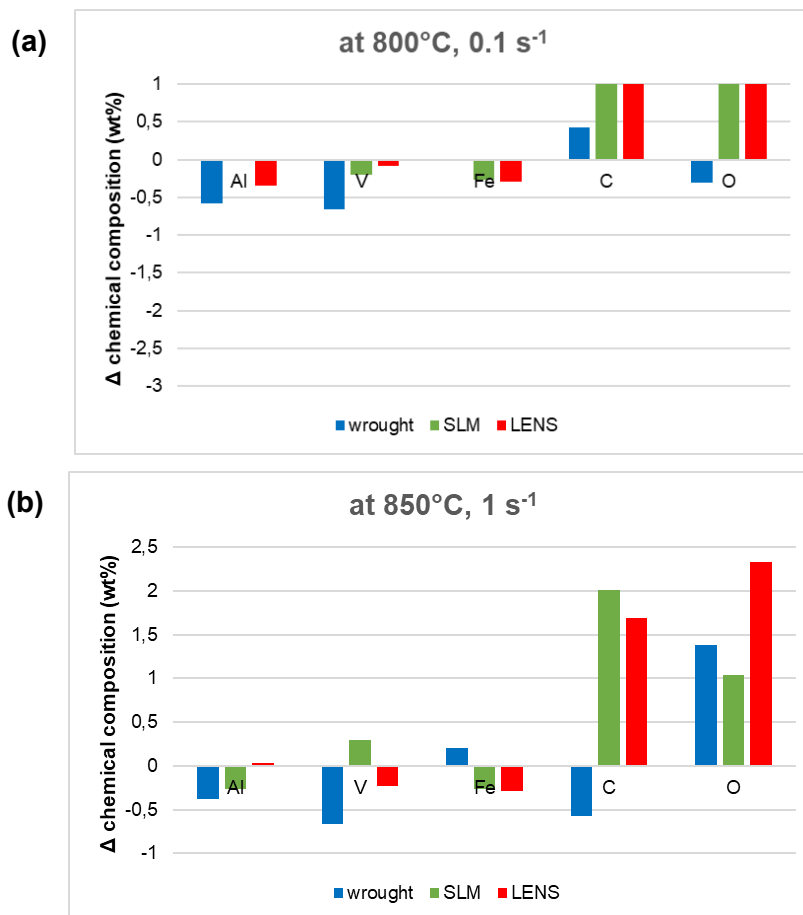


Figure 4.15: Difference in wt% of each element in the wrought, SLM, and LENS Ti6Al4V samples for deformation conditions: a) 800°C 0.1 s⁻¹ and b) 850°C and 1 s⁻¹.

The specified aluminium content for Ti6Al4V alloys was between 5.50 and 6.75%. The aluminium content for all the Ti6Al4V samples after hot compression testing was mainly within

the specified aluminium content, with a minimum percentage of 5.17% found in the SLM samples. This was less than the measured 5.65% aluminium found in the SLM sample before deformation. All samples before and after deformation showed vanadium percentages between the specified content of 3.50 and 4.50%.

Some iron percentages, 0.20–0.26%, were seen in the wrought, SLM, and LENS samples after hot compression testing at a temperature of 800 and 850°C. The iron compositions found in the samples fell below the specified maximum of 0.4%. The wrought, SLM, and LENS Ti6Al4V samples showed higher percentages of oxygen compared to the pre-deformation samples. This was also observed for the carbon percentages between 2.65 and 1.71% in the wrought, SLM, and LENS Ti6Al4V samples. This was above the specified carbon content of 0.08%.

4.3.2 Hardness tests

Figure 4.16 presents the percentage change in hardness values of the wrought and AM Ti6Al4V samples after hot deformation at 900°C with varying strain rates. The negative values indicate a decrease in hardness compared to a positive value showing an increase in hardness. Therefore, a decrease in hardness was observed for the AM Ti6Al4V, whereas the wrought Ti6Al4V samples showed an increase in hardness values.

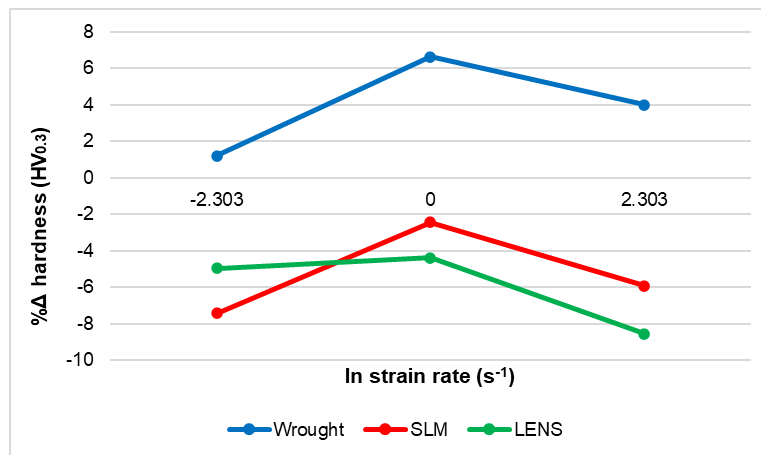


Figure 4.16: Percentage change in average hardness ($VH_{0.3}$) of the wrought, SLM, and LENS Ti6Al4V samples for each strain rate at a temperature of 900°C.

At lower strain rates, the hardness of the AM samples was approximately 18 HV higher than the wrought Ti6Al4V samples, while at higher strain rates the hardness values were similar, with a difference of 4 HV. The change in hardness for both the wrought and AM samples compared to the pre-deformed sample showed relatively small differences, with the largest change of about 8.5% observed with the LENS sample at 10 s⁻¹.

4.3.3 Microstructural analysis

The microstructures of the wrought and AM Ti6Al4V samples after deformation were examined for a strain rate of 0.1 and 10 s⁻¹ at the various deformation temperatures. These microstructures are presented in Figures 4.17 to 4.18. It was expected that the β -phase would increase with an increase in temperature, with temperatures closer to the β -transus temperature of 960 to 990°C exhibiting greater percentages of the β -phase. Microstructures between temperatures of 750, 800, and 850°C were expected to be dominantly α -phase, while they were predominantly β -phase at higher temperatures of 900°C.

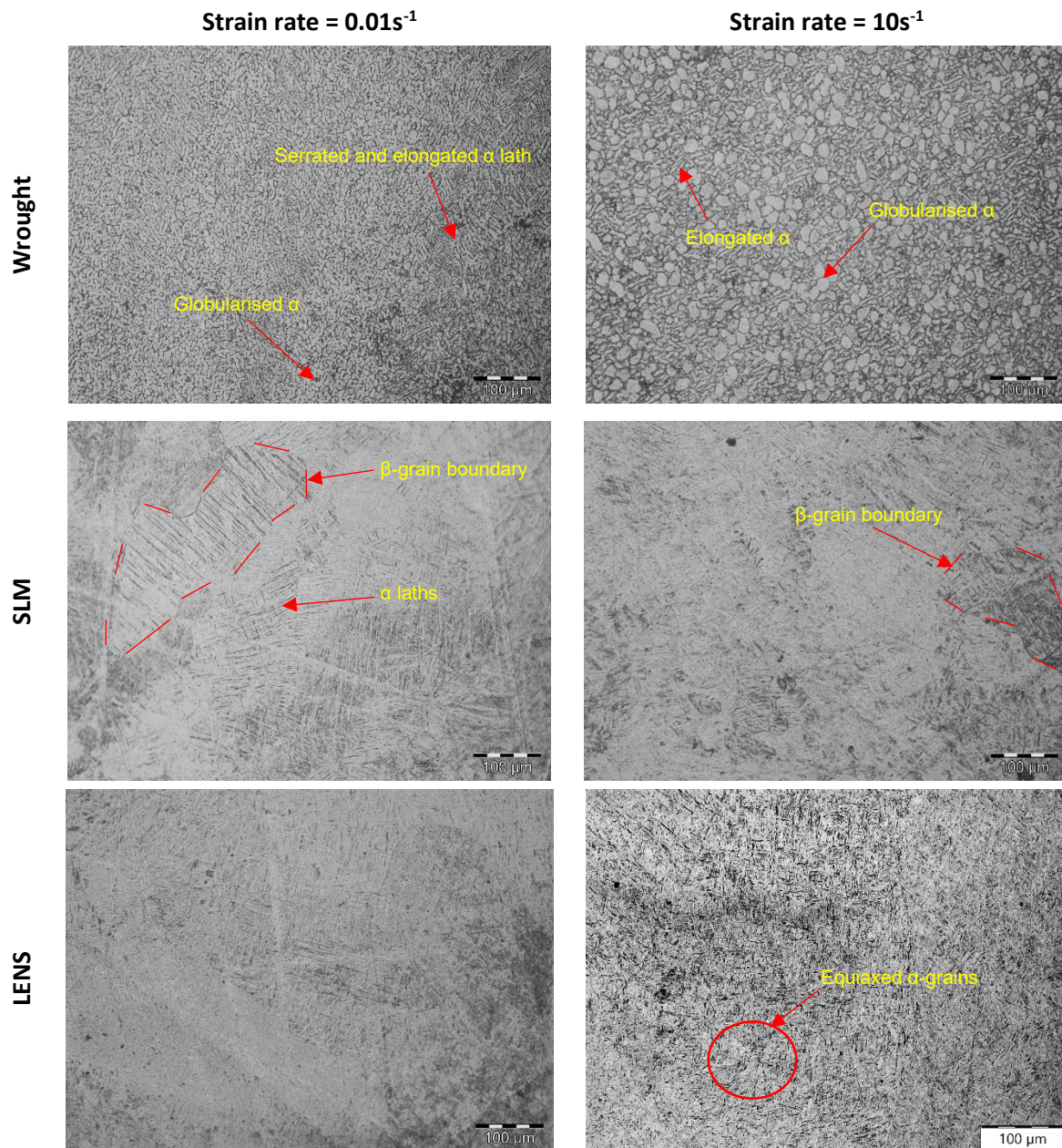


Figure 4.17: Optical micrographs of the wrought, SLM, and LENS Ti6Al4V microstructures at 750°C and 0.1 and 10 s⁻¹.

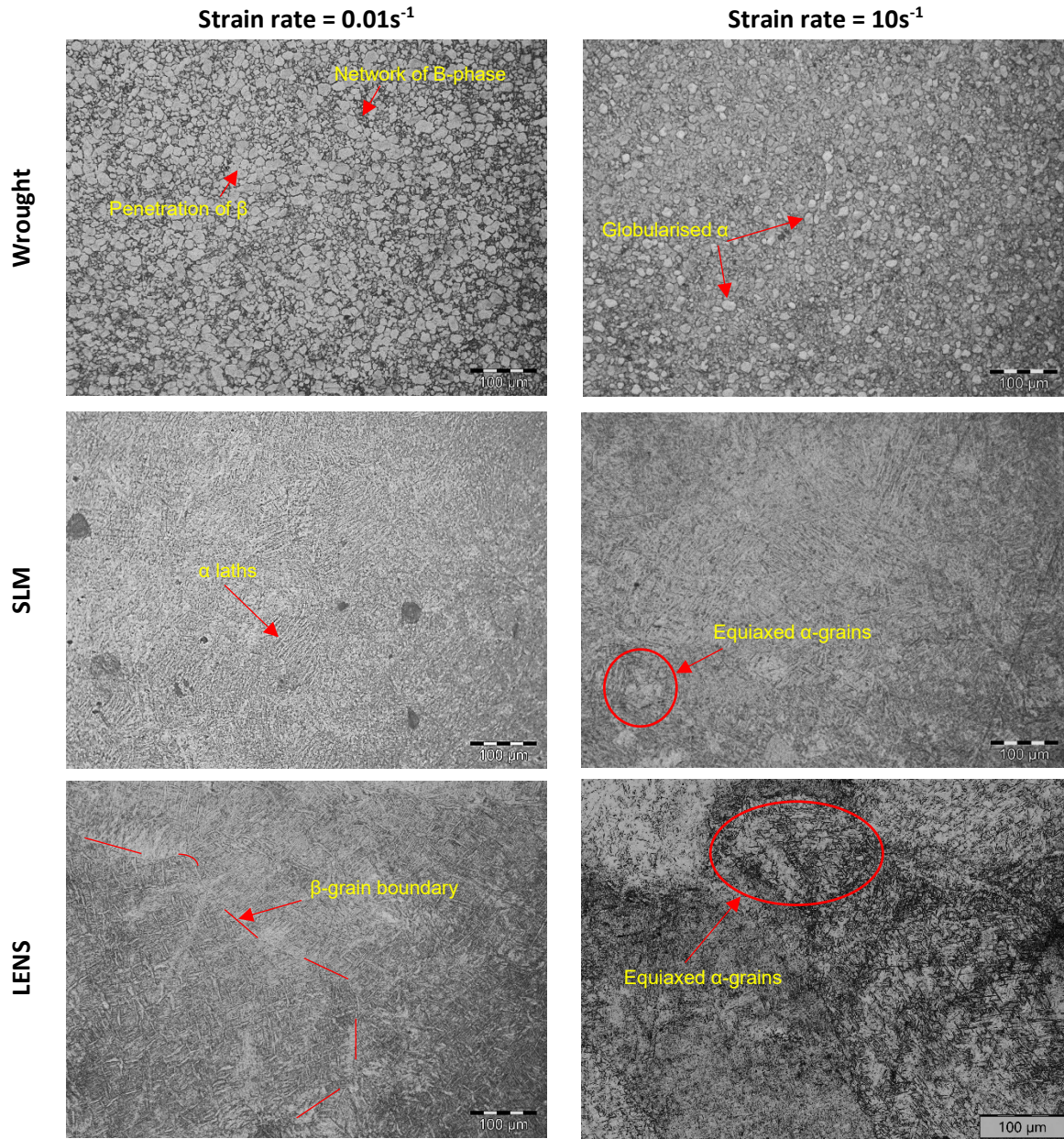


Figure 4.18: Optical micrographs of the wrought, SLM, and LENS Ti6Al4V microstructures at 900°C and 0.1 and 10 s^{-1} .

The initial microstructure of the SLM Ti6Al4V samples was acicular-type α' martensite formed within the elongated β -grains, whereas the LENS Ti6Al4V samples showed α -Widmanstätten with intersecting lamellae of α with prominent β -grain boundaries. As the temperature increased, the lamellar type of structure for both AM microstructures started to convert to a fine globularised or equiaxed structure with α -laths formed inside these smaller grains. Additionally, as the strain rate increased, an increase in alloy globularisation could be observed (Figure 4.18). The decomposition of AM microstructures through dynamic globularisation, or DRX, indicates a reduction in dislocation density (Motyka, 2021). However,

the globularisation process occurred slower in the SLM samples, as observed visually in Figure 4.18, where fewer globularised α -grains were formed.

The initial microstructure of the wrought Ti6Al4V samples was a complex microstructure with equiaxed α , elongated α , and intergranular β . As the temperature increased, the serrated and elongated α -grains formed finer globular α -grains, a characteristic of DRX which separates the elongated α into smaller grains (Jo et al., 2022). The increase in strain rate, as shown in Figure 4.18, led to a fully globularised α , while an increase in temperature led to an increase in the volume fraction of the β -phase which promoted the penetration of the β -phase into the α/α boundaries, thereby making dynamic globularisation occur readily (Bodunrin et al., 2019).

4.4 Discussion

4.4.1 Characterisation of Ti6Al4V alloys

During high-temperature compressive deformation, an increase in temperature was expected to cause a decrease in the α -phase and an increase in the β -phase, as discussed in Section 2.4.1 and as observed in Figures 4.9 to 4.11. This is because the Ti6Al4V transitions from a structure stabilised by aluminium to one stabilised by vanadium at higher temperatures but with no significant effect on the overall composition of the Ti6Al4V samples (Despax et al., 2020). Therefore, no significant changes in the chemical composition of titanium, aluminium and vanadium after deformation were expected, consistent with the results presented in Figure 4.15.

There were notable amounts of oxygen after deformation (Figure 4.15). Titanium is a passive metal, which tends to form a thin, tenacious titanium oxide layer when exposed to a corrosive environment (Prando et al., 2017). The size and growth rate of the oxide layer is a function of temperature and time (Li et al., 2024), and in Ti6Al4V may comprise aluminium oxide as well. This affinity for oxygen may explain the high oxygen contents noted in the Ti6Al4V samples after deformation at 750 to 900°C. Interestingly, the amounts of oxygen were much higher in AM Ti6Al4V samples, which could be related to the fine grain structure of the samples (Figures 4.5, 4.17, and 4.18). Fine grain structures, such as those typical in AM alloys, are highly reactive (Kumar et al., 2010; Mohamed et al., 2021).

The increased oxygen levels were not seen in the powders and, therefore, not likely present during the AM process due to the carrier gas utilised in the operations. Although, even in a vacuum, some degree of contamination is likely to be present. In addition, the formation of the oxidation layer could have been mitigated by applying surface coatings such as glass and graphene to minimise oxidation and contamination, as well as lubrication during hot deformation (Zafar et al., 2020; Kulakov et al., 2022).

The addition of 0.2 to 0.5 wt% carbon to Ti6Al4V leads to an increase in strength, hardness, and oxidation and creep resistance but could cause a reduction in ease of hot plastic deformation due to grain refinement and the formation of TiC, which hinders dislocation slip (Szkliniarz & Szkliniarz, 2020; Wang et al., 2022). However, the presence of carbon was attributed to contamination by carbon-rich materials when sample preparation and characterisation were performed, as the presence of carbon before deformation was not observed in the AM samples. The high carbon content in the AM Ti6Al4V samples also confirms the relatively higher reactivity of AM alloys. Such an increase in carbon content was not observed on the wrought Ti6Al4V samples.

Generally, the mechanical properties of AM alloys are significantly influenced by the presence of porosities, as demonstrated previously (Sola & Nouri, 2019). Pores act as stress concentrators and crack initiation sites, and mechanical properties such as strength, ductility, and hardness will decrease with an increase in porosity (Kan et al., 2022). In this study, the AM Ti6Al4V samples had < 0.15% porosity, which was unlikely to be the main factor to influence the mechanical properties of the AM Ti6Al4V samples and hence their deformation behaviour during high-temperature compression as shown in Figures 4.9 to 4.11. It therefore means that any variations in the mechanical properties and deformation behaviour of the AM Ti6Al4V samples were primarily due to phase and/or microstructural evolution in the materials rather than manufacturing defects. du Plessis et al. (2020) showed that small pores up to 0.5 mm and total porosity up to 1% made no difference in mechanical properties, while surface roughness and part geometry were much more important; failure mechanisms are mainly driven by microstructure but may depend on the size and type of defect.

The hardness values presented in Figure 4.8 agreed well with those in Table 2.3. Hardness in Ti6Al4V is frequently attributed to the microstructure of the alloy (Nguyen et al., 2022). The wrought Ti6Al4V samples exhibited a complex microstructure (Figure 4.5) and a lower hardness compared to the SLM and LENS samples that exhibited α' -martensitic and Widmanstätten microstructures, respectively. Martensitic and Widmanstätten microstructures typically show higher strength compared to equiaxed microstructures, as they offer higher resistance to dislocation movement (Beyl et al., 2019; Nguyen et al., 2022). AM Ti6Al4V samples therefore showed high flow stress in most of the cases studied (Figures 4.9 to 4.11). At 900°C, the wrought and AM samples showed similar hardness values, suggesting that the martensitic structure of the AM samples may have decomposed into an $\alpha+\beta$ microstructure. This was also observed by Motyka (2021), where the α' decomposed into an $\alpha+\beta$ microstructure, where hardness and strength decreased.

A peculiar feature of AM metals is columnar grains brought on by heat discharge in a single direction during solidification (Kim et al., 2018:35; Shi et al., 2023). Partially remelted areas in the upper region of the previously deposited layer are promoted by epitaxial growth, which results in the formation of elongated columnar grains (Kim et al., 2018:35). Figure 4.6 reveals the presence of columnar grains in the AM Ti6Al4V samples used in this study. Dislocation movement in lamellar grains is increased compared to dislocation movement in equiaxed grains (Bodunrin, 2018). During deformation, AM Ti6Al4V would therefore be expected to exhibit a higher flow stress than wrought Ti6Al4V, as observed in Figures 4.9 to 4.11.

At room temperature (Figure 4.5), the wrought Ti6Al4V consisted of the primary α -phase, with the β -phase located at the grain boundaries. As the temperature increased within the $\alpha+\beta$

region, the proportion of β -phase increased, forming more continuous networks and laths. This was shown by Bodunrin et al. (2019), where the volume fraction of β -phase increased from 37 to 64% when deformed at 900 and 950°C, respectively. The increase in volume fraction of the β -phase resulted in easier deformation, where a decrease in flow stress was observed in Figures 4.9 to 4.11. Similar behaviour was seen in the AM Ti6Al4V flow curves, where the β -phase increased and more globular α -grains formed.

In SLM Ti6Al4V samples, the microstructure evolved with temperature, transitioning from a predominantly α' -martensitic structure to a mixed $\alpha+\beta$ phase structure at higher temperatures (Figures 4.17 and 4.18). The Widmanstätten structure in the LENS Ti6Al4V samples was characterised by α -phase laths within a β -phase matrix. At lower temperatures, the microstructure predominantly exhibited a Widmanstätten pattern with fine α -laths and minimal transformation to β -phase. As the temperature increased, the α -laths formed more globalised laths, decreasing the dislocation movement in the AM Ti6Al4V samples (Lv et al., 2022), leading to similar flow stress values between the AM and wrought Ti6Al4V samples.

Low strain rates in the SLM samples facilitated DRX, refining the grain structure and transitioning from martensitic needles to fine equiaxed grains with martensitic needles inside (Figures 4.17 and 4.18). High strain rates in SLM samples restricted recrystallisation, preserving the elongated martensitic structure, possibly with high dislocation density (Tao et al., 2019). This resulted in higher flow stresses than those observed when deformed at lower strain rates, due to the preservation of the martensite structure's high strength (Beyl et al., 2019). At lower temperatures, the SLM microstructure was predominantly α' -martensite, which decomposed into a mixed $\alpha+\beta$ phase structure with increasing temperature (Motyka, 2021), increasing the presence of β -phase and transforming α' into α , as explained in Section 2.4.1. Similarly, in LENS samples, low strain rates refined the grain structure, transitioning from a lath-like Widmanstätten to an α -lamellar and equiaxed grain structure.

Serrated stress-strain curves were observed on the Ti6Al4V samples tested at a strain rate of 1 s^{-1} , where the serrations represent the replacement of the primary yield point by numerous localised yield points (Rym et al., 2020). This is typical of the Portevin-Le Chatelier effect and became more pronounced at higher temperatures and lower strain rates (Figure 4.11). The Portevin-Le Chatelier effect may have been due to dynamic strain ageing of Ti6Al4V, where dislocation mobility is disrupted by interstitial atoms, possibly vanadium, or the α -phase (Villa et al., 2019; Rym et al., 2020). As temperature increases, the diffusion of interstitial atoms and the dissolution of the α -phase also increase, and at low strains, the α -phase is expected to grow coarser, as clearly seen in Figure 4.19 on the wrought Ti6Al4V tested at 900°C (Villa et

al., 2019). The Portevin-Le Chatelier effect is indicative of inhomogeneous deformation and instability as observed in the power dissipation maps in Figure 4.14.

The power dissipation maps, values between 20-50% of power dissipation efficiency, η , are typically associated with stability, heat dissipation via microstructural evolution and deformation by recrystallisation (DRX), globularisation, and dynamic recovery (DRV) (Prasad, 2003; Prasad et al., 2015). The red and orange domains in Figure 4.14 indicate contours with high η . AM constricted these domains at lower strain rates and reduced the range of the highest η of Ti6Al4V, making the AM Ti6Al4V samples less prone to DRX. Therefore, at higher strain rates and temperatures, the AM samples showed higher dissipation efficiency values, indicating DRX and globularisation, consistent with the globularised α -grains in Figure 4.18. At lower strain rates and temperatures, the efficiency of power dissipation was below 20%, suggesting unstable flow and lower flow stresses (Figures 4.11 and 4.14).

The wrought samples showed high power dissipation efficiency values at all strain rates and higher temperatures, indicative of DRX and DRV, while at lower temperatures unstable flow was observed. Therefore, the flow stress was lower compared to the AM samples at lower temperatures (Figures 4.9 to 4.11). Unstable flow regions may have presented mechanisms such as wedge cracking, void formation, intercrystalline cracking, prior particle boundary cracking, and flow instabilities during hot deformation, which was considered to cause inhomogeneities or microstructural damage and is typically avoided (Prasad 2003; Prasad et al., 2015).

5 Conclusions and Recommendations

The target applications for Ti6Al4V alloys are essentially high-temperature applications, such as the aerospace and aircraft industries. In this research, the deformation behaviour of as-built AM Ti6Al4V at high temperatures was studied using the Gleeble 3500® thermomechanical simulator. Selective laser melting (SLM) and laser engineered net shaping (LENS) AM techniques were used to prepare the samples, while wrought Ti6Al4V was used as a control for all tests.

5.1 Conclusions

The hot deformation behaviour of the wrought, SLM, and LENS Ti6Al4V was investigated isothermally at temperatures of 750 and 900°C and strain rates between 0.1 and 10 s⁻¹. The flow stress of the AM samples decreased rapidly at higher strain rates and lower temperatures. As the temperature increased, the stress-strain curves showed some strain hardening or steady-state behaviour. The wrought stress-strain curves displayed a broader peak with some flow softening or strain hardening. Serrated stress-strain curves were observed on the Ti6Al4V samples tested at strain rates of ≤ 1 s⁻¹. This is typical of the Portevin-Le Chatelier effect and became more pronounced at higher temperatures and lower strain rates.

Notably, the peak stress of the AM Ti6Al4V samples was generally higher than the wrought samples. This was consistent with the initial finer microstructure of the AM samples compared to the coarser wrought microstructure. At higher strain rates and temperatures, the AM samples showed higher dissipation efficiency values, indicating DRX and globularisation, consistent with the globularised α -grains that formed after deformation. The wrought samples showed high power dissipation efficiency values throughout all strain rates and higher temperatures, indicative of DRX and DRV. At lower strain rates and temperatures for both the wrought and AM samples, the power dissipation efficiency was below 20%, suggesting unstable flow and lower flow stresses. In general, the AM Ti6Al4V samples showed higher peak stress, with a drastic decrease in flow stress as the strain increased. This concluded that the AM samples had a higher strength with decreased ductility compared to the wrought samples at elevated temperatures.

5.2 Recommendations for future research

Recommendations for further research are:

1. High-temperature tensile testing of AM Ti6Al4V to enable a comprehensive comparison with compression deformation behaviours and mechanisms.
2. In situ analysis of phase and/or microstructural evolution at the test temperatures and strain rates to provide further insight into thermo-mechanical material behaviour.

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Appendix A – Supplementary results for hot compression testing of the Ti6Al4V samples

A-1: Flow stress values of the wrought Ti6Al4V alloy as a function of strain, temperature, and strain rate.

Strain rate (s ⁻¹)	Strain	Temperature (°C)			
		750	800	850	900
10	0.1	400	375	275	180
	0.2	423	378	287	195
	0.3	430	371	275	192
	0.4	425	360	265	187
	0.5	411	346	257	177
	0.6	397	330	246	162
1	0.1	458	278	256	137
	0.2	479	284	252	137
	0.3	461	281	242	137
	0.4	428	270	235	136
	0.5	393	260	228	138
	0.6	368	250	222	139
0.1	0.1	349	222	140	76
	0.2	378	244	151	83
	0.3	403	281	166	92
	0.4	404	286	176	101
	0.5	395	265	179	110
	0.6	383	282	186	114

A-2: Flow stress values of the SLM Ti6Al4V alloy as a function of strain, temperature, and strain rate.

Strain rate (s ⁻¹)	Strain	Temperature (°C)			
		750	800	850	900
10	0.1	554	497	297	197
	0.2	515	440	285	186
	0.3	472	406	279	176
	0.4	422	367	270	170
	0.5	373	335	257	162
	0.6	337	311	241	147
1	0.1	334	353	252	132
	0.2	325	319	237	129
	0.3	308	287	225	125
	0.4	285	278	211	121
	0.5	274	265	206	116
	0.6	262	249	197	111
0.1	0.1	408	319	173	100
	0.2	399	320	162	96
	0.3	391	271	155	99
	0.4	378	241	154	104
	0.5	359	214	154	108
	0.6	350	237	150	110

A-3: Flow stress values of the LENS Ti6Al4V alloy as a function of strain, temperature, and strain rate.

Strain rate (s ⁻¹)	Strain	Temperature (°C)			
		750	800	850	900
10	0.1	566	423	355	164
	0.2	516	380	315	167
	0.3	477	352	288	164
	0.4	439	319	262	157
	0.5	395	285	241	152
	0.6	354	257	222	146
1	0.1	450	320	199	153
	0.2	407	286	191	135
	0.3	356	256	185	116
	0.4	339	236	174	91
	0.5	318	223	167	78
	0.6	297	203	165	69
0.1	0.1	369	247	159	100
	0.2	354	242	153	92
	0.3	324	244	148	104
	0.4	311	236	152	110
	0.5	276	227	148	96
	0.6	269	214	137	110