

Microstructural Evolution of Ti-5Al-5Mo-5V-3Cr

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Abstract

The high specific strength and corrosion resistance of titanium alloys provide an attractive alternative to steels when selecting materials for aerospace applications. High strength beta titanium alloys, for example Ti-10V-2Fe-3Al, have replaced steels for large components such as those found in landing gear assemblies. Economic production of these components is achieved through hot near net shape forging. However, the mechanical properties of titanium alloys are highly dependent on the final microstructure, which is developed during the manufacturing processes. Ti-10V-2Fe-3Al exhibits a high level of microstructural sensitivity to hot working variables, often leading to complications in achieving the desired microstructure.

Ti-5Al-5Mo-5V-3Cr is a recently developed high strength beta titanium alloy which is claimed to offer similar mechanical properties as those of Ti-10V-2Fe-3Al in addition to a greater tolerance to process variables. This thesis characterises the effect of initial microstructure and process variables on the flow behaviour and final microstructures of Ti-5Al-5Mo-5V-3Cr during subtransus isothermal forging. The morphology of the alpha precipitates in the microstructure prior to forging influences the peak stress and the plastic flow behaviour. Acicular alpha particles lead to a higher peak stress and a large extent of flow softening, whereas spheroidal alpha particles act as witnesses to the deformation of the beta phase. Regardless of starting microstructure a steady state flow regime is achieved by a strain of 0.35 during which deformation is dominated by dynamic recovery of the beta phase.

The formation of the metastable omega phase in Ti-5Al-5Mo-5V-3Cr and its influence on subsequent microstructural developments has also been investigated. In-situ X-ray diffraction data has shown that embryonic omega forms well above the omega start temperature during rapid cooling. Aging material which contains the omega phase at industrial alpha aging temperatures results in the rapid formation of acicular alpha precipitates. The speed of formation and the even distribution of the alpha is believed to be influenced by the omega phase, thereby offering a potential mechanism to engineer the final microstructure.

Preface

This dissertation is submitted for the degree of Doctor of Philosophy at Imperial College London, University of London. It describes research carried out in the Department of Materials under the supervision of Professor Richard Dashwood, Dr David Dye and Dr Martin Jackson.

Part of this dissertation have either been published, or are currently under review for publication:

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1 Introduction

Titanium alloys are high strength, light weight materials with an excellent resistance to corrosive environments. Titanium is commonly alloyed with aluminium and other transition metals, combinations of which provide a wide spectrum of different alloys, each with specific benefits. However, the widespread use of titanium alloys in engineering applications has been restricted by the high cost of material production. The lucrative, high use markets, for example the automotive industry, are unable to justify the cost benefit of titanium over other structural metals, such as aluminium alloys or steels. Thus the use of titanium alloys has been limited to areas where the inherent major benefits are required by design, the most notable example of which is the aerospace industry. The continuing economically driven effort to improve efficiency by reducing weight has seen an increasing percentage of a modern airframe being made from titanium alloys, which in turn requires better understanding of titanium alloy metallurgy [1].

Beta titanium alloys are arguably the most versatile class of titanium alloys, offering the highest strength to weight ratio and a good combination of toughness and fatigue resistance [2]. The mechanical properties of β titanium alloys are governed by the final microstructure, which in turn is determined by the processing history of the material. One problem when working with β titanium alloys is the relatively small processing window available, which therefore requires careful process control in order to obtain the optimal property combinations [3].

In the early 1990s high strength steels were replaced by the β titanium alloy Ti-10V-2Fe-3Al for the large truck beam forgings on the Boeing 777 airframe. This resulted in a significant weight saving (approximately 40%) and a reduction in the risk of stress corrosion related failure [4]. Economic production of these large components is achieved through near net shape forging, which has to be conducted isothermally due to the sensitivity of the microstructure to temperature fluctuations [5]. Over the last twenty years the behaviour of Ti-10V-2Fe-3Al during isothermal forging has been extensively investigated, with a view to optimising both the developed microstructure for specific applications and also the processing route. Despite the significant level of

research conducted, Ti-10V-2Fe-3Al exhibits some inherent detrimental features, such as a small processing window, solute segregation and a high microstructural sensitivity to small temperature fluctuations, which have ultimately led it being superseded.

In 1997 Timet developed TIMETAL 555 (Ti-5Al-5Mo-5V-3Cr), a new β titanium alloy based upon the Russian alloy VT22 (Ti-5Al-5Mo-5V-1Cr-1Fe). It was claimed that Ti-5Al-5Mo-5V-3Cr offered simpler processing and a higher strength than Ti-10V-2Fe-3Al whilst having an identical density of 4.65 gcm^{-3} [6]. These beneficial properties, which overcome many of the industrial difficulties of Ti-10V-2Fe-3Al, have rapidly led to the selection of Ti-5Al-5Mo-5V-3Cr for the large section forgings in the Boeing 787 airframe [7]. Limited work has been published concerning Ti-5Al-5Mo-5V-3Cr and studies which are in the public domain have focussed on microstructural development through varying heat treatments. Little data concerning the hot working of Ti-5Al-5Mo-5V-3Cr [8], and in particular the effects and deformation mechanisms of isothermal forging, has been published to date, an area which is addressed in this thesis.

This thesis is divided into six major chapters. A review of titanium metallurgy, phase transformations and deformation behaviour is presented in Chapter 2. Particular focus is given to the ω phase and the microstructural evolution during deformation. Chapter 3 presents a fundamental study into the specific metallurgy of Ti-5Al-5Mo-5V-3Cr and the establishment of key information such as the β transformation temperature. The flow behaviour and microstructural evolution during subtransus isothermal forging is discussed in Chapter 4. The effect of initial microstructural condition is investigated and suggestions made as to the dominating deformation mechanisms. Chapter 5 investigates the formation of athermal omega in Ti-5Al-5Mo-5V-3Cr and its influence upon subsequent α phase nucleation and growth through a combination of interrupted testing and in situ techniques. It is believed that data presented in this chapter represents the first time the athermal omega transformation has been studied using an X-ray synchrotron. The major findings of the research are drawn together in Chapter 7, along with suggested areas for further work.

2 Literature Review

This chapter presents an overview of published material concerning the metallurgy of titanium and its alloys. The review is subdivided into five major sections with a progressive focus towards β titanium alloys. Section 2.1 discusses the fundamental aspects of titanium alloy metallurgy, including the role of different alloying additions, alloy classifications and the development of both equilibrium and non equilibrium phases. The formation mechanisms of the non equilibrium phase omega (ω) are the topic of a more detailed discussion in section 2.2. The section also considers the effect of the ω phase on the mechanical properties of an alloy and its influence on subsequent phase transformations. The role of processing on the microstructural evolution of β titanium alloys is discussed in section 2.3. The possible deformation mechanisms during hot working, the relevant constitutive laws and empirical modelling methods are all considered in relation to β alloys. Section 2.4 considers some specific features of the most extensively used β alloy, Ti-10V-2Fe-3Al before introducing its supposed replacement alloy, Ti-5Al-5Mo-5V-3Cr in section 2.5.

2.1 Titanium Alloys: Metallurgy and Microstructures

Titanium is the world's seventh most abundant metal, after aluminium, iron, calcium, sodium, potassium and magnesium [9]. It has two primary beneficial properties, a high strength to weight ratio and a good resistance to corrosion. However, titanium alloys did not find an engineering application until the development of the gas turbine engine and the growth of the aerospace industry during the middle of the twentieth century [10]. Up until that point in time, titanium had been little more than a laboratory curiosity because of the difficulty and cost of obtaining the metallic element from the relevant ores [11].

Titanium is naturally found in two predominant forms, rutile (TiO_2) and ilmenite ($\text{FeO} \cdot \text{TiO}_2$). Both these ores are remarkably difficult to decompose in order to produce metallic titanium and even today the only commercially viable production method is through laborious and expensive batch processing. The Kroll process (developed by William Kroll in 1937) remains the major method of producing pure titanium. The metallothermic process involves the chlorination of titanium containing ores at 1300°K to produce TiCl_4 . Subsequent reduction of TiCl_4 with molten magnesium under an argon atmosphere produces metallic titanium in the form of a porous sponge. Sponge is removed from the reaction vessel and compacted into an electrode which then undergoes vacuum arc remelting to produce a conventional ingot of solid titanium [12]. Production of titanium via the Kroll process costs 70% more per ton than the production of aluminium and 14 times as much as steel [11].

The high raw cost of titanium (when compared to steels or aluminium alloys) has restricted its use to applications where its two major benefits are required by design, such as in the aerospace industry or in high corrosion components in power generators. The limited application made in the automotive industry is purely due to financial considerations. Many current research efforts are attempting to overcome this issue by utilising solid state processing, often involving titanium powders [13, 14], or through novel production methods from TiO_2 [12, 15].

The aerospace industry remains the largest consumer of titanium alloys, with each successive generation of airframes and gas turbine engines showing an increase in usage (Figure 1) [1, 16, 17]. The range of mechanical properties obtainable from titanium alloys through the modification of alloying additions (discussed in section 2.1.1) or through the close control of processing and forming operations (discussed in section 2.3) has led to the selection of titanium alloys for components throughout a modern aircraft. The high temperature capabilities of some alloys are utilised in the compressor section of a gas turbine engine, whilst the ability to produce high strength properties throughout a large component make other alloys suitable for use in landing gear assemblies [17].

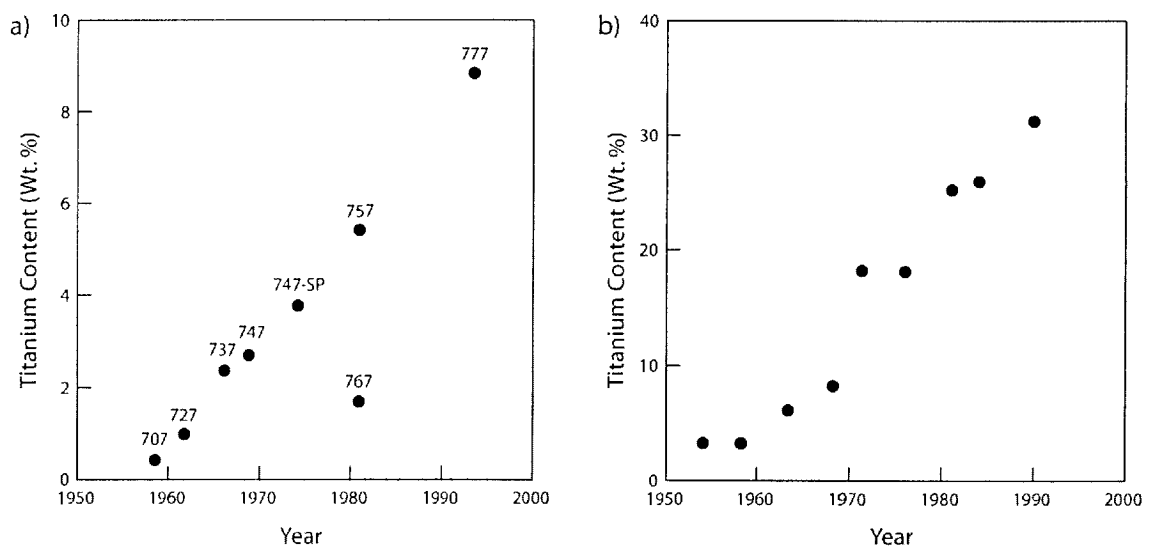


Figure 1: The increasing use of titanium products by the aerospace industry over the last 50 years; a) titanium content in commercial Boeing airframes (adapted from [1]) and b) titanium usage in Rolls – Royce gas turbine engines (adapted from [16]).

The inherent production costs of titanium alloys, when compared to cheaper competing materials, dictates a need for a more complete understanding of the fundamental metallurgy and interplay between processing and properties [10]. The development of process models for large components, based upon material descriptions from small scale studies has become key in reducing production costs, and now forms a large part of titanium research.

2.1.1 Alloying Titanium

Titanium metallurgy is dominated by the allotropic transformation of the high temperature body centred cubic (bcc) crystal structure (beta, β) to the low temperature close packed hexagonal (hcp) structure (alpha, α). The temperature at which this transformation occurs is known as the β transus temperature and in pure titanium occurs at $\sim 883^\circ\text{C}$. All commercial alloys contain additions intentionally chosen to alter the thermodynamic stability of the two phases, thereby changing the transformation temperature [18]. The manipulation of the transus and therefore the fraction of each crystal structure is the direct cause of the wide range of properties offered by different titanium alloys.

Alloying additions can raise, have no effect, or lower the transus temperature depending on which crystal type they stabilise. Elements which are soluble in the hexagonal phase increase the transformation temperature (Figure 2a) and are known as α stabilisers. This range of additions includes aluminium as a substitutional alloying element and interstitial elements such as oxygen, nitrogen and carbon. Aluminium is the most commonly employed α stabiliser as it is the only element for which significant level of solubility are observed in both bcc and hcp crystal structures.

Elements such as zirconium and tin essentially have no effect on the β transformation temperature (Figure 2b) and are therefore referred to as neutral alloying elements. The metallurgy of zirconium is identical to titanium and as such complete solubility is observed between the two. A tin-titanium intermetallic compound can form if tin is present in sufficient concentration, however, the β transformation temperature remains unaffected [10].

Alloying additions which lower the β transus do so by stabilising the bcc crystal structure and are known as β stabilisers. There are two classes of β stabilisers, either isomorphous or eutectoid forming depending on their relevant binary phase diagram. Commonly used isomorphous stabilising elements are molybdenum, vanadium, niobium and tantalum, all of which have a bcc structure. Isomorphous additions (Figure 2c) are completely soluble in titanium system and simply stabilise the cubic structure so

that it exists at lower temperatures. Eutectoid forming elements (Figure 2d) show solubility in the titanium system up to a given concentration above which intermetallic compounds will begin to form. Common examples of eutectoid forming additions are iron, chromium and silicon.

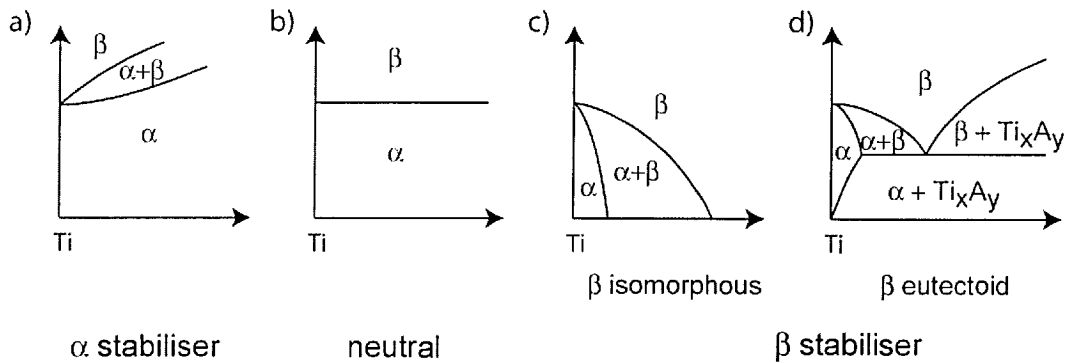


Figure 2: Schematic representation of the effect of alloying elements on the phase diagrams of titanium alloys (adapted from [10]).

An understanding of the individual binary systems belonging to titanium and its alloying elements provides the basis for the metallurgy of the more complex multi component commercial alloys. However, the influence of each individual stabilising element varies and therefore different alloy compositions result in a greater or lesser stabilisation. A working relationship for equating alloying and the relative stability of a given system was outlined by Bania [2]. The strength of a given β stabiliser can be determined by evaluating the level of that stabiliser required to suppress the β transus sufficiently such that 100% β phase is retained upon quenching to room temperature (Table 1). Molybdenum is used as a standard reference from which all other β stabilising elements are equated. A similar system for α stabilising elements was outlined by Rosenberg [19] using aluminium as its standard reference. Aluminium has a β equivalence exactly inverse to that of Mo, thus the two systems can be combined to provide a qualitative view of a given alloys stability. For a given alloy system the level of β stability ('moly equivalence' or Mo Eq) can be calculated by the summation of all the alloying element's contribution, which is their weight fraction multiplied by their stabilising equivalence.

Table 1: Concentration of common titanium stabilising elements required to retain β upon quenching to room temperature and equated to produce relative moly equivalence (Mo Eq - data adapted from [2,19][19]).

Element	Wt.% to retain β	Mo Eq
Mo	10.0	1.00
V	15.0	0.67
Cr	6.5	1.54
Fe	3.5	2.86
Nb	36	0.28
Ta	45	0.22
Al	-10.0	-1.00
O, C, N	-	-10.00

The moly equivalence system provides a simple way to compare complex titanium alloy systems and to gauge the relative level of β stability. An alloy with a higher moly equivalence value has a greater level of β phase stabilisation and therefore the β phase field extends to lower temperatures. Changes to the β transition temperature will alter the equilibrium microstructures of an alloy and when combined with thermomechanical processing enables titanium alloys to offer the wide range of properties mentioned in the previous section [20].

Titanium alloys can be divided into five general categories determined by the contribution of the two phases in the microstructure. The alloy categories; (i) α , (ii) near α , (iii) $\alpha+\beta$, (iv) metastable β and (v) β , can be seen superimposed on a pseudo binary phase diagram in Figure 3 and a brief overview of each will be given in turn.

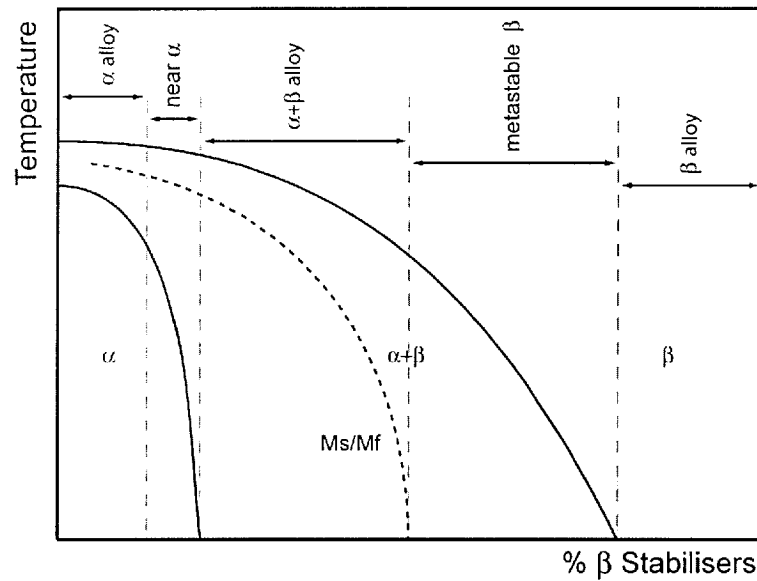


Figure 3: Schematic representation of a titanium pseudo binary phase diagram with the general alloy categories. Ms and Mf represent the martensitic transformation start and finish lines respectively (adapted from [20, 21]).

The α class of alloys are defined by an inability to retain the β phase in any form at room temperature [20]. Cooling α alloys through the β transus results in a complete transformation from a cubic structure to a hexagonal structure. These alloys show good ductility down to low temperatures and are readily weldable. However, the hexagonal structure and a high level of work hardening results in poor malleability [11]. Common examples of this type of alloy are the commercially pure grades of titanium, strengthened by interstitial α stabilising elements (e.g. oxygen). These alloys are utilised in sheet form where excellent corrosion resistance is required, such as tubing for the fuel and hydraulic systems in an aircraft [16, 17].

Near α alloys offer higher room temperature strength than α alloys and display the best creep properties of any titanium alloys [11]. The addition of up to 2 wt.% β stabilising elements widens the $\alpha+\beta$ region which improves alloy formability by enabling hot working in both the $\alpha+\beta$ and the β phase fields. Additionally small amounts of metastable β phase can be retained if sufficient solute enrichment occurs during processing. The performance of near α alloys in the 400°C to 600°C temperature range has led to the extensive use of alloys such as TIMETAL834 (Ti-5.8Al-4Sn-3.5Zr-

0.7Nb-0.5Mo-0.35Si-0.06C) throughout the compressor section of a gas turbine engine [17, 20].

The $\alpha+\beta$ alloys are contained in a region between the $\alpha/\alpha+\beta$ boundary and the martensitic transformation line (Ms/Mf) [20, 21]. Flower [20] noted that the martensitic transformation start and finish lines in titanium alloys lie very close together and therefore alloys either undergo a full martensitic transformation or retain a fully β structure when rapidly cooled. Therefore, since $\alpha+\beta$ alloys are defined as compositions left of this line, they will undergo a full martensitic transformation when quenched. The increased level of β stabilisers (4 – 6%) extends the $\alpha+\beta$ phase field to room temperature allowing a wide range of microstructures and volume fractions to be developed through different processing routes [11]. This class of alloy provides the bulk of commercial titanium use through the versatile alloy Ti-6Al-4V. The alloys offer a good balance of mechanical properties and formability due to the increased presence of the β phase. However, the increasing the β phase fraction is deleterious to creep resistance and therefore restricts $\alpha+\beta$ alloys to applications where working temperature are less than 400°C, such as forged fan blades for a gas turbine engine [17].

Metastable β alloys (sometimes referred to as near β alloys) are $\alpha+\beta$ alloys with sufficient β stabiliser concentrations to depress the martensitic transformation line below room temperature. Thus metastable β alloys retain a fully β microstructure following rapid cooling from the β phase field. Martensitic phases (discussed in section 2.1.2) have been observed at room temperature in some metastable β alloys but detailed examination has shown that these transformations are caused by the application an external stress [22, 23]. The level of β stability in metastable β alloys is such that α phase nucleation and growth processes are sufficiently retarded to enable the retention of metastable β at room temperature following slow cooling. The depression of the β transus allows the processing of metastable β alloys at temperatures well below that of $\alpha+\beta$ alloys. For example Ti-10V-2Fe-3Al is processed at ~800°C, with similar loads to that required for Ti-6V-4Al when processed at ~950°C [5]. Metastable β alloys can be processed to give the highest strength of all titanium alloys, well in excess of 1GPa [24]. The excellent workability of near β alloys, combined with deep hardenability and corrosion resistance has made this alloy class ideal for large section applications. Small

amounts of Ti-10V-2Fe-3Al were used in the Boeing 757 airframe, whilst both the Boeing 777 (introduced in 1995) and the Airbus A340 -500 and -600 (introduced in 2002 and 2001 respectively) airframes utilised Ti-10V-2Fe-3Al rather than steel for the large truck beam forgings in landing gear assemblies [17, 25, 26].

A true definition of a β alloy would require sufficient β stabilising additions to make the β phase thermodynamically stable at room temperature [11]. Only a few commercial alloys of this type exist, one example being the burn resistant system Alloy C (Ti-35V-15Cr), and so the term is widely expanded to incorporate metastable alloys where a martensitic transformation does not occur, either from rapid cooling or under applied load. The high level of β stabilisation is often enough to effectively eliminate the β to α transformation, even during industrial heat treatments [20]. This allows the production of uniform thick section material which can be easily cold worked. β alloys have been used for high strength applications, for example Beta III (Ti-11.5Mo-6Zr-4.5Sn) as fasteners in many airframes. Despite the numerous commercial β alloys available the total market usage is very small. However, the low elastic modulus and high yield stress offered by β alloys are seeing an increase in their popularity for applications such as springs [21].

For the sake of simplicity throughout the rest of this thesis the term β alloy will be used generically to represent any alloy with a composition such that it is able to retain a β microstructure following rapid cooling.

2.1.2 Microstructural development in β alloys

The development of microstructures in titanium alloys is dependent on the alloy chemistry, the thermal history and the processing history [27]. Deviations to any of these process variables, for example cooling rate, can lead to the development of completely different microstructures. In addition to the equilibrium α and β phase briefly mentioned already, β titanium alloys can form a number of non equilibrium phases such as the solute lean beta phase (β'), the martensites (α' and α'') and the omega phase (ω). This section briefly discusses the development of α , the martensitic phases

(α' and α'') and β' , whilst section 2.2 describes the formation and effect of the omega phase in more detail.

The transformation of the high temperature cubic structure into the lower temperature hexagonal structure can either occur through a diffusion based nucleation and growth process, or through a martensitic transformation when cooled rapidly. Regardless of the mechanism a basic orientation relationship exists between the two structures: $(110)\beta \parallel (0001)\alpha$; $[111]\beta \parallel [11\bar{2}0]\alpha$ (Figure 4). This relationship was first characterised by Burgers [28] when working with zirconium, but it was also later found to correspond to titanium [29]. Inline with this relationship there are twelve individual hcp variants which can form from each parent bcc crystal.

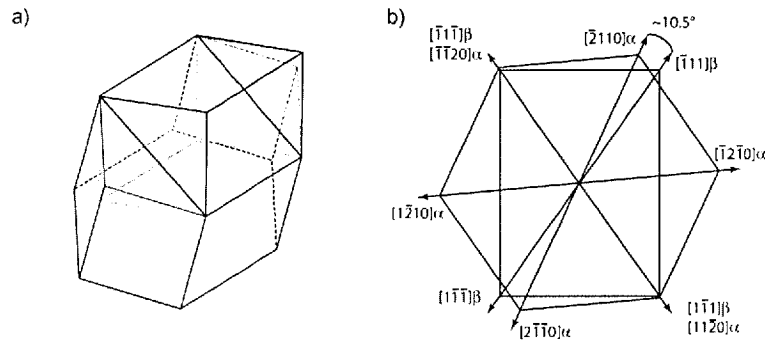


Figure 4: Schematic representations of a) the relationship between α and β crystal structures, and b) a detailed view of the Burgers relationship looking down the $[0001]\alpha \parallel [110]\beta$ (adapted from [30]).

Transformation of the β phase to the α phase under equilibrium conditions occurs via nucleation and growth, controlled by the diffusion of the different alloying elements [31]. When cooled very slowly α is first formed at the β grain boundaries, rapidly becoming a complete layer. From this layer, colonies of parallel plate like α precipitates sympathetically nucleate and undergo slow diffusional growth towards the grain centre (Figure 5a). Individual α plates are separated by thin layers of retained β , further stabilised by solute rejection during α growth. The α plates maintain an adherence to the same Burgers variant as that of their parent grain boundary layer [30].

Increasing the cooling rate decreases both the colony size and the individual α plate width. Faster cooling also encourages further nucleation and new colonies form on both the grain boundary layer and existing colonies. New plates minimise their formation energy by orientating the major axis perpendicularly to the broad face of the existing colony [21]. This formation mechanism is a form of variant selection and combined with the thinner plates gives rise to the characteristic basketweave structure (Figure 5b). Each colony acts as a single structural unit since the low misorientation between α plates facilitates easy slip transfer [20].

Further increasing the cooling rate gives rise to increased nucleation, a finer α plate size and the appearance of more α variants (Figure 5c). This microstructure is a true representation of the Widmanstätten morphology where multiple plates can no longer be thought of as a single structural unit [20].

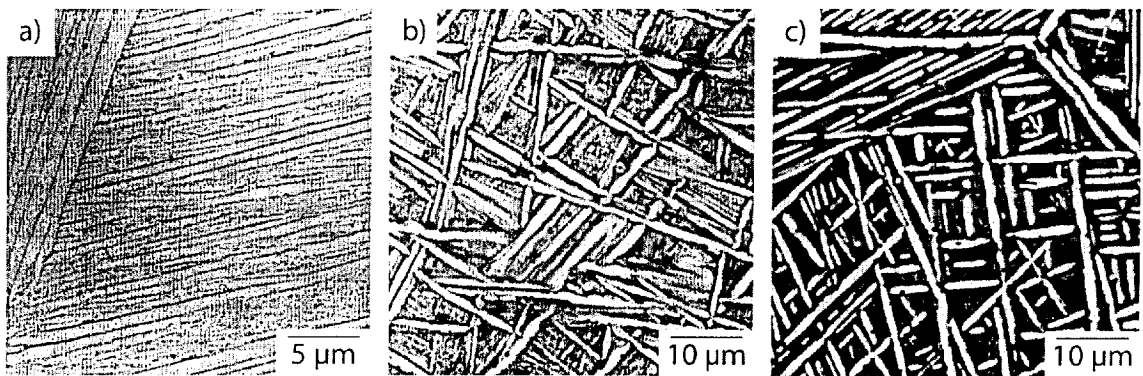


Figure 5: Light micrographs illustrating the different microstructures achievable by varying cooling rate, when cooling titanium alloys from the β phase field; a) furnace cooled, showing a large colony of aligned α plates, b) air cooled, showing a typical basketweave structure and c) rapidly air cooled the Widmanstätten α structure (all adapted from [20]).

When cooling rates become rapid and diffusion driven processes are insufficient to relieve the necessary reduction in system energy, transformations occur by massive cooperative atomic movement. The nature of the martensitic transformation in titanium alloys is dependent upon the level of β stabilisers. Alloys with a leaner solute content

transform to the hexagonal close packed α' phase (Figure 6b) while alloys rich in β stabilising elements transform to the orthorhombic α'' martensite (Figure 6c).

The α' martensitic transformation in pure titanium occurs as a massive transformation, producing a microstructure consisting of large irregular zones approximately 50 – 100 μm in size. Each zone consists entirely of parallel martensitic plates less than a micron wide [31]. As system solute content increases the martensitic colony size decreases and at sufficiently high solute concentrations the colonies degenerate entirely into individual acicular plates. The onset of this colony degeneration has allowed the determination of the orientation relationship between the α' martensite and its parent β phase, which is found to uphold that determined by Burgers (i.e. $(110)\beta \parallel (0002)\alpha'$; $[111]\beta \parallel [11\bar{2}0]\alpha'$) [31]. Once complete degeneration has occurred the acicular martensite is generally found to adopt a $\{334\}\beta$ habit plane [32].

The α'' martensite occurs in alloys with a higher level of β stabilising elements than those that form α' . The α'' martensite has been reported in many binary systems (e.g. Ti-Mo and Ti-Nb) but interestingly not in the Ti-V system unless Al is added [31]. This type of martensite has an orthorhombic crystal structure (Cmcm) which can be described as a slightly distorted hexagon with lattice parameters between those of the bcc parent matrix and the hexagonal α' [22]. By describing both bcc parent and orthorhombic martensite in orthorhombic space (Figure 6c) a generic orientation relationship can be produced which is synonymous of the Burgers relationship. The lattice parameter of α'' martensite has been found to be highly dependent on alloy composition. In many systems the $a_{\alpha''}$ has been observed to increase with increasing solute content whilst the $b_{\alpha''}$ and $c_{\alpha''}$ both decrease [31, 33]. An important feature when considering the formation of α'' is the almost vertical nature of the martensitic transformation line below temperatures of $\sim 400^\circ\text{C}$. Alloys with nominal compositions close to this line may well fluctuate between a martensitic microstructure or a retained β microstructure following rapid cooling dependent on by local chemistry [22].

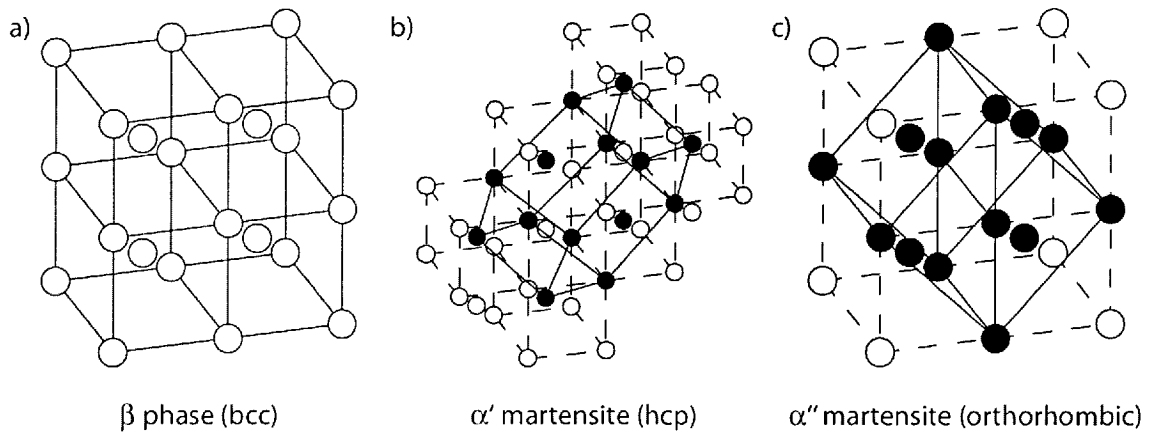


Figure 6: Schematic representation of martensitic crystal structures in relation to the β phase a) parent β phase (bcc), b) α' martensite (hcp) and c) α'' orthorhombic martensite.

β alloys, with a greater concentration of β stabilisers, suppress the martensitic transformation line below room temperature. As such the martensitic transformations (discussed above) are avoided when rapidly cooling from the β phase field. However, the retained metastable β structure is also susceptible to decomposition into other metastable phases upon relatively low temperature aging. Figure 7 shows a schematic phase diagram incorporating the metastable phase fields of $\omega + \beta$ and $\beta + \beta'$. Alloys with compositions close to the martensitic transformation can form the ω phase whilst those with increased β stability undergo a separation of the β phase into β and β' .

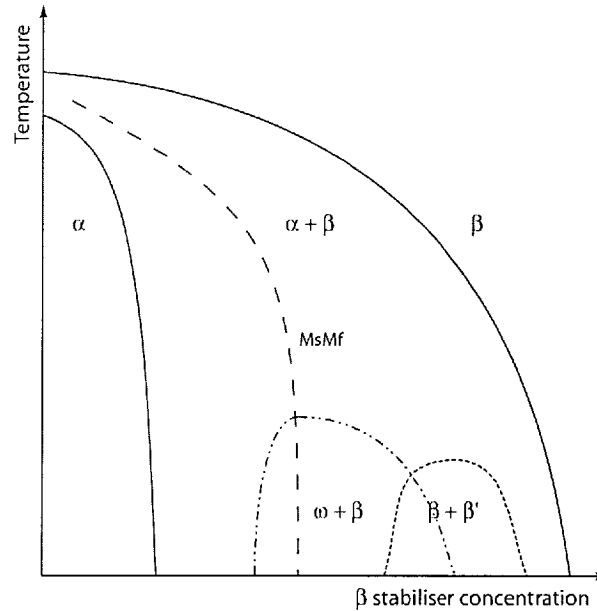


Figure 7: Schematic pseudo binary phase diagram showing metastable $\omega + \beta$ and $\beta + \beta'$ phase fields (adapted from [22]).

The $\beta \rightarrow \beta + \beta'$ transformation occurs during low or medium temperature aging (between 200 – 500°C) in alloy systems which exhibit a miscibility gap [21, 31]. Upon aging in the $\beta + \beta'$ phase field the β phase separates by solute rejection into solute lean regions (β') within a solute rich β matrix [34]. The majority of reported β' observations have been made using transmission electron microscopy on binary Ti alloys [35-39], although some evidence has also been found using high angle X-ray diffraction [40]. Several morphologies of β' have been observed, ellipsoidal, cuboidal and disc shaped, depending on the alloying additions [34, 37, 38]. The $\beta \rightarrow \beta + \beta'$ transformation is an important decomposition mechanism for metastable β alloys of composition too rich to decompose through the $\beta \rightarrow \omega$ transformation [34]. The formation of the β' regions prior to the formation of the ω phase has been reported [39], which in combination with other data lead to the suggestion that β' may be a precursor to the ω phase. However, the work of Chandrasekaran *et al.* [39] on a Ti-Cr alloy found that the isothermal ω phase nucleated in the matrix rather than from the β' precipitates. It has also been hypothesised that the solute lean precipitates will act as favourable nucleation sites for the α phase due to the low solute content [34, 37].

2.1.3 Secondary processing and mechanical properties of beta titanium alloys

The fact that β titanium alloys can be cooled rapidly without undergoing a martensitic transformation enables a significant ability to achieve a wide range of properties through subsequent (secondary) processing. The wide $\alpha+\beta$ region allows lower temperature processing than the other classes of titanium alloys and attractive combinations of strength, toughness and fatigue resistance can be achieved [3]. The source of these properties is the precipitation of small α phase particles within an inherently ductile matrix [41]. The highest strength alloys are those with compositions just to the right of the martensitic transformation line (Figure 3), as these alloys can precipitate the highest volume fraction of the α phase.

Secondary processing of β alloys usually involves a hot working operation and heat treatment, followed by accelerated cooling and subsequent aging. During the first stage of this processing the heat treatment temperature controls the fraction of primary α developed, whilst the level of deformation imparted during hot working controls the α morphology. Without deformation a needle morphology is developed, which becomes more globular with increased working [3]. During the lower temperature aging, typically carried out in the 400°C – 600°C range, secondary α is precipitated within prior β grains. The careful control of the processing route enables the desired β grain size and the volume fraction, type and morphology of both primary and secondary α precipitates to be achieved. The hot working of β titanium alloys will be covered in more detail in section 2.3.

In general the best overall combination of mechanical properties is achieved with a duplex structure combining globular primary α with acicular secondary α throughout all prior β grains. However, for some applications specific properties or combinations of properties may be desired. Strength can be increased by decreasing the fraction of primary α and by obtaining a well dispersed microstructure of fine scale acicular α [22]. Conversely the ductility of the material will decrease with a reduced level of primary α [42]. These two properties are highly interrelated since a change to the volume fraction of one will inversely effect the potential volume fraction of the other. Fracture

toughness and fatigue crack growth are both improved by the more tortuous crack path created by an increased content of acicular morphology α and a larger β grain size. However, an overall improvement in fatigue resistance is not necessarily gained by an acicular morphology as the nucleation level of fatigue cracks is higher [20].

It is clear that understanding the complex correlations between mechanical properties and microstructure is essential to achieving an optimised state for a given application. The unique microstructural response of each alloy in relation to hot working conditions means that each system must be evaluated prior to producing technically reasonable processing routes [3].

2.2 The Omega Phase (ω)

The ω phase was first identified by Frost *et al.* [43] when aged β phase titanium alloy samples were found to be unexpectedly brittle. X-ray diffraction of the brittle material revealed the presence of a secondary phase, which was designated as the ω phase. Subsequently the ω phase has been reported in many group IV transition systems and has generated a lot of interest both experimentally and theoretically [44].

The $\omega+\beta$ phase field exists over a compositional range with a relatively high concentration of β stabilizers (Figure 7). Within this field the local chemical composition is thought to influence the mechanism by which ω is formed. For alloys with compositions close to the lower limit for β retention ω phase formation is observed athermally during rapid cooling from the β phase field [31, 44]. In more heavily stabilised alloys the ω phase forms during low to medium temperature aging (300 – 500°C) and is termed isothermal ω . Whilst the formation mechanisms are different the final structures of the two forms are the same and both forms are coherent with the matrix [21, 45]. Early work investigating the ω phase proposed several different crystal structures, however, it has now established that ω either exists as a simple hexagonal structure (P6/mmm) or a trigonal structure (P-3m1) [46].

2.2.1 Athermal Omega

Athermal formation of ω was initially thought to be a martensitic type reaction as it could not be suppressed by ultra high cooling rates (11000°C sec⁻¹) [47]. However, transmission electron images of the athermal ω showed a high density of small ellipsoidal particles, reported in the range of 2 - 40 nm [22, 45, 48]. The small size and the morphology of the observed ω particles prevents accepted theories regarding martensitic transformations from accounting for athermal formation [31]. This inability to describe athermal ω formation mechanism led de Fontaine to propose a new class of displacement-controlled transformations [49].

The ω transformation as described by de Fontaine [49] is a series of longitudinal displacements in the form $2/3 \langle 111 \rangle$, which involves small atomic shuffles of $\pm a_{\beta} \sqrt{3}/12$

where a_β is the β lattice parameter [22, 44]. This mechanism is more easily understood as the regular collapse of two out of every three consecutive $(111)\beta$ planes leaving the third plane unaltered (Figure 8). Complete collapse of these planes results in the formation of the ideal hexagonal structure ($\omega_{(3)}$) with $a_\omega = \sqrt{2}a_\beta$ and $c_\omega = \sqrt{3/2}a_\beta$. Incomplete, or partial collapse, where atomic shuffle $< \pm a_\beta \sqrt{3}/12$, leads to the trigonal structure [44]. The displacement mechanism is not only capable of describing the correct structural change it is also in accord with the experimentally determined orientation relationship between β and ω , $\{111\}\beta \parallel (0001)\omega$; $[110]\beta \parallel [11\bar{2}0]\omega$ [46]. This orientation relationship between observed between ω and β gives rise to four different possible ω variants (parallel to the four $[111]\beta$) which are equally populated unless the material has been deformed [50].

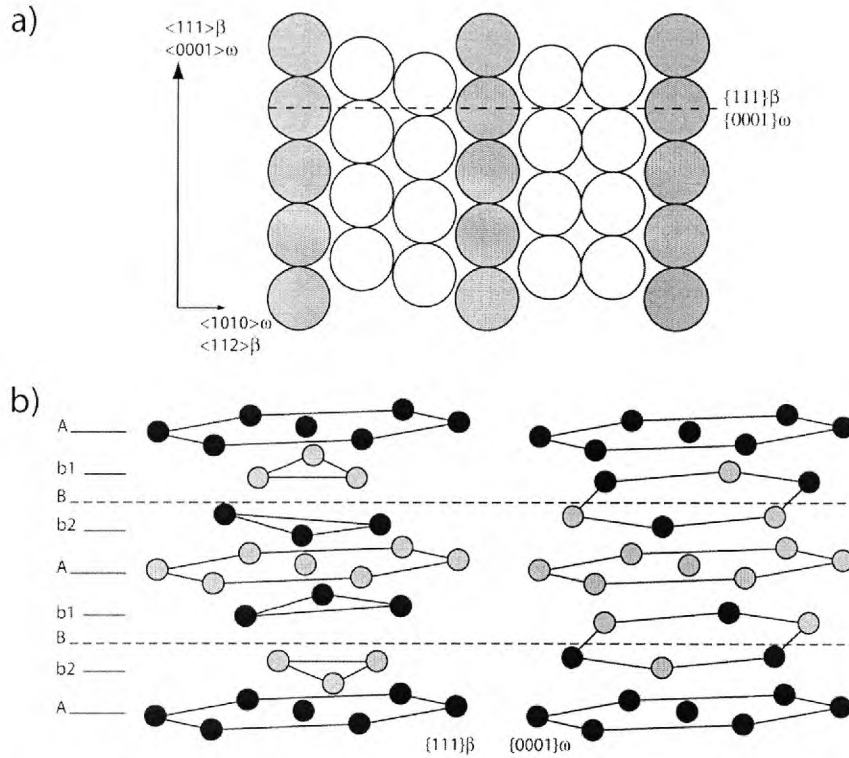


Figure 8: Schematic representations of the $\beta \rightarrow \omega$ transformation; a) through section of β (left) and ω (right) crystal structures showing the $\{111\}\beta$ collapse mechanism (adapted from [47]), and b) hexagonal structure of ω phase indicating the ω_3 structure by the creation of plane B from planes b1 and b2. Different colours are used to aid interpretation.

It has been suggested that at high temperatures, well above the supposed ω start temperature (300 - 500°C in near β alloys [22]), embryonic ω or “ ω -like fluctuations” are present [44, 51, 52]. These structures can be thought of as a bcc lattice defect produced by atomic jumps in the $\langle 111 \rangle$ direction causing local plane collapse. The highly correlated displacements give significant concentrations, even at high temperatures, where the structure is maintained for longer than a typical atomic vibration [48]. At higher temperatures the embryos persist as uncorrelated line defects aligned with the $\langle 111 \rangle$ direction, meaning that each diffracts differently until cooled closer to the ω transition temperature, when the correlation increases and discrete ω particles are formed [48].

The complete reversibility of the athermal transformation was established by de Fontaine *et al.* investigating a Ti-Mo alloy [53]. When studying the alloy with cold stage microscopy the ω phase was observed to form upon cooling and then revert to its parent β when returned to room temperature. This observation confirmed the diffusionless nature of the transformation and added further support to the displacement mechanism. In the course of this investigation the authors also noted diffuse streaking (Figure 9b and c), between β matrix reflections in electron diffraction patterns rather than distinct reflections (Figure 9a) from the ω phase, a feature also observed in other studies and characteristic of the athermal transformation [45, 54, 55].

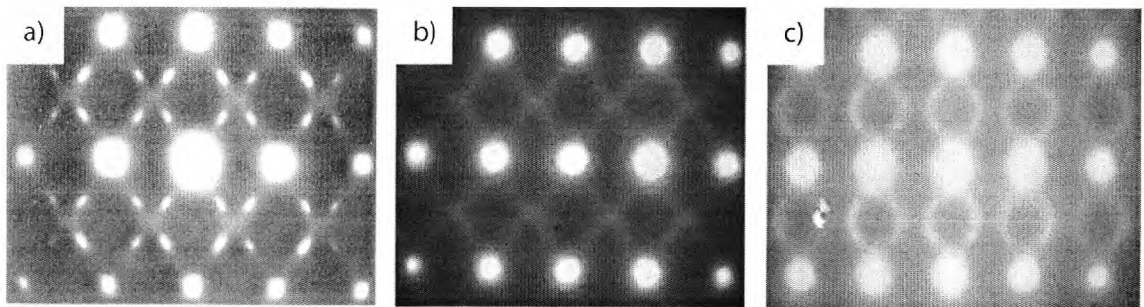


Figure 9: Typical electron diffraction patterns from the $[110]\beta$ zone axis of a) commensurate ω [54], b) diffuse streaking attributed to incommensurate ω formed via the athermal transformation [56] and c) curved diffuse streaking as seen by de Fontaine [53].

Sass [57] proposed that sharp reflections resulted from diffraction from clusters of ω rows, while diffuse scattering was the result of individual ω structures, producing sheets of intensity perpendicular to $\langle 111 \rangle \beta$. However, this explanation is unable to account for the curved nature of the streaking observed in some systems (Figure 9c) [53]. Instead de Fontaine proposed that the local strain fields associated with each omega variant interact with each other, causing the variants to become coupled. As solute content is increased the extent of variant coupling also increases leading to streaking which becomes less rectilinear and more circular [53].

Dawson [54] observed that ω reflections progressively changed from sharp reflections to diffuse scattering as the solute content was increased when working with Zr-Nb alloys. Subsequently several alloy systems have been used to study the as quenched ω form and solute type as well as concentration level have been identified as affecting ω diffraction characteristics [58]. High resolution transmission electron microscopy of the Ti-Mo system has shown that in alloys with leaner concentrations, ω particles form parallel to the $\langle 111 \rangle \beta$ and with diffraction peaks in commensurate positions. At higher solute contents the reflections are found to be incommensurate and ω particles deviate from the $\langle 111 \rangle \beta$ by up to 10° , rotating towards the $[110] \beta$ [59]. This observation has been rationalised by coalescence of multiple ω embryos. The selection of the collapsing planes in a given embryo is random and for each individual case will be aligned with $\langle 111 \rangle \beta$. If two variants, which did not form along a common $\langle 111 \rangle$, coalesce then their combined orientation will not align with any given $\langle 111 \rangle$, instead their alignment will be observed as a rotation towards $[110] \beta$ (Figure 10). Sinkler and Luzzi [58] suggested a structural model capable of describing the observed shifts of the diffuse ω streaking away from commensurate Bragg positions. Creating the $\omega_{(5)}$ or $\omega_{(7)}$ structures by the addition of collapsed plane pairs (Figure 11), they showed that combinations of these structures could account for the observed shifts in diffraction intensity maxima.

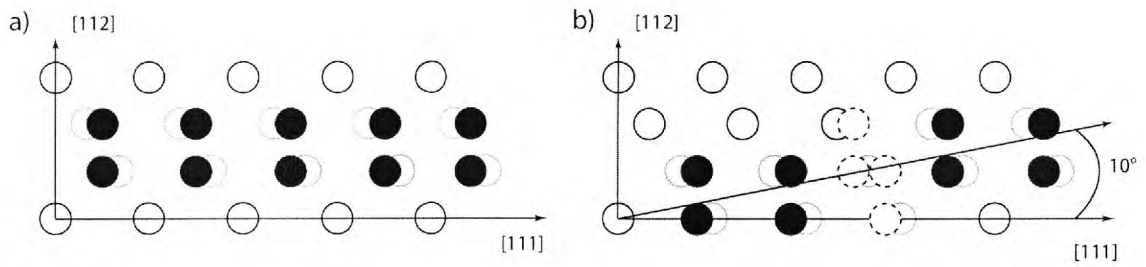


Figure 10: Atomic models proposed for a) commensurate ω single modulation along $\langle 111 \rangle_\beta$ and b) coalescence of two similar embryos producing a 10° variation from a single $[111]_\beta$ (incommensurate ω) [59].

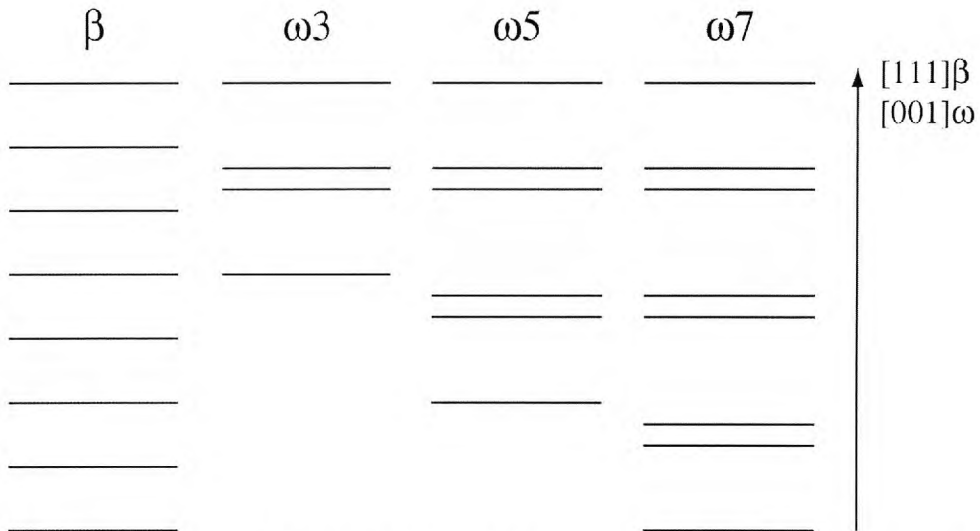


Figure 11: Structural model proposed by Sinkler and Luzzi [58] of inserting a collapsed plane pair to create the $\omega_{(5)}$ and $\omega_{(7)}$ structures, thereby accounting for the incommensurate positions of electron diffraction patterns.

2.2.2 Isothermal Omega

The fundamental mechanism of ω formation, the collapse of $(111)_\beta$ planes, is identical for both isothermal ω and athermal ω . However, isothermal formation is governed by diffusion and therefore differences in particle morphology and ordering length scale can be found when compared to athermal ω [48]. Low to medium temperature aging (in the

300 – 500°C range) offers favourable conditions for diffusion and concentration fluctuations develop, similar to those of characteristic of spinodal decomposition. Continuous segregation leads to the development of solute lean regions within an enriched matrix [60]. Solute lean regions are then able to transform to the ω structure through the plane collapse mechanism discussed in section 2.2.1. The diffusional nature of the precursive segregation is evident through the changing lattice parameter of the β phase during aging [60]. The level of segregation is such that even in heavily stabilised alloys, the composition of isothermal ω will eventually be in the same range as that of an athermally formed particle [44, 45]. The isothermal ω transformation is the most rapid of all the diffusional β phase decomposition modes and hence why it is observed in so many systems [22].

The morphology of the isothermal ω phase is dictated by misfit of the alloying elements. High misfit systems, such as titanium alloyed with Fe, V or Cr, produce a cuboidal ω morphology, with the faces of the cubes aligned to $\{100\}\beta$ [37]. Low misfit systems, for example titanium alloyed with Mo or Nb, produce an ellipsoid morphology with the major axis aligned to $\langle 111 \rangle \beta$ [31]. Isothermal ω is generally larger in size than athermal ω , with particle sizes of ~ 200 nm having been reported [37], and due to the nature of its formation only commensurate diffraction peaks are observed from the ideal hexagonal structure. The formation of isothermal ω has been likened to that of β' , with the major difference between the two being the greater level of misfit between the matrix and the β lean regions of the ω phase [21].

2.2.3 Influence of alloying elements

The ω phase has been reported in many binary titanium systems, when alloyed with a β stabilising element in sufficient concentration. However, most commercial alloys are multi-component systems, and are likely to contain some level of α stabilising elements. Williams *et al.* [34] considered the effect of the two most common α stabilising elements (Al and O) on binary systems of Ti-Mo and Ti-V. It was found that both Al and O suppressed the stability of the ω phase by accelerating the kinetics of α phase nucleation. The effect of the neutral stabilisers Zr and Sn was also investigated. These additions also decreased the ω stability, however, the suppression mechanism was

different to that of the α stabilisers. Rather than encourage the $\beta \rightarrow \alpha$ transformation, it was hypothesised that Zr and Sn acted to enhance the stability of the β phase, therefore suppressing the decomposition into the ω phase.

2.2.4 Mechanical influence of the omega phase

The precise effect of the ω phase on the mechanical properties of β titanium alloys is not well understood. Several investigations considering the effect of ω particles in various alloy systems have failed to provide a conclusive or corroborative description. It is generally thought that the presence of ω particles is embrittling and therefore is avoided by industrial thermal regimes [61].

The β phase will deform through slip along the $\langle 111 \rangle$ commonly on $\{110\}$ or $\{112\}$, whilst hexagonal structures will slip either along $\langle 11\bar{2}0 \rangle$ on $\{0001\}$, $\{10\bar{1}0\}$ and $\{10\bar{1}1\}$ or $\langle 11\bar{2}3 \rangle$ on $\{11\bar{2}2\}$. The four ω variants lie parallel to the $\langle 111 \rangle_\beta$ and therefore for any given cubic slip system only one of the ω variants slip system will be coincident, with the other three acting as barriers to dislocation movement [46]. However, the coherent nature of the interface between the ω and β phases will allow the shear of ω particles [21]. The required stress associated with cutting ω particles has been shown to be lower than that required for a bypass mechanism [62]. Thus, during deformation dislocations shear the ω particles, effectively destroying their structure and create localised slip bands with limited resistance to further dislocation movement. The formation of slip bands leads to the nucleation of cracks, as voids created at the intersection of slip bands act as crack nuclei [62]. Conversely to this, dislocation loops indicating particle bypass have been reported in Ti-Mo and Ti-V systems [63].

The size and volume fraction of the ω particles has also been identified as being pertinent to the mechanical response of an alloying system. Lin *et al.* [64] observed a brittle tensile failure in a rapidly cooled Ti-7.5Mo-1Fe alloy whilst a Ti-7.5Mo-2Fe alloy with the same thermal history exhibited ductile behaviour. The difference in behaviour between the two alloys was attributed to the reduction of dislocation slip caused by the larger ω particles observed in the 1 wt.% Fe alloy.

Experimental results have shown that ω containing microstructures are not necessarily brittle following rapid cooling. However, subsequent aging in the $\beta + \omega$ field was accompanied by ω growth and corresponding embrittlement [46, 62]. Bowen [65] summarised that larger ω particles (for a given volume fraction) would interact with a greater number of slip systems than smaller particle sizes. Increased slip interaction would be deleterious to cross slip and would lead to a greater dislocation pile up, which would result in void formation at lower strains. Congruent to these findings, Williams *et al.* [63] identified a critical volume fraction of 25%, below which ω particles appear to have no major influence on either strength or ductility.

A conclusive description of the effect of the ω phase on the mechanical properties of β titanium alloys or the relevant deformation mechanisms has still yet to be found. The general acceptance that ω embrittles the material, as reported by most studies, essentially eliminates the concept of an ω hardened engineering alloy. Therefore, continued interest in the ω phase lies primarily in its ability to influence subsequent precipitation sequences [22, 66].

2.2.5 The influence of the omega phase on subsequent microstructural development

Metastable β alloys have the highest strength of all titanium alloys (see section 2.1.3) due to the ability to precipitate harden the β phase. Exceptional strengths in excess of 1.4 GPa have been reported in some alloys and one proposed source of this strength is the development of fine scale α precipitates (~ 20 nm by 200nm) [22, 67]. The development of a microstructure which contains fine, well distributed α precipitates requires multiple site nucleation and limited growth. The ability to use metastable phases as nucleation sites for equilibrium phases is well established in aluminium alloys [11]. Therefore, many studies have investigated the $\beta + \omega \rightarrow \beta + \alpha$ transformation, in an attempt to engineer an improved α distribution and morphology.

Different investigators have reached opposing conclusions as to whether the α phase nucleates from ω or if the ω particles become the α phase. X-ray diffraction studies concerning the changes in lattice parameter during the aging of a Ti-V alloy led

Hickman [60] to suggest that α formed by direct conversion of ω particles. The α phase was observed to have nucleated with a composition similar to that of the ω phase and subsequently rejected V until reaching its equilibrium composition. Flower and Swann [68] reported the formation of the α phase preferentially on stacking faults within previously formed ω particles. Conversely Ageyev *et al.* [69] reported that the ω phase in a Ti-Mo alloy dissolved back into the β matrix prior to α phase formation. It was hypothesised that when the ω particles revert to the β phase, titanium rich regions remain, which encourage the nucleation of the α phase.

Williams [31] suggests that formation of α in ω containing microstructures appears to be related to the misfit of the system caused by its alloying elements. In low misfit systems (Ti-Mo, Ti-Nb) the ω phase appears to have no role in α nucleation, however in high misfit systems (Ti-V) the β/ω interface is observed to act as a nucleation point for α laths. The α precipitates in the latter case were observed to have a much more uniform distribution and a higher precipitate density when compared to α precipitated in low misfit systems. However, aging at temperatures very near to the ω start temperature, where the ω phase persists for only a short amount of time, α precipitates appear to form directly from an ω particle regardless of system misfit. Precipitates formed by such a mechanism appear to adopt the morphology of the prior phase [70].

More recently, Ohmori *et al.* [71] have employed high resolution transmission electron microscopy to investigate the effect of the ω phase on subsequent phase transformations. Evidence was found for α lath nucleation at the interface between the ω and β phases. It was also reported that the growth of such a nucleated α precipitate often penetrated and consumed the nucleating ω region. Sakedai [72] has also recently reported the formation of a region (with lattice spacing consistent with the α phase) adjacent to an ω particle and concurred that the α crystal grew from the ω particle. However, a 'transitional' region was also observed adjacent to an ω particle, with a lattice spacing in-between that of either ω or α . The author postulated that this region might transform to the α phase, which would not follow the nucleation and growth mechanism which had seemingly become accepted.

2.3 *Hot working of β titanium alloys*

Titanium alloys are more difficult to process than other engineering materials, such as steels or aluminium alloys. Typical processing of β titanium alloys can be subdivided into two major operations, primary and secondary processing. Primary processing breaks down an ingot to achieve an improved microstructural and chemical homogeneity and to produce usable product forms, such as bar, plate or billet. Secondary processing achieves the desired microstructural condition and the final shape of a component such that minimal finishing machining is required. This section provides a brief overview of primary processing, before focussing on the deformation mechanisms and process modelling relative the commonly employed secondary processing method of isothermal forging.

2.3.1 Primary Processing

As cast vacuum arc re-melted ingots are inherently non uniform with a microstructure consisting of equiaxed, columnar and dendritic grains [73]. Since material properties are dependent on microstructure, it is essential that during final processing a material behaves and responds in a uniform manner. Thus, the aim of primary processing is to create an intermediate product form of a suitable shape suitable for further processing and with a uniform structure.

Typically this is achieved through preheating the ingot in the β phase field, followed by hot working. Ingots are cogged using an open forging press into billet form. Between each bite the ingot is rotated by 90° around its major axis and moved forwards, thus producing longer smaller diameter billets [5]. The deformed ingots are cooled rapidly using fan assisted cooling to minimise the thickness of grain boundary α . Deformed ingots are heated high in the $\alpha+\beta$ field ($\sim 30 - 50^\circ\text{C}$ below the β transus) where further deformation can take place to ensure that full recrystallisation takes place during subsequent static annealing in the β phase field [74]. Normal practice is to cool as quickly as possible from the recrystallisation step as this produces finer scale intragranular α precipitates which are more readily converted into an equiaxed morphology. Depending on the desired final grain size further deformations sequences

can take place, prior to a final $\alpha+\beta$ field deformation step which typically produces a fine equiaxed α microstructure [74].

2.3.2 Secondary Processing

The secondary processing of β titanium alloys creates a finished product which is very close to final component shape and with the desired microstructure condition. Thermomechanical processes are capable of achieving a wide range of microstructures by a combination of mechanical working under controlled thermal conditions, which could not be achieved via heat treatment alone [18]. Several hot working processes, such as extrusion or rolling, are utilised to produce final components, however the most cost effective method of achieving final form and microstructure is through isothermal forging [75].

During isothermal forging the dies and workpiece are heated to a constant temperature prior to deformation. The isothermal conditions eliminate die chill, allowing the initial billet temperature to be reduced [75]. Additionally as the billet temperature is maintained throughout the forming process lower strain rates ($10^{-1} - 10^{-5} \text{ s}^{-1}$) can be used, when compared to those typical of traditional forging ($10^2 - 10^3 \text{ s}^{-1}$) [76]. Slower strain rates lead to lower forging loads, the elimination of adiabatic heating effects, better die filling and increased microstructural homogeneity [76, 77]. The closer conformance to final component shape offered by isothermal forging reduces both the level of required final machining and the loss of material [75]. The effect of deformation and temperature can have a marked effect on the microstructural development of β titanium alloys. Two major thermal processing regimes exist, forging either above or below the β transformation temperature, leading to a difference in the deformed microstructure which will affect the forging characteristics and the evolved microstructure.

Final forging above the β transformation temperature (super transus) allows lower forging loads and the ability to successfully form complex shapes. Flow behaviour in this regime is typified by an initial sharp peak followed by an almost constant stress [78]. The peak stress has been found to increase at faster strain rates and when decreasing the forging temperature and the initial β grain size [79]. The steady state

flow stress has been found to increase when forged at faster strain rates and at lower temperatures [80, 81]. The microstructural deformation mechanism is dominated by dynamic recovery (discussed in section 2.3.3) at strains less than 1, after which point dynamic recrystallisation is observed. Supertransus deformation can lead to the development of a mixed grain size microstructure, caused by selective recrystallisation [73, 82]. Once established a mixed grain size microstructure cannot be reversed by thermal treatment alone [78].

Forging below the β transformation temperature (subtransus) normally requires higher forging loads and results in reduced material flow due to the presence of the α phase. Flow behaviour is typified by a high yield stress followed by a period of flow softening to steady state flow. As with supertransus forging the peak stress is reduced at higher temperatures and the softening effect has been ascribed to dynamic changes in the α phase and texture [78, 83]. High peak stresses have also been associated with an acicular α phase morphology [78]. The presence of the α phase also has an effect on the β phase, as it acts as a hard particle in a soft matrix. The α precipitates act as strain concentrators, restricting β flow and creating small high angle subgrains [41]. Both the size and the volume fraction of primary α precipitates can be controlled by the forging temperature, whilst secondary α precipitates are controlled by the subsequent cooling rate [84].

The process control facilitated by isothermal forging is highly desirable, however, there are limitations in capability. The capital cost of an isothermal forging unit is high and the size of component restricted. Tooling is exposed to the same thermal regime as that of the workpiece and therefore the materials utilised become more expensive. Material suitable for isothermally forging titanium needs to have a flow stress three times that of the workpiece at temperature thereby limiting the possible materials to molybdenum or nickel based alloys [75]. To minimise potential problems during forging operations, forging loads, die stresses and ultimately microstructure evolution need to be predicted by modelling techniques [76]. Modelling processes require accurate description of material behaviour, based upon small scale experimental data, an understanding of the relevant deformation mechanisms and relevant constitutive relationships.

2.3.3 Dynamic deformation mechanisms

One of the major advantages of thermomechanical processing (TMP) is the ability to produce microstructures not achievable through conventional thermal and mechanical treatments [18]. During hot working mobile dislocations are rapidly generated and are readily able to glide in active slip bands. As the level of work imparted on the workpiece is increased the dislocation density rises, leading to a higher strain energy in the system due to dislocation interaction and entanglement. A higher system strain at the elevated temperature provides sufficient driving force for dynamic restoration processes, such as recovery and recrystallisation [5].

Recovery processes reduce the total system energy by decreasing the dislocation density and is typical in materials with high stacking fault energies [85]. Dislocations of opposing sign move to a common slip plane and annihilate, while those of the same sign align to form substructure (a process known as polygonisation). These processes are governed by the cross slip of screw dislocations and at higher temperatures the climb of edge dislocations. Cross slip is a form of glide and therefore dependent upon shear stress, irrespective of whether this stress is externally applied or caused by the dislocation structure itself. Thus, recovery tends to occur when a characteristic level of work in the system is reached. Conversely climb depends on the diffusion of vacancies and therefore has a characteristic temperature (~ 0.3 of the materials melting temperature) which must be surpassed prior to the mechanism becoming active. The climb process does not rely upon deformation for its driving force and therefore can provide a more extensive softening than that possible from the cross slip [85].

For a given temperature and strain rate a steady regime can be achieved where the rates of dislocation creation and annihilation are equal. The size of the polygonised substructure varies inversely with strain rate (e.g. higher strain rates result in a smaller subgrain size) since the generation of dislocations is dependent on strain rate, whilst the recovery processes are thermally dominated. Raising the working temperature increases dislocation movement, by enabling slip along slip systems with higher activation energies, thereby encouraging annihilation and increasing the substructure size [86, 87].

Recrystallisation is a radical form of restoration in which deformed grains are replaced by new strain free crystals with high angle boundaries [85]. Similarly to recovery, recrystallisation requires both an elevated temperature (~ 0.5 of the materials melting temperature) and a critical dislocation density. Unlike static recrystallisation in which new grains grow until mutual impingement, dynamic recrystallisation proceeds by repeated nucleation and limited growth of new grains [88]. Nucleation of recrystallisation occurs preferentially at existing grain boundaries and the growth of the new grains is limited by immediate deformation, which acts to form a driving force for subsequent nucleation.

2.3.4 Dynamic globularisation

Globularisation in titanium alloys, creates a more equiaxed α phase morphology from acicular precipitates commonly formed by slow cooling. The microstructure and deformation conditions can both have a significant effect on globularisation and both the initiation and subsequent mechanisms have been studied [89-93].

The mechanisms involved in globularisation of the α phase in Ti-6Al-4V (Ti-6-4) were characterised by Margolin [89]. A surface tension argument was proposed to explain the growth and separation of recrystallised α grains, both within an α plate and at grain boundaries. Following recrystallisation the α/α interfacial energy is reduced by the movement of the β phase between the two α crystals. This movement results in rounded α phase boundaries and leads to the mechanism being referred to as 'pinch off' (shown schematically Figure 12). Weiss [90] observed α lamellae breaking up during deformation by intense shear localisation or substructure formation, developed by both recovery and recrystallisation. The interfaces created within the α phase were penetrated by the β phase, severing the α lamella.

The mechanisms of dynamic globularisation are hypothesised to be analogous to that of static globularisation. Whilst undergoing hot deformation, shear bands develop within primary α crystals, creating a substructure of misorientated grains [92]. At a critical strain the β phase is able to penetrate the α/α interfaces created, splitting the α lath (pinch off) [89, 90]. The initiation of dynamic globularisation was attributed to the

break up of the α phase at grain boundaries and within α platelets preferentially orientated for localised shear effects [91]. The process of globularisation in Ti-6-4 was deemed to have two constituent stages, (1) the break up of the α structure through deformation and (2) diffusion based coalescence. Experimental findings have indicated that at low strain rates and temperatures, thermal activation alone was not enough to cause spheroidisation [91].

Semiatin [93] observed globularisation initiating at prior β grain boundaries and also suggested that kinks in the hard α laths could be nucleation sites. The localised shear within a plate or at surface steps, which is related to line tension effects, causes the break up of the prior α structure into a roughly equiaxed morphology. The authors hypothesised that the globularisation at low strain rates (10^{-3} s^{-1}) was dominated by dislocation glide, as there was little sensitivity of the kinetics to temperature. They also noted that at the onset of globularisation a large proportion of the α plates had orientated themselves normal to the compression axis. The development of such a texture may increase the localised shearing effect through the loss of active slip systems.

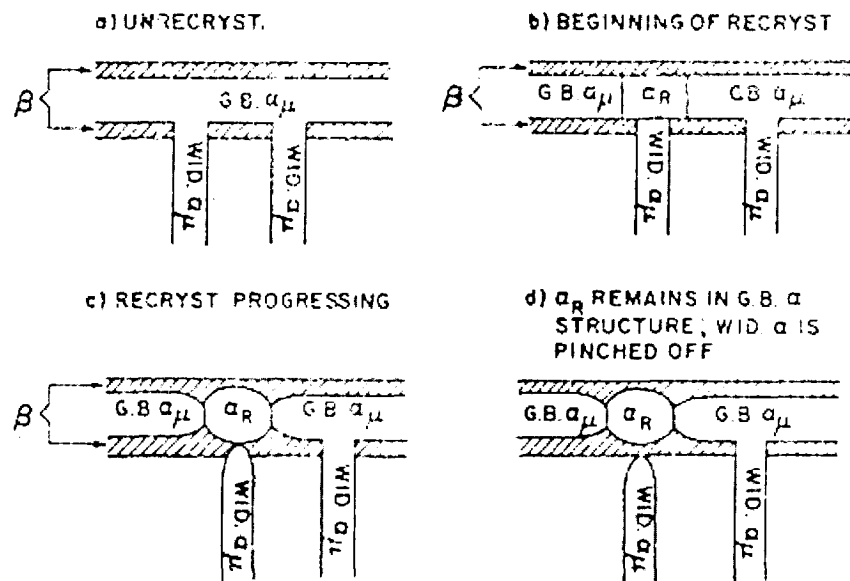


Figure 12: Schematic representation of the 'pinch off' mechanism causing α phase globularisation [89].

2.3.5 Flow Softening

The flow softening phenomenon mentioned in section 2.3.2, is commonly observed during the hot working of Ti alloys. The process is controlled by a combination of adiabatic heating effects and changes to the deformed microstructure. When working at high strain rates (above 1 s^{-1}) and relatively low temperatures the effect is thought to be dominated by adiabatic heating effects [5]. At lower strain rates (below 1 s^{-1}) the controlling mechanism changes to a regime governed by microstructural development [78]. The softening in these conditions is attributed to the formation and changes to substructures, phase morphology evolution (for example dynamic globularisation), and the development of texture.

Semiatin *et al.*[93] found that the rate of softening appeared to be independent of initial α phase colony size or β grain size, but that the plastic behaviour was controlled by dislocation glide and climb. Shell *et al.*[94] working with $\alpha+\beta$ alloy Ti-6-4 confirmed that the dynamic globularisation initiated at kinks seen in α laths, as proposed by Semiatin *et al.*[93], as well as at prior β grain boundaries. It was suggested these areas developed higher localised strains, due to deformation incompatibilities, and therefore favoured initiation. It was also found that the strain required to initiate dynamic globularisation was significantly higher than that observed at the onset of flow softening. From these results a mechanism of α platelet bending/buckling prior to dynamic globularisation was proposed to explain the source of the flow softening.

A problem associated with modelling processes such as flow softening is the influence of crystallographic texture. Texture evolution during testing, and flow inhomogeneities tend to be overlooked in the models and investigations. Recent work by Bate [95] and Sarma [96], divided grains into smaller regions so that local plastic deformation and lattice rotation could be incorporated. However, insufficient knowledge of the physical mechanics occurring during hot deformation of two phase materials such as titanium alloys has limited the application of this technique [97]. As such, current research efforts are attempting to understand the complex reaction of the different phases in titanium alloys during hot deformation, so that increasing accurate modelling descriptions can be compiled.

The work of Semiatin and Bieler [97] found the plastic flow behaviour of Ti-6-4 was influenced by a colony type microstructure. The variation of the average effective slip lengths present (effectively the thickness of an α phase plate), and interaction at the α/β interfaces, influenced the flow behaviour. They cite the work of Armstrong [98] and Eshelby [99] to quantify the effect of the Hall-Petch effect, Eqn 1, on the flow stress of a textured alloy.

Eqn 1
$$\sigma = M(\tau_0 + k_s l^{-\frac{1}{2}})$$

Where M is the Taylor Factor associated with orientation, τ_0 is the resolved shear stress, k_s is the Hall-Petch constant linked to the action of the α/β boundaries, while l is the slip length equivalent to α plate thickness. It was proposed that the critical stress required to ensure that slip crosses the α/β interface corresponded to the peak stress observed on the relevant flow curve. Therefore, once the critical stress has been surpassed flow softening can be rationalised by the corresponding loss of Hall-Petch strengthening offered by the α/β interfaces acting as a slip barrier. The far superior strength of the α phase at hot working temperatures would imply that Hall-Petch type relationship proposed would be controlled by the properties and thickness of the α plates [78]. The hot deformation of the softer β phase is characterised by recovery and substructure formation; therefore the interfaces between the α and β phases can be thought of as grain boundaries [100].

Further investigation indicated that strain rate also had an effect on the Hall-Petch style relationship. At high strain rates (above 10^{-1} s^{-1}) the highest peak strain was found when the α plate was thinnest. At very low strain rates (10^{-3} s^{-1}) the thinnest α plate microstructure produced the lowest peak strain. It was proposed that in this slower deformation regime a diffusional creep process competed with the dislocation glide and climb mechanism that dominates the high strain rates. The hypothesis of the loss of interface (Hall-Petch) strengthening [97], was confirmed to be the cause of flow softening as a first order relationship existed between experimental flow curves and mechanistic Eshelby grain size strengthening analysis [99]. It was also found that the Hall-Petch constants have a temperature dependence that can also be rationalized by the

Eshelby analysis. Semiati and Bieler [101] also investigated the effect of existing texture on flow softening, especially pertinent with respect to titanium alloys, where significant texture can be developed in primary ingot breakdown. It was concluded that the anisotropy in flow softening rate during deformation could be ascribed to different rates of texture hardening.

The concept of flow softening by loss of Hall-Petch interface strength, as proposed by Semiati and Bieler, was applied to the metastable β titanium alloy Ti-10V-2Fe-3Al. During subtransus forging, Ti-10V-2Fe-3Al had exhibited significant flow softening, however, the Hall-Petch approximation was unable to successfully describe the observed behaviour [5].

The α volume fraction present in the initial microstructure of Ti-10V-2Fe-3Al was considerably lower than that of Ti-6Al-4V and therefore the α/β interfacial area was insufficient to account for the observed softening. Instead, the peak stress and subsequent flow softening prior to steady state was attributed to the break up of acicular α precipitates into more equiaxed morphology and the development of β substructure [5]. Strain rate was found to influence the critical strain at which spheroidisation processes occur and the level of β grain misorientation. Decreasing the strain rate lowered the strain at which spheroidisation occurred and extended total time at the forging temperature. Extending time at elevated temperatures allows processes such as recovery to lower the dislocation density, creating larger more coherent substructures. This effect was further observed in the globularisation kinetics. At faster strain rates (e.g. 10^{-1} s^{-1}) evidence for α plate ‘pinch off’ was observed and deformation appeared to be controlled by a glide/climb processes. At slower strain rates (e.g. 10^{-2} and 10^{-3} s^{-1}) coarsening style globularisation was observed, suggesting that diffusional processes were the dominating mechanism. The change in mechanism during deformation at 760°C was found to be at a strain rate of 10^{-2} s^{-1} [5].

2.3.6 Empirical relationships

The isothermal open die forging of a uniaxial short cylinder is a standard test to determine the workability, or flow stress, of a material. A servo controlled testing

machine can be used to obtain constant strain rates up to a rate of 10 s^{-1} , providing the specimen is lubricated [102]. Basic mechanical theory states that for specimen with cross sectional area A , under a uniaxial force F , the flow stress σ , is given by the following equation:

$$\sigma = \frac{F}{A}$$

Eqn 2

As the geometry of the specimen is cylindrical the law of constant volume can be introduced. From an initial diameter of d_0 and height h_0 , the specimen will spread outward a under compressive force. At any given instance the specimen diameter will be d and height h . These instantaneous dimensions can be related to the initial dimensions as follows:

$$d_0^2 h_0 = d^2 h$$

Eqn 3

Thus during compression the stress can be defined as follows:

$$\sigma = \frac{4Fh}{\pi d^2 h}$$

Eqn 4

True compressive strain ϵ , for this system is given below:

$$\epsilon = \ln\left(\frac{h_0}{h}\right)$$

Eqn 5

The use of lubricants is essential during compression testing to help maintain uniform deformation. Without sufficient lubrication, frictional forces between the specimen surface and die build, opposing the outward movement of the metal, whilst material in the centre of the specimen is still able to flow. These conditions lead to the sample bulging at the mid height (barrelling, Figure 13a) and the formation of dead zones of undeformed material near the die surface (Figure 13b). Homogenous deformation can be achieved by using a glass lubricant to prevent the onset of barrelling during upsetting, providing that the sample is subjected to a strain of less than one [102].

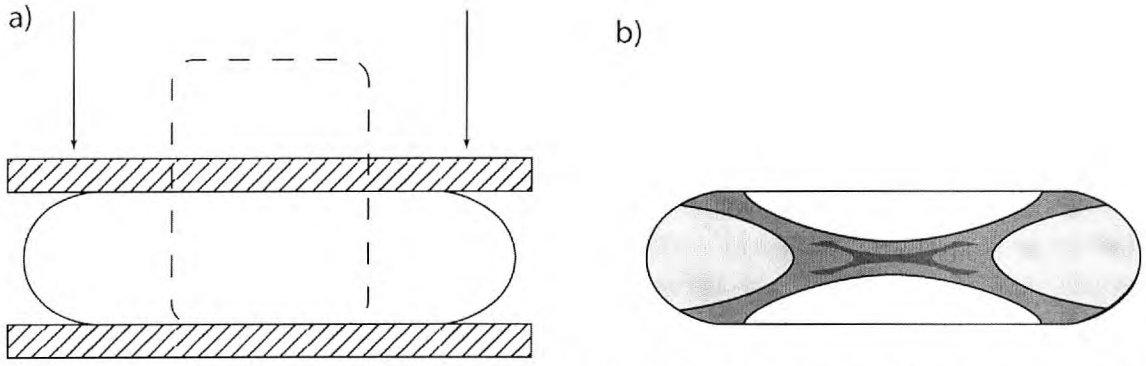


Figure 13: Schematic representations of the effect of friction during forging; a) sample barrelling during compression due to inadequate lubrication, b) strain profile of barrelled sample with intense shear banding (darker shade) and undeformed dead zones (lightest shade).

The frictional forces are caused by development of shear stresses at the specimen surface, which are directed towards the centre. The exact nature of the relationship between the shear stress (τ) and the stress normal to the interface (P), depends on the determined level of friction. Low friction conditions assume that there is no noticeable barrelling of the specimen and that Coulomb friction applies (Eqn 6). In this regime it is assumed that there is sliding between the specimen and the die, and is therefore also known as sliding friction. The other extreme is sticking friction, which is more realistic for hot metal working where there is no relative movement between workpiece and die. The interface shear stress (τ_i) in this regime is constant at the interface and by implementing the von Mises yield criterion, can be said to be equal to the flow stress in shear (k) (Eqn 7) [102].

Eqn 6

$$\tau = \mu P$$

Eqn 7

$$\tau_i = k = \frac{\sigma}{\sqrt{3}}$$

Often a condition exists where sliding occurs near the edge of the specimen while the centre obeys sticking behaviour. If it is assumed that the workpiece in contact with the

tooling is represented by a constant shear strength τ_i , which rather than being equal to yield stress in shear is related through p , the interface friction factor, Eqn 8. Then a range of frictions can be obtained from $p = 0$, perfect sliding, through to $p = 1$, sticking.

$$\tau_i = pk = \frac{p\sigma}{\sqrt{3}}$$

Eqn 8

Constitutive equations, relating material responses to external variables, are required before any sort of predictive modelling can be commenced. Of the two approaches, engineering and microstructural, the forging community has adopted an empirical curve fitting method based on the engineering variables [20]. As such, the constitutive equation for flow stress should relate it to hot working parameters such as temperature T , strain ϵ , and strain rate $\dot{\epsilon}$, as given by the general form in Eqn 9.

$$\sigma = f(T, \epsilon, \dot{\epsilon})$$

Eqn 9

This general form can be reduced by applying two well known relationships. The Zener-Hollomon parameter (Z) provides a single factor that includes the influences of both temperature and strain rate (Eqn 10). Where Q is the activation energy and R is the universal gas constant. The Norton-Hoff rule (Eqn 11) relates flow to the Zener-Hollomon parameter, using the strain rate sensitivity (m), strain hardening factor (n) and constant (C).

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right)$$

Eqn 10

$$\sigma = \sqrt{3}^{m+1} C \dot{\epsilon}^m \epsilon^n \exp\left(\frac{Q}{RT}\right)$$

Eqn 11

Sellars and Tegart [103, 104] suggested that a relationship linking strain rate and temperature (Eqn 12), which is based on creep, could also be applied to steady state hot deformation of metals, as an approximation to the Zener-Hollomon parameter. The parameters A , α and n are all experimentally derived constants.

$$\dot{\varepsilon} = A(\sinh \alpha \sigma)^n \exp\left(\frac{-Q}{RT}\right)$$

Eqn 12

$$Z \approx A(\sinh \alpha \sigma)^n$$

Immarigeon [105] noted that none of these constitutive laws considered microstructure evolution as a dynamic variable. Observation of various alloys during hot deformation at a constant temperature and strain rate noted a steady state region, which often correlates with the desired (target) microstructural state [106]. When dynamic recovery is the only deformation mechanism the equilibrium steady state can be expressed in an exponential manner (Eqn 13); where state flow stress (σ_{ss}) and the constant (β) are determined by temperature and strain rate [107].

$$\frac{\partial \sigma}{\partial \varepsilon} = \beta(\sigma_{ss} - \sigma)$$

Eqn 13

Immarigeon [106] showed that when dynamic recrystallisation occurs, a target steady state microstructure (defined by grain size) is again determined by temperature and strain rate. In this regime these variables can be combined through the Zener-Hollomon equation (Eqn 12).

To tackle the problem cited by Immarigeon [105], Bate [108] and Blackwell [107] introduced a generalised structure based internal variable (λ) to relate instantaneous target stress (σ_t) to Z by a power law (Eqn 14; k being a constant). This structural parameter was also found to vary during processing as the microstructure evolved and was assumed to reach equilibrium in an analogous manner to flow behaviour (Eqn 15: ς is a constant).

$$\sigma_t = k\lambda Z^m$$

Eqn 14

$$\frac{\partial \lambda}{\partial \varepsilon} = \varsigma(\lambda_{ss} - \lambda)$$

Eqn 15

The steady state internal variable is also related to Z by a power law relationship, where λ_0 and q are constants.

Eqn 16

$$\lambda_{ss} = \lambda_0 Z^q$$

This constitutive model based upon two exponential constitutive relationships has become known as the Bate-Brooks model, after its major founders; and it has proved to be very effective in predicting the microstructural evolution of thermally processed metals [5].

2.4 Metastable β alloy Ti-10V-2Fe-3Al

Ti-10V-2Fe-3Al (Ti-10-2-3) is a metastable β alloy developed by TIMET in the 1970's as a high strength forging alloy [26]. Ti-10-2-3 lies just to the right of the MsMf line in Figure 3 and has a β transformation temperature around 800°C. When correctly processed Ti-10-2-3 can achieve the best combination of mechanical properties, when compared to the other high strength β alloys [3, 78]. Ultimate tensile strengths of over 1.2GPa have been reported and the deep hardenability of Ti-10-2-3 led to its use for large truck beam forging in the landing gear assemblies of a Boeing 777 [109]. The selection of Ti-10-2-3 produced significant weight saving (~40%) compared to a steel of the same strength, and decreased the risk of stress corrosion cracking [1, 109, 110].

2.4.1 Specific metallurgy of Ti-10V-2Fe-3Al

Ti-10-2-3 is a leanly stabilised β alloy, with V and Fe stabilising the β phase whilst Al stabilises the α phase. The formation of the α phase during slow cooling from the β phase field proceeds as outlined in section 2.1.2 with the segregation of different alloying elements to stabilise the relevant phases. Rapid cooling from the β phase field results in the formation of the α'' martensite, however, close examination of the transformation has determined that it is driven by a quench induced strain, rather than thermally [22, 23]. Martensitic plates tend to extend across prior β grains, terminating at the grain boundary [111]. The ω phase was also found to form athermally during quenching from the β phase field. Ellipsoidal ω particles, approximately 5nm in size, were observed in a uniform distribution throughout the retained β matrix [112]. Athermal ω was also observed to form in β solution treated material with up to 30% volume fraction of primary α [111].

Low temperature aging (below 400°C) of the β solution treated and quenched material produces isothermal ω which rapidly grows from an initial ellipsoidal morphology to a cuboidal morphology. Further aging in this temperature regime results in the nucleation of extremely fine α in a nondescript morphology, which is thought to have formed from the ω particles [111]. Above 400°C the α phase is able to form as high aspect ratio plates in tight clusters, which predominantly nucleate from grain boundaries. Increasing

aging temperature to 650°C limits the α phase to thick grain boundary layers with sporadic formations in a grain interior.

2.4.2 Hot working of Ti-10V-2Fe-3Al

Primary processing of Ti-10-2-3 largely follows the generic processing route outlined in section 2.3.1, however, a significant level of macro and micro segregation were noted in Ti-10-2-3 ingots [113, 114]. Macro segregation leads to regions enriched in β stabilisers, commonly known as β fleck, which will lead to an inhomogeneous microstructural response.

The increased use of Ti-10-2-3 by the aerospace industry has ensured that the secondary processing of the alloy been extensively studied, with particular focus on optimising mechanical properties through microstructural control [67, 83, 84, 115, 116]. Key factors identified from these efforts included initial microstructural condition (including β grain size, α volume fraction and α phase morphology), strain rate and in which phase field the primary forging operation occurred.

Higher peak flow stresses are observed in material which has been β forged (resulting in a microstructure of large β grains with Widmanstätten type α) when compared to flow data for $\alpha+\beta$ forged material (globular primary α and a transformed matrix) [5, 83, 116]. At higher strains the flow stresses for the two microstructural conditions become similar. The effect of strain and strain rate on $\alpha+\beta$ forged was found to have little effect on the microstructure [117]. The α phase volume fraction and morphology remained constant and the β phase exhibited a constant subgrain size. Contrastingly β forged material exhibited significant microstructural change during forging, with strain rate influencing the scale of the newly formed α laths [117]. The $\alpha+\beta$ forged condition material adopts a much coarser β subgrain size than that observed in the β forged material. It is hypothesised that this difference is influenced by the difference in the initial α phase morphologies observed in the two conditions. The globular morphology of the $\alpha+\beta$ forged material is more compact and as such imposes less constraint on the β phase during deformation than the Widmanstätten α in the β forged material, thus allowing a coarser substructure to develop.

The possible deformation mechanisms occurring during isothermal forging of Ti-10-2-3 have been studied by many authors [5, 116, 118]. Microstructural observations in combination with experimental flow data and empirical analysis led to the hypothesis that that dynamic recovery is the dominating restoration mechanism during forging at 760°C. The flow softening exhibited by β forged material has been assigned to dynamic globularisation [5].

Forging temperature has been found to have a significant effect on the microstructure evolution. Slight increases from the optimal 760°C (775°C) the observed microstructure and globularisation mechanisms were found to be analogues of that seen at 760°C. The higher temperature decreased the volume fraction of the α phase and coarsened the globular primary α . At lower strain rates the extended time at temperature has a greater effect as an increased chemical equilibrium effect reduces the stability of the β phase and hence the volume fraction of martensite increases. At a higher temperature of 790°C, just below the transus temperature of 795°C, the fraction of α is further reduced, decreasing β stability, meaning that a total martensitic transformation occurs upon quenching, despite other literature suggesting the athermal transformation was suppressed [115]. Small decreases in the forging temperature, for example to 750°C, changed the dominant recovery mechanism from recovery to recrystallisation [5].

2.5 Metastable β alloy Ti-5Al-5Mo-5V-3Cr

Ti-5Al-5Mo-5V-3Cr-0.5Fe (Ti-5-5-5-3) is a recently developed (1997) high strength metastable β alloy based on the Russian alloy VT-22 (nominally Ti-5Al-5Mo-5V-1Cr-1Fe) [6, 7, 119]. The segregation problems observed in Ti-10-2-3, coupled with the relatively small processing window ($\sim 760 - 780^\circ\text{C}$) led to the need to develop an alloy of similar mechanical properties but with better processing characteristics. The reduction of Fe as an alloying addition (compensated by increasing Cr content) reduces macro segregation and the formation of β fleck [120]. Ti-5-5-5-3 has been reported to have a wider processing window than Ti-10-2-3 and also a deeper hardenability [6, 7].

2.5.1 Specific Metallurgy of Ti-5-5-5-3

Ti-5-5-5-3 has a β transformation temperature around 855°C , some 55°C higher than Ti-10-2-3 [119]. Slow cooling from above the transus results in a fully lamella α structure within the prior β grains, accompanied by grain boundary α [56, 120]. Cotton *et al.* [120] note that some of the benefits of Ti-5-5-5-3 over Ti-10-2-3 are achieved through the high alloying levels of slow diffusers. As such the onset of the $\beta \rightarrow \alpha$ transformation does not occur until about 650°C , approximately 200°C below the β transformation temperature. It has also been found that cooling rates higher than 0.25°C per second are sufficient to suppress the $\beta \rightarrow \alpha$ transformation entirely. Clement *et al.* [56] also report the slow nature of the $\beta \rightarrow \alpha$ transformation, although intragranular nucleation of α plates in addition to a grain boundary α film is observed at a temperature of 700°C . Rapid cooling from the β phase field retains a fully β structure, with reports of the athermal ω phase following quenching [56, 120].

Deformation prior to aging can enable subtransus recrystallisation provided that sufficient deformation energy is retained in the material. Subsequent two step aging approaches (sometimes referred to as duplex aging) can be used to produce bimodal microstructures [56, 120]. The first aging step is high in the $\alpha+\beta$ region (typically $40 - 50^\circ\text{C}$ below the β transus) and produces a globular morphology primary α at β grain boundaries. The temperature employed for the first aging, dictates the volume fraction of primary α in final microstructure. The second aging temperature is much lower in

the $\alpha+\beta$ phase field and forms small α plates within the β grains. The final aging temperature appears to be critical to the balance of strength and ductility in bimodal Ti-5-5-5-3. Aging at temperatures lower than 500°C was reported to produce a very high strength condition (~ 1.5 GPa), however material in this condition suffered brittle failure [56]. Aging above 500°C progressively decreased strength but exhibited a corresponding increase in ductility [56, 121, 122]. The high strength and brittle failure achieved by low temperature aging has been attributed to the formation of isothermal ω , although no direct experimental evidence exists [56].

2.5.2 Hot Working of Ti-5-5-5-3

Little information has been published detailing the hot working of Ti-5-5-5-3. Boyer and Briggs [7] state that the processing of Ti-5-5-5-3 will be simpler than that of Ti-10-2-3 as it can take place entirely in the $\alpha+\beta$ field, rather than a combination of β and $\alpha+\beta$ field forging. Venkatesh *et al.* [8] considered isothermal forging of Ti-5-5-5-3 over a range of strain rates from 10^{-3} s^{-1} to 10 s^{-1} and at temperatures of 760°C (the industrial forging temperature of Ti-10-2-3) and 983°C. A level of flow softening was reported when working at 760°C but the material was also found to have undergone non-uniform deformation. Despite this study, little information pertaining to the effect of initial microstructure or hot working parameters on the microstructural evolution of Ti-5-5-5-3 has been published.

2.5.3 Applications of Ti-5-5-5-3

Boeing has announced that it plans to use Ti-5-5-5-3 for the truck beam forgings of landing gear assemblies on the 787 airframe [7]. For such an application the solution heat treated and aged (STA) condition (duplex heat treatment) would be used as it produces microstructures with the highest strength. Material in this condition is also being tested for use in high strength fasteners [121, 122]. Slow cooling from the β phase field produces a more damage-resistant microstructure (increased fracture toughness compared with STA) and material is being considered for application in nacelles, fuselage and wings [7].

2.6 Summary

Titanium alloys are an important structural metal since they offer a high strength to weight ratio and a good resistance to corrosion. Such properties are of great interest to the aerospace industry where both specific strength and corrosion are key issues. Therefore, it is of no surprise that use of titanium alloys both in gas turbine engines and throughout the airframe has increased over the last 60 years.

Titanium is allotropic and much of its metallurgy is dominated by the transformation of the high temperature body centred cubic phase (β) to the low temperature close packed hexagonal structure (α). The addition of different alloying elements stabilises either one of the two phases creating a range of possible microstructure, each with unique benefits.

The properties of titanium alloys are directly related to the microstructural features that develop during processing. The relevant volume fractions and morphologies of both α and β phases influence the resulting mechanical response. Microstructural development can be varied through both thermal regimes and by thermomechanical processing, thus requiring an intimate knowledge of influence of process parameters.

β titanium alloys are the most versatile class of titanium alloys offering a good balance of mechanical properties. For high strength applications metastable β alloys are chosen as they offer the highest strength available from all titanium alloys and uniform properties can be achieved throughout large sections. Billets of material are produced from vacuum arc melted ingots by primary forging operations usually in the β phase field. Hot working the ingots not only produces an intermediate product form, but acts to homogenise the material such that its response to further forming will be uniform. The economic production of the final product is achieved through hot near net shape forging and subsequent aging. The final microstructure is determined by the billet microstructural condition and the temperature, strain rate and strain encountered during secondary forging. Large scale components, such as landing gear truck beams, have complex shapes and therefore process modelling occurs long before production to optimise process parameters. Such modelling requires a detailed knowledge of the

relevant materials flow behaviour, deformation mechanisms and microstructural response.

In the mid 1990s Boeing used the metastable β alloy Ti-10V-2Fe-3Al to make components of the undercarriage assemblies on the 777 airframe. The weight saving and corrosion resistance offered by this alloy made it a preferable choice to the more traditional steel. Extensive research has been conducted using this alloy's response to thermomechanical processing variables and process optimisation has been achieved, if largely empirically through small scale trials. Whilst a good balance of properties can be achieved from Ti-10V-2Fe-3Al, the alloy suffers from segregation problems and has been shown to be very sensitive to forming variables, allowing only a small processing window. The recently developed Ti-5Al-5Mo-5V-3Cr is another metastable β alloy, but it has been observed to be easier to process, less prone to segregation and can achieve a higher strength than Ti-10V-2Fe-3Al. Thus great interest has developed around using this new alloy in the next generation of airframes.

Work conducted with Ti-5Al-5Mo-5V-3Cr to date has mainly focused on the development of microstructure with respect to thermal regimes. Little has been published regarding the microstructural development during isothermal forging, (an area which is critical to final product fabrication) or analysis made of the alloy's flow behaviour. This thesis addresses the lack of information by investigating the flow behaviour, dominating mechanisms and microstructural response of Ti-5Al-5Mo-5V-3Cr over a range of applicable process variables. The work presented will also discuss the influence of the ω phase upon microstructural evolution in Ti-5Al-5Mo-5V-3Cr.

3 Initial characterisation of Ti-5Al-5Mo-5V-3Cr

This chapter presents a basic characterisation of the metastable β alloy Ti-5Al-5Mo-5V-3Cr. Prior to investigating the effect of temperature on the microstructure, the as received material was evaluated for chemical composition and the initial micro and macro structures were established.

The β transformation temperature, and the volume fraction of the α phase when approaching this temperature, have been established and are compared to those of Ti-10V-2Fe-3Al.

The effect of cooling rates (slow furnace cooling, air cooling and water quenching) on microstructural evolution has been investigated from temperatures both above and below the β transformation temperature. Additionally the precipitation and dissolution of the low temperature α phase with respect to both temperature and time spent at temperature has been investigated. From these results insights have been gained into the microstructural development and diffusion characteristics of the alloy.

3.1 *Experimental Methods*

3.1.1 Effect of time at temperature

The dissolution of the α phase with time spent at temperature as the equilibrium volume fraction is established was studied using 10 mm cube samples. Samples were coated in Deltaglaze 3418 to minimise oxidation during thermal treatment in a Lenton furnace. Samples were placed in a pre heated furnace at temperatures of 750°C, 800°C and 850°C for 5, 15, 60 and 600 minutes. Following heat treatment samples were immediately water quenched, and prepared for characterisation using a scanning electron microscope.

3.1.2 Determination of the β approach curve

The β transformation temperature and approach curve was determined using small cube samples, approximately 1 cm³ in size. Samples were heat treated at a range of temperatures between 800°C and 870°C for 60 minutes in a Lenton furnace. Following heat treatment samples were immediately water quenched to retain the high temperature microstructure. To prevent the absorption of oxygen during heat treatment all samples were coated in Achesons Deltaglaze 3418. Metallographic determination of the β approach curve was conducted using optical microscopy.

3.1.3 Effect of cooling rate

The effect of cooling rates, from both the β and $\alpha+\beta$ phase fields, was investigated using small cube samples, approximately 1 cm³ in size. Samples were heat treated to 900°C (super transus) and 800°C (subtransus) in a Lenton furnace for 60 minutes before cooling in the furnace ($\sim 0.025^\circ\text{Cs}^{-1}$), in air ($\sim 10^\circ\text{Cs}^{-1}$) or via water quenching ($>100^\circ\text{Cs}^{-1}$). Again, oxygen absorption at temperature was minimised by coating the samples in Deltaglaze 3418. Characterisation of these samples was carried out using scanning electron microscopy and laboratory based X-ray diffraction.

3.1.4 Slow cooling and aging

The effect of slow cooling on the microstructure following a solution heat treatment above the β transus was carried out on ~ 1 cm³ cubes coated in Deltaglaze 3418.

Samples were solution heat treated in the β phase field at 900°C for 60 minutes prior to being cooled at 1°Cmin⁻¹. Cooling was discontinued once the target aging temperature was reached and samples were held for a further 60 minute period. Aging temperatures of 800°C, 700°C, 600°C and 500°C were all investigated as well as direct quench from 900°C. Following the completion of the thermal treatment all samples were water quenched to preserve the higher temperature microstructure for investigation using electron microscopy.

3.1.5 Metallography and Analysis

Metallographic preparation was carried out by mechanical grinding using water lubricated silicon carbide paper. Samples for light microscopy inspection were subsequently mechanically polished using a quarter micron colloidal silica suspension before being etched using a solution of 2% HF and 10% HNO₃ in water. Samples for inspection using a scanning electron microscope (SEM) were prepared either by electro polishing in a solution of 3.2% perchloric acid, 40.3% butan-1-ol, 56.5% methanol at a temperature of -40°C or by mechanical polishing with a quarter micron colloidal silica suspension containing 10% hydrogen peroxide. Backscattered electron images (BSEI) were obtained using a Jeol 840A SEM. Thin foils for inspection using a transmission electron microscope (TEM) were created by mechanical grinding to a thickness of 0.2 mm before final thinning using a Struers Tenupol with a solution of 6.9% perchloric acid and 34.3% butan-1-ol in methanol at -40°C. The freely available software package ImageJ¹ was used to perform quantitative image analysis, determining data such as the α volume fraction and precipitate size.

¹ ImageJ (version 1.37) W. Rasband, National Institutes of Health, USA

3.2 As Received Material

A billet of Ti-5-5-5-3 manufactured by Verkhnyaya Salda Metallurgical Production Association (VSMPO) was supplied for the project by Messier-Dowty. The chemical composition of the billet is shown in Table 2.

Table 2: Chemical composition of Ti-5-5-5-3 in weight%, balance Ti.

	V	Fe	Al	Mo	Cr	C	O	N	H
Ti-5-5-5-3	5.25	0.35	4.91	5.22	2.98	0.008	0.135	0.006	0.001

The level of β phase stability ('Moly equivalence' (Mo Eq)) of an alloy can be calculated from standard data from the literature [2] using the following equation:

$$\text{Mo Eq} = 1.000\text{Mo} + 0.667\text{V} + 1.538\text{Cr} + 2.857\text{Fe} - 1.000\text{Al} - 10.000(\text{O} + \text{C} + \text{N} + \text{H})$$

The nominal composition of the alloy gives an Mo Eq of 9.38 which is exactly the same value for nominal composition Ti-10-2-3. The Mo Eq for the actual composition as given in Table 2 is ~7.89.

The as-received material had been subjected to a number of prior processing steps, including hot forging in both the β and $\alpha+\beta$ regimes. The final forging step occurred in the $\alpha+\beta$ region and produced elongated ovoid β grains, ~500 μm long, with an aspect ratio ~40:1 (Figure 14a). The initial microstructure (Figure 14b) consisted of an even distribution of small (~3 μm) globular α particles in a retained β matrix, with an α volume fraction of $10\pm1\%$.

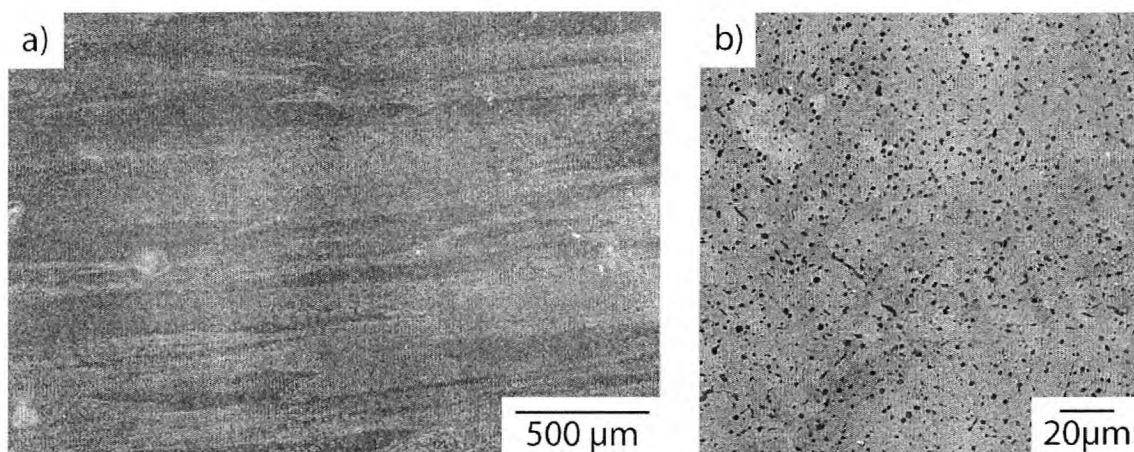


Figure 14: a) Light micrograph of Ti-5-5-5-3 as received macrostructure, b) backscattered electron image showing the microstructure of the same material section.

3.3 *Effect of Temperature on Microstructure*

3.3.1 Effect of time at temperature on microstructure

For any alloy system it is critical to understand the influence of time spent at a given temperature on the microstructure, especially when investigating the effects of hot working. Cotton [120], mentioned that the alloy design of Ti-5-5-5-3 purposely included ‘slow diffusers’ and therefore microstructural changes over a relatively long period (600 minutes) were considered. Figure 15 shows representative backscattered electron micrographs illustrating the microstructures obtained following aging Ti-5-5-5-3 for 5, 15, 60 or 600 minutes at temperatures of 750, 800 and 850°C. All samples were water quenched after heat treatment to retain the high temperature microstructure. Quantitative data, such as α phase volume fraction and particle sizes, were obtained using the image analysis software ImageJ. Volume fractions are given to an accuracy of $\pm 1\%$ and particle size to the nearest $0.5\mu\text{m}$.

Aging at a temperature of 750°C (approximately 100°C below the reported transus temperature [119]), clearly shows that the α microstructure is evolving between 5 and 600 minutes (Figure 15a-d). Following 5 minutes at temperature, the microstructure resembles the as received condition, with small globular α particles at a volume fraction

of ~9% within a β matrix. As the time at temperature increases, an enlargement of the α particles can be seen and the volume fraction is increased to ~12% after 15 minutes and to ~17% after 60 minutes. After 60 minutes of aging, secondary α begins to form between the primary precipitates. The nucleation and growth of secondary α continues during further aging and after 10 hours the α phase volume fraction has reached ~26%. The observed increase in α phase volume fraction by the formation of secondary α precipitates would seem logical as 750°C is only 70°C above the reported industrial α aging range [119].

The effect of time when Ti-5-5-5-3 is aged at a temperature of 800°C is shown in Figure 15e-h. As with aging at 750°C, the quenched microstructure observed following 5 minutes aging is similar to that of the as received billet, with an α volume fraction of ~10%. Further aging, for 15 minutes, reduced the size of the globular α precipitates and correspondingly the α volume fraction decreased to approximately 7%. Increasing the aging time at 800°C to 60 minutes does not result in any discernable change in the microstructure, when compared to the 15 minutes aged sample. The globular α particles are approximately the same size (~2.5 μm) and the volume fraction remained at ~7%. Following aging for an extended period of 600 minutes the α precipitates can be seen to be coarsening, with the average α particle size increasing to ~4 μm . However, the volume fraction was found to be ~8% which is not significantly different to the volume fraction found following 15 or 60 minutes at 800°C.

Aging close to the reported β transus at 850°C causes the most obvious change to the microstructure of the alloy (Figure 15 i-l). Unlike aging at 750°C and 800°C, there is a distinct change in the α microstructure after 5 minutes at 850°C. The α precipitates have decreased in size and the volume fraction has decreased to ~4%. Further aging close to the transus reduced the volume fraction of the α phase to ~1% after 15 minutes at 850°C. Extended aging for 60 or 600 minutes results in the complete transformation of the α phase into the β phase, resulting in an entirely retained β microstructure upon quenching. This observation would suggest that the β transformation temperature for the Ti-5-5-5-3 billet provided for this study lies below 850°C.

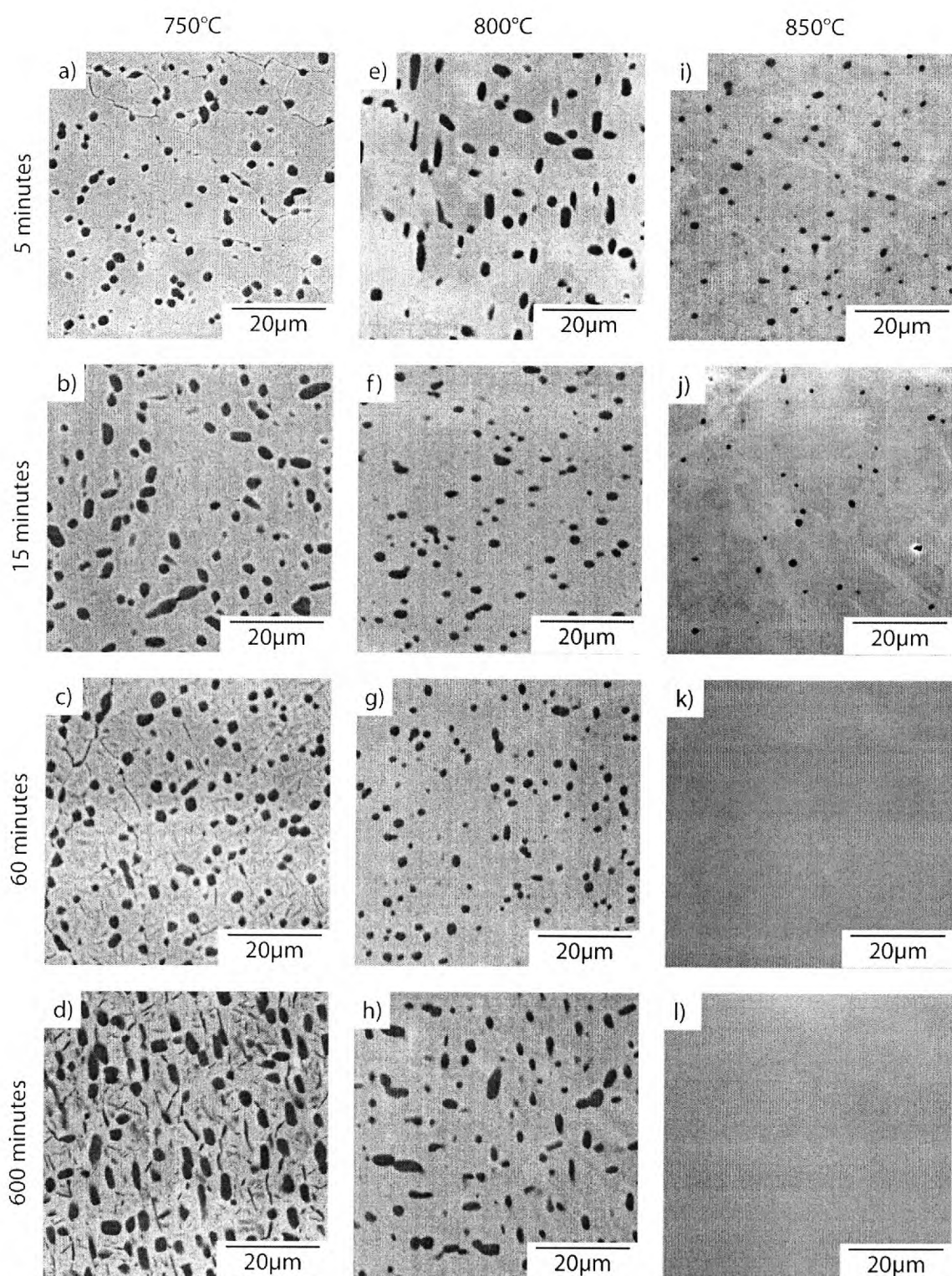


Figure 15: Backscattered electron micrographs showing the effect of time at temperature on the volume fraction of the α phase Ti-5-5-5-3. All samples were cooled via water quenching.

3.3.2 Beta Approach Curve

The data presented in Section 3.1.1 suggests that the β transformation temperature for the billet of Ti-5-5-5-3 used in this study lies between 800°C and 850°C. It would appear that the equilibrium microstructure within this temperature range is achieved by aging for 60 minutes at the relevant temperature. Therefore a more detailed study of the β approach curve was obtained by evaluating the α phase volume fraction of small samples which were water quenched having been aged for 60 minutes at temperatures between 800°C and 870°C. The resultant approach curve is shown in Figure 16, with the β transus temperature found to be ~845°C, consistent with that reported by Fanning for TIMETAL 555 [119].

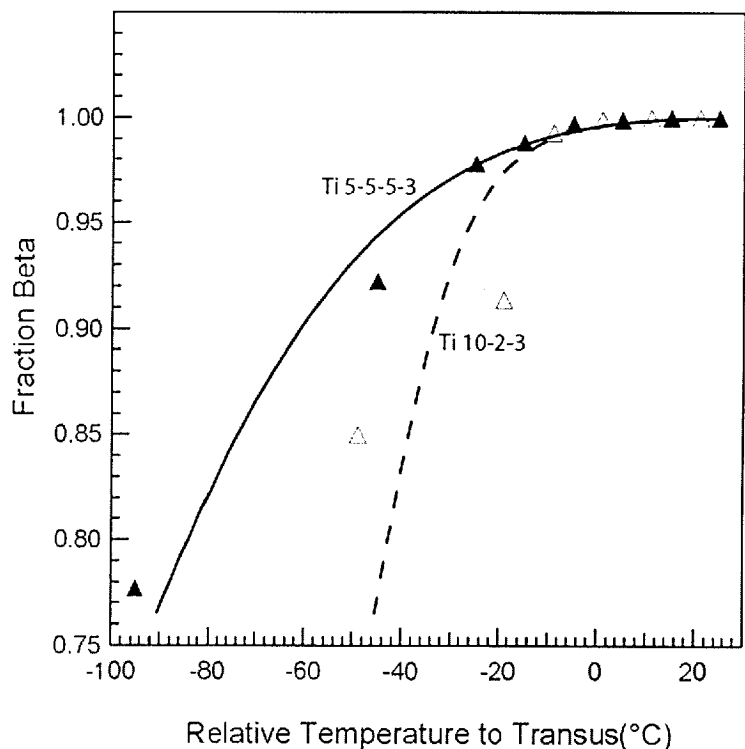


Figure 16: β Transus approach curves for Ti-5-5-5-3 and Ti-10-2-3 (lines are provided as guides to the eye).

For comparison the approach curve of Ti-10-2-3 is also shown in Figure 16. The β transus temperature for Ti-10-2-3 is ~790°C, which is ~50°C lower than that of Ti-5-5-5-3, and it is important to note the difference in approach gradient between the two alloys. Ti-10-2-3 has a steeper β transus approach gradient than Ti-5-5-5-3, implying

that Ti-10-2-3 will have a higher microstructural temperature sensitivity. Flow behaviour and microstructural evolution are both influenced by the α phase [78] and therefore its control during forging can be critical. Previous work has shown that small temperature variations (e.g. due to die chill or adiabatic heating) during processing can have a dramatic effect on the resultant microstructure in Ti-10-2-3 [123]. The shallower approach curve of Ti-5-5-5-3 suggests that the level of process control can be reduced, as a wider or “friendlier” processing window is available [7].

3.3.3 Cooling Rates

The effect of cooling rate upon the microstructure is very important in industrial forgings where the size of the workpiece can prevent controlled cooling. Figure 17 shows the microstructures corresponding to small samples ($\sim 1\text{cm}^3$) cooled from 800°C (transus – 45°C) and 900°C (transus +55°C) by furnace cooling, air cooling and water quenching.

Furnace cooling from the $\alpha+\beta$ region (Figure 17a) produces an α volume fraction of $\sim 20\%$. The slow cooling rate allowed sufficient time for diffusion to produce small globular α particles, $\sim 3 - 5\ \mu\text{m}$ in size, and thin α precipitates in between the globular primary α particles. Air cooling from subtransus temperatures (Figure 17b) produces a globular α structure in a β matrix. The α particles were slightly smaller than when furnace cooled, $\sim 2 - 4\ \mu\text{m}$, and the α volume fraction dropped to approximately 10%. Quenching the material from the $\alpha+\beta$ field (Figure 17c) produced a microstructure very similar to that of air cooling (Figure 17b); a β matrix containing $\sim 10\%$ volume fraction of $\sim 3\ \mu\text{m}$ diameter globular α particles.

Slow cooling from the β phase field (Figure 17d) produces a continuous layer of α on the β grain boundaries. In addition to the grain boundary layer, colony-like α has nucleated from the boundaries and grown inwards towards the grain centre, consistent with the generalised microstructure evolution summarised by Flower [20]. Air cooling and quenching from the super transus temperature produced apparently 100% β microstructures, with no grain boundary α precipitation (Figure 17e and f).

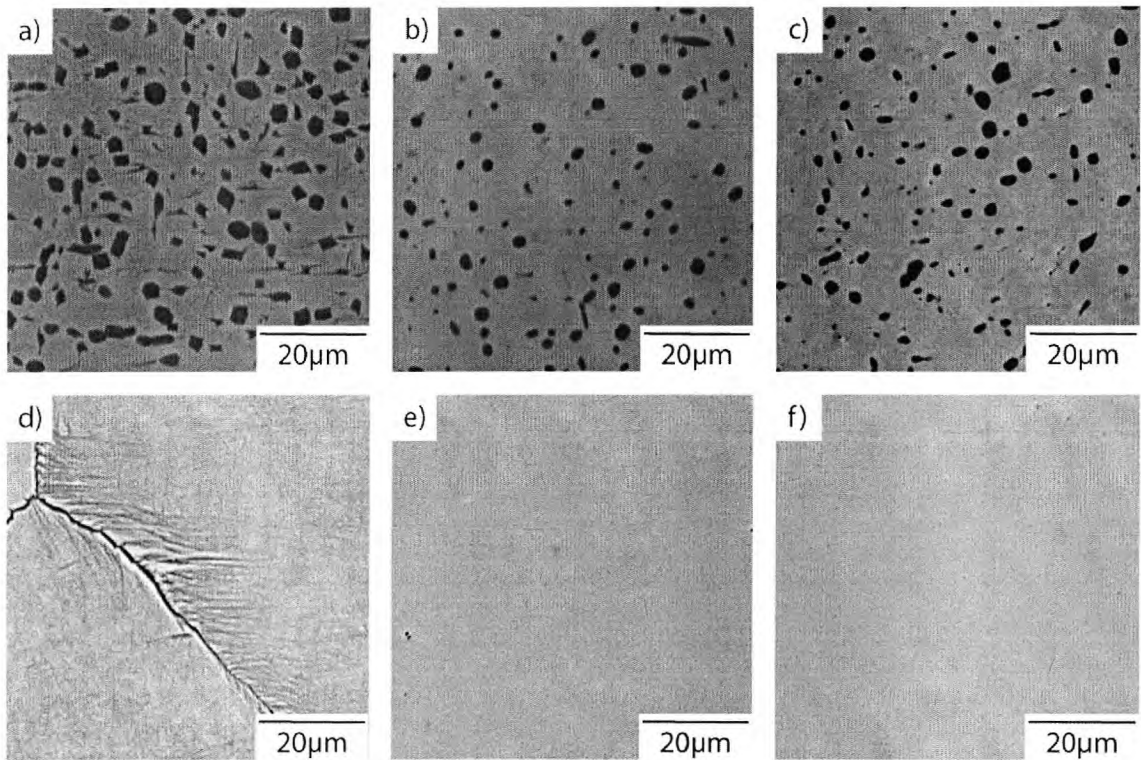


Figure 17: Backscattered electron micrographs of Ti-5-5-5-3 microstructures obtained when furnace cooling from a) 800°C and d) 900°C, air cooling from b) 800°C and e) 900°C, and water quenching from c) 800°C and f) 900°C .

X-ray diffraction data from both the air cooled and water quenched super transus samples is shown in Figure 18. The spectra peaks correspond solely to the bcc β phase, confirming that the α phase is not present following air cooling or water quenching from the β phase field. Around the base of each β peak there is noticeable broadening which could suggest the presence of a small second phase. The broadening appears around each of the β reflections which could correspond to the ω phase, previously reported in Timetal-555 [120, 124]. The broadening of the X-ray diffraction reflections can be seen in Figure 19a, and the supposition of these being caused by the ω phase is supported by the streaking observed between the bcc β spots in Figure 19b.

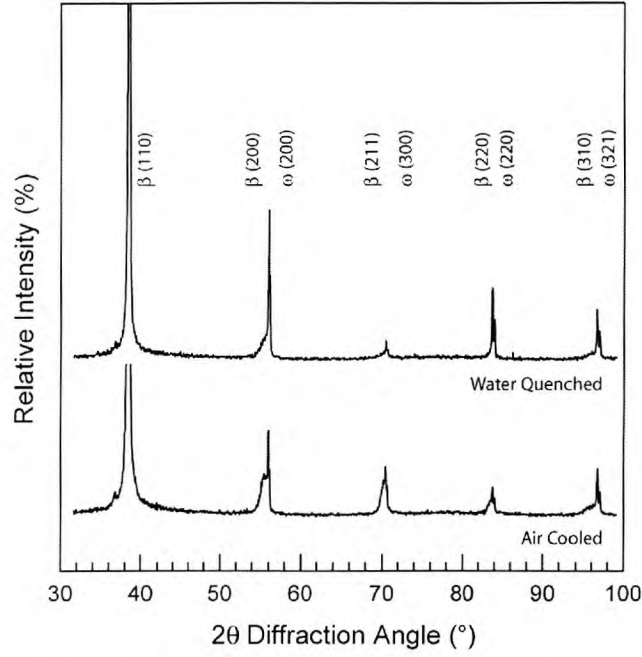


Figure 18: X-ray diffraction spectra for Ti-5-5-5-3 cooled from 900°C by water quenching and air cooling.

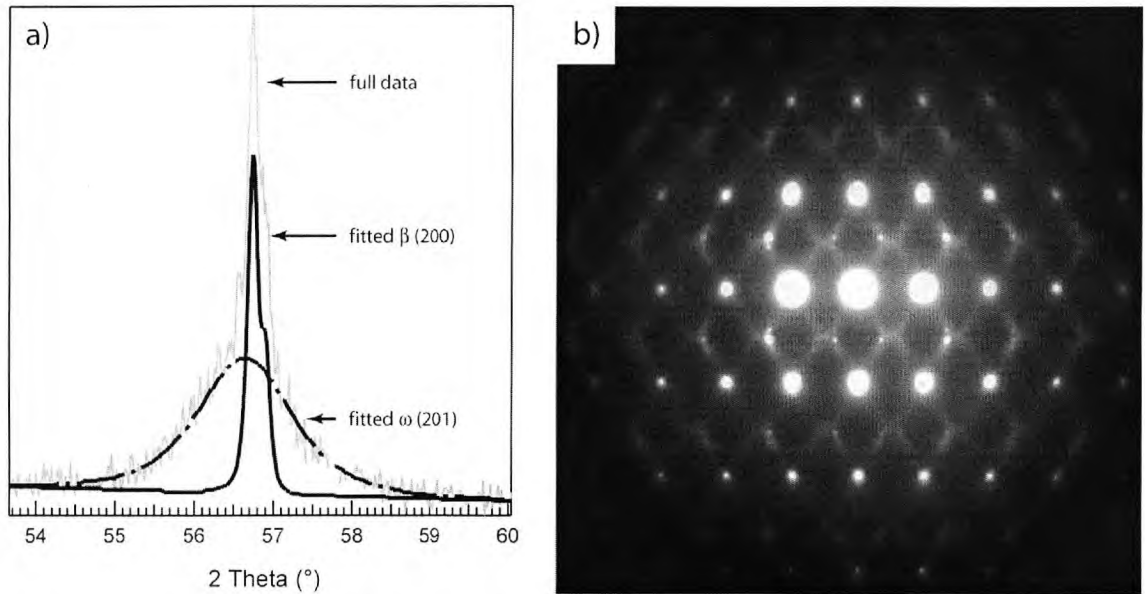


Figure 19: Evidence of ω phase in Ti-5-5-5-3 following quenching from 900°C a) conventional X-ray diffraction showing broad peaks with β and ω phases fitted, b) electron diffraction pattern from β [110] zone axis showing diffuse streaking typical of the ω phase.

3.3.4 Slow Cooling and Aging

The microstructures developed by slow cooling and aging can be seen in Figure 20. Treatment above the transus results, unsurprisingly, in a microstructure consisting entirely of the β phase, Figure 20a. Slow cooling and holding approximately 50°C below the transus at 800 (Figure 20b) begins to precipitate α as small globular particles. The observed volume fraction of precipitated α (< 1%) is far lower than that predicted by the β approach curve (~8%), further alluding to the slow diffusional nature of this alloy system and the need to produce approach curves relative to the process time frame.

Cooling and aging at 700°C, ~150°C below the transus temperature, precipitates α as a continuous layer along the parent β grain boundaries. Independent nucleation observed within the parent β grains as acicular precipitates, ~10 - 15 μ m in length, surrounded by a β matrix.

At 600°C (Figure 20d) the microstructure consists of grain boundary α layers from which fine colonies of α , ~15 - 20 μ m in length, have nucleated and grown inwards towards the parent grain centre. The intragranularly nucleated precipitates have grown to become high aspect ratio particles, ~30 μ m in length by ~5 μ m wide. It would appear that these precipitates have grown from an initial globular precipitate, and subsequently fine colony α structures have initiated and grown from these precipitates via sympathetic nucleation.

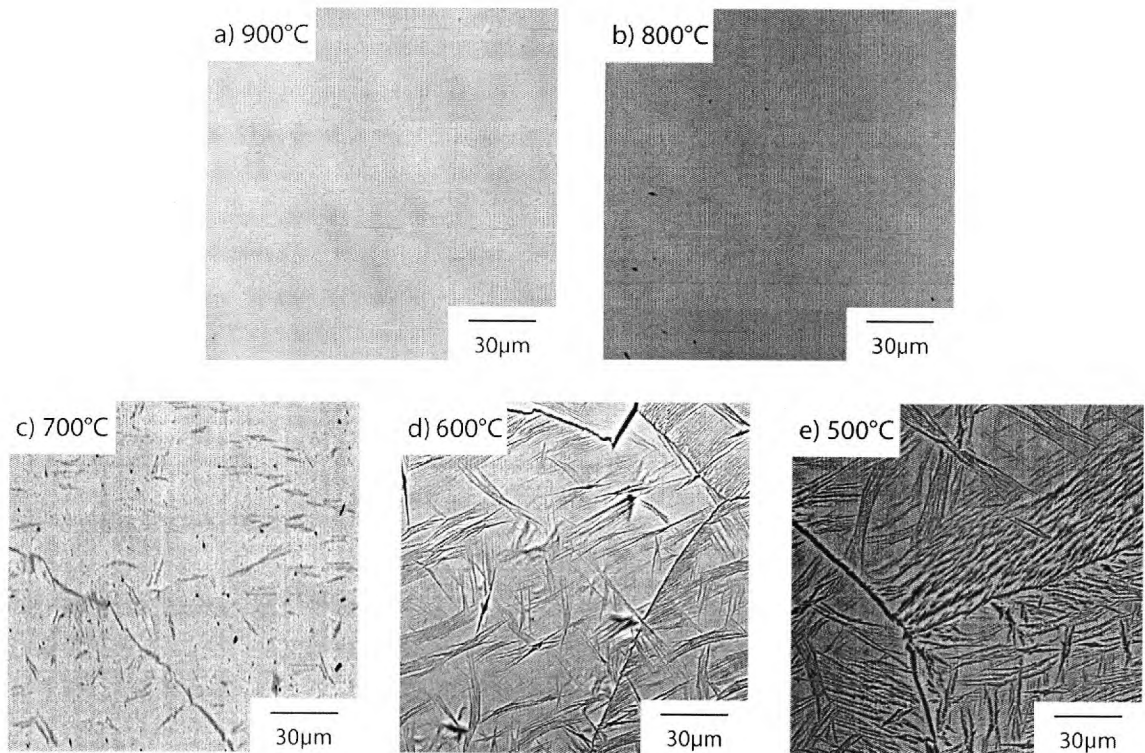


Figure 20: Backscattered electron images of microstructures developed by a 60 minute β field heat treatment followed by slow cooling to a) 900°C, b) 800°C, c) 700°C, d) 600°C and e) 500°C followed by a further 60 minute hold.

Slow cooling from above the transus to 500°C produces a microstructure dominated by colonies of α precipitates, Figure 20e. Formation of this microstructure appears to have followed a similar path to that of the 600°C sample, with colonies initiating from grain boundary α or from larger acicular precipitates within the parent β grains. The additional time at temperature has allowed the fine colonies to further develop and thicken the α precipitates within these structures.

3.4 Summary of Results

The initial characterisation of Ti-5Al-5Mo-5V-3Cr has shown some significant variations from its predecessor alloy Ti-10V-2Fe-3Al. The observed differences between the alloys indicate that Ti-5-5-5-3 microstructures would be more controllable during industrial scale thermal processes.

Ti-10-2-3 has been shown by previous work [5] to be very sensitive to small fluctuations in temperature during subtransus processing. Results presented in this chapter show a shallower gradient for the β approach curve of Ti-5-5-5-3 when compared with that of the Ti-10-2-3. Therefore, the volume fraction of α , which dominates the mechanical properties of β titanium alloys, in Ti-5-5-5-3 is less prone to significant change during hot working and thus the alloy would have a wider processing window than Ti-10-2-3.

Despite the two alloys having an identical Mo Eq (9.38, based upon nominal compositions) Ti-10-2-3 has a β transition temperature $\sim 50^\circ\text{C}$ lower than that of Ti-5-5-5-3. This difference would indicate that the level of β stability in Ti-5-5-5-3 is less than that of Ti-10-2-3, contrary to what might be concluded from the Mo Eq data alone. However, Ti-5-5-5-3 shows a low quench sensitivity, compared to Ti-10-2-3, which will be industrially significant as there appears to be no obvious microstructural difference between water quenched and air cooled conditions. Additionally no martensite is observed following quenching, indicating that Ti-5-5-5-3 lies to the right of the martensitic transformation (Ms/Mf) line as described by Flower [20]. There is also evidence to support the observations [125] of the ω phase in Ti-5-5-5-3 following quenching from the β phase field.

The ability of Ti-5-5-5-3 to retain a β structure following super transus quenching would suggest that the β phase is actually more stable than that of Ti-10-2-3, with a higher level of deformation required to induce a stress induced martensitic transformation. An interesting question which arises from this situation would be what mechanism is suppressing the martensitic transformation in Ti-5-5-5-3. One possible answer is the presence of the large alloying element Mo, which is known for its slow

diffusion in Ti alloys. It can be hypothesised that no reduction in packing density can occur without being able to locally reject Mo atoms. As such during the rapid process of quenching no such change is possible due to the inability of Mo atoms to diffuse a significant distance

Work presented in this chapter regarding both the formation and dissolution of the α phase has exhibited the reported slow diffusion kinetics in Ti-5-5-5-3, an apparent design feature of the alloy [120]. α phase precipitates were able to persist for more than 15 minutes at a temperature just above the transus temperature. Furthermore when cooling through the transus to 45°C below the transformation temperature, α precipitation produced a volume fraction less than 1% following a 60 minute hold at 800°C. Slow microstructural response makes exact prediction of α volume fractions for a given time and temperature difficult but is likely to facilitate a wider processing window, significant for the forger working with the large components made using this alloy.

4 Subtransus Forging of Ti-5Al-5Mo-5V-3Cr

Ti-5-5-5-3 is widely reported to be superseding Ti-10-2-3 for high strength aerospace applications, such as truck beams in landing gear assemblies on large aircraft [7]. Economic manufacture of these large components is achieved via hot near net shape forging [75]. The mechanical properties of these components are entirely reliant upon the final microstructure and therefore an understanding of the relationships between processing and microstructure is essential. Due to the microstructural sensitivity of Ti-10-2-3 to hot working variables, forgers have had to spend time optimising their procedures to meet required mechanical properties. Initial attempts to forge Ti-5-5-5-3 based on knowledge of Ti-10-2-3 have resulted in the successful achievement of the required mechanical properties [6]. However no specific study of the deformation mechanisms or microstructural evolution when forging Ti-5-5-5-3 has been reported. In order to enable a direct comparison between the two alloys, forging conditions employed in this study were chosen based upon relevant literature concerning Ti-10-2-3 [123, 126].

A comprehensive study concerning the microstructural evolution during subtransus forging of $\alpha+\beta$ condition Ti-10-2-3 was carried out by Jackson [5]. The work employed a similar experimental configuration as that used for the current study (described in Section 4.1.1) with forging conducted at the industrially significant temperature of 760°C. This temperature is approximately 35°C below the β transus where the α phase volume fraction is ~15%. The data presented in this chapter describes work conducted to evaluate the effect of isothermal forging on microstructural evolution in Ti-5-5-5-3. Starting microstructural conditions are referred to by the phase field in which the preceding processing step occurred; for example the as received material was last processed in the $\alpha+\beta$ field and hence is labelled as $\alpha+\beta$ condition .

Ti-5-5-5-3 was isothermally forged at temperatures of 785°C (chosen to match the α phase volume fraction of Ti-10-2-3 at 760°C), 810°C (chosen as the same relative temperature below the β transus temperature as 760°C is for Ti-10-2-3) and 835°C (investigate forging high in the $\alpha+\beta$ phase field). The industrial processing

development for Ti-5-5-5-3 has been largely based upon the existing knowledge and experience of Ti-10-2-3. Initially therefore, Ti-5-5-5-3 is being forged approximately 35°C below is β transition temperature, the same relevant temperature as used for Ti-10-2-3 [119].

The effect of temperature on the microstructure at the relevant forging temperatures and testing times are investigated such that thermally influenced changes can be distinguished from those which are deformation driven. The flow behaviour of Ti-5-5-5-3 is evaluated over a range of strain rates between 10^{-3} s^{-1} and 10^{-1} s^{-1} and for three different initial material conditions. The effect of both strain rate and strain on the microstructural evolution is considered and from this data suggestions as to the dominating deformation mechanism have been made. Comparison of the flow behaviour and microstructural observation following forging of the different initial materials has enabled the development of an empirical relationship to be determined between the morphology and volume fraction of the α precipitates prior to forging and the extent of flow softening.

4.1 *Experimental Methods*

4.1.1 Isothermal Compression Testing

Isothermal forging was carried out using a Mayes 100 kN servo-hydraulic testing machine with a Zwick-Roell HW9610 controller. The controller is capable of producing constant strain rate conditions using a derived control channel, calculated using the instantaneous ram position. Thermal conditions were achieved using a Severn Furnaces 1000°C radiant furnace controlled using a K-type thermocouple connected to a Eurotherm 902P controller. The thermocouple was located 2 mm into the bottom ram and 5 mm below the platen face. The samples were loaded at ~400°C before being heated to the testing temperature at a rate of 25°Cmin⁻¹. During heating, a clamping load of 0.1 kN was used, which maintained sample position whilst also accommodating thermal expansion of both the sample and the system.

Uniaxial compression samples (8 mm in diameter by 10 mm in height) were forged at the subtransus temperatures of 785°C, 810°C and 835°C, and at strain rates of 10⁻³ s⁻¹, 10⁻² s⁻¹ and 10⁻¹ s⁻¹. Double truncated cone specimens, with geometry detailed in [126], were forged at a strain rate of 10⁻² s⁻¹ at temperatures of 785°C, 810°C and 835°C. Samples were also tested without applying deformation to assess the effect of time at temperature. All samples were coated in a boron nitride (BN) release agent and received an immediate water quench following compression. Metallographic techniques used to prepare samples were the same as outlined in section 3.1.5 and microstructural characterisation was carried out using optical and electron microscopy.

4.1.2 Finite Element Modelling

The forging process was simulated using the Transvalor (France) software package Forge2005 [127]. Tooling and specimen geometries, material specific rheology data and interface friction parameters were all included in the modelling input. The package uses the Norton-Hoff constitutive law, shown below, to relate flow stress σ to strain ϵ , strain rate $\dot{\epsilon}$ and temperature T .

$$\sigma = \sqrt{3}^{m+1} K \dot{\epsilon}^m \epsilon^n \exp\left(\frac{Q}{RT}\right)$$

Where m is the strain rate sensitivity, n the work hardening exponent, Q an activation energy, R the universal gas constant and K a material constant. The Norton-Hoff constitutive model is appropriate in materials that show no flow softening, such as $\alpha+\beta$ condition Ti-10-2-3 [5].

4.1.3 Image Analysis

Imaging of fine scale α precipitates requires the use of high magnification backscattered electron microscopy. When imaging using backscattered electrons, the conditions required to resolve these small structures often results in a loss of phase contrast. The reduced contrast difference between the α and β phases was insufficient for automatic differentiation using the thresh-holding capability of the image analysis software (ImageJ). Therefore, BSEI images were digitally enhanced before being accentuated manually such that an outline image of the α precipitates could be constructed and analysed, schematically represented in Figure 21.

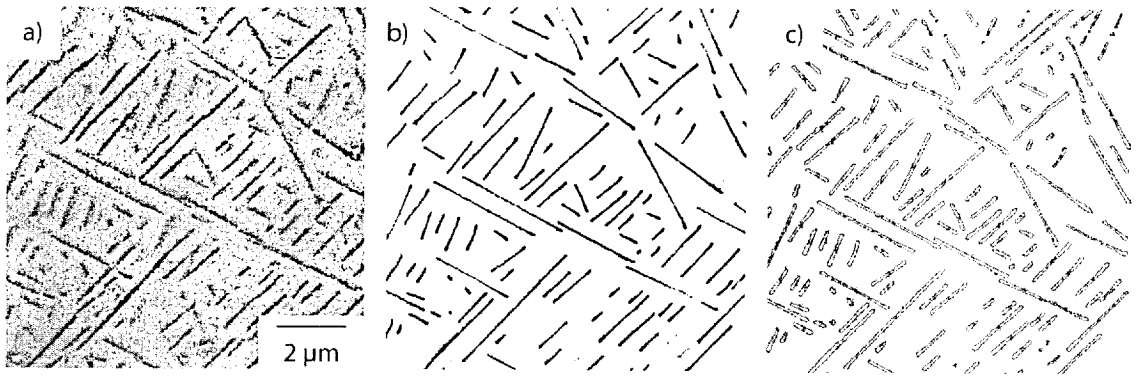


Figure 21: Schematic representation of the image analysis approach used to characterise the α phase precipitates in β condition Ti-5-5-5-3 a) digitally enhanced original backscattered electron image micrograph, b) manual accentuation of the laths, and c) image analysis software outline of α precipitates.

4.2 Forging of $\alpha+\beta$ Condition Ti-5-5-5-3

4.2.1 Witness microstructures

Prior to investigating microstructural development during hot forging, it was important to understand any thermal effects on the microstructure so that they could be distinguished from deformation effects. Figure 22 shows the microstructures of samples held at the subtransus temperatures of 785°C, 810°C and 835°C, for 120 seconds (which represents the experimental soak time prior to testing) and 920 seconds (which represents the soak and forging time when testing at the lowest strain rate of 10^{-3} s^{-1}). Unlike the microstructures shown in Figure 15, these samples were produced using the same equipment and procedure as used for isothermal forging, thereby producing true witness microstructures. Image analysis was used to quantify the α phase volume fractions of the witness sample microstructures.

At 785°C, Figure 22a and b, the α volume fraction compares favourably with that which would be predicted by the β approach curve. There is little variation between the two samples, with an α phase volume fraction of ~9% after 120 seconds and ~8% after 920 seconds. The morphology of the α precipitates after 120 seconds appears to be more elongated and coarser than that of 920 seconds, consistent with normal spheroidisation effects as precipitates reduce their interfacial area. This slight variation and the volume fractions would appear to correspond with the microstructures presented in Figure 15. Thus, heating to 785°C and holding for a 120 second soak period prior to forging does establish a stable α phase volume fraction, however the α precipitates are spheroidising during the testing period. Despite the spheroidisation, it is anticipated that any significant change in the microstructure observed following forging would be attributable to deformation not thermal effects.

Samples held at 810°C, Figure 22c and d, show a slight reduction in the volume fraction of the α precipitates with increasing time at temperature. Following heating and a 120 second soak period there is an α volume fraction of ~8.5%, which over the following 800 seconds decreases to ~6%. This change in the α phase volume fraction is consistent with that observed in Figure 15 and is approaching the volume fraction predicted from

the β approach curve. There is little change to the morphology of the α phase, although it would appear that the number of precipitates and their individual size is slightly reduced during the time period considered. However, it is not expected that such small changes are likely to cause any significant effect on the microstructural response during forging.

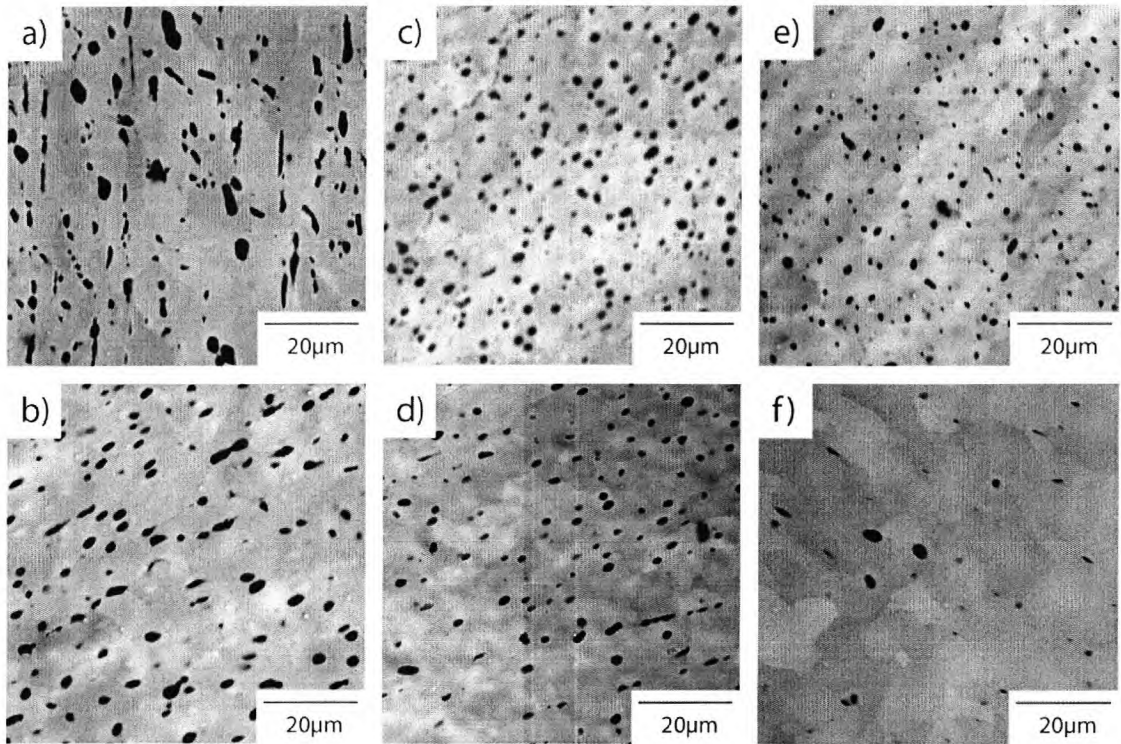


Figure 22: Backscattered electron microstructures of Ti-5-5-5-3 following heat treatment at 785°C for a) 120 seconds and b) 920 seconds, 810°C for c) 120 seconds and d) 920 seconds, and 835°C for e) 120 seconds and f) 920 seconds.

When Ti-5-5-5-3 is heat treated at 835°C there is a marked effect on the α phase volume fraction within the slowest forging time period. After heating and a 120 second soak the α volume fraction is ~5% which further reduces to ~1% within the testing time. This change to the microstructure is not unexpected as the α volume fraction predicted by the β approach curve is less than 1%. As the α precipitates dissolve back into the β matrix their morphology does not appear to be changing significantly such that a small volume fraction of globular α particles will be present during forging at 835°C. With the decrease in volume fraction of α precipitates during the testing period an associated growth of β grains could be expected due to a reduction in grain boundary pinning.

The reduction of interfaces, both through α dissolution and β grain expansion, could be significant in hot working behaviour. Semiatin and Bieler [128] proposed that the presence of α/β interfaces were significant to the peak stress and the magnitude of observed flow softening in the $\alpha+\beta$ alloy Ti-6Al-4V, through a Hall-Petch style strengthening mechanism. However when the concept was transferred to a β alloy, Ti-10-2-3, with a much lower α volume fraction ($\sim 13\%$ as opposed to $\sim 50\%$ in Ti-6V-4Al), the approximation failed to predict experimental observations [129]. When incorporating the lower volume fraction of thinner α plates (observed in the Ti-10-2-3 microstructure) into the Hall-Petch model, it was found that the estimation of flow softening was almost 10 times larger than that found experimentally.

4.2.2 Flow behaviour

The subtransus flow behaviour of $\alpha+\beta$ condition Ti-5-5-5-3 (Figure 23) is typified by elastic deformation to a peak yield stress followed by a steady state flow regime. As temperature decreases, limited flow softening is observed at higher strain rates before the steady state condition is reached; however, the magnitude of this effect is minimal ($\sim 10 - 15$ MPa) when compared to that seen in β condition Ti-10-2-3 (~ 60 MPa)[5]. The peak stress magnitude also decreases as the temperature approaches the transus temperature.

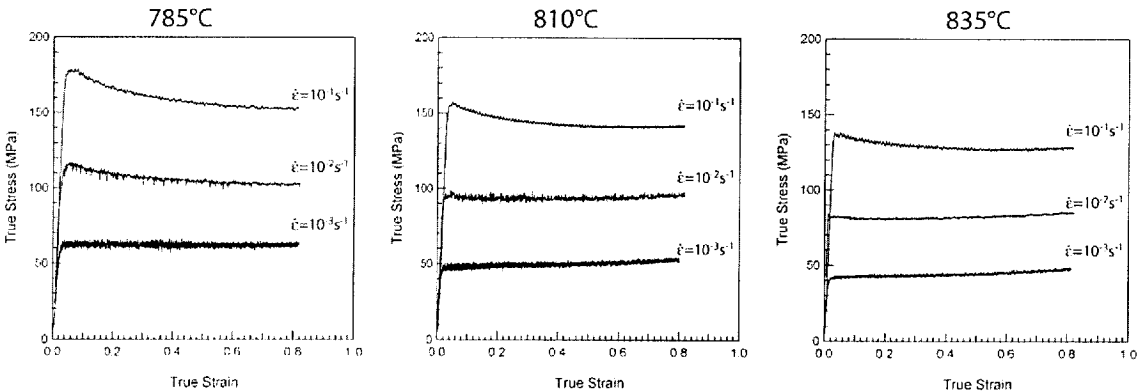


Figure 23: Isothermally forged $\alpha+\beta$ condition Ti-5-5-5-3 flow curves.

The observed flow behaviour of Ti-5-5-5-3 in the $\alpha+\beta$ condition is very similar to that seen with Ti-10-2-3 in a similar condition; whereby steady state is achieved at low

strains following a similar magnitude of flow softening [5, 130]. Rheology coefficients for Ti-5-5-5-3 can be obtained from the flow curves utilising a regression study detailed in [5]. As with work on Ti-10-2-3 the strain hardening coefficient, n , is assumed to be zero, as negligible work hardening is seen with increasing strain at isothermal forging temperatures. The strain rate sensitivity, m , and the material constant, K , are found from the regression study and are 0.22 and 4.0 MPa respectively. The activation energy for steady state flow is discussed in the next section.

One hypothesis suggested for the flow softening observed in β condition Ti-10-2-3 is based on the change in the morphology of the α phase. Previous work [5] suggests that the flow softening can be accounted for by the break-up of the α laths into several smaller globular particles. Microstructural evidence for the break up of the laths can be found in β condition Ti-10-2-3, where the laths break up into a necklace of small α particles, Figure 24 [129]. The α morphology in the initial $\alpha+\beta$ condition Ti-5-5-5-3 microstructure was found to consist of small globular precipitates. Such an α morphology would not present a significant barrier to dislocation movement during forging. The small level of flow softening observed in Figure 23 is most likely to be caused by elongated α particles which remain from the dissolution of the original grain boundary layer.

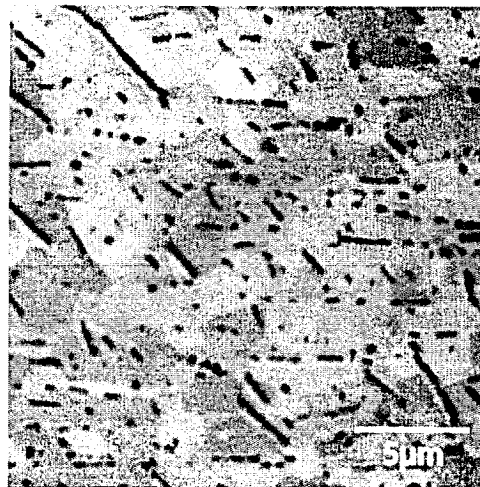


Figure 24: Backscattered electron image of Ti-10-2-3 showing evidence of α lath break up (sample forged at 0.1s^{-1} to a strain of 0.35 then to final strain of 0.4 at a rate of 0.01s^{-1}) from [129].

4.2.3 Effect of strain rate

The microstructural evolution of $\alpha+\beta$ condition Ti-5-5-5-3 following forging in the $\alpha+\beta$ region is shown in Figure 25. The globular α phase morphology observed in the initial microstructure (Figure 14b) has been maintained with little or no alteration. The retained β matrix is still present following quenching, with no evidence of a martensitic transformation. This would suggest that while processing in the $\alpha+\beta$ region, both strain rate and small changes to the working temperature have little effect on the microstructure of $\alpha+\beta$ condition Ti-5-5-5-3. Industrially this observation is highly significant as temperature fluctuations due to adiabatic heating will not have an undesirable effect on the microstructure, unlike Ti-10-2-3 where small temperature fluctuations can cause significant microstructural changes [123].

Quantitative image analysis was used to produce data on the development of the α phase during subtransus forging. Figure 26 shows the distribution of α particle size, quantified using an equivalent radius analysis. The distributions across all the strain rates show very little deviation in the positive skew, essentially indicating that there is no effect of strain rate on the α particle size.

Examination of the microstructures in Figure 25 and the flow data from Figure 23 gives some indication of the likely dominating deformation mechanism. Using steady state flow stresses and the analogy with steady state creep, outlined by Sellars [103], a regression study can be employed to find an activation energy for the process mechanism. Taking flow data from Figure 23 the activation energy is calculated to be 183 kJmol^{-1} , which is similar to published values for the self diffusion of (unalloyed) β titanium (153 kJmol^{-1}) [78].

The process of recovery requires dislocation to cross slip and climb, the latter of which can also be described as the diffusion of vacancies, and therefore the activation energy for the process would be similar to that of self diffusion in the β phase. A slightly higher activation energy would be expected for an alloy when compared to a pure metal, due to the increased solute content of the alloy. Whilst the alloy received a final forging step in the $\alpha+\beta$ region and the microstructure does contain both phases, the volume

fraction of alpha phase is small. Thus the majority of the deformation takes place in the β matrix. Hence the magnitude of the activation energy would suggest that the dominating mechanism is dynamic recovery of the β phase.

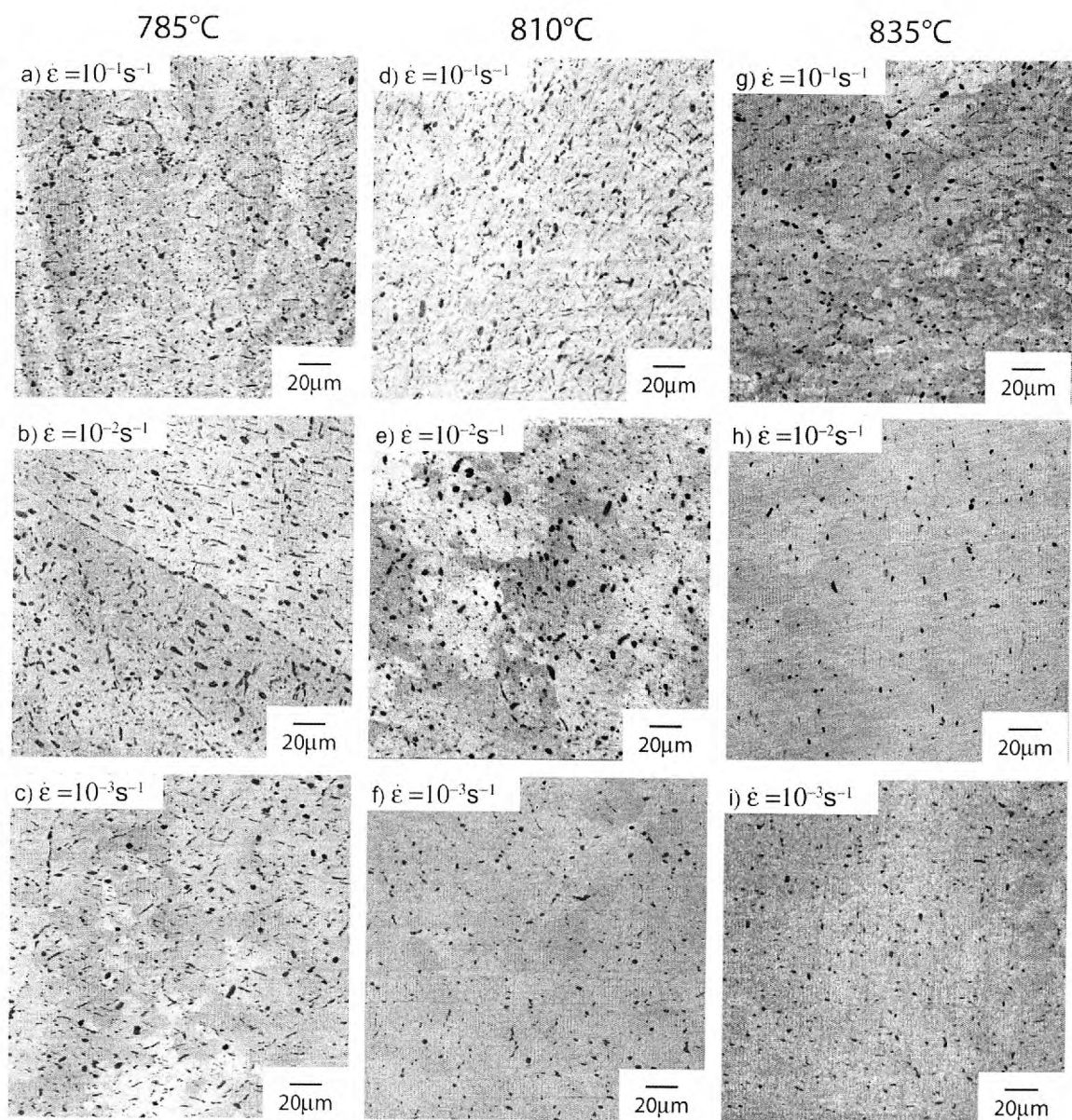


Figure 25: Backscattered electron images of $\alpha+\beta$ condition Ti-5-5-5-3 microstructures obtained after subtransus forging at different temperatures and strain rates.

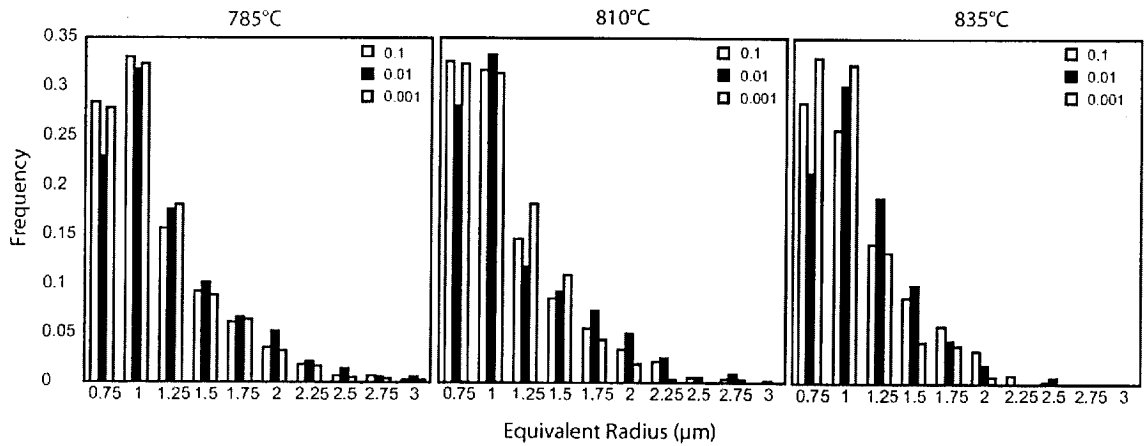


Figure 26: Equivalent radius distributions for α phase particles in Ti-5-5-5-3 following forging at different temperatures and strain rates.

4.2.4 Effect of strain

The effect of strain on the microstructure was investigated using double truncated cone specimens which when forged produce a controlled strain profile, as can be seen in Figure 27. Strain appears to have little effect on the volume fraction of the α phase at the temperatures investigated. The micrographs in Figure 27 do indicate an effect of the localised strain mode on α precipitate morphology. At the centre of the forging, which is similar to a uniaxial compression test in strain state, the α phase morphology is slightly elongated perpendicular to the forging axis. At the mid-radius location a more equiaxed α precipitate morphology was found, whilst 12 mm from the centre a hoop-strain dominated strain state gave rise to α elongated parallel to the forging axis. It would appear from Figure 27 that the extent of this effect is accentuated when deformation occurs at lower temperatures. The rotation of the α precipitates is consistent with the findings of the previous section, which suggested that α particles act as a witness to the flow of the β phase. As the matrix material flows during deformation the α phase precipitates are simply manipulated to cause the least resistance to the bulk flow. Thus suggesting that the majority of the flow occurs within the softer β phase with little load transfer to the hard α phase particles.

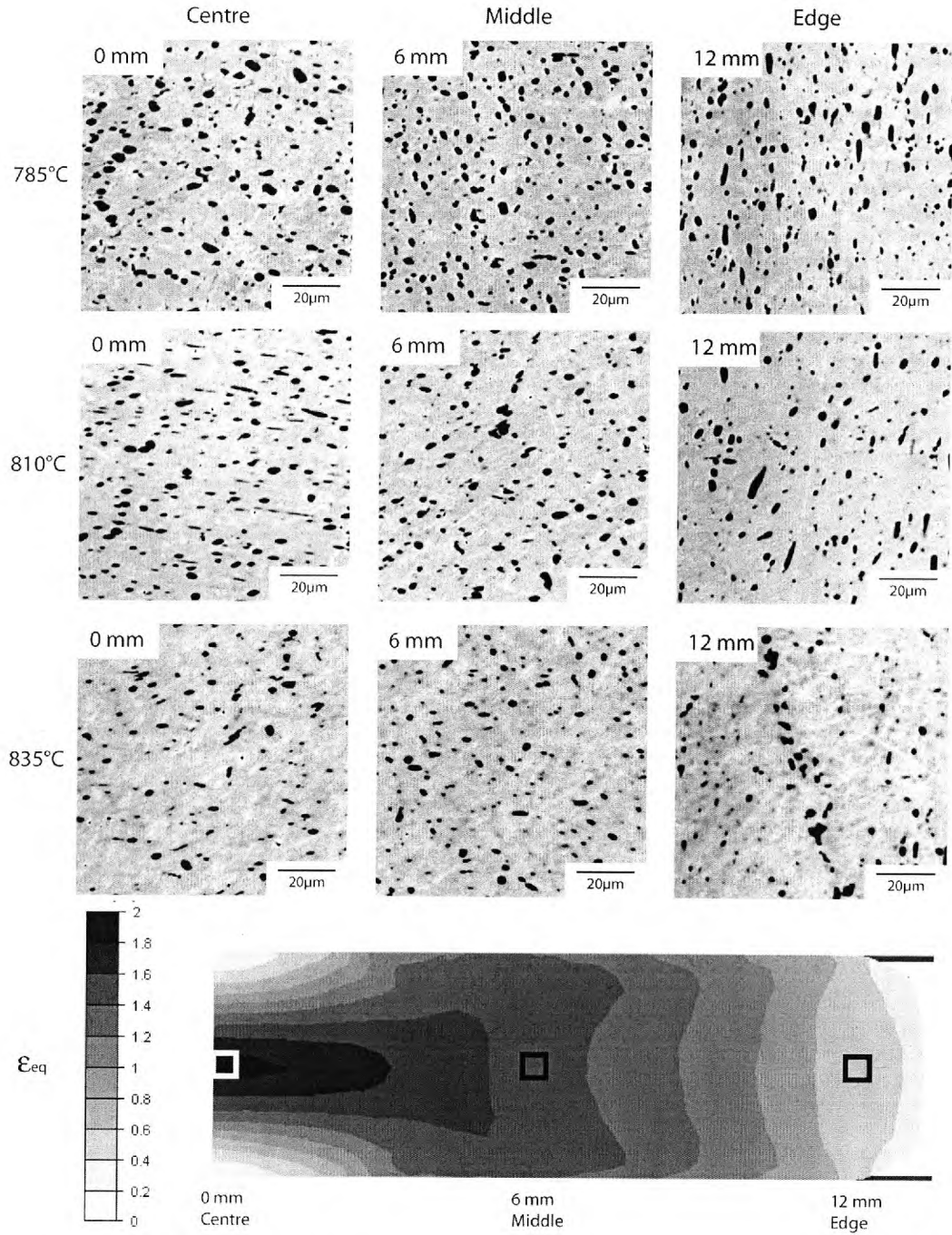


Figure 27: Backscattered electron images, of the microstructural evolution along the centre line of a double truncated cone following forging to a final height of ~ 4.5 mm at temperatures of 785°C, 810°C and 835°C and Norton-Hoff finite element model showing strain variation and sample location in the final forged state. The forging axis of all the micrographs is down the page.

(Equivalent strain is given by $\epsilon_{eq} = \frac{4}{3} (0.5((\epsilon_{xx} - \epsilon_{yy})^2 - (\epsilon_{zz} - \epsilon_{xx})^2 - (\epsilon_{yy} - \epsilon_{zz})^2 + 3\epsilon_{xy}^2))^{0.5}$)

4.3 Forging of Retained β Condition Ti-5-5-5-3

A cuboid sample, approximately 95 x 75 x 25 mm in size, was machined from the as received billet and heat treated above the β transus at 900°C for 60 minutes. Following heat treatment the billet was removed from the furnace and allowed to air cool. To minimise oxygen pick-up during the heat treatment the billet was coated in Achesons Deltaglaze 3418.

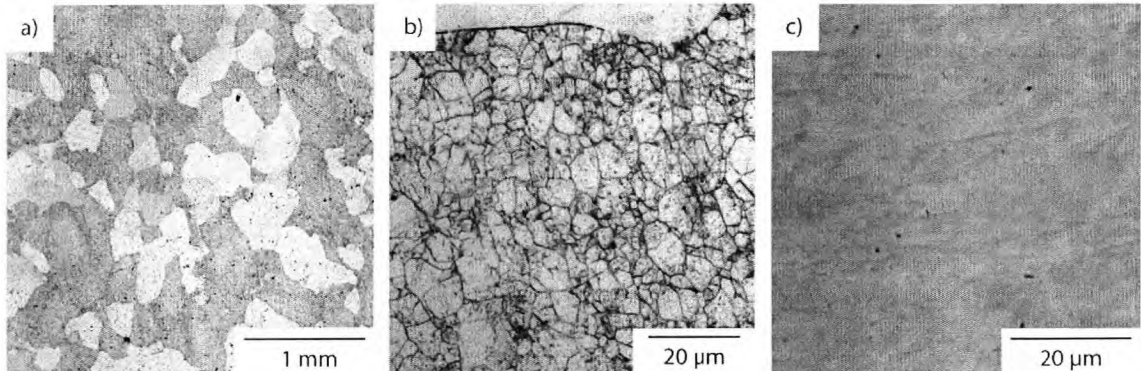


Figure 28: a) Light micrograph of retained β starting macrostructure, b) light micrograph of smaller grains inside those seen in macrostructure and c) backscattered electron image of the retained β microstructure from the same area as b).

The macrostructure and microstructure developed by super transus heat treatment and air cooling can be seen in Figure 28. The macrostructure (Figure 28a) consists of predominantly equiaxed β grains, with an average grain size of $\sim 300 \mu\text{m}$. Within these larger grains smaller equiaxed subgrains can be seen (Figure 28b) with an average size of $\sim 10 \mu\text{m}$. The formation of these smaller subgrains following a super transus heat treatment suggests that a polygonisation recovery process has occurred within the material. The microstructure appears to be fully retained β (Figure 28c), which is consistent with the cooling data presented in section 3.3.3.

4.3.1 Witness microstructures

The effect of the heating schedule, the pre-compression 120 second soak period and the maximum testing time on the retained β microstructure prior to isothermal forging at 785°C, 810°C and 835°C is shown in Figure 29.

At 785°C (Figure 29a) the subgrain structure can still be seen with the boundaries becoming delineated by the precipitation of an α layer following heating and pre test soaking. During the sample heating very small acicular α precipitates, less than 0.5 μ m long, have formed within the β grains. After 920 seconds at 785°C both the grain boundary layer and acicular α precipitates have coarsened (Figure 29d).

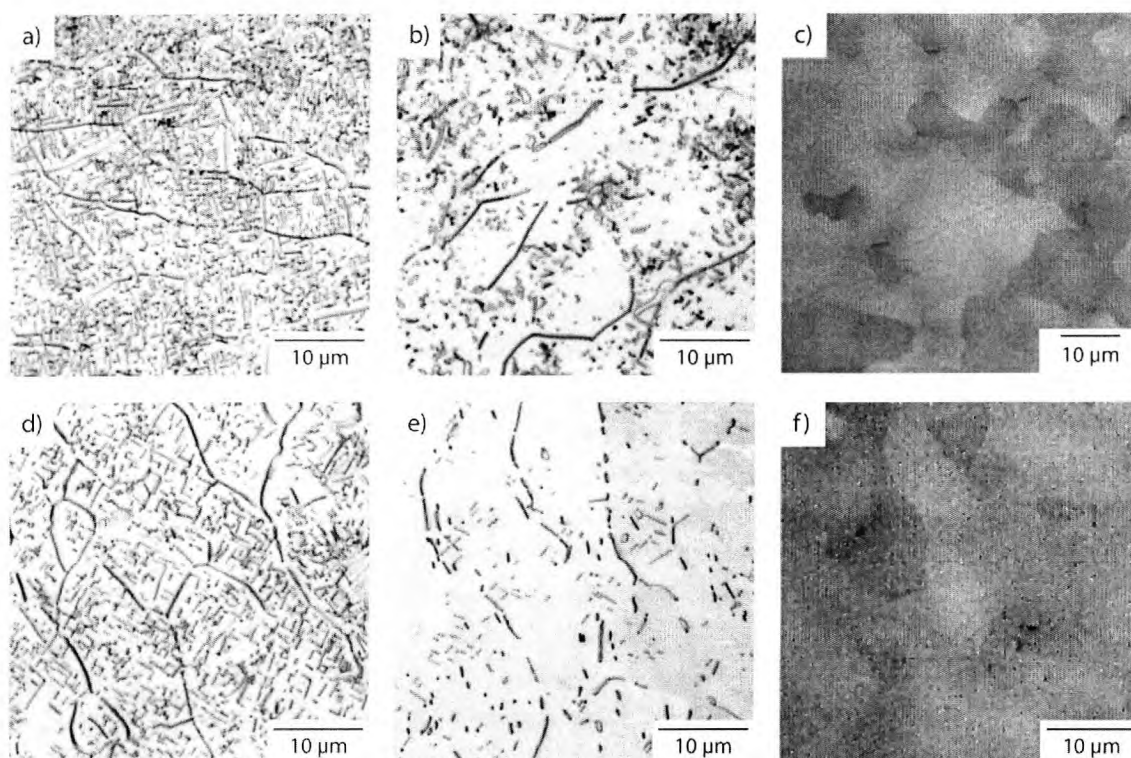


Figure 29: Light (a, b, d, e) and backscattered electron (c, f) micrographs of retained β witness structures following heating to a) 785°C, b) 810°C, c) 835°C, for a 120 second soak period and d) 785°C, e) 810°C, f) 835°C, for a 920 second period.

Following heating at 810°C for 120 seconds, the subgrain boundary decoration is less pronounced and less continuous than at 785°C (Figure 29b). Within the β grains the α precipitates are coarser than those observed at 785°C and have adopted a more globular morphology. Heating for 920 seconds (Figure 29e) reduces the volume fraction of α , most noticeably the small acicular precipitates within the β grains. The grain boundary layer has become even less continuous as it slowly dissolves developing into a globular morphology (Figure 29e). The microstructure developed when heating to 835°C, just below the β transus, consists solely of β grains (Figure 29c). The β grains are found to have grown when compared to the initial microstructure, however with no evidence of α

phase precipitates growth of β grains is not unexpected as the β grain boundaries are unconstrained. Further heating at 835°C results in the continued growth of the β grains, without any sign of α phase nucleation.

4.3.2 Flow behaviour

The flow behaviour of retained β condition Ti-5-5-5-3 during subtransus forging is presented in Figure 30. The behaviour can be characterised by elastic deformation to a yield point followed by steady state flow. As the forging temperature decreases and the strain rate increases, the peak stress increases and a small amount of flow softening (maximum of ~25MPa) is observed immediately following yield, before the system reaches a steady state regime. The magnitude of this softening, and non uniformity of flow behaviour across the temperatures investigated, is greater than observed in the $\alpha+\beta$ condition material. The variation is probably linked to the difference in volume fraction and morphology of the α precipitates in the high temperature microstructures (Figure 29).

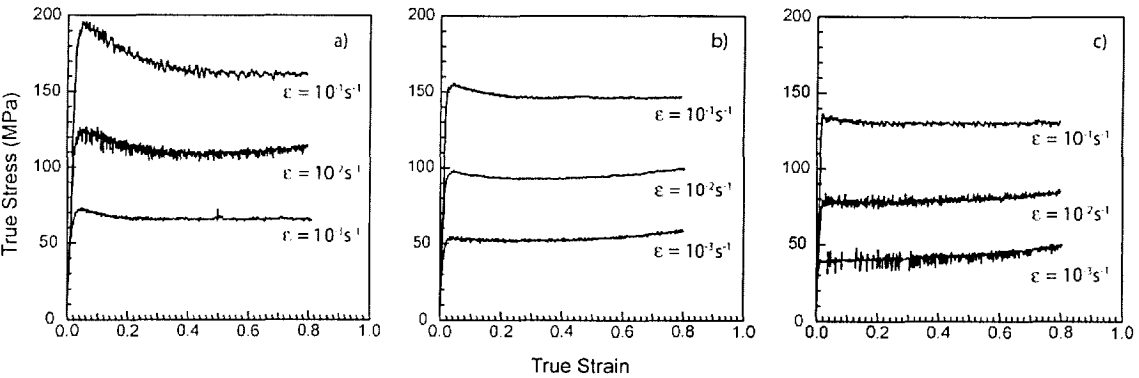


Figure 30: Flow curves of retained β condition Ti-5-5-5-3 isothermally forged at strain rates of 10^{-1}s^{-1} , 10^{-2}s^{-1} and 10^{-3}s^{-1} and temperatures of a) 785°C, b) 810°C and c) 835°C.

Comparison of the flow behaviour for the retained β condition material and the as received $\alpha+\beta$ condition is shown in Figure 31. The flow curves are largely the same and show good agreement with regard to steady state flow at a strain of 0.4, suggesting that an equilibrium microstructure has been developed at this point. As such it can be assumed that dynamic recovery is the dominating restoration mechanism in the retained β condition material once steady state has been reached.

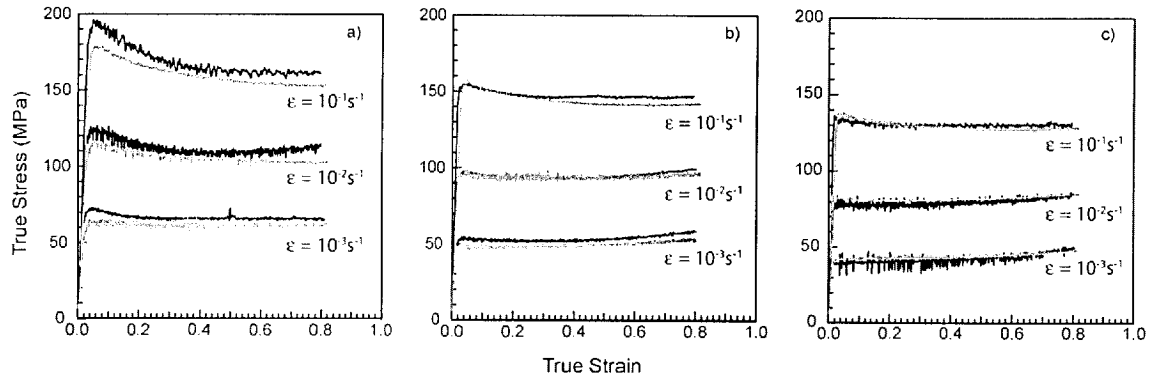


Figure 31: Comparison of the flow curves generated by $\alpha+\beta$ condition (grey) and retained β condition (black) during isothermal forging at strain rates of 10^{-1} s^{-1} , 10^{-2} s^{-1} and 10^{-3} s^{-1} and temperatures of a) 785°C, b) 810°C and c) 835°C.

4.3.3 Effect of strain rate

The effect of sub-transus forging on the microstructural development of retained β condition material can be seen in Figure 32. When forging at 785°C and 810°C, evidence of the α phase grain boundary layer, present at the onset of forging can be seen in Figure 32a, b and d. The grain boundary α layer appears to have been distorted perpendicularly to the compression axis during hot working and is particularly evident in Figure 32d. Figure 32a shows evidence that the grain boundary α layer fragments into smaller particles, slightly larger than the acicular precipitates observed in Figure 29a. Decreasing the strain rate leads to a more evenly distributed microstructure, with the α phase morphology becoming progressively more globular, presumably due to normal spheroidisation effects. The fragmentation of grain boundary α could account for the flow softening observed, through a similar mechanism as proposed for the flow softening in β condition Ti-10-2-3 [131]. Such an attribution would also appear to account for the relative magnitudes of flow softening at different temperatures. The volume fraction of grain boundary α developed during the pre-compression heating and aging is greatest at 785°C where a continuous layer was observed. At 810°C the grain boundary layer was less well defined and not continuous. Thus at higher forging temperatures there is a lower volume of α to fragment, equating to a lower level of observed flow softening during forging.

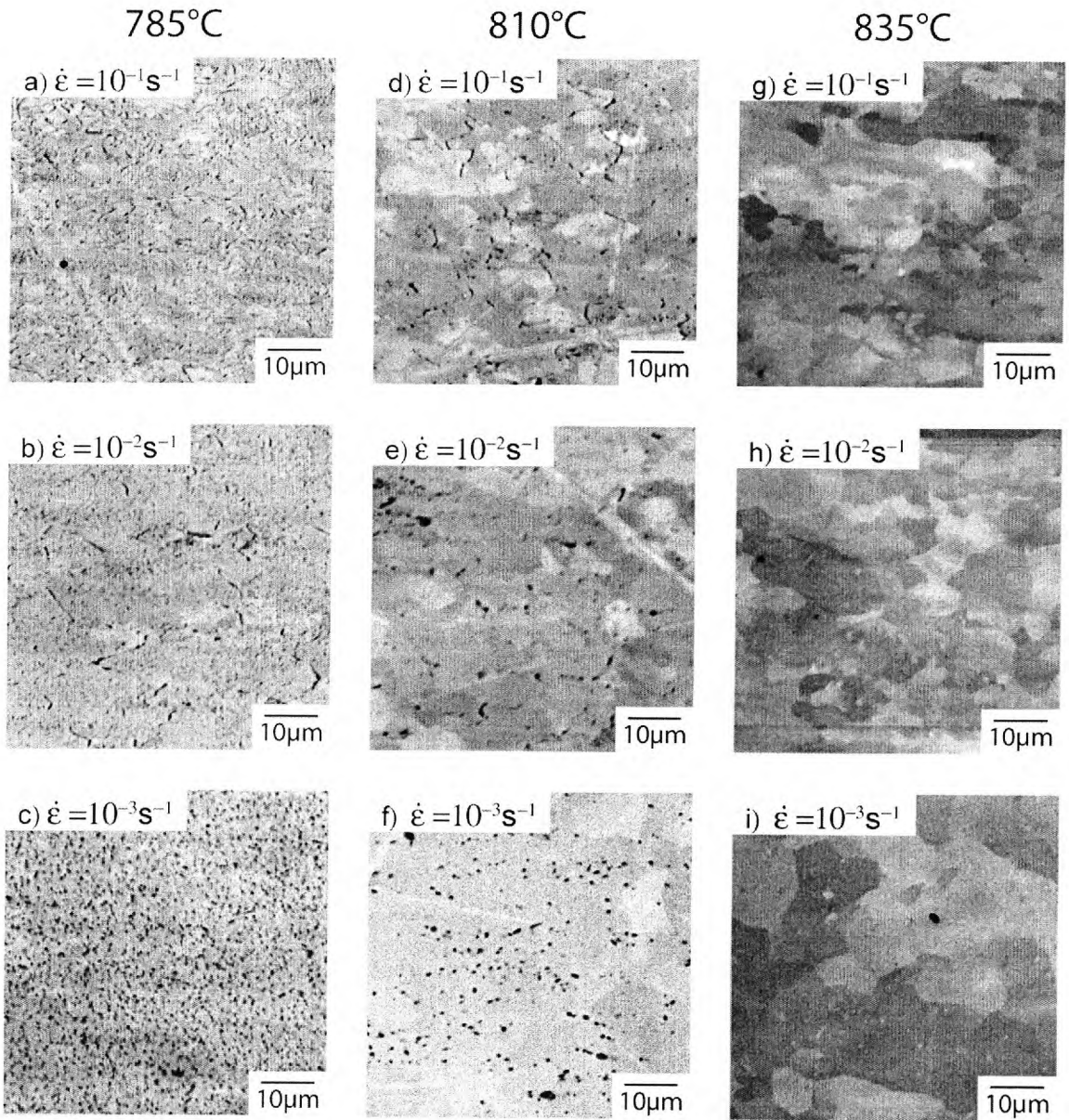


Figure 32: Backscattered electron images of microstructural evolution following isothermal forging of retained β condition Ti-5-5-5-3 at a range of temperatures and strain rates. The forging axis for all micrographs runs down the page.

The microstructural evolution of retained β material when forged at 835°C exhibits a very different microstructural response when compared to working at the lower temperatures of 810°C and 785°C. In the absence of any α phase precipitates the β grains are free to deform, grow and develop without constraint. As such a different grain size can be observed at each of the forging temperatures, with larger grains being observed when worked at lower strain rates. The larger grain size would be indicative of the lower dislocation density present at lower strain rates, as implied by the Zener-Holloman parameter. The increased times spent at temperature when forging at slower strain rates would also allow greater dislocation movement (by vacancy diffusion) and hence the formation of larger sub grains.

Another significant observation is the very similar flow behaviour of the retained β condition material and $\alpha+\beta$ condition material at 810°C and 835°C, despite having entirely different microstructures. This point is most prominent at the forging temperature of 835°C. The $\alpha+\beta$ condition material has a microstructure which prior to testing features small ($\sim 4\ \mu\text{m}$) globular α precipitates with a volume fraction of 5% (Figure 22). During heating equivalent to the longest forging time the α phase precipitates persist despite reductions in both volume fraction and particle size. In contrast, at the same temperature (835°C), the retained β condition material exhibits only β grains following heating and a 120 second soak period. During subsequent forging there is no evidence of α precipitation and as such it can be assumed that the deformation takes place entirely in the β phase. When the flow behaviour of the two microstructures is compared (Figure 31) it is found that the flow curves are very similar. Therefore, since the α phase cannot have influenced the flow behaviour of the retained β condition material, it can be suggested that small globular α precipitates have no effect on the subtransus flow behaviour of Ti-5-5-5-3 during hot working. The flow behaviour of Ti-5-5-5-3 in both the $\alpha+\beta$ and the retained β condition is representative of β phase deformation. This observation would appear to be consistent with the findings of section 4.2.4, where α phase precipitates were observed to be a witness to the flow of the β phase.

4.4 Forging of β Condition Ti-5-5-5-3

A cuboid shaped sample, approximately 35 x 35 x 75 mm in size, was isothermally forged above the β transformation temperature at The University of Birmingham. The sample was upset forged at a strain rate of 10^{-1} s^{-1} to a final height of 40 mm, equivalent to a true strain of approximately 0.56. Forging was conducted in an argon filled environmental chamber at a temperature of 950°C. Following upsetting the sample was slowly cooled to room temperature inside the environmental chamber.

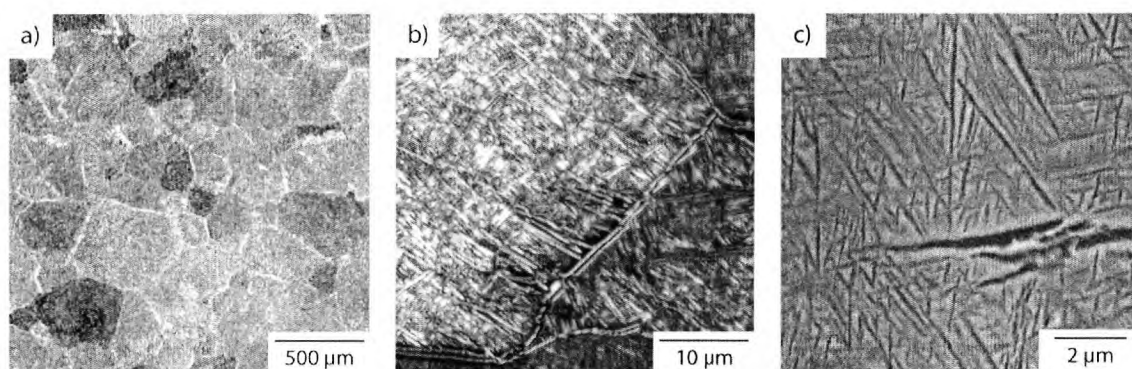


Figure 33: Light micrographs of the Ti-5-5-5-3 following β forging and slow cooling, showing a) parent β grain structure, b) grain boundary α phase from which laths have nucleated and grown, and c) backscattered electron image of the fine Widmanstätten type α microstructure within the prior β grains (electron).

The macrostructure and microstructure developed by the super transus forging to a strain of 0.56 can be seen in Figure 33. The macrostructure consists of predominantly equiaxed prior β grains which have an average grain size $\sim 100 \mu\text{m}$ (Figure 33a). The boundaries of each parent β grain are delineated by a continuous layer of α phase from which some laths have originated and grown towards the grain centre (Figure 33b). Within each parent β grain a fine Widmanstätten α morphology has evolved, with a high density of α laths (Figure 33c). Individual laths range in length from sub micron to $\sim 10 \mu\text{m}$, the largest of which have thicknesses of $\sim 0.1 \mu\text{m}$.

4.4.1 Witness microstructures

The effect of the thermal conditions on the microstructure of β condition Ti-5-5-5-3 following heating and the 120 second pre-compression soak period for the three

temperatures investigated are shown in Figure 34. Also shown in Figure 34 is the effect of time at each temperature for the same period as that required to forge a sample at the slowest strain rate of 10^{-3} s^{-1} .

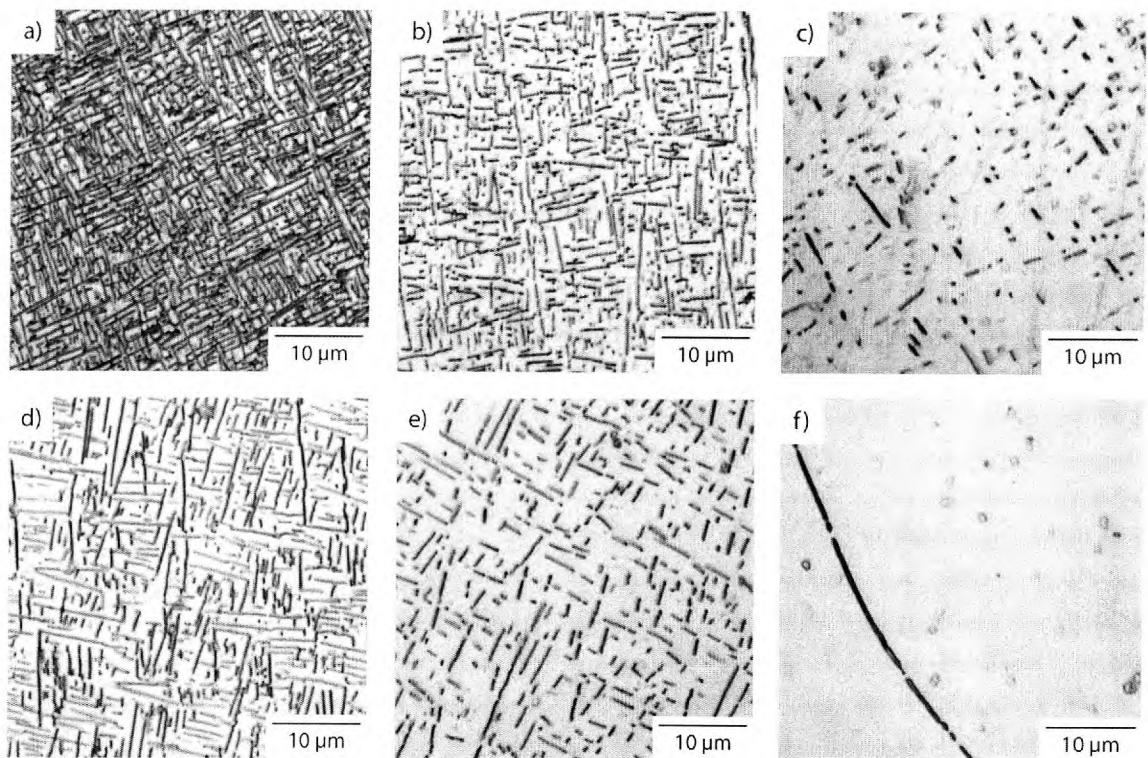


Figure 34: Light micrographs of β condition witness structures following heating (at a rate of $25^{\circ}\text{Cmin}^{-1}$) to temperatures of a) 785°C , b) 810°C , c) 835°C for a 120 second hold period and d) 785°C , e) 810°C , f) 835°C for a 920 second hold period.

Following heating to 785°C and holding for 120 seconds (Figure 34a), a high density of fine α laths remains but individual laths have coarsened such that the average precipitate length and thickness has increased when compared to the initial condition. Subsequent time spent at 785°C decreases the lath density and further coarsens the microstructure, increasing the lath thickness (Figure 34d). Heating to 810°C and holding for 120 seconds (Figure 34b) reduces the α lath density when compared to the initial condition. The volume fraction of α laths further decreases when held for 920 seconds, with smaller laths appearing to lower their aspect ratio. This process is probably a mechanism of surface interface energy reduction whilst the equilibrium volume fraction is achieved by α phase dissolution. Heating to 835°C has a dramatic effect on the

microstructure of β condition Ti-5-5-5-3. The α volume fraction is much lower than that of the initial condition and little evidence of the α lath morphology remains (Figure 34c). The α phase within the prior β grains exists as small globular particles within a β matrix. Continued aging at 835°C (Figure 34f) further reduces both the α phase volume fraction and number of α particles within the prior β grains, with evidence of disruption of the continuous grain boundary layer.

4.4.2 Flow behaviour

The flow behaviour of β condition Ti-5-5-5-3 during isothermally forging at subtransus temperatures can be seen in Figure 35. The flow behaviour when working at 785°C and 810°C is typified by elastic deformation to yield at a low strain, followed by a period of pronounced softening before achieving steady state flow. A steady state flow stress is achieved by a strain of approximately 0.35. Higher peak stresses are observed at lower forging temperatures and the magnitude of flow softening increases at lower forging temperatures and at higher strain rates. Forging β condition Ti-5-5-5-3 at 835°C exhibits little to no flow softening, with the steady state flow being achieved almost directly after yielding.

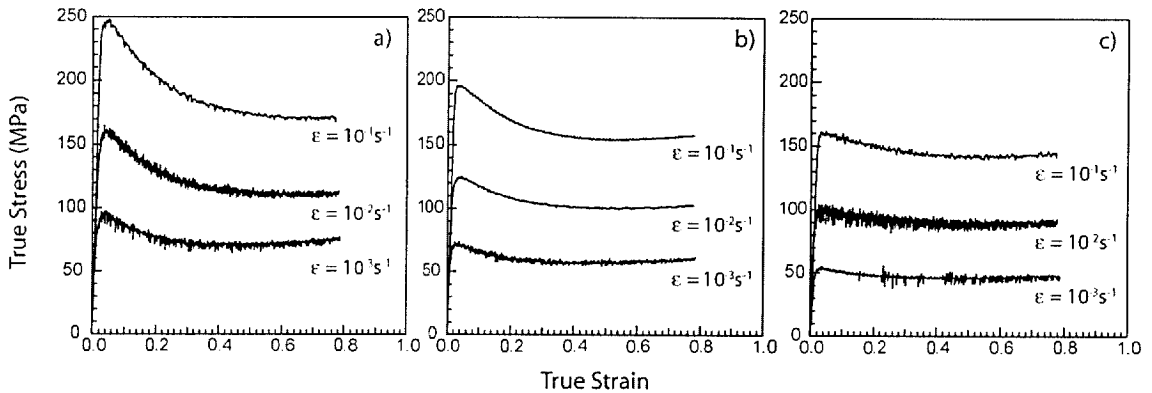


Figure 35: Flow curves of β condition Ti-5-5-5-3 forged at strain rates of 10^{-1} , 10^{-2} and 10^{-3} s^{-1} at temperatures of a) 785°C, b) 810°C and c) 835°C.

There is an obvious difference in flow behaviour of β condition material and that of both the $\alpha+\beta$ and retained β conditions, as only the β condition material shows a significant level of flow softening at 785°C (~30 - 70MPa) and 810°C (~20 - 40 MPa).

The behaviour of β condition Ti-5-5-5-3 at 835°C more closely matches the behaviour of $\alpha+\beta$ and retained β condition material at the same temperature. This observation can be rationalised by the microstructural features observed at the forging temperatures, apparent in the 120 second witness samples (Figure 34). The largely reduced volume fraction and aspect ratio of the α phase present following heating to 835°C produces a high temperature microstructure which resembles that of the $\alpha+\beta$ condition material, as opposed to the fine α lath dominated microstructures developed at 785°C and 810°C.

4.4.3 Effect of Strain Rate

The microstructural development during subtransus isothermal forging of β condition Ti-5-5-5-3 can be seen in Figure 36. Forging at 835°C produces a microstructure consisting of well distributed globular α particles within a β matrix. Changing strain rate has little effect on the evolved microstructure within the grains but does alter the extent to which the continuous grain boundary α layer remains. When forging at higher strain rates the grain boundary α layer can be seen to become fragmented (Figure 36a), however, minimal spheroidisation of these fragments has occurred. As the strain rate decreases the fragments become progressively less correlated and more rounded, resulting in little evidence of their former presence when forged at a strain rate of 10^{-3}s^{-1} . This is in contrast to the almost perfect continuous layer observed in the 920 second witness sample (Figure 34f).

Flow softening is observed when forging at 810°C and direct evidence of microstructural effects can be seen (Figure 36d-f). When forged at a strain rate of 10^{-1}s^{-1} a mixed microstructure of lath and globular α precipitates is developed (Figure 36d). At lower strain rates (for example 10^{-2}s^{-1}) the microstructure becomes more globular during forging (Figure 36e), with less evidence of the initial lath structures. Further reduction in strain rate leads to the formation of a well distributed globular α microstructure within a β matrix (Figure 36f). The globular α precipitates have visibly coarsened, becoming $\sim 2\mu\text{m}$ in diameter, which is significantly larger than the small fragments seen at the higher strain rates. This microstructure is extremely different to that developed by thermal treatment alone (Figure 34e), thus suggesting that the deformation has caused the transition from acicular to spheroidal α particles.

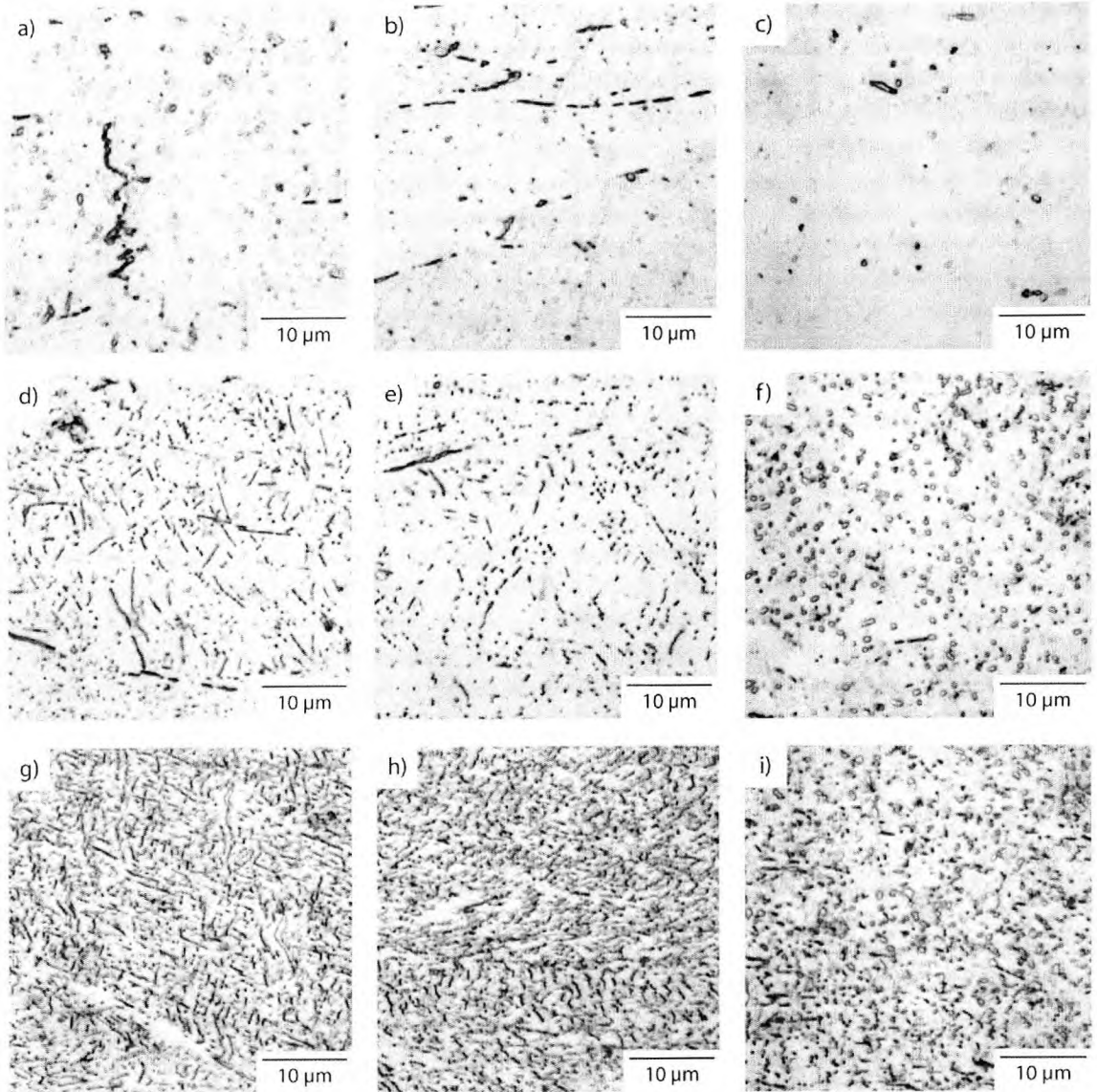


Figure 36: Light micrographs of the microstructural development following subtransus forging of β condition Ti-5-5-5-3 to a final true strain of 0.8. Forging conducted at a temperature of 835°C and a strain rate of a) 10^{-1}s^{-1} , b) 10^{-2}s^{-1} , c) 10^{-3}s^{-1} . Forging conducted at a temperature of 810°C and a strain rate of d) 10^{-1}s^{-1} , e) 10^{-2}s^{-1} , f) 10^{-3}s^{-1} . Forging conducted at a temperature of 785°C and a strain rate of g) 10^{-1}s^{-1} , h) 10^{-2}s^{-1} , i) 10^{-3}s^{-1} . The forging direction runs vertically with the page.

Forging β forged condition material at 785°C exhibits a similar microstructural evolution pattern to that of 810°C. The microstructure observed following compression at the fastest strain rate shows direct fragmentation of α laths lying parallel to the forging direction (Figure 36g). Individual laths appear to have been fragmented in multiple locations but remain largely correlated in their original positions. Decreasing the strain rate leads to increased spheroidisation, with evidence of lath fragmentation still visible but a greater displacement between individual pieces (Figure 36h). At the slowest strain rate a significant level of spheroidisation has occurred with some globular α particles having a diameter of $\sim 2\mu\text{m}$.

The source of the flow softening behaviour observed in β condition Ti-5-5-5-3 when isothermally forged at 785°C and 810°C would appear to be related to the high temperature microstructure. As previously discussed the lack of flow softening when forging β condition material at 835°C and the difference observed in the witness microstructures would seem to indicate that the α morphology and volume fraction influence the flow behaviour. Flow softening effects due to adiabatic heating which increases the sample temperature significantly during compression, thereby lowering the flow stress can be discounted following work with Ti-10-2-3 [5]. Data obtained with an identical experimental setup showed that internal sample temperature rose no more 4°C during testing at a strain rate of 10^{-1}s^{-1} , thus the observed level of softening is not due to adiabatic heating. Similar flow softening behaviour has been observed in β condition Ti-10-2-3 [5, 131]. The authors suggested that the softening is caused by the break up of high aspect ratio α laths which were acting as barriers to dislocation movement. The fragmented laths are subsequently spheroidised and delineated by the penetration of the β phase. The fragmentation of α laths seen in Figure 36 would appear to support this mechanism and furthermore the lack of flow softening when forging at 835°C, where no α precipitates exist, further strengthens this hypothesis.

4.5 Flow Softening in Ti-5-5-5-3

The three initial microstructural conditions ($\alpha+\beta$, retained β and β forged) exhibit a similar steady state flow stress, achieved by a strain of approximately 0.35, which would suggest a common deformation mechanism is reached. The activation energy for the restoration process at steady state is found to be similar to that of self diffusion in β titanium. Therefore the dominating deformation mechanism in this regime is thought to be dynamic recovery of the β phase. Thus the flow softening behaviour exhibited by Ti-5-5-5-3 in both the retained β condition and in the β forged condition appears to be linked to the fragmentation of either acicular α particles or grain boundary α layers. Therefore some critical microstructural parameters relating to the α phase must exist which will describe the extent of flow softening that will occur.

The morphology of the α phase is obviously of key importance to flow behaviour, as small globular particles have been shown to act as witnesses to the deformation of the β phase, whilst high aspect ratio α precipitates are thought to act as barriers. The size of the α precipitates is also a relevant factor, as shown when forging the retained β condition material. In that case it was assumed that whilst the intragranular α precipitates were acicular their sub micron size (Figure 29) was such that they would not act as a significant barrier to dislocation movement. Instead the observed flow softening was attributed to the fragmentation of grain boundary α which had a length greater than 10 μm . Therefore a critical length parameter for flow softening can be proposed, against which the maximum axis length of any given α precipitate can be compared. The $\alpha+\beta$ condition material, which showed little to no softening effect, had high temperature microstructures with an average globular α particle axis of $\sim 4 \mu\text{m}$ (Figure 22). Thus, it is proposed that α precipitates with an axis length greater than 4 μm will be able to act as dislocation barriers and hence cause flow softening.

The α phase precipitates in the high temperature β witness microstructures were characterised using image analysis software. Following characterisation the α precipitate data was filtered, such that only those particles which had an axis greater than 4 μm (referred to as α^*) were included in any subsequent data manipulation. When forging at a strain rate of 10^{-1}s^{-1} the microstructure can be assumed to be identical to that

of the witness structure as there is insufficient time for diffusional changes during the upset forging process. Therefore the extent of flow softening ($\Delta\sigma$ - defined as the difference between the peak stress and the steady state flow stress) exhibited by the β condition material when forged at a strain rate 10^{-1}s^{-1} was compared to the volume fraction of α precipitates hypothesised to act as dislocation barriers (α^*) for each of the three investigated testing temperatures. A linear relationship is found to exist between the two parameters (Figure 37a), suggesting that the volume fraction α^* particles is directly related to the extent of flow softening observed when forging at a strain rate of 10^{-1}s^{-1} . The extent of softening observed when there is no α^* present would represent the softening of the grain boundary layer. The parent β grain size in each of the three forging temperatures investigated (Table 3) is essentially identical following heating and the pre-compression soak period. A continuous layer of the α phase was observed along the grain boundary of each sample and therefore it is assumed that the softening contribution of this layer is constant across all temperatures.

Table 3: Characteristic microstructural measurements obtained from β condition Ti-5-5-5-3 witness samples in the condition immediately before forging.

Temperature	(°C)	785	810	835
$\Delta\sigma$	(MPa)	80	40	15
Volume fraction α^*	(%)	14.6	5.2	0
β grain size	(μm^2)	18125	20234	19726
Average β grain diameter	(μm)	76	80	79

It is anticipated that a change in β grain size would result in a related change to the observed flow softening contribution, providing that a continuous α boundary layer is present. The witness microstructures representative of the retained β condition (Figure 29) feature both small acicular intragranular α particles and discontinuous grain boundary α . As previously hypothesised the intragranular α is very fine, falling outside the α^* selection criteria and therefore does not contribute to the observed softening. There are indications in the 785°C and 810°C witness microstructures (Figure 29 a and b) that the small β grain size observed in the initial microstructure (Figure 28) appears not to have grown significantly during heating and the pre-compression soak period.

However this supposition is based upon the approximation of grain size from the boundaries which are delineated by an α layer. As the grain size in the retained β witness structures cannot be accurately defined the influence of each of the individual discontinuous α segments was evaluated as if they were independent intragranular particles (Table 4). For comparison the extent of softening for the β condition microstructure was corrected to only incorporate an intragranular effect and plotted on the same graph (Figure 37b). A linear correlation is found and from this relationship the influence of the volume fraction of α^* particles on the extent of flow softening is found to be approximately $4.5 \text{ MPa.\%}^{-1}$.

Table 4: Analysis of discontinuous grain boundary α against the α^* criteria for retained β condition Ti-5-5-5-3.

Temperature	(°C)	785	810	835
$\Delta\sigma$	(MPa)	30	5	0
Volume fraction α^*	(%)	5.6	2.8	0

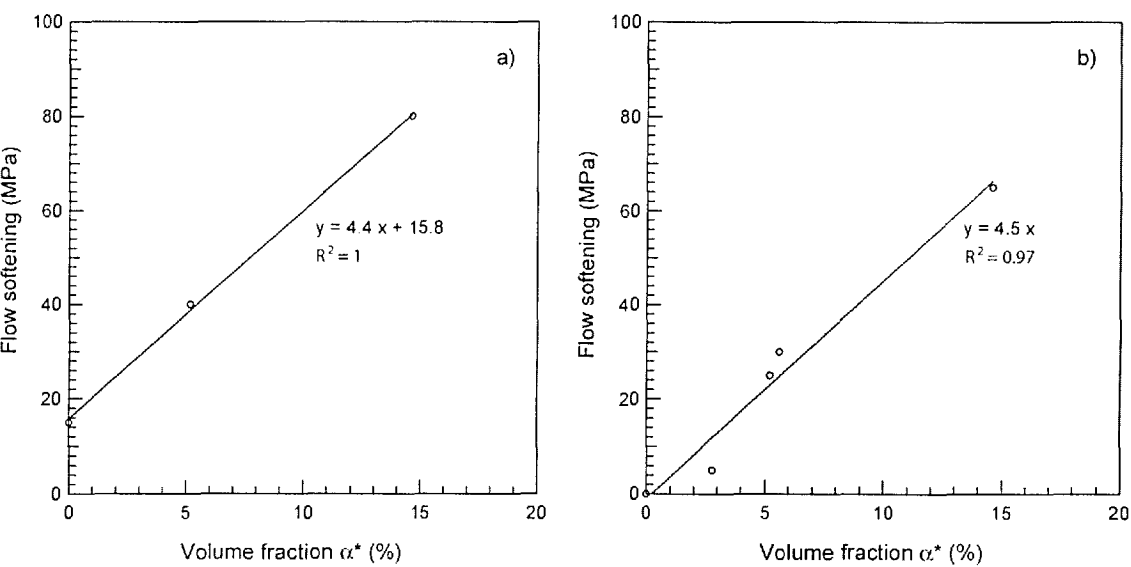


Figure 37: Plots showing a) the extent of flow softening as a function of α^* volume fraction for β condition Ti-5-5-5-3 forged at 10^{-1} s^{-1} , and b) the extent of softening as a function of the volume fraction of the intragranular α^* particles in both β forged and retained β condition Ti-5-5-5-3.

4.6 Summary of Results

The flow behaviour, dominating deformation mechanisms and microstructural evolution during subtransus forging of Ti-5-5-5-3 have been evaluated over a range of temperatures, strain rates and initial microstructural conditions.

$\alpha+\beta$ condition Ti-5-5-5-3, which has an initial microstructure consisting of small globular α particles within a β matrix, is largely insensitive to forming variables. The flow behaviour of material in this condition can be described by elastic deformation to yield at low strains (< 0.1), followed by a steady state flow regime. Using a regression study and a creep based analogy, an activation energy for steady state flow of 183 kJmol^{-1} is derived. This value is similar to that of self diffusion in β titanium [78], and thus it is suggested that the dominating deformation mechanism during forging of $\alpha+\beta$ condition Ti-5-5-5-3 is dynamic recovery. The deformation appears to have little effect on the resulting microstructure, increasing strain rate results solely in an increased flow stress. The chosen forging temperature dictates the volume fraction and size of the primary globular α phase. This observed insensitivity to forging temperatures will be significant in obtaining homogenous microstructures in large forged components. Evidence is found which suggests that the majority of the deformation is absorbed by the β phase matrix with the hard α particles merely acting as a witness to the flow of the β phase.

Forging retained β condition Ti-5-5-5-3, which has a microstructure consisting entirely of metastable β grains, produces almost identical flow behaviour to that of $\alpha+\beta$ condition material. During pre compression heating, grain boundary α was observed to form and it is hypothesised that the fragmentation of these layers is responsible for the slightly higher peak stresses observed in the retained β condition when compared to the $\alpha+\beta$ condition. Forging at lower temperatures and strain rates gives rise to a very evenly distributed microstructure of small globular α precipitates. Forging at increased strain rates results in less rounded fragmented grain boundary α particles in closer proximity to each other, presumably as less time for diffusional processes exists. Increasing temperature decreases the volume fraction of α precipitates in the microstructure, such that at 835°C the material appears to remain fully β . The very similar flow behaviour of

retained β condition and $\alpha+\beta$ condition material would suggest that small globular α particles have no effect on the flow behaviour of Ti-5-5-5-3, with the deformation being characteristic of the β phase. It is also assumed that since the steady state flow is the same between the retained β and the $\alpha+\beta$ condition material the dominating mechanism of dynamic recovery is also the same.

Forging β condition Ti-5-5-5-3, which has a microstructure consisting of a high density of Widmanstätten α plates, produces a very different characteristic flow curve. The behaviour can be described by elastic deformation to yield at low strains (< 0.1), followed by a softening regime before reaching steady state flow at a strain of ~ 0.35 . Following pre-compression heating it was observed that the lath morphology α precipitates had almost entirely dissolved at a temperature of 835°C , whilst still remaining at 785°C and 810°C . Subsequent forging of the material at 835°C did not exhibit flow softening at any strain rate, leading to the conclusion that flow softening is governed by α phase morphology. Deformed microstructures obtained following forging at 785°C and 810°C show evidence of α lath fragmentation and spheroidisation. Increasing the strain rate results in a less well distributed microstructure and smaller α particles, whilst increasing temperature decreases the α volume fraction.

The mechanism of flow softening in β condition Ti-5-5-5-3 is thought to be identical to that proposed for β condition Ti-10-2-3[131]. High aspect ratio α laths act as barriers to dislocation movement resulting in pile up at the α/β interface. The local strain eventually fragments the lath and allows β phase penetration, creating lower energy dislocation paths and hence reducing the global flow stress. Microstructural evidence of α lath fragmentation is found in β condition Ti-5-5-5-3 and the proposed mechanism appears to be consistent with the other microstructural observations. A simple empirical relationship has been developed which relates the observed extent of flow softening to the high temperature microstructure prior to forging. It is anticipated that other factors, such as the volume fraction of grain boundary α layers, will also influence the extent of flow softening. However, insufficient experimental data exists in the current study to allow a meaningful evaluation of their contribution.

5 The influence of the ω phase on microstructural development

The mechanical properties of β and near β titanium alloys are largely governed by the volume fraction, morphology, distribution and size of the hexagonal α phase precipitates. Great interest lies in understanding and controlling the microstructural development, such that alloy properties can be tailored for specific applications.

A particular area of interest is the influence of the metastable ω phase during microstructural development, since ω precipitates have been shown to act as potent α lath nucleation sites [22]. One of the proposed origins for the high strength of the β alloys is the development of fine scale (~ 200 nm) acicular α particles during aging. It is thought that these acicular structures nucleate from ω precipitates, formed both athermally and isothermally [22, 67]. Whilst several studies have investigated the $\omega \rightarrow \alpha$ transformation [71, 72], the precise characteristics are yet to be determined. Therefore, before any level of microstructural tailoring can be developed, a better understanding of the transformations that occur, the relevant timescales and the dominating mechanisms needs to be gained. This chapter presents and discusses results obtained whilst investigating the $\beta \rightarrow \omega \rightarrow \alpha$ transformations in Ti-5-5-5-3.

In-situ X-ray diffraction using synchrotron radiation has allowed the time scales involved in the relevant phase transformations to be evaluated during rapid cooling from the β phase and subsequent aging in the $\alpha+\beta$ field. A comparative ex-situ study using transmission electron microscopy, in conjunction with the laboratory X-ray diffraction, has enabled the characterisation of the evolving microstructure of Ti-5-5-5-3 and has corroborated the experimental findings of the in-situ study.

The microstructural evolution of Ti-5-5-5-3 was also investigated when subjected to direct aging (or step quenching) at 400°C having been solution heat treated in the β phase field. Unique chevron like α precipitates were found to develop in aligned strings during aging, having nucleated intragranularly. Within the chevron precipitates a latticework structure was found, consisting of fine scale acicular α particles.

5.1 *Experimental Methods*

A cylindrical sample (67 mm in diameter by 79 mm in height) of Ti-5-5-5-3, was solution heat treated at 980°C for 60 minutes. The hot billet was transferred to an ENEFCO 5 MN press and extruded into a 20 mm round bar form using a reduction ratio of 10:1. All samples used this chapter were machined from this bar stock.

5.1.1 In-situ Synchrotron Diffraction

The operation of a synchrotron is based on the production of radiation by bending the path of electrons, moving at speeds approaching the speed of light, by the application of a magnetic field. The energy of the radiation is far greater than that of laboratory based X-ray source, and the beam divergence is much smaller. This leads to a higher spectral brightness when compared to conventional X-ray diffraction, a higher signal to noise ratio and a narrower half height full width for a given diffraction peak [132].

In-situ synchrotron experiments were carried out on ID15b of the European Synchrotron Radiation Facility in Grenoble, France. The narrow monochromated beam (~500 by 500 μm) was focused on the specimen and atomic planes fulfilling the Bragg criterion produced characteristic Debye-Scherrer cones which are collected as concentric diffraction rings. Diffraction patterns, were recorded using a two dimensional Pixium 4700 area detector, located ~647 mm from the sample. Calibration of instrument parameters, such as the position of the beam centre and tilt angle between the sample and the detector, was achieved through the analysis of diffraction patterns from a standard powder sample (either LaB_6 or Al). The calibration file is then applied to experimental diffraction patterns prior to analysis using the ESRF developed software Fit 2D [133, 134].

Small bar samples, 1mm x 1.75mm x 36mm, were held in the beam path by an Instron Electro-Thermal Mechanical Testing machine (ETMT). Samples were heated into the β phase field (950°C) inside an argon filled enclosure using resistance heating. Temperature was monitored and controlled via an R type thermocouple, spot welded to the sample surface directly above the beam position. Samples were homogenised in the β phase field before being cooled by conduction through the ETMT grips to room

temperature. Rapid cooling rates of $\sim 1800^{\circ}\text{C min}^{-1}$ were obtained during the study. Following cooling samples were heated to 570°C , which is within the industrial α aging range for the alloy [119], at a rate of $\sim 950^{\circ}\text{C min}^{-1}$. Samples were aged for a total of 15 minutes, allowing the observation of initial α phase nucleation and growth. Full ring diffraction data was obtained every 2 seconds during cooling and every 5 seconds during heating to, and aging at, 570°C .

Whilst in-situ experiments provide an excellent opportunity to witness events in real time, the ability to utilise other methods of characterisation, such as SEM or TEM when employing interrupted testing is lost. Therefore an ex-situ study was also employed, designed to mirror the procedures from the in-situ experiments as closely as possible, to enable further interrupted testing and investigation of the transformation behaviour.

5.1.2 Ex-situ Aging

A 20 mm diameter by 100 mm height bar section was taken from the extruded material and solution heat treated at 900°C for 30 minutes followed by a water quench. Small disc samples, 1.5 mm thick, were suspended in the middle of a Severn Furnaces 1000°C infrared furnace driven by a Eurotherm 902P controller. Sample temperature was controlled using a K type thermocouple spot welded to the sample surface, relaying back to the controller unit. Heating to the aging temperature of 570°C was carried out at $300^{\circ}\text{Cmin}^{-1}$, again sufficient to bypass the isothermal ω forming region, before being aged for 1, 3, 5, 10, 15 and 30 minutes. Samples were immediately water quenched following aging to preserve the developed microstructure. Sample characterisation was conducted by conventional laboratory based X-ray diffraction, transmission electron microscopy and Vickers hardness indentation.

5.1.3 Direct Aging

Thin disc samples, 1.5 mm high with a diameter of 20 mm, were machined from the extruded bar. Discs were solution heat treated in a Severn Furnaces 1000°C infra-red furnace for 30 minutes at 900°C . Following solution treatment the samples were subjected to 'direct aging' by rapid air cooling to a target aging temperature of 400°C .

Samples were aged for times of 0, 1, 4 and 8 hours before final air cooling to room temperature. Throughout the thermal treatments the sample temperature was controlled using a K type thermocouple spot welded to the disc surface relayed into the Eurotherm 902P controller unit sample temperature. Characterisation of the resulting microstructures was conducted using laboratory based X-ray diffraction, scanning electron microscopy, transmission electron microscopy and Vickers hardness indentation.

5.2 *In-Situ study of phase transformation in Ti-5-5-5-3*

In-situ X-ray synchrotron diffraction data examining the phase transformations occurring in Ti-5-5-5-3 during rapid cooling from the β phase field and subsequent aging at 570°C (an industrially employed aging temperature for Ti-5-5-5-3) from a 10° azimuthal ring section is summarised in Figure 38. The figure shows the experimental time-temperature scheme and diffraction patterns corresponding to four selected experimental points

The diffraction pattern corresponding to the β phase achieved following a 600 s solution heat treatment at 950°C is shown in Figure 38a. After rapid cooling to room temperature secondary peaks are observed in the diffraction pattern, as shoulders to the β phase reflections (Figure 38b). The appearance of the secondary shoulder peaks during rapid cooling in Figure 38b would appear be consistent with the formation of the athermal ω phase following quenching from the β phase field, as discussed in section 3.1.3.

The accuracy and homogeneity of the temperature achieved by resistive heating in the analysed volume, can be established by comparing the variation of the cubic lattice parameter with values of thermal expansion from the literature. At room temperature the β phase lattice parameter is found to be 3.22Å and at 950°C is 3.25Å, giving a change of ~1%, which agrees with the thermal expansion data presented by Fanning [119]. A simple calculation of the steady state temperature gradient within the sample, using a parabolic function, indicates that the central 2 mm are within 3°C of the measured temperature. The peak widths of the β phase observed in Figure 38a are similar to those of the aluminium calibration standard, therefore suggesting that within the analysed volume the sample is homogeneous. The use of a 10° azimuthal ring section discounts diffraction signals caused by a Poisson's ratio type stress gradient, which could arise when integrating the full diffraction ring [135]. Thus it can be concluded that the asymmetry of the peak must correspond to the development of a new phase within the material.

Heating this structure to the aging temperature of 570°C at a rate of $\sim 950^{\circ}\text{Cmin}^{-1}$ resulted in a decrease in the intensity of the shoulder peaks (Figure 38c). Rapid heating was employed to avoid the potential formation of isothermal ω . Recent work [136] investigating various β alloys found that the rate at which the material was heated to final aging temperature influenced the size and distribution of the α precipitates. The authors hypothesised that at slow heating rates ($\sim 0.25^{\circ}\text{Cmin}^{-1}$), sufficient time is spent in the $\beta+\omega$ regime to form isothermal ω particles, which then acted as α phase nucleation sites. In the current study aging the material for 900 seconds at 570°C, resulted in the loss of the shoulder peaks and the appearance of new peaks corresponding to the hexagonal α phase (Figure 38d).

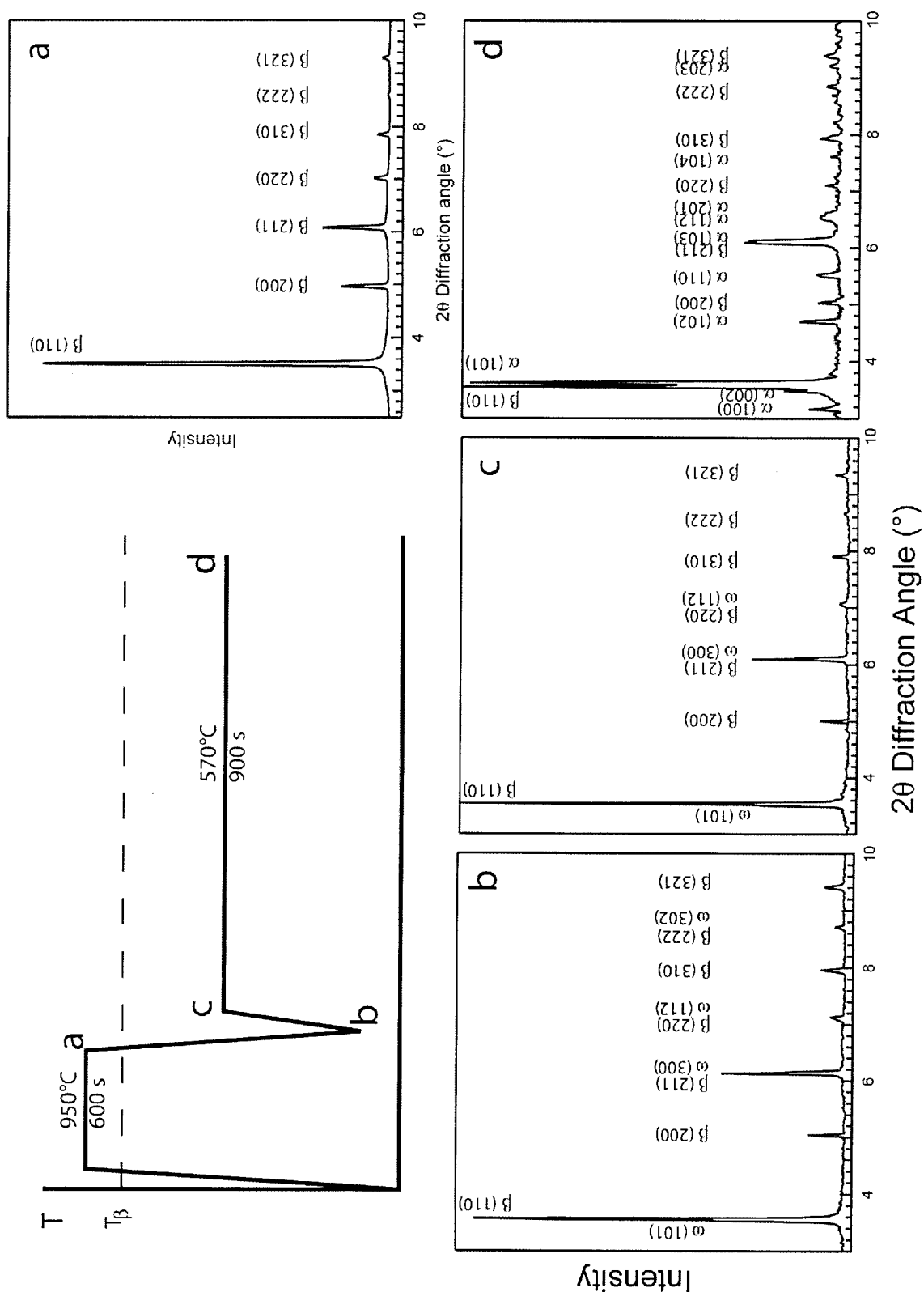


Figure 38: Schematic time-temperature plot of the quench and aging performed during the synchrotron X-ray diffraction experiment and corresponding diffraction data obtained at selected points in the thermal treatment sequence.

5.3 Rapid cooling of Ti-5-5-5-3

The formation of secondary shoulder peaks around the β matrix reflections during rapid cooling is further examined in Figure 39. The figure shows synchrotron diffraction data at two second intervals during rapid cooling from the β phase field around the β (110) peak. At 950°C the material is ~100°C above its β transus and a single distinct peak corresponding to the (110) β was observed. Soon after commencing rapid cooling a shoulder to the left hand side of the main (110) β peak appeared which becomes a distinct peak between 624°C and 470°C. This observation is consistent with the formation of embryonic ω as discussed in the section 2.2.1. High in the β phase field (950°C) uncorrelated linear (111) β defects exist but diffract independently and therefore a single distinct β peak is observed. During rapid cooling the defects become more correlated, forming ω embryos which diffract to produce the peak shoulder seen at 885°C and 624°C. It is inferred that the ω start line is crossed during continued rapid cooling, by the appearance of a second peak in the place of the shoulders at 470°C. The presence of ω particles at this temperature is consistent with reported ω start temperatures (~500°C) for similar alloy systems [22].

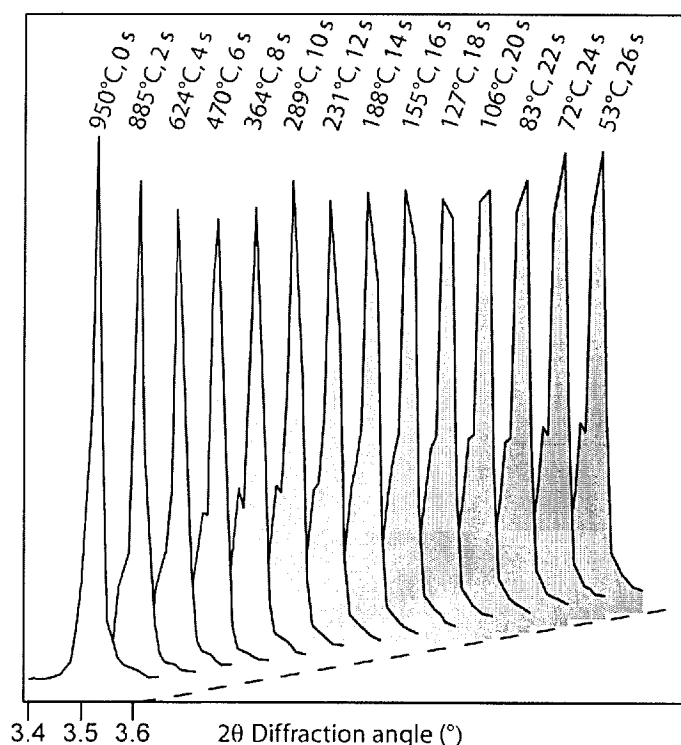


Figure 39: In situ X-ray diffraction data obtained during rapid cooling of Ti-5-5-5-3 from 950°C to ~50°C around the (110) β peak.

Several studies have considered the decomposition of the β phase in the binary systems of Ti-V, Ti-Mo and Ti-Cr during cooling and low temperature aging [37-39, 137-139]. In each case the authors have reported a miscibility gap in the phase diagram which could lead to phase separation and the precipitation of bcc titanium rich regions, known as β' . As the constituent β stabilising alloying elements in Ti-5-5-5-3 exhibit β phase separation, it is plausible that Ti-5-5-5-3 would also exhibit the same behaviour.

Recent research concerning TIMETAL-5553 [140] suggests that β phase separation does occur in this system, including over a very short length scale following rapid cooling. Therefore the shoulder peak observed in Figure 39 could be a result of β' rather than of the ω phase. However, if β phase separation occurs during the rapid cooling of Ti-5-5-5-3 then each diffraction peak would broaden and split as cooling proceeds. It would seem unlikely that the onset of β phase separation would occur whilst still in the β phase field, as seen in Figure 39. Additionally each split peak would consist of two maxima, corresponding to two similar bcc phases with different lattice parameters caused by compositional variance. As the splitting of each high temperature β peak is directly related to the difference in lattice parameter between β and β' , the observed splitting would occur in the same direction for every peak. Figure 40 shows the development around the $(211)\beta$ peak during rapid cooling. A broadening effect and the development of a shoulder at higher angles of 2θ than the $(211)\beta$ peak is clearly visible. The secondary peak developed around the $(110)\beta$ peak, seen in Figure 39, occurred at a lower 2θ angle and therefore the observed behaviour cannot be explained by a β phase separation argument.

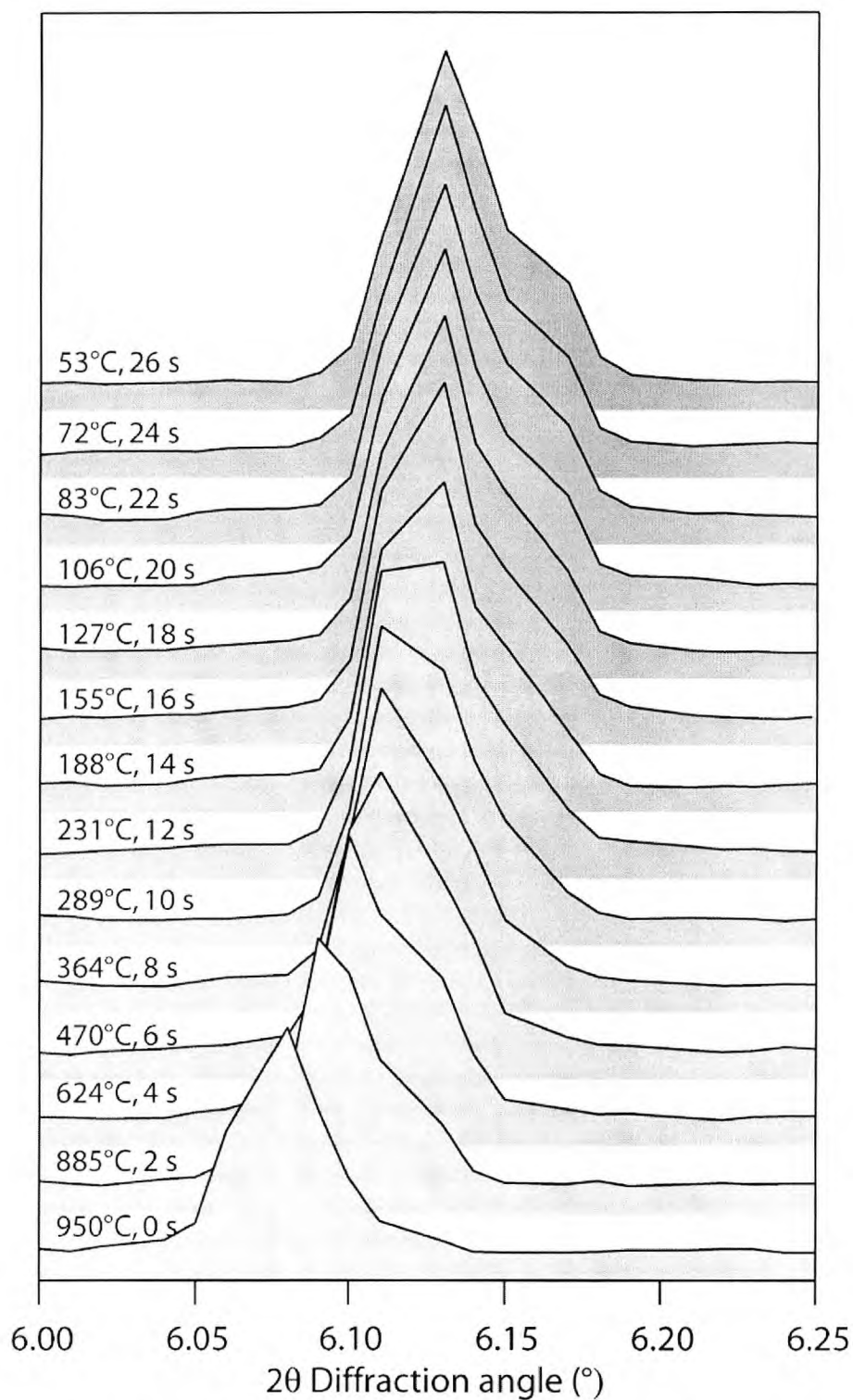


Figure 40: In situ X-ray diffraction data obtained during rapid cooling of Ti-5-5-5-3 from 950°C to ~50°C around the (211) β peak.

Further evidence of the ω phase following rapid cooling can be found from ex-situ testing. Figure 41a shows an electron diffraction pattern of the $[110]\beta$ zone axis from a sample which has been water quenched from the β phase field. The typical diffuse streaked ‘x’ pattern is present between β reflections upon which intense ω maxima can be identified. There are four intensity maxima corresponding to the $(10\bar{1}1)\omega$, $(\bar{1}010)\omega$ and $(\bar{1}011)\omega$ planes lying on the diffuse streaks, however they do not appear in the commensurate positions of $1/3$ and $2/3$ $[112]\beta$ which corresponds to the ideal ω structure (Figure 41b). Instead the reflections from the two variants, $(10\bar{1}1)\omega / (\bar{1}010)\omega$ and $(\bar{1}011)\omega / (\bar{1}010)\omega$, are shifted towards each other (Figure 41c).

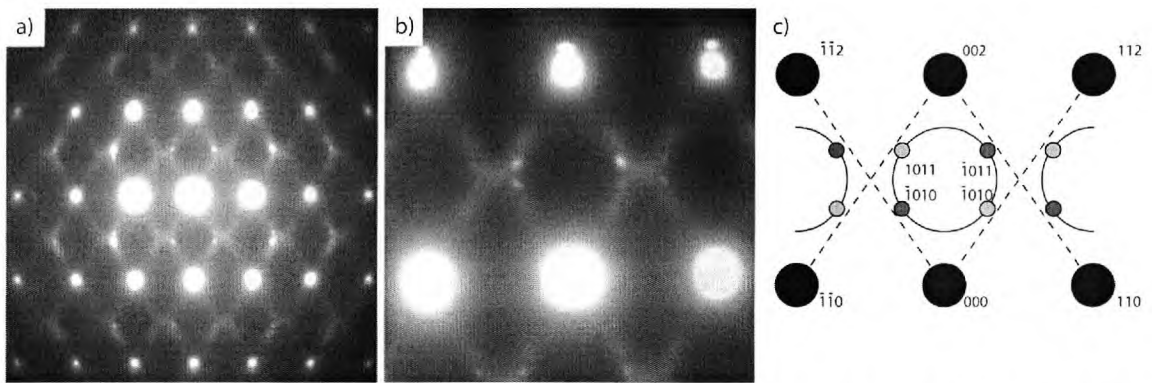


Figure 41: a) Electron diffraction pattern from $[110]\beta$ zone axis (Ti-5-5-5-3 quenched from 900°C , camera length $D = 800\text{mm}$), b) electron diffraction pattern from the $[110]\beta$ zone ($D = 2000\text{ mm}$) and c) schematic representation of the diffraction pattern showing movement of ω reflections towards each other (adapted from [53]).

Similar shifting has been observed in other Ti and Zr alloys [54] and a rationalisation proposed by De Fontaine [53]. An ω particle, when initially forming, can be thought of as a small transformed disc like region, surrounded by a parent β matrix (Figure 42a). The interface between the two structures generates a strain as two bcc planes bend to together to form a single collapsed plane. When close to the ω formation temperature the displacement wave persists for only a few iterations within a given region, and the collapsed $(111)\beta$ planes are only correlated locally. There are four independent ω variants which can form and while the correlation of collapsed planes is only on the local scale it is proposed that the strain fields relating to each of the variants tangle and interact. This interaction effectively couples the variants, with the effect of joining

intersecting linear processes. When two linear processes are combined the resultant effect is a hyperbolic function. In three dimensions, relevant to the four ω variants, the resultant function is spherical. In the context of bcc reciprocal space this theory can be visualised as a sphere bounded by the $\{111\}\beta$ planes (Figure 42b) which correspond to a plane of diffracted intensity for an uncoupled variant. As solute content is increased the variant coupling also increases resulting in the movement of ω $(10\bar{1}1) / (\bar{1}010)$ and $(\bar{1}011) / (\bar{1}010)$ reflections towards each other on the in-sphere shown in Figure 41c.

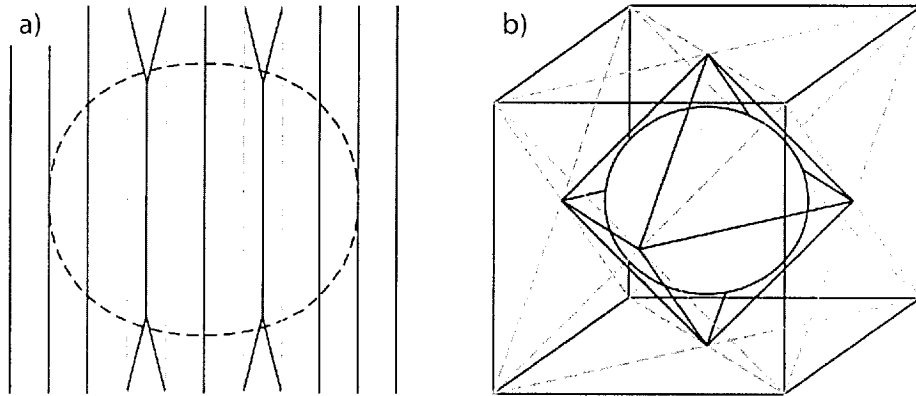


Figure 42: Schematic representation of a) $(111)\beta$ plane collapse to form an ω particle, and b) diffraction intensity insphere for coupled ω variants (both adapted from [53]).

Sinkler and Luzzi [58] also reported a similar shifting of ω reflections whilst investigating Ti-Cr alloys. They suggested that the shifts in position were due to variations to both the chemical ordering and the ratio of collapsed to uncollapsed $(111)\beta$ planes in the local ω structure. By inserting extra pairs of collapsed planes the ω structure can be expanded from the expected $\omega_{(3)}$ to $\omega_{(5)}$ or $\omega_{(7)}$ (see Figure 11), causing reflections to move towards each other along the $[111]\beta$. It was also noted that the stronger β stabilising elements (Fe, Cr and Mo) cause a greater shift in position of the ω reflections than weaker stabilising elements (Nb and V). This was thought to stem from short range chemical ordering which when decreased returned the ω reflections to the expected Bragg positions.

In a Ti-15Mo alloy the diffuse streaking was initially observed, at temperatures close to the ω transformation temperature, to form in a circular pattern and became progressively more linear as the temperature decreased [53]. This was attributed to a

change from short range order to long range order and therefore to a reduction in the coupling effect. In the current study neither the distinct reflections, nor the diffuse streaking, were observed to be rectilinear to the matrix reflections at temperatures far below the ω start temperature. The higher solute content of Ti-5-5-5-3 (~23 at. %) when compared to Ti-15Mo (~8 at. %), would be expected to increase the strain field coupling effect [53]. This would therefore suggest that the ordering in the system remains short range and the ω structures very small, consistent with the weak reflections observed in Figure 41.

From a theoretical standpoint the $(\bar{2}021)\omega$ and $(30\bar{3}0)\omega$ reflections would be obscured by the $(200)\beta$ and $(211)\beta$ reflections respectively and in addition the coupling effects discussed above can make determining the ω lattice parameters difficult. However, distinct reflections can be seen in Figure 41b outside the β matrix reflections. Using Figure 41b a β lattice parameter of 3.21 Å was obtained, which exhibits close correspondence to the 3.22 Å obtained from the major β peak in Figure 39 when fitted using a Voigt function. From the β lattice parameter theoretical lattice parameters for ω can be calculated, $a_\omega = 4.55$ Å and $c_\omega = 2.79$ Å, from well established geometric assumptions, $a_\omega = \sqrt{2}a_\beta$ and $c_\omega = \sqrt{3}/2a_\beta$ [44]. Using the $(\bar{2}021)\omega$ and $(30\bar{3}0)\omega$ reflections from Figure 41b, experimental lattice parameters can be calculated, $a_\omega = 4.37$ Å and $c_\omega = 2.65$ Å, which show good correlation with the theoretical values.

It is interesting to note that the $(10\bar{1}1)\omega$ interplanar spacing derived from the in-situ X-ray diffraction varies when compared to that derived from the ex-situ electron diffraction patterns. The spacing between $(10\bar{1}1)\omega$ planes is larger in the ex-situ study by ~0.3 Å. This variation is thought to be related to the cooling rate and suggests that the local ω structure is not identical in the two studies.

Previous research investigating isothermal ω report electron diffraction patterns with ω reflections at the commensurate positions with no diffuse streaking. From these studies it can be concluded that when ω is isothermally precipitated by low temperature aging there is no variation from the $\omega_{(3)}$ structure. However, when produced via rapid cooling variations to both structure and orientation occur due to the random nature of the local plane collapse, for example the $\omega_{(5)}$ and $\omega_{(7)}$ structures [58].

While a rapid cooling rate was achieved during the in-situ experiments it is anticipated that the water quenching employed in the ex-situ study provided faster cooling. Sinkler and Luzzi [58], show that as the ratio of collapsed to un-collapsed plane pairs within the ω structure increases the distance, in reciprocal space, between (0001) and (0002) planes decreases. Thus the larger $(10\bar{1}1)\omega$ inter planar spacing observed following a faster cooling rate would suggest a larger average ω structural unit size, which would be reduced by slower cooling.

5.4 Influence of athermal ω on α nucleation

The time scales corresponding to α formation during aging can also be evaluated using data around the $(110)\beta$ peak, as both the 100% peaks for ω and α phases occur in this region. Synchrotron diffraction from aging an as-quenched sample of Ti-5-5-5-3 at 570°C data is shown at 30 second intervals in Figure 43. The first observation of the α phase occurs after 120 seconds, manifested as a small perturbation to the right of the main β peak corresponding to the $(10\bar{1}1)\alpha$. Peak intensity increases as the aging proceeds and more α nucleates and grows. After 300 seconds of aging at 570°C appearance of the $(0002)\alpha$ peak was observed to the left of the $(110)\beta$. During the initial stages of aging the $(10\bar{1}1)\omega$ peak, present following the rapid cooling, decreases in intensity, becoming less discrete and finally being indistinguishable after 210 seconds.

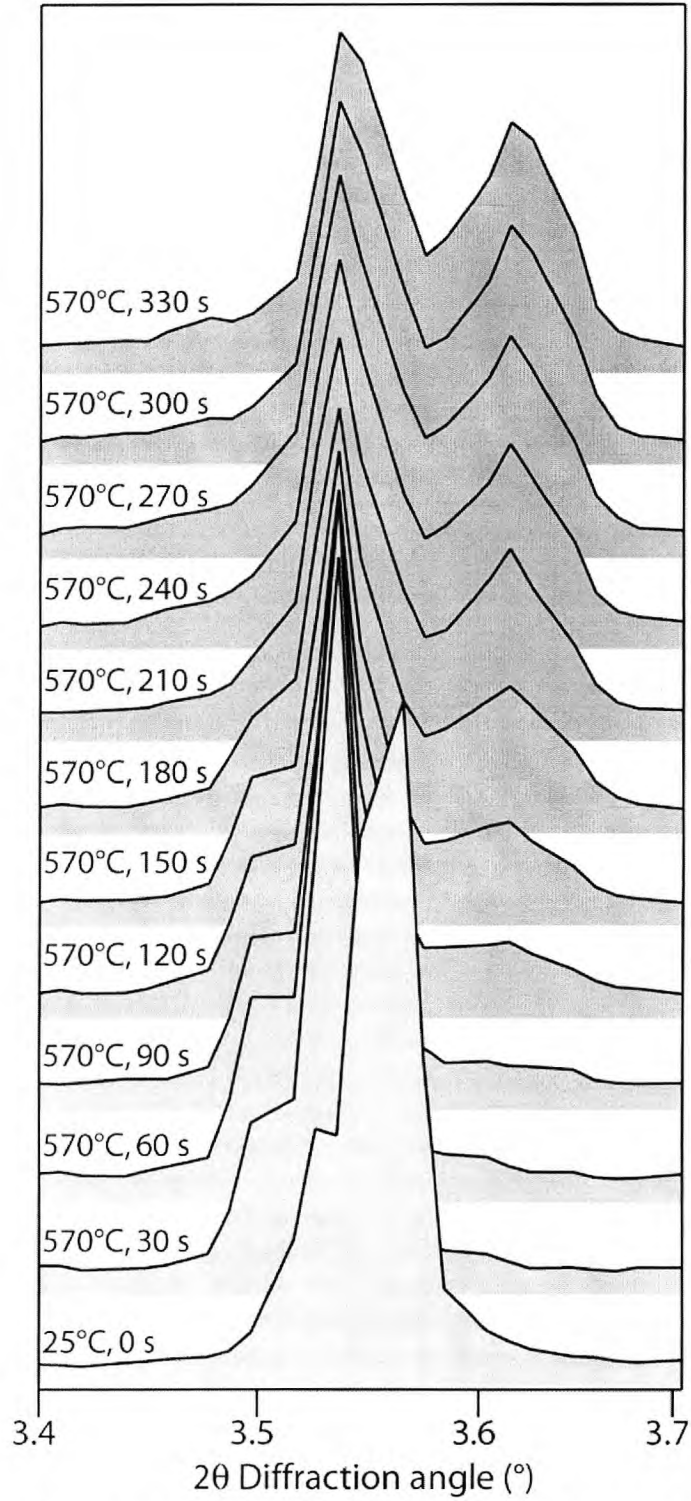


Figure 43: In situ X-ray diffraction data around the (110) β peak, obtained during the aging of Ti-5-5-5-3 at 570°C following rapid cooling from 950°C.

Conventional X-ray diffraction data from the ex-situ study support the findings of the in-situ experiment as can be seen in Figure 44. The greater energy and angular divergence utilised compared to the synchrotron experimentation does not allow the $(10\bar{1}1)\omega$ reflection to be resolved, however, the nucleation of the α phase is detected after 5 minutes aging. The slight time difference, between the two studies before distinct α peaks become apparent can be accounted for by the time spent at the aging temperature due to different heating rates (~ 90 s for in-situ and between 70 and 190 s for ex-situ) and the higher resolution of the synchrotron measurements.

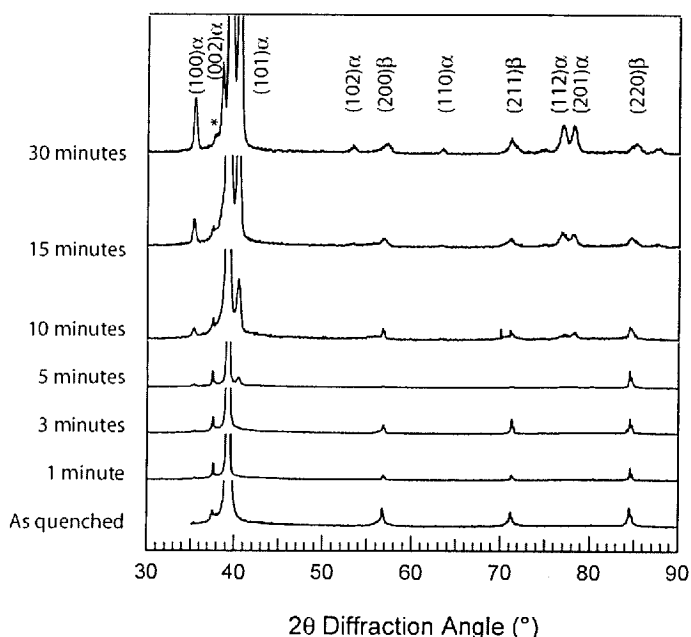


Figure 44: Ex situ laboratory X-ray diffraction results for Ti-5-5-5-3 quenched from 900°C and aged at 570°C for 1, 3, 5, 10, 15 and 30 minutes. The peak labelled (*) is a reflection caused by tungsten contamination related to the $[110]\beta$ peak.

Aging for 5 minutes resulted in the precipitation of small, ~ 150 nm long by 10 nm wide, lath structures in the β matrix (Figure 45b). The intensity of the diffuse streaking in the associated diffraction pattern (Figure 45a) decreased, as did the intensity of the ω reflections when compared to the as-quenched sample. In addition, new regions of increased intensity were observed at the $2/3 \langle 112 \rangle \beta$ position. An example is ringed in Figure 45a. The lath morphology of the structures shown in Figure 45b, combined with both in-situ and ex-situ X-ray diffraction data would suggest that these new reflections correspond to α phase particles.

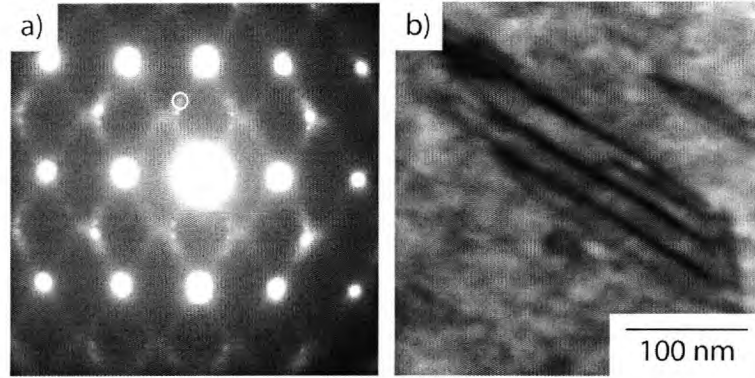


Figure 45: a) Electron diffraction pattern from $[110]\beta$ zone axis (Ti-5-5-5-3 quenched from 900°C and aged for 5 minutes at 570°C , $D = 800$ mm) and b) corresponding bright field image showing several α lath structures.

A similar observation was made by Sakedai in a Ti-20Mo alloy [72]. High resolution TEM studies showed the lattice around a needle like ω particle contained regions of both α and a transitional state within a β matrix. In Sakedai's study the ω reflection was in a commensurate position and therefore overlapped with the developing α reflection. The reflections reported in the current study do not overlap with the α reflections and therefore add support to the in-situ diffraction data which indicated that ω is present when α first begins to form.

The lath-shaped precipitates continue to develop when aged for 10 minutes into particles approximately 300 nm long by 20 nm wide (Figure 46a). The continued growth of the precipitates at the aging temperature employed in this study correlates well with the ex-situ X-ray diffraction data presented in Figure 44 and is consistent with the hypothesis that these precipitates are the α phase. Furthermore, electron diffraction from the $[110]\beta$ zone axis (Figure 46b) shows reflections in addition to those of the matrix and diffuse streaking, which correspond to one of the Burgers variants of the α phase, illustrated by the corresponding key diagram (Figure 46c). The laths shown in Figure 46a are orientated with their major axis aligned to the $[112]\beta$, which is the normal growth direction for the α phase [72]. Why one particular variant would form preferentially during the early stages of development is not clear at this stage and forms the subject of further investigation.

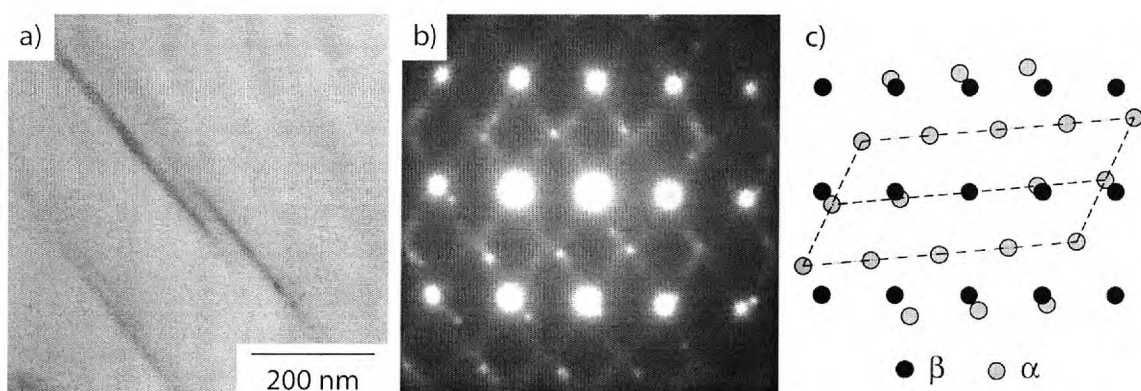


Figure 46: a) Bright field image of α laths observed in a Ti-5-5-5-3 quenched from 900°C and aged for 10 minutes at 570°C, b) corresponding electron diffraction pattern from the $[110]\beta$ zone axis, $D = 800$ mm and c) key diagram for the $[110]\beta$ zone axis diffraction pattern with reflections from a single α variant overlaid.

Following 15 minutes of aging the α laths were observed to increase in size (Figure 47a) to ~ 500 nm in length by 25 nm in width. The bright field image appears to show two orientations of α laths, however a β grain boundary can be identified running across the image, with each orientation contained within separate grains. The corresponding diffraction pattern (Figure 47b) still shows diffuse streaking, however the intensity maxima remain in positions corresponding to a single Burgers variant of α . The continued presence of diffuse streaking suggests that some ω -like structures persist for a significant time period and as observed by Sakedai [72], transitional states between ω and α may well be present.

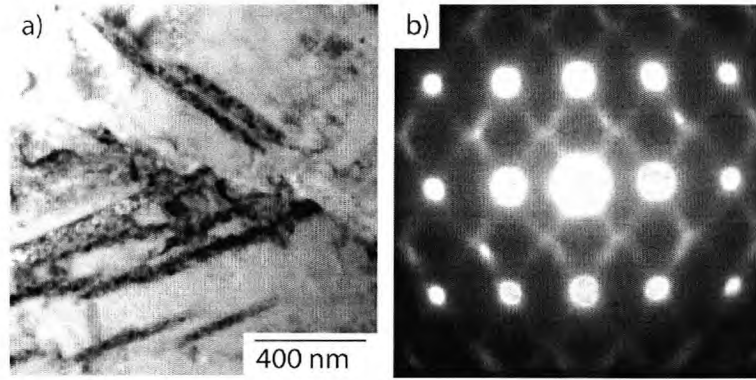


Figure 47: a) Bright field image of α laths in a sample quenched from 900°C and aged for 15 minutes at 570°C (note a β grain boundary traversing the image from left to right), b) corresponding electron diffraction pattern from the upper grain along the $[110]\beta$ zone axis, $D = 800$ mm.

The α phase laths continued to develop during aging to 30 minutes, to ~ 1.5 μm in length by 200 nm in width (Figure 48a). The α laths are evenly distributed and two distinct orientations can be seen within the microstructure. This observation is supported by diffraction from the $[110]\beta$ zone axis (Figure 48b), which shows reflections consistent with both the expected α variants obeying the Burgers relationship as indicated in the key diagram (Figure 48c). The extra reflections that are observed in Figure 48b, when compared to the key diagram, are due to double diffraction. An α phase basal lattice parameter of 2.88 Å can be calculated from Figure 48b, which is in good agreement of that from the in-situ data, which when approximated by a Voigt function produces an α phase basal lattice parameter of 2.83 Å.

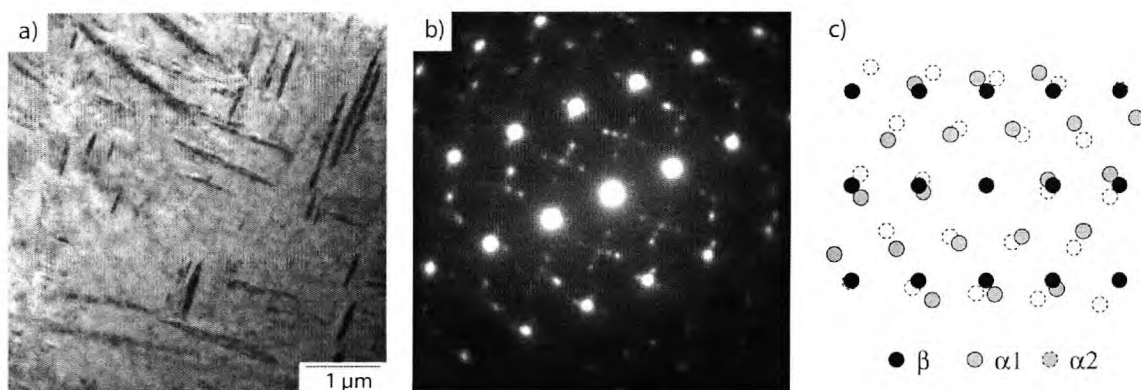


Figure 48: a) Bright field image showing two α lath variants in a sample quenched from 900°C and aged for 30 minutes at 570°C, b) corresponding electron diffraction pattern from the $[110]_\beta$ zone axis, $D = 800$ mm and c) key diagram of $[110]_\beta$ zone axis with reflections corresponding to two α variants overlaid.

The development of the α precipitates during aging results in an associated change in the mechanical properties. Figure 49 shows representative mechanical data, in the form of Vickers hardness, at each aging step. As α develops the hardness increases, suggesting that the final properties of the material are dependent on the α structure. It would also indicate that with control of the aging regime further optimisation is possible through the control of the α development from the pre-existing ω phase.

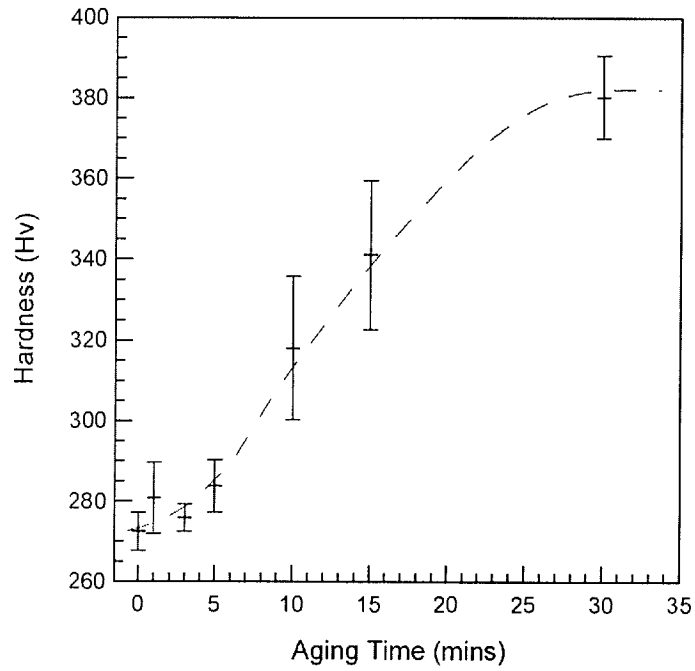


Figure 49: Evolution of Ti-5-5-5-3 hardness with aging time following quenching from the β phase field (the line is a guide to the eye).

5.5 Direct Aging of Ti-5-5-5-3

The microstructure achieved following rapid cooling of a thin disc samples to room temperature from the β phase field can be seen in Figure 50a. An equiaxed β grain structure can be observed with an average grain size of $\sim 120\ \mu\text{m}$. Such a microstructure is consistent with the results of section 3.3.3 in which air cooling Ti-5-5-5-3 from the beta phase field resulted in a retained β microstructure, with no evidence of either α or α'' precipitates. It is therefore assumed that when directly aging Ti-5-5-5-3 at 400°C by air cooling from 900°C the microstructure at the start of the aging process would also be entirely β . This supposition is supported by Figure 50b and Figure 51. Figure 50b indicates that direct aging to 400°C and holding for 1 hour produces a similar microstructure to that of the sample air cooled to room temperature. Light microscopy does not show any evidence of α phase precipitation or β grain growth following aging at 400°C for 1 hour. Laboratory based X-ray diffraction data (Figure 51) also exhibits little evidence of the α phase after 1 hour of aging at 400°C . Peaks corresponding to the α phase are only observed following 4 hours aging, when the $(100)\alpha$ and $(101)\alpha$ plane reflections become apparent. Following 8 hours of aging at 400°C multiple α phase plane reflections can be seen in Figure 51.

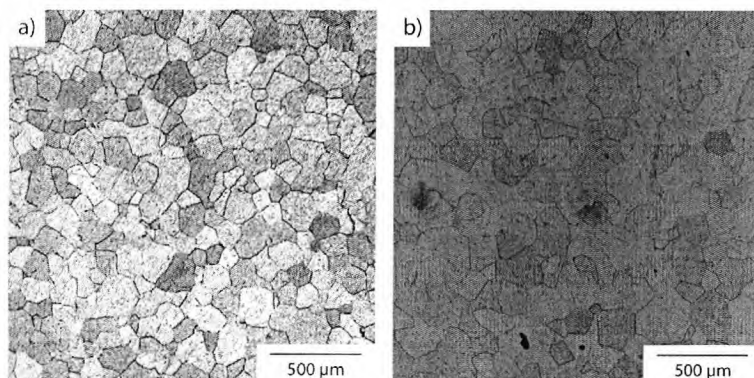


Figure 50: Light micrographs of a) Ti-5-5-5-3 rapidly cooled to room temperature following 30 minute solution heat treatment at 900°C , and b) Ti-5-5-5-3 rapidly cooled from 900°C to 400°C and aged for 1 hour.

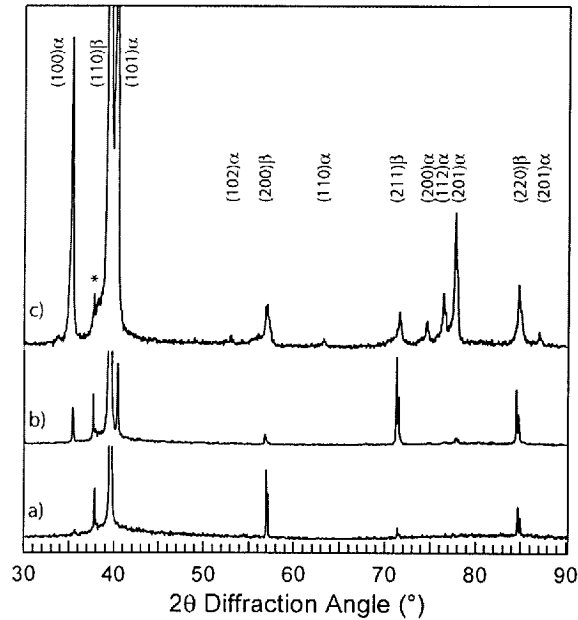


Figure 51: X-ray diffraction data for Ti-5-5-5-3 directly aged at 400°C for a) 1 hour, b) 4 hours, and c) 8 hours. The peak labelled (*) is a reflection caused by tungsten contamination related to the $[110]\beta$ peak.

Further characterisation of the microstructural development during direct aging was investigated using backscattered electron microscopy. Micrographs taken from samples following 4 and 8 hour ages can be seen in Figure 52. The α phase is observed as strings (approximately 15 μm in length) of aligned chevron shaped precipitates which are $\sim 1 - 3 \mu\text{m}$ in diameter. The β grain boundaries are clearly visible, decorated with serrated α precipitates surrounded by a precipitate free zone. This zone is presumably formed due to solute rejection of β stabilising elements, associated with α growth.

After 4 hours of aging the serrated grain boundary α particles appear to have reached maturity (Figure 52a), as little difference in morphology or size is observed having been aged for 8 hours at 400°C (Figure 52d). However, the intragranular chevron precipitates are clearly in a state of development following 4 hours of aging. The chevron morphology (Figure 52b) is not as clearly developed as that of the 8 hour aged sample (Figure 52d) and much smaller precipitates can be observed within a given string. The apparent density of α precipitate strings is also much lower after 4 hours than that observed following 8 hours of aging, as evident from Figure 52a and Figure

52c. Additionally large regions, and in some cases entire prior β grains, remain as retained β following 4 hours at 400°C.

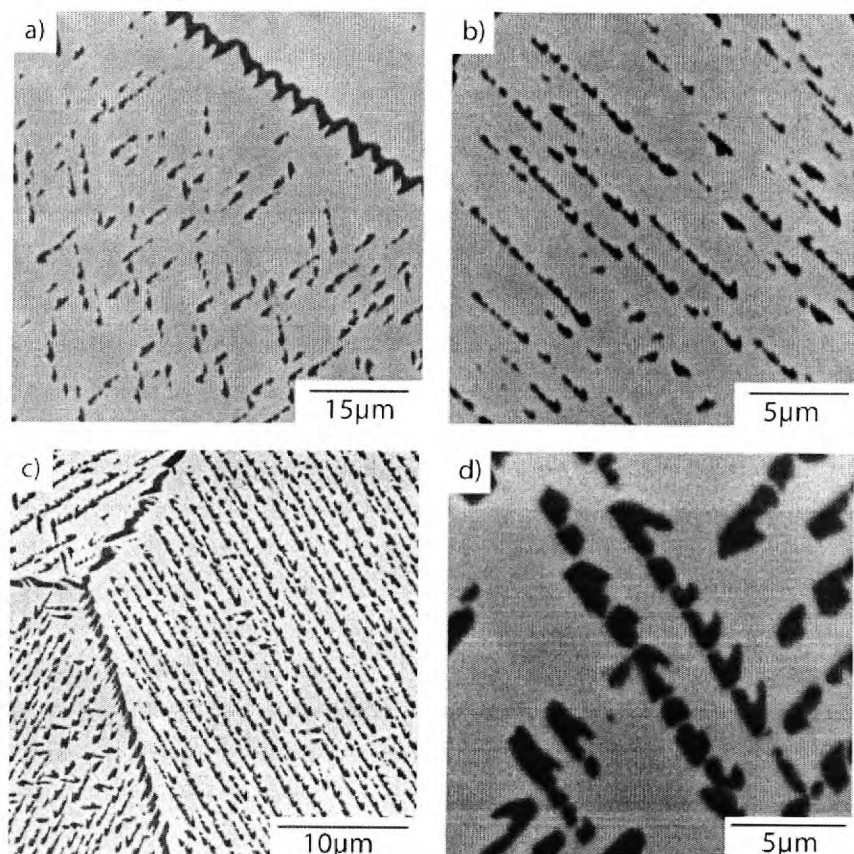


Figure 52: Backscattered electron micrographs of Ti-5-5-5-3 following direct aging at 400°C for 4 hours a) and b), and 8 hours c) and d).

Within the prior β grains the α precipitate strings are mainly observed to share a common morphological orientation, Figure 52b and c. A far lower number of prior β grains have also been observed with two or three distinct string orientations, as can be seen Figure 52a and d. The angle between different string orientations is always found to be $\sim 60^\circ$, consistent with formation of α on $\{110\}\beta$ habit planes.

Electron backscatter diffraction pole figures (Figure 53), obtained from one grain in Figure 52c, show a clear correlation between the matrix and the α precipitate strings, which obeys the Burgers relationship. The relative positions of the $\{0001\}\alpha$ poles directly overlay those of two of the observed $\{110\}\beta$ poles, confirming that the basal hexagonal planes are aligned with the $\{110\}\beta$ of the cubic structure. Using the same

principle an exact alignment can be found between one of the $\{11\bar{2}0\}\alpha$ poles and one of $\{111\}\beta$ poles, which fulfil the directional alignment component of the Burgers relationship. The $\{11\bar{2}0\}\alpha$ poles for any given crystal should lie on a great circle at 90° to that of the related $\{0001\}\alpha$. Thus is it that the $\{11\bar{2}0\}\alpha$ poles observed in Figure 53 all correspond to the more intense $\{0001\}\alpha$ pole. The relevant alignment of the three $\{11\bar{2}0\}\alpha$ poles with $\{111\}\beta$ poles (one pole fully aligned, one $\sim 10^\circ$ misaligned and one with no relation) is consistent with the selection of a single α variant for the given β plane (Figure 4).

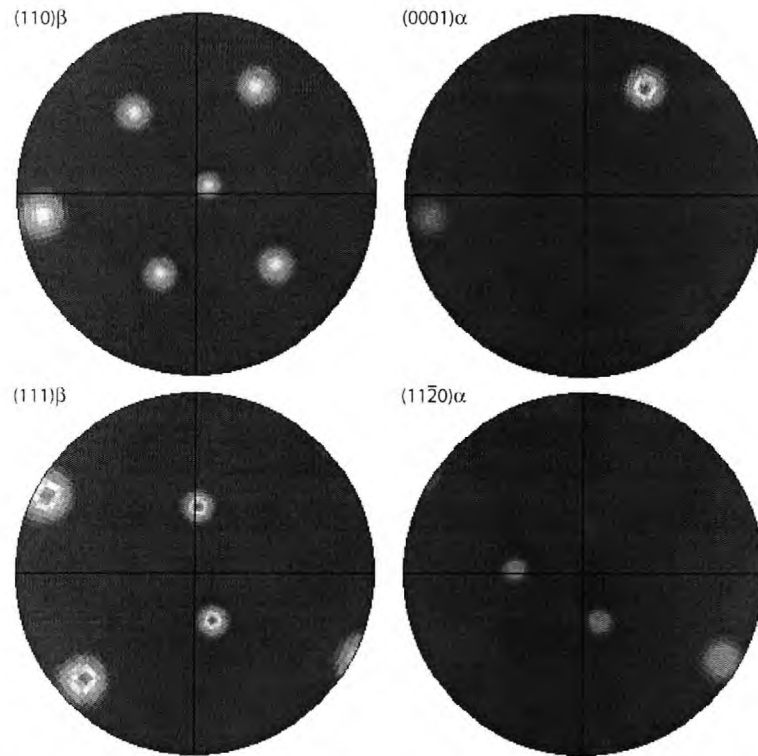


Figure 53: Pole figures obtained using electron backscatter diffraction showing the adherence to the Burgers relationship between the α and β phases in Ti-5-5-5-3 when directly aged at 400°C for 8 hours.

Individual α precipitates develop into a chevron-like morphology clearly visible in Figure 52d. The two arms of the chevrons are approximately $1 - 3\ \mu\text{m}$ in length and within a string each precipitate appears to have grown until mutual impingement occurs. Higher magnification imaging using TEM was employed to further investigate the chevron precipitates. Each individual chevron was found to consist of a series of paired laths diverging from a common point (Figure 54). The laths are approximately $1 - 3\ \mu\text{m}$

in length, consistent with the length scales of the chevrons observed from the SEM images, and create the two arms of the chevrons, seen in Figure 52.

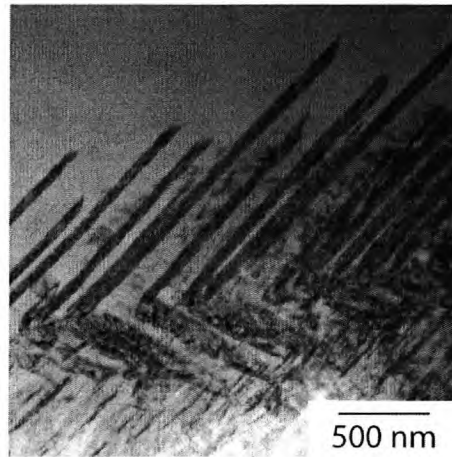


Figure 54: Bright field TEM image of Ti-5-5-5-3 following direct aging at 400°C for 8 hours showing a chevron shaped precipitate.

Higher magnification imaging (Figure 55) revealed the complex internal structure of a chevron precipitate in greater details. Electron diffraction from within an α chevron in the $[110]\beta$ zone axis is shown in Figure 55a. Two distinct α variants are clearly present in both the diffraction pattern and the bright field image of the chevron microstructure, Figure 55c. The internal structure of a chevron (Figure 55c) consists of major parallel α laths, 1 – 3 μm long by approximately 30 nm wide. Smaller α laths, presumably formed via sympathetic nucleation from the major laths, have grown across the interlaying β matrix creating a series of β islands. Further nucleation has produced fine scale α laths within the β islands with dimensions approximately 100 nm in length by 10 nm in width, visible in Figure 55 d.

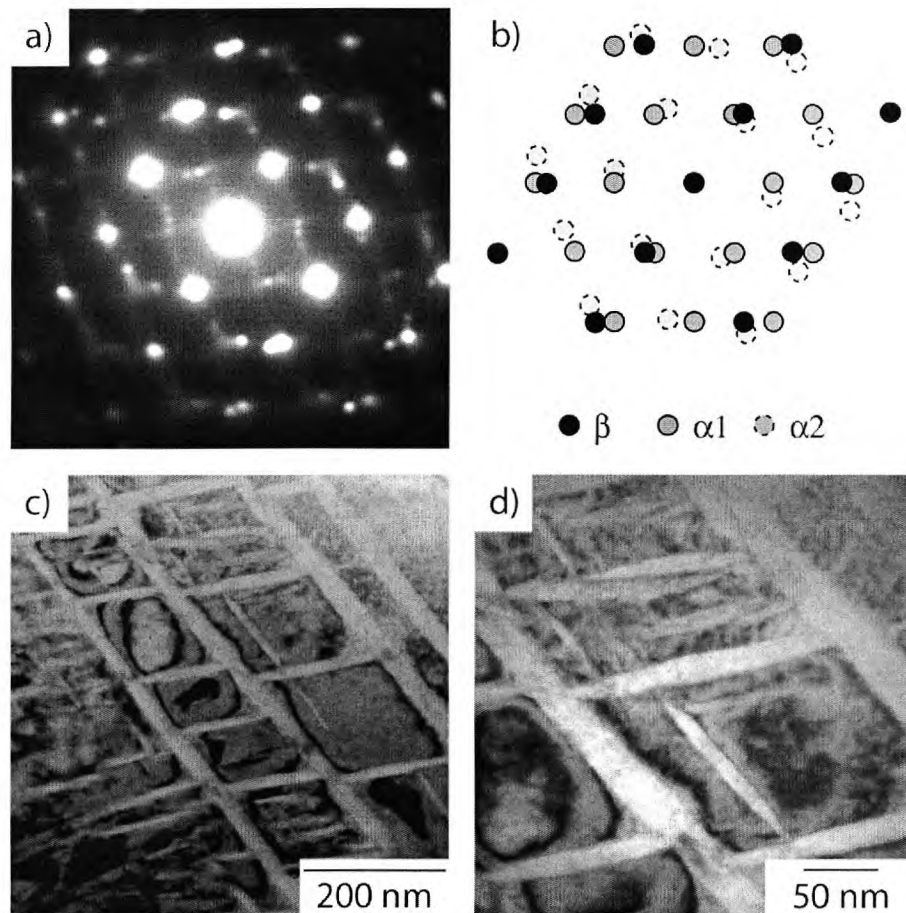


Figure 55: TEM data from Ti-5-5-5-3 following direct aging at 400°C for 8 hours, a) diffraction pattern from the $[110]\beta$ zone axis, b) diffraction pattern key diagram, c) relevant bright field image of the internal structure of an α chevron, and d) bright field image of fine scale α laths within β islands.

Each chevron however, is isolated from its neighbours in any given string and therefore development can only have occurred by independent nucleation within each prior β grain. Furthermore, grain boundary α has not acted as a nucleation site, in contrast to the commonly observed Widmanstätten or basket weave structures. One possible source of the high number of nucleation points could be ω particles produced during the rapid cooling which presumably coarsen during the early stages of low temperature aging. An electron diffraction pattern obtained from the $[110]\beta$ zone axis following one hour of aging at 400°C, Figure 56, shows diffuse streaking between the strong matrix reflections. This streaking is typical of the ω phase and could imply the presence

of a non ideal structure where $[111]\beta$ plane collapse has not completed, as discussed in section 5.3.

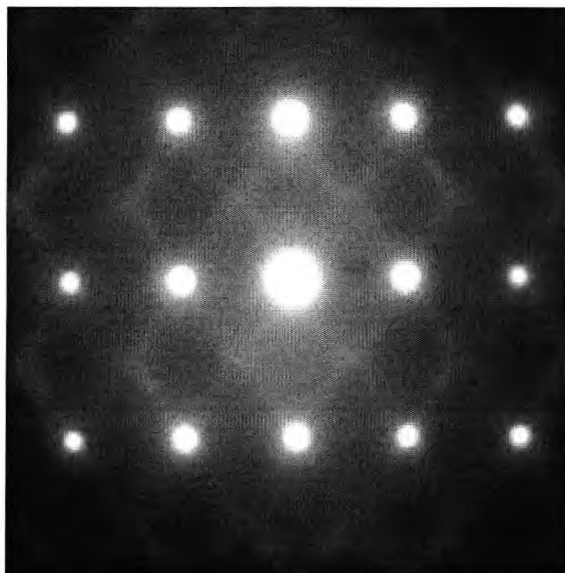


Figure 56: Electron diffraction pattern following aging for 1 hour at 400°C from $[110]\beta$ zone axis, showing streaking typical of the ω phase.

The development of α precipitates during aging is accompanied by a general increase in sample hardness, Figure 57. However, despite the presence of the fine scale α laths within the β islands the hardness developed is not as high as other microstructures in the same alloy, for example the hardness shown following 30 minutes of aging at 570°C in Figure 49. It would appear that direct aging for 1 hour at 400°C decreases the sample hardness when compared to the air cooled to room temperature sample. A similar effect was seen by Ohmori [71] in Ti-10-2-3 when direct aging from 1000°C to 350°C, however no comment was made by the authors. The ω phase itself, despite forming as small precipitates, is not thought to provide any strengthening effect to the parent material until the volume fraction is above 25% [52].

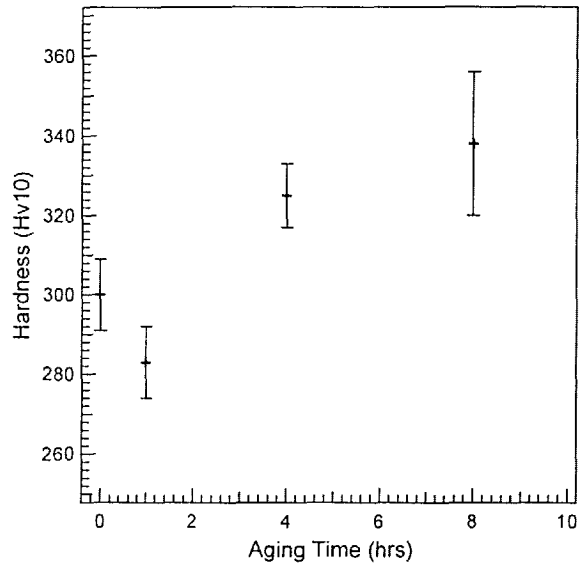


Figure 57: Evolution of Ti-5-5-5-3 hardness with aging time when directly aged to 400°C.

5.6 Summary of Results

In-situ synchrotron X-ray diffraction in combination with ex-situ transmission electron microscopy studies has provided new insights concerning the decomposition of the β phase during rapid cooling. When Ti-5-5-5-3 is rapidly cooled from the β phase field ω particles are formed. Shoulder diffraction peaks found in synchrotron diffraction data suggests that embryonic ω structures exist above the β transformation temperature, supporting the theory of Williams *et al.* [52]. Upon further cooling the shoulders develop into distinct peaks at temperatures corresponding to ω start temperatures reported for similar alloys (approximately 500°C) [22]. Despite recent research reporting β phase separation with a very similar alloy system, data presented here appears not support these findings.

Investigation of the quenched material using transmission electron microscopy also suggested that the ω phase was present. Diffuse streaking approximately along the $[112]\beta$ was observed and four intensity maxima lay on these streaks in between strong matrix reflections. The intensity maxima however, were not in the commensurate positions of $1/3$ and $2/3$ $[112]\beta$ which correspond to the ideal ω structure. Similar observations have been made by several other investigators [53, 54, 58] and based on the work of Sinkler and Luzzi [58], it is hypothesised that the local 111 plane collapse does not create the ideal $\omega_{(3)}$ structure but instead creates combinations of $\omega_{(5)}$ and $\omega_{(7)}$ structures.

Rapid heating of an as quenched microstructure into a commercially used $\alpha+\beta$ aging range led to rapid nucleation and growth of the α phase in a lath morphology. In-situ diffraction data indicates that the α phase has begun to form following as little as 90 seconds at the aging temperature of 570°C. The apparent speed of this nucleation would seem to contradict the sluggish nature of kinetics discussed in Chapter 3. It is thought that the nucleation of the α phase laths is promoted by the presence of the ω particles, providing multiple nucleation sites. Ex-situ studies are in close agreement with the in-situ findings in terms of the timescales of α phase formation. Lath precipitates are observed in the matrix within 180 seconds of reaching the aging temperature, whilst evidence of the ω phase is still visible. Initially it would appear that

the α phase laths form and grow as only one of the Burgers variants, reaching a size of 500 nm in length by 25 nm in width. Following aging for longer than 15 minutes the second Burgers variant for a given (110) β plane appears in the microstructure. By an aging time of 30 minutes the material has developed a well distributed microstructure of small α laths, approximately 1.5 μm in length by 0.2 μm in width, within a β matrix. Developments in the mechanical properties of the alloy have been shown by the increase of Vickers hardness concurrently with the development of the α phase structures.

The direct aging of Ti-5-5-5-3 at 400°C following solution heat treatment in the β phase field results in a unique microstructure consisting of aligned 15 μm strings of α chevrons within parent β grains. The parent β grain boundaries are decorated by serrated α precipitates and it is thought that these precipitates form before the α chevrons. Each chevron appears to have nucleated independently and develops to be approximately 1 – 3 μm within a string, before mutual impingement occurs. It is believed that the high density of small α precipitates is due to multiple site nucleation initiated by ω particles formed during rapid cooling.

The α chevrons have a complex internal structure consisting of α laths ranging in size from 3 μm to 100 nm in length and from 25 to 5 nm in width. It is thought that α precipitates nucleate and grow on (110) β habit planes (60° apart). In between neighbouring primary laths secondary and tertiary laths nucleate sympathetically creating a tight lattice work within the α chevrons. However, despite the formation of very fine scale α precipitates within the chevron structures the corresponding increase in hardness is not as significant as that achieved by quenching and higher temperature aging.

6 Conclusions and Further Work

Work presented in this thesis has characterised the microstructural evolution of the recently developed metastable β alloy Ti-5Al-5Mo-5V-3Cr during simple heat treatments and subtransus isothermal forging. The formation of the athermal ω phase during rapid cooling from the β phase field has also been studied and investigation into the influence this phase has on the formation of the α phase upon subsequent aging has been made.

When comparing Ti-5-5-5-3 to the precursor alloy Ti-10V-2Fe-3Al several marked differences can be observed. The β approach curve of Ti-5-5-5-3 is shallower than that of Ti-10-2-3 which will correspond to a reduced microstructural sensitivity to small temperature fluctuations during processing. Quenching Ti-10-2-3 from the β phase field leads to a stress induced martensitic transformation, where as Ti-5-5-5-3 is able to retain a fully β structure when rapidly cooled. It would also appear that the kinetics of Ti-5-5-5-3 are slower than those of Ti-10-2-3, reportedly an intentional feature of the alloy's design [120].

The flow behaviour of Ti-5-5-5-3 has been found to be related to the initial microstructural condition. Material which had previously been processed in the $\alpha+\beta$ phase field was observed to have a microstructure consisting of small globular α precipitates in a β matrix. Flow behaviour of this material during subtransus isothermal forging was characterised by elastic deformation to yield, followed by steady state flow. Such behaviour would enable simple process modelling using the Norton-Hoff equation incorporating the process variables of strain rate sensitivity, $m = 0.22$ and the material constant, $K = 3.95$ MPa. Little microstructural change was observed following deformation, suggesting that $\alpha+\beta$ condition Ti-5-5-5-3 is relatively insensitive to forming variables such as strain rate, whilst temperature was found to only effect the α volume fraction. Double truncated cone specimens, which produce a graduated strain profile when forged, showed that strain has little influence of the morphology of the globular α precipitates but rotation of slightly elongated precipitates suggests that the α precipitates are merely witnesses to β flow.

Isothermal subtransus forging of material with a fully retained β structure was found to exhibit similar flow behaviour to that of $\alpha+\beta$ condition material. Thus it was concluded that small globular α precipitates have no influence on the flow behaviour of Ti-5-5-5-3, which is dominated by dynamic recovery in the β phase. The microstructural development of retained β condition material when forged at temperature of 785°C and a strain rate of 10^{-3}s^{-1} produced a homogenous distribution of small globular α precipitates. The evolution of this microstructure could be beneficial in reducing the subsequent aging treatments required to achieve the bimodal microstructure commonly used in large components from a two step process to a single step process.

Material which had previously been worked above the β transus displayed a microstructure containing very fine scale Widmanstätten α laths. The flow behaviour during subtransus forging of this microstructure was characterised by elastic deformation to a peak yield stress followed by a period of softening before reaching steady state flow. This behaviour was not observed when forging at 835°C, however, witness microstructures showed that the lath morphology had almost entirely dissolved during pre-compression heating. The acicular α precipitates in the initial microstructure displayed evidence of fragmentation following forging at lower temperatures. The fragmentation produced a microstructure containing small globular α precipitates, similar to the morphologies observed in retained β and $\alpha+\beta$ condition material. All three initial microstructural conditions displayed a similar steady state flow stress at strains ≥ 0.35 , suggesting a common deformation regime had been achieved. Thus the flow behaviour of β forged Ti-5-5-5-3 at low strains is thought to be dominated by dislocation pile up at the matrix / lath interface, leading to a stress concentration and subsequent fracture of the α plates. The resultant equiaxed α precipitates have little further effect on the flow behaviour of the alloy and the steady state flow regime is dominated by the dynamic recovery of the β phase. These observations, appear to corroborate the flow softening mechanism of α lath break up and spheroidisation proposed by Jackson *et al.* for Ti-10-2-3 [5, 131].

During rapid cooling the formation of the athermal ω phase had been established. X-ray synchrotron data presented in Chapter 5 shows the formation of an embryonic form of ω well above normally reported ω start temperatures, consistent with a mechanism

proposed by Williams *et al.* [52]. The structure of the athermal ω phase in Ti-5-5-5-3 is found to be incommensurate by the position of intensity maxima in electron diffraction patterns obtained during TEM studies. It is hypothesised that the athermal ω phase observed consists of $\omega_{(5)}$ and $\omega_{(7)}$ structures, after the work of Sinkler and Luzzi [58].

The α phase is observed to form within 90 seconds following rapid heating of quenched material to 570°C. In situ studies show that the ω phase is still present at this moment and thus it is thought that it influences the nucleation of the α phase. The initial growth of the α phase is observed as a lath morphology aligned to only one of the possible Burgers variants for a given (110) β . Following 30 minutes aging at 570°C both expected α variants for a given (110) β are observed as well distributed lath precipitates approximately 1.5 μm long by 0.2 μm wide.

A unique chevron morphology α phase precipitate has been observed following direct aging of Ti-5-5-5-3 at 400°C from the β phase field. The chevrons appear to form in aligned strings approximately 15 μm long. Each individual chevron nucleated independently and then grew until mutual impingement occurred, reaching a size of approximately 1 - 3 μm . Within each chevron a complex lattice structure of α laths existed, with evidence of sympathetic nucleation of smaller laths on the broad face of the larger laths. The high level of intragranular nucleation would suggest the presence of a nucleation enhancer and it is suggested that the presence of the ω phase following rapid cooling could provide potential nucleation sites.

6.1 *Areas for Further Work*

The characterisation of the microstructural development of Ti-5-5-5-3 during isothermal forging presented in Chapter 4 has shown that prior processing can have an effect on the flow behaviour and the resulting microstructure. Since it is likely that material supplied for secondary working will be in the $\alpha+\beta$ condition a combination of empirical studies and process modelling can be employed to optimise the process to obtain the desired microstructure. Flow behaviour of $\alpha+\beta$ material should be simple to model as the insensitivity of microstructure enables the use of a Norton Hoff relationship. The correct balance between temperature, which influences the volume fraction of primary α , and strain rate, which dictates the required flow stress in combination with temperature, for an optimised process should not be difficult to define.

Despite the flow softening mechanism of α lath precipitate break up [131] accounting for the observed microstructural features, a more detailed study needs to clarify the precise nature of the materials response. Lath precipitates need to be observed both prior to, and in the process of fragmentation such that the proposed dislocation pile ups can be imaged. This could be achieved by forging samples to very low strains, for example between 0.05 and 0.1, where the material is in the softening regime. Imaging this structure should allow the location of α precipitates prior to and in the process of fragmentation. Such microstructural features could then be selectively removed using focussed ion beam milling and thinned into sections suitable for examination in a transmission electron microscope. This study should focus on the dislocation structure in and around the α lath as well as investigating the active slip systems and the progression of the break up sequence.

The influence of the ω phase on the microstructural development of complex metastable β alloys remains poorly understood, despite some recent studies [71, 72]. A greater understanding of the formation behaviour needs to be developed for multi component alloy systems, determining characteristic parameters such as the critical cooling rate above which the ω phase will form. Data of this type could be achieved by employing a similar study as to that used in Chapter 5 but incorporating a range of controlled cooling rates. If the athermal ω phase can be formed at industrially viable cooling rates, then

there is potential to utilise it in developing and tailoring the final component microstructure. Some work already exists which has investigated how to control the ω phase volume fraction precisely for this reason [125]. The ability to use in situ techniques, such as an X-ray synchrotron, capable of short time scale resolution over the wide temperature range required to study the athermal ω transformation will enable a deeper understanding as to the formation mechanisms.

It is widely accepted that ω phase particles act as potent α phase nucleation sites, however the mechanisms of this influence remain unclear. Despite recent studies using lattice imaging [71, 72], whether the ω phase rearranges to become the α phase or if the ω particle heterogeneously nucleates the α precipitate before being consumed during growth remains unresolved. The characterisation of transformation timescales available from synchrotron diffraction studies should enable better selection of interrupted testing sample conditions such that material before, during and after the initial formation of the α phase can be studied using high resolution techniques. Understanding the α phase formation mechanism through detailed high resolution studies would also aid the interpretation of the observation of single variant formation during the early stage of aging. The slow kinetics exhibited by Ti-5-5-5-3 may offer the possibility of a cooling regime capable of retaining β at room temperature without forming any other phase. The development of the α phase in such material would be very useful to investigate as a comparison to ω influenced material.

The development of the α chevron structures following direct aging to 400°C is an interesting observation in terms of the mechanisms behind its formation. The timescales involved in the formation could easily be characterised by an in situ synchrotron study, which would also confirm the presence of any intermediate phase. Combining this information with electron microscopy of carefully selected interrupted testing samples should enable a full description of the transformation sequence to be developed.

7 References

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