Variant selection and its effect on texture in Ti-6Al-4V

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### Abbreviation

<table>
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<th>Description</th>
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<tbody>
<tr>
<td>[Al]eq</td>
<td>Aluminum Equivalent (ASTM E112).</td>
</tr>
<tr>
<td>Bcc</td>
<td>Body Centred Cubic</td>
</tr>
<tr>
<td>CCD</td>
<td>Charged Couple Device</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>Commercially Pure Titanium</td>
</tr>
<tr>
<td>EBSP</td>
<td>Electron Backscatter Diffraction Pattern</td>
</tr>
<tr>
<td>(φ₁ Φ φ₂)</td>
<td>Euler angles</td>
</tr>
<tr>
<td>Fcc</td>
<td>Face Centred Cubic</td>
</tr>
<tr>
<td>FEGSEM</td>
<td>Field Emission Gun Scanning Electron Microscope</td>
</tr>
<tr>
<td>GEM</td>
<td>General Material Diffractometer</td>
</tr>
<tr>
<td>hcp</td>
<td>Hexagonal Close Packed</td>
</tr>
<tr>
<td>IPF</td>
<td>Inverse Pole Figures</td>
</tr>
<tr>
<td>M</td>
<td>Transformation Matrix,</td>
</tr>
<tr>
<td>[Mo]eq</td>
<td>Molybdenum Equivalent</td>
</tr>
<tr>
<td>Mrd</td>
<td>Multiples of Random Distributions</td>
</tr>
<tr>
<td>ND</td>
<td>Normal Direction</td>
</tr>
<tr>
<td>ODF</td>
<td>Orientation Distribution Function</td>
</tr>
<tr>
<td>PF</td>
<td>Pole Figure</td>
</tr>
<tr>
<td>RD</td>
<td>Rolling Direction</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>TD</td>
<td>Transverse Direction</td>
</tr>
<tr>
<td>TTT</td>
<td>Time Temperature Transformation</td>
</tr>
<tr>
<td>TOF</td>
<td>Time-of- Flight</td>
</tr>
<tr>
<td>β HT</td>
<td>β Heat Treatment</td>
</tr>
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Variant selection and its effect on texture in Ti-6Al-4V

Abstract of thesis submitted by Gideon Obasi to the School of materials, The University of Manchester for the Degree of Doctor of Philosophy 2011

Titanium alloys are strong candidates for the aerospace industry and biomaterial applications because of their low density, high strength-to-weight ratio and very high strength even at temperatures up to 600°C. Like many other engineering alloys, titanium alloys are prone to strong preferred crystallographic orientation development during thermomechanical processing. Part of the titanium processing route is to heat treat the material above the β transus for the purpose of homogenization and associated phase transformation. This heat treatment dramatically affected the microstructure and texture evolution. Theoretically, such heat treatment should result in a nearly random texture if all variants during α→β→α phase transformation are active. In reality, significant textures are observed after such a heat treatment process. The present project aims at developing a detailed understanding of the root cause for this relatively strong texture by means of EBSD and in-situ neutron diffraction studies.

The effect of β grain growth on variant selection during β to α phase transformation has been investigated by using two variants of Ti-6Al-4V with and without 0.4 wt% yttrium addition. The aim of adding yttrium was to control β grain growth above the β transus by pinning grain boundaries with yttria. Both materials were first thermomechanically processed to generate a similar starting microstructures and crystallographic textures. Subsequently, both materials were solution heat treated above the β transus followed by slow cooling to promote growth of the α lath structure from grain boundary α. Detailed EBSD and in-situ neutron diffraction analysis were carried out to study microstructure and texture evolution. The variant selection calculation suggests that more variant selection occurred in convectional material with a large grain size compared to material with yttrium addition. In-situ measurements showed that β texture strengthened significantly above the β transus with increasing β grain size. There was no significant variant selection during α→β transformation; variant selection noticeably increased during β→α transformation with increasing β grain size. Additional interrupted cooling experiments followed by EBSD analysis showed early nucleation of α variants with a “butterfly morphology” from β grain boundaries that have a pair of β grain with a common <110> pole. These observations suggest reduced nucleation energies for α formation in such circumstances allowing extensive growth of these α variants into unoccupied β grains making it a dominant variant.

The influence of rolling temperatures (i.e. at 800 ºC and 950 ºC) to produce different starting texture, on texture evolution and variant selection during α→β→α transformation was also investigated. Laboratory X-ray, EBSD and in-situ neutron diffraction texture analyses were carried out. Even though the transformation texture is stronger at 800 ºC, the degree of variant selection is stronger in materials rolled at 950 ºC compared to material rolled at 800 ºC. Here, the enhanced variant selection for the material rolled at 950 ºC was related to the different β texture. It is suggested that the combination of a particular β texture components promote variant nucleation that can increase the likelihood of having β grain pairs with a common <110> pole.
DECLARATION

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Finally, I thank God for his mercy, grace and protection throughout the duration of this project.
This work is dedicated to my family
Introduction

Titanium alloys have received considerable attention for decades due to their excellent properties, such as low density of about 4.5 g/cm$^3$, high strength-to-weight ratio, high corrosion resistance in different environments and excellent biocompatibility. Due to these excellent properties, Ti-alloys are widely used for many structural applications, for example in automotive, aerospace, chemical, power and biomedical industries (Froes, S., 2002). In the aerospace industry, Ti-6Al-4V is mainly used in the fan and compressor section of aero-engines including disks and blades, which operate at temperatures up to about 600 °C depending on the loading conditions. The thermomechanical processing route determines the microstructure and texture and as a consequence the mechanical properties. During thermomechanical processing, Ti-alloys undergo phase transformation from low temperature hexagonal close packed (hcp) $\alpha$ phase to high temperature body centred cubic (bcc) $\beta$ phase and back to low temperature hexagonal close packed (hcp) $\alpha$ phase ($\alpha \rightarrow \beta \rightarrow \alpha$). This transition also affects the microstructural and texture evolution. The transformation is governed by the so-called Burgers orientation relationship $\{0001\}_\alpha \parallel \{110\}_\beta$ and $<11-20>\_\alpha \parallel <111>\_\beta$. As a result of this relationship, only six possible $\beta$ variants can grow from the daughter phase ($\alpha$) and twelve $\alpha$ variants from the mother phase ($\beta$) grain (Burgers, W. G. 1934). If all variants occur during transformation should lead to randomisation of any existent $\alpha$ texture. But this is not the case, even though the orientation relationship is obeyed not all the variants are generated due to the so-called variant selection (Lonardelli, I. 2007, Jourdan, C. 1991 and Romero, J. 2009). Variant selection is the preferential occurrence of certain variants during phase
transformation. This phenomenon has been investigated both in bcc materials and hcp materials. The root cause for such microtextural development is still unclear but might be related to texture evolution at very early stages of transformation leading to specific crystallographic morphology and grain orientation relationship. This has been link to degrade mechanical properties of this alloy (Wojcik, C.C.et al., 1988). It is believed that any strong texture generated during the early stage of thermomechanical processing has a strong effect on the texture of the final product (Zhu Z. S et al., 2000).

It has been reported (Lonardelli, I. 2007, Jourdan, C. 1991 and Romero, J. 2009) that β heat treatment followed by displacive or diffusional β → α phase transformation in titanium and zirconium alloys, can lead to strong texture memory i.e. starting texture is retained. The phenomenon was related to retain α during heat treatment suggesting that the β transus was not exceeded during such investigation (Romero, J. 2009)

However, other studies have also shown significant textures after β heat treatments that are linked to variant selection (Vogel, S. C et al., 2005, Preuss et al., 2007). To date, there is no concensus about the fundamental mechanisms responsible for variant selection in Ti and Zr alloys.

In order to achieve an effective control of the texture and microstructural evolution in Ti-6Al-4V, it is important to have a detailed understanding of the variant selection mechanism during the thermomechanical process and phase transformation. The aim of this project was to ascertain the degree of variant selection occurrence during the β to α phase transformation and the associated mechanisms. The new understanding would be utilised to optimise processing routes of titanium alloys to limit any microtexture development. The objectives of this study are itemised below:
(1) To characterise the starting materials in terms of microstructure and texture using Electron Backscatter Diffraction (EBSD) and laboratory x-ray (LXRD).

(2) To understand the influence of β grain growth on the variant selection and texture memory since β grains of conventional Ti-6Al-4V grow very fast once the β transus temperature is exceeded.

(3) To investigate the effect of deformation temperature on texture evolution and variant selection during phase transformation.

(4) To perform in situ investigations of the evolution of β and α texture during heating and cooling and variant selection studies by means of neutron diffraction.

In the current study, Optical Microscopy (OM), Scanning Electron Microscopy (SEM), Electron BackScatter Diffraction (EBSD), laboratory X-ray diffraction and neutron diffraction were used for microstructural and micro- and macrotexture characterisations. The α orientation maps recorded by EBSD were reconstructed to obtain the high temperature β phase using β reconstruction code (Davies, P. S 2007). The α textures were predicted from reconstructed β texture (ambient EBSD measurement) and measured β texture (neutron diffraction) using TEXTAN III (Bate, P 1990) based on Burgers orientation relationship without variant selection. The predicted textures were compared with the measured α texture to ascertain the extent of variant selection occurrence for each thermomechanical processing condition.
Introduction

This thesis has been written in alternative format. The main justification for opting to submit this thesis in alternative format is the desire to publish the results of this study in journal papers and an opportunity to get comments not only from the external and internal examiners but also from peer reviewers. Most PhD works with important findings end up in the library shelves without been published in journals where the academic communities can have full access to these works. In this work, I am the main contributor and my project supervisor, who read through the manuscript. Other co-authors in both the journal and conference papers are colleagues that I had useful discussion with or that accompanied me for neutron diffraction experiments at Science and Technology Facilities Council Rutherford Appleton Laboratory ISIS Facility, UK and as well as the beam scientist for his help during the experiment. It is necessary to point out that due to the nature of such experiments more than one person are required to perform the experiment.

The thesis has been divided into eight chapters and two appendix chapters. The first and second chapters review important concepts of the thermomechanical processing, texture evolution and variant selection necessary to understand the results presented subsequently. Chapter 1 deals with the physical metallurgy and deformation modes of Ti alloys. Chapter 2 covers principle aspects of texture representation, texture evolution during processing and principle aspects of variant selection and \( \beta \) reconstruction. The experimental methods are presented in Chapter 3. This includes microstructural analysis by optical microscopy (OM), texture measurement and analysis using different diffraction techniques (laboratory X-ray diffraction, EBSD and neutron diffraction) and chapter 4 presents detailed description of post data processing methods developed to analyse the results presented in this study. The
results and discussion are presented in form of journal papers in Chapters 5, 6 and 7 and additional results and discussion in form of conference papers are presented in appendix 1 and 2. A detail description of the matlab code developed for the analysis of the result presented in the study is found in appendix 3. Finally, in chapter 8, the conclusions of the work are presented, summarizing the knowledge gained from this work. It is hoped that the conclusions drawn from this work will not only contribute to a better understanding of the root cause of variant selection and dominant mechanisms of variant selection, during $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation, but that these new findings can be utilised in order to develop a processing route for Ti to minimized occurrence of macrozones in the final product.
Chapter 1  **Physical Metallurgy of Titanium Alloys**

1.1  *Titanium and its Alloys*

The attention attracted by titanium alloys stem from their mechanical properties at both room and elevated temperatures and their critical application as structural components in aerospace industries. This section will cover the development of titanium, as well as the physical metallurgy and deformation mechanisms of titanium alloys.

1.1.1  **Development of Titanium alloys**

Titanium constitutes about 0.6 % of the earth’s crust and is the fourth most abundant structural metal. Due to its high affinity for oxygen, titanium occurs mostly in the form of oxides, ilmenite (FeTiO₃) and rutile (TiO₂). Ilmenite (FeTiO₃) and rutile (TiO₂) are the most sought after oxides due to their economic value. The metal was first discovered in 1791 by Gregor and in 1795, Klaproth named the metal titanium after Greek mythology, the Titans. Several attempts to develop cost-effective commercially available titanium metal proved difficult until (1937-1940) when a commercially attractive process was developed by Kroll (Lütjering and Williams 2003). Kroll demonstrated that TiCl₄ could be used to extract titanium commercially using magnesium as a reducing agent. To date, this process remains the most viable commercial means of extracting titanium. Interest in this metal and its alloys began in the late 1940s and early 1950s (Lütjering and Williams 2003) due to their excellent properties, such as high strength-to-weight ratio, corrosion resistance, creep resistance...
and biocompatibility. Due to these excellent properties, the metal is today widely used in the manufacturing of critical components in aerospace and chemical industries. The aerospace industries accounted for about 55% of titanium use in the USA in 2002 (Gambogi J., 2004). It is used in both the air frames and aero-engines. The corrosion resistance property of this metal is exploited in the chemical industries, oil and gas offshore structures and its biocompatibility are utilized in surgical implants like the hip joints and dental parts. The application of titanium in the areas of consumer products is making great impacts, including golf club heads, watches, tennis rackets and bicycle frames. However, the application of this metal has been limited due to high raw material and production costs relative to other structural materials. The production sequences from the ore to semi-finished products of the titanium are depicted in Figure 1-1.
Figure 1-1 Production sequence of titanium
1.1.2 Alloaying Elements

Titanium exists in more than one crystallographic form. At room temperature, pure titanium exhibits a hexagonal close packed (hcp) crystal structure; known as the “alpha” (α) phase and transforms from the α phase (hcp) to body centered cubic (bcc) called “β phase” once the temperature is raised above 882.5 ºC. The temperature at which this transformation occurs is called the β transus temperature and it is defined as the lowest equilibrium temperature at which the material consists of 100 % β phase (Boyer, R., 1985). The transformation temperature of pure titanium can be raised or lowered by alloying element additions. The alloying elements can be classified into three groups depending on their effect on the β transus temperature. Elements such as aluminum, gallium, germanium, carbon, oxygen and nitrogen that raise the β transus temperature are referred to as α stabilizers. These elements increase the temperature at which the α phase is stable, consequently, raising the β transus temperature. In contrast, elements such as vanadium, hydrogen, molybdenum, tantalum and niobium lead to expansion of the β phase field, thus lowering the β transus temperature and are referred to as β stabilizers (Donachie M. J. Jr., 2004, Joshi, V A., 2006). However, other elements such as yttrium and zirconium do not have significant influence on the β transus temperature and are therefore classified as neutral elements. The effects of these elements on titanium phase diagrams are illustrated in Figure 1-2.
Moreover, apart from altering the β transus temperature, the alloying elements contribute to the mechanical properties of titanium and its alloys and can be grouped into three categories:

- interstitial elements
- substitutional elements
- metallic impurities

The α stabilizing alloying elements such as oxygen, nitrogen and carbon form interstitial solid solutions with titanium; but display a limited solubility. The interstitials are much smaller than the host atoms resulting in a significant increase in the strength of alloy by solid solution strengthening. The oxygen level is a standard used to determine different grades of commercial pure titanium. Aluminum is considered the most important α stabilizing alloying element due to its low density-to-weight ratio, increase in creep properties and moduli (Joshi, V A., 2006). The limit to the amount of α stabilizing element in titanium alloys can be calculated using the aluminum equivalent [Al]eq; if the [Al]eq exceeds 9% in titanium, it results in the formation of α₂ alloy and embrittling. The aluminum equivalent can be calculated using the empirical formula (Leyens and peters, 2003).
\[ [Al]_{eq} = [Al] + 0.33 [Sn] + 0.17 [Zr] + 10 [O] \] (1.1)

The content of hydrogen, which is a $\beta$ stabilizing element in Ti alloys, is strictly limited owing to hydrogen embrittlement which tends to degrade mechanical properties (Bhadeshia, H. K., 2004).

The substitutional $\beta$ stabilizing alloying elements such as V, Mo and Nb have a similar atomic size as the host atom. These elements play a significant role in improving the mechanical properties by altering the microstructure. Sufficient amounts of these elements stabilize $\beta$ phase at room temperature and increase the ductility of the material. The required amount of $\beta$ stabilizing elements in titanium can be determined by using the molybdenum equivalent. This is calculated with the following empirical formula (Bieler T. R., 2005).

\[ \text{Mo(eq)} = \%\text{Mo} + 0.67 (\%\text{V}) + 1.25 + 2.9 (\%\text{Fe}) + 1.6 (\%\text{Cr}) - \%\text{Al} \] (1.2)

In the molybdenum equivalent equation, the different constants are related to the ratio of the $\beta$ composition for molybdenum divided by the $\beta$ composition for the particular element. Higher values for the molybdenum equivalent would lead to the decrease in the $\beta$ transus temperature.

Zr, Hf, Sn and Y are regarded as neutral alloying elements or metallic impurities because they do not strongly increase or reduce the $\beta$ transus temperature. These elements have considerable solubility in both the $\alpha$ and $\beta$ phases and are useful strengthening agents (Joshi, V A., 2006). In addition to strengthening of the alloy, increased concentration of yttrium resulted in considerably reduction of $\beta$ grain...
Variant selection and its effect on texture in Ti-6Al-4V growth. This is due to the precipitation of yttria (Y$_2$O$_3$) at the prior β grain boundaries (Hotta, S. et al., (2006), which retard β grain growth based on the so called Zener grain boundary pinning. The β grain refinement resulting from Y addition is a function of volume fraction of Y and oxygen that forms Y$_2$O$_3$ precipitates. The precipitation of Y$_2$O$_3$ in the β phase retards the onset temperature of β grain growth. According Hotta et al, (Hotta, S. et al., (2006) yttrium addition can result in microstructural inhomogeneity with very large grains surrounded by very small grains when the temperature is raised above the β transus. This abnormal grain growth is found in localized areas where rapid coarsening due to re-dissolution of precipitates occurs. However, as the temperature is raised further above 1300 °C, a homogeneous microstructure with coarse grain size is achieved due to dissolution of yttria into the β matrix.

### 1.1.3 Classification of Titanium alloys

Titanium alloys are classified according to their chemical composition and microstructure at room temperature. There are five major groups of titanium alloys, namely: α alloys, near-α alloys, α+β alloys, metastable β alloys and β alloys. A schematic binary phase diagram containing both α and β stabilising elements is shown in Figure 1-3. The upper part of the diagram, indicated with an arrow, shows the range of each of the various groups of the alloys. The dashed line marked M$_s$/M$_f$ indicates the martensitic start and finish line.

**1) α alloys**

The α alloys range cover compositions in which the β phase cannot be retained at room temperature as the composition of β is on the right of the M$_f$ line at room
temperature. The α alloy microstructure are composed of α or α’ (martensite) after transformation depending on the cooling rate. Very fast cooling rates (water quenched) produce α’ whereas controlled cooling rates produce the α microstructure. α alloys have lower strength compared to other titanium alloys but have high corrosion and creep resistance properties.

(2) Near-α alloys
Unlike α alloy near-α alloys contain small amount of β stabilising. The forging heat treatment processes of the near-α alloy within α + β phase field usually produce β with composition to the right of the Mₜ/Mₜ line on phase diagram at room temperature. Consequently, the microstructure comprises retained β in metastable state at room temperature. It is ideal for high temperature applications in the range of 600 °C, where its excellent creep resistance can be combined with high strength due to small amounts of dispersed β phase compared to the α alloy. The examples of this alloy is the Timetal 834 and Timetal 1100, these alloys are forged within the α + β and resultant microstructure displays a mixture different α morphologies, which provides compromise for good creep and low cycle fatigue resistance (Polmear, 1995).

(3) α + β alloy
The α + β microstructure is composed of α + β at room temperature; it intersects the boundaries of Mₜ/Mₜ line at room temperature, thus transforming martenstically upon quenching from the high temperature single phase β to room temperature. α + β alloys are rich in β stabilizing elements and contain β phase at all temperatures. The strength of this alloy can be improve from medium to high strength level by solution
heat treatment, quenching and aging processes (Bieler T. R., 2005). Ti-6Al-4V, which is the most widely used of the $\alpha + \beta$ alloys, will be discussed in detail in section 1.1.4 since it is the alloy used in this study.

(4) Near- $\beta$ alloy

The near $\beta$ composition range lies to the right of the $M_s/M_f$ line at room temperature. The microstructure comprises highly metastable retained $\beta$ which can transform to $\alpha'$ upon quenching. Near $\beta$ alloys have more $\beta$ stabilizing elements than the convectional $\alpha + \beta$ alloys, but not sufficient enough to retain 100 % $\beta$ phase upon air cooling of a thin section. The strength level is high due to the precipitation of fine $\alpha$ precipitates during aging heat treatment (Donachie M. J. Jr., 2004).

(5) $\beta$ alloy

The $\beta$ alloys contain enough $\beta$ stabilizing elements to avoid cooling through the $M_s$ line, consequently creating a microstructure with 100 % retained $\beta$ at room temperature. These alloys generate high strength levels with good ductility and displays excellent cold formability due to high volume fraction of the softer $\beta$ phase. (Donachie M. J. Jr., 2004).
1.1.4 The $\alpha + \beta$ Ti-Alloys

Since the present work is concerned with Ti-6Al-4V, some more details regarding $\alpha + \beta$ titanium alloy are given here. Two phase $\alpha + \beta$ titanium alloys have high strength and good formability and contain about 4-6 wt % of $\beta$ stabilizers which allow substantial volume fractions of $\beta$ to be retained on cooling from the $\beta$ phase field. Different microstructures of these alloys can be obtained by varying thermomechanical processing parameters and/or $\beta$ heat treatments.

The commonly used class of $\alpha + \beta$ titanium alloys are Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo-0.1Si and Ti-6Al-2Sn-4Zr-6Mo. Ti-6Al-4V is the most widely used $\alpha + \beta$ alloys. At 800 °C Ti-6Al-4V contains about 15 vol.% of the $\beta$ phase (Lütjering and Williams 2003). The $\alpha$ phase is the dominant phase at room temperature but exists as a single phase $\beta$ above 995 °C (Ahmed and Rack, 1998).
Aluminum is added to promote α stabilisation and to increase the strength of the alloy by solid solution hardening. Vanadium stabilises β and significantly improves the room temperature ductility to obtain a balance of α and β phase mechanical properties. The addition of β stabilisers can retard the formation of α phase so that β can transform to martensite or retained β phase. Its relatively good creep resistance makes it suitable for use in high temperature applications up to 400 ºC. Other characteristics of this alloy are good weldability and formability. However, the creep properties of this alloy is lower than the near-α alloy, due to the presence of the bcc β phase, which has higher diffusivity and more slip systems (Bieler T. R., 2005).

Similar variants of Ti-6Al-4V produced by powder metallurgy processes contain additions of boron and carbon compared to conventional ingot processed variants of Ti-6Al-4V. Powder metallurgy processed alloys have been developed for specific properties; it has about 25 % higher strength and modulus but with lower ductility compared to ingot processed Ti-6Al-4V. The boron content forms stable TiB precipitates along the grain boundaries, which retard grain growth during themomechanical processing (Bhadeshia, H. K., 2004, Tamirisakandala, S., 2005).

1.2 **Crystallographic structure and deformation mechanism**

1.2.1 **Crystallographic structure**

Ti-6Al-4V exhibits both hexagonal (α) and body centered cubic (β) phases at room temperature. The α unit cells and their lattice parameters (a and c) are shown in Figure 1-4. The top and bottom faces of the unit cell consist of six atoms that form
the regular hexagonal structure with a single atom at the centre. The c/a ratio is slight lower (1.59 nm) than the ideal ratio for hexagonal crystal structure (1.6633 nm) (Lütjering and Williams 2003). Also indicated in Figure 1-4 are the three main crystallographic planes, basal (0002) plane, two types of prismatic planes, (10 10) and (11 20) and the pyramidal (1011) planes. The three axes, a₁, a₂, and a₃ are close packed directions represented by [1120] as shown in Figure 1-4.

![Hexagonal close packed (hcp) structure showing the close packed planes and directions (Lütjering and Williams 2003)](image)

In the β unit cell shown in Figure 1-5 the atoms are located at all eight corners and a single atom at the centre. The corner and centre atoms touch one another along the diagonals. The most densely packed planes in the bcc structures are the 6 {110} planes whereas the four <111> directions are the most closely packed directions. Figure 1-5 indicates only one of six close pack planes of the bcc crystal structure.
1.2.2 Deformation mechanism

In hcp metals, the number of active slip systems is more limited compared to cubic metals due to lower symmetry. Consequently, twinning is often an important deformation mode in hcp materials. The strict crystallographic orientation relationship (Burgers, W. G., 1934) and low numbers of easy slip systems result in the formation of strong texture during deformation. The texture, on the other hand, is responsible for strong anisotropy in the mechanical properties of the materials (Hosford and Backofen, 1964).
As reviewed in Lütjering and Williams (Lütjering and Williams 2003), the three $<11ar{2}0>$ slip directions shown in Figure 1-6 are the main closed packed directions and have $\bar{a}$ type Burgers vectors. The slip planes are the (0002) planes, the three prismatic $\{10\bar{1}0\}$ planes and the six pyramidal $\{10\bar{1}1\}$ planes.

According to Leyens and Peters (Leyens and Peters, 2003), the basal and prismatic planes have three slip systems each, but only two of the slip systems are independent of each other. Hence, only four independent slip systems are available in hcp materials. In the other slip system, the pyramidal slip plane does not contribute to the
number of independent slip planes since the combined effect of the prismatic and basal slip components give rise to pyramidal glide and as such cannot be considered as an independent slip system (Leyens and Peters, 2003). The various slip systems found in titanium alloys are shown in Table 1-1.

### Table 1-1 Types of slip systems active in titanium alloys (Lütjering and Williams 2003)

<table>
<thead>
<tr>
<th>Slip System type</th>
<th>Burgers Vector type</th>
<th>Slip direction</th>
<th>Slip Plane</th>
<th>No. of slip systems</th>
<th>Total</th>
<th>Independent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basal</td>
<td>$a$</td>
<td>$\langle 11 \overline{2} 0 \rangle$</td>
<td>(0002)</td>
<td>3</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Prismatic</td>
<td>$a$</td>
<td>$\langle 11 \overline{2} 0 \rangle$</td>
<td>${10 \overline{1} 0}$</td>
<td>3</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Pyramidal</td>
<td>$a$</td>
<td>$\langle 11 \overline{2} 0 \rangle$</td>
<td>${10 \overline{1} 1}$</td>
<td>6</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Pyramidal</td>
<td>$c + a$</td>
<td>$\langle 11 \overline{2} 3 \rangle$</td>
<td>${11 \overline{2} 2}$</td>
<td>6</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

Each of these systems require different critical shear stress to be activated. The slip is easiest at the prismatic plane at low temperatures due to smallest resolve shear stress occurring at the prismatic slip plane (Rosi et al., 1953). The plastic deformation as a result of the prismatic slip leaves the crystal shape along the c-axis and c-axis orientation unchanged. The second easiest slip plane is the basal slip plane, again the crystal shape along the c-axis remains unchanged but it causes crystal rotation about the prism plane normal axis (Bieler T. R., 2005). Also, slip can occur on the pyramidal planes. The basal, prismatic and pyramidal slip in the $\langle a \rangle$ close packed direction, which is perpendicular to c-axis, cannot produce strain along the c-direction. Therefore, additional slip systems are needed for homogenous deformation to occur. According to the Von Mises criterion (Von Mises and Angew, 1928), five
Variant selection and its effect on texture in Ti-6Al-4V

Independent slip systems are required for homogeneous plastic deformation of polycrystals (Von Mises and Angew, 1928). However, since only four $<1\overline{1} 0>$ slip systems are available in hcp metals, additional slip systems, either $<c + a>$ slip on pyramidal planes or twinning, need to be activated (Paton and Backofen, 1970, Anand, 2002). In pure titanium twinning is more readily activated than $<c + a>$ slip due to smaller critical resolve shear stress associated with twin systems compared to $<c + a>$ slip (Bieler T. R., 2005). The active deformation systems changes with temperature; at elevated temperature the deformation twinning is not activated and therefore cannot be the dominant deformation mechanism at high homologous temperature (Paton and Backofen, 1970, Anand, 2002). Therefore, slip in the $<a>$ and $<c + a>$ are dominant deformation mechanisms at elevated temperature (Paton and Backofen, 1970). On the other hand, at low homologous temperature twin systems are activated and become the dominant deformation mechanism. The slip systems and twinning are shown in Figure 1-7 and Figure 1-8.
Variant selection and its effect on texture in Ti-6Al-4V
1.2.3 Microstructural evolution of $\alpha + \beta$ alloy

The microstructures that are created during processing depend greatly on the processing temperature, annealing treatment and applied cooling rate with respect to $\beta$ transus (Lutjering, G, 1998). The heat treatment conditions are chosen based on the following reasons:

- Dislocation annihilation processes, which reduce internally generated stress and improve machinability and dimensional stability.
- Annealing and $\beta$ heat treatment followed by either fast cooling or using relatively very slow cooling rates aimed at optimising specific mechanical properties of this alloy, such as the fracture toughness and fatigue strength.
- A quench from the high temperature single $\beta$ phase provides a thermodynamic driving force for nanoscale rearrangement at low aging temperatures (Bieler, T. R 2005).

It is important to point out that commercial pure titanium CP Ti can be stress relieved but the strength level is not improved by heat treatment (Bieler, T. R 2005). In this section, annealing and $\beta$ heat treatment processes will be discussed.

1.2.3.1 Annealing

The annealing of titanium alloys within the $\alpha + \beta$ phase field can be classified into three types: mill annealing, duplex annealing and recrystallisation annealing:

Mill annealing

Mill annealing is generally carried out on every mill product in order to relieve internal stresses. However, the annealing process is usually not long enough to completely recrystallise the material (Lutjering, G, 1998) and most often for heavily...
worked sheet product traces of cold or hot worked microstructures are left behind, even after mill annealing (Donachie M. J. Jr., 2004).

**Duplex annealing**

In duplex annealing, the material is heated high enough into the $\alpha + \beta$ phase field and on cooling the $\beta$ phase at that temperature transforms to a lamellar $\alpha/\beta$ structure (Donachie M. J. Jr., 2004) generating significant volume fraction of the two phases to form a duplex microstructure. The resulting microstructure has improved damage tolerance over mill annealed microstructure (Donachie M. J. Jr., 2004).

**Recrystallisation annealing**

Recrystallisation annealing of Ti-6Al-4V alloy is done at temperatures high enough within the $\alpha + \beta$ phase field and held for hours in order to ensure development of equiaxed grains. It is then slowly cooled from the annealing temperature to grow the equiaxed primary $\alpha$ with some of the $\beta$ being retained at the triple junction and $\alpha-\alpha$ boundaries. The duplex annealing and recrystallisation annealing is very similar expect that the cooling rate is faster in duplex annealing compared to recrystallisation annealing. This recrystallisation annealing produce microstructure with improve fracture toughness and crack growth resistance of this alloy, damaged tolerance, fatigue performance and ductility compared to duplex annealed microstructure ((Donachie M. J. Jr., 2004, Bieler, T. R 2005). An example of an annealed equiaxed microstructure is illustrated in Figure 1-9.
The β heat treatment is done by heating the material above the β transus temperature into a single phase β; the α phase is completely dissolved at this temperature. During cooling, the high temperature β phase transforms back to α and part of the β phase is retained. The transformation can be either martensitic or diffusional depending on the cooling rate applied. The cooling rates for the transition from diffusional to martensitic varies from 1 °C/min, typical furnace cooling for diffusional transformation to very high cooling rate (water quenching), which is ~ 8000 °C/min for relatively thin size (< 10 mm thickness) and about 1000 °C/min (thick section) for martensitic transformation (Lutjering and Williams, 2003). Thus, the cooling rate required for martensitic transformation of large thickness section is rarely achieved during commercial production of Ti-6-Al-4V. However, medium cooling rate of ~
100 °C/min is typical for commercial cooling rates resulting in Widmanstätten microstructure. Details of these microstructures are discussed in the following section.

### 1.2.3.2 Martensitic Transformation

The martensitic transformation from the single β phase field can be achieved by applying very high cooling rates (water quenching) for relatively thin size (< 10 mm thickness). At relative high cooling rates (water quenching), from the β phase field, the β (bcc) to α (hcp) phase transition occurs by displacive transformation to form martensite (α′). Depending on the alloy content, the morphology of the α’ can be classified into two types, for example massive (packet) or acicular martensite (Lütjering, G 2003). The massive martensite microstructure comprises large irregular regions without any observable features using optical microscopy. Indeed, these regions consist of packets of small parallel α’ belonging to the same α variant. In contrast, a mixture of individual α’ plates each belonging to different Burger orientations dominate the acicular martensitic microstructure (Lütjering, G 2003). In addition, in the two phase alloy with high β stabilizing solutes, the transformation produces fundamental differences in the crystallographic orientation of the α product hexagonal structure with the orthorhombic structure (α’’). A typical martensitic microstructure is illustrated in Figure 1.10. The exact cooling rate that could lead to martensitic transformation in Ti-6Al-4V was identified by Ahmed and Rack (Ahmed and Rack 1998).
The modified Jominy method (ASTM; 1995) was used to study the deviation in the properties of Ti-6Al-4V resulting from different cooling rates applied from the single β phase field. The microstructures were analysed using optical and electron microscopy and the results obtained were utilised to design the α Time Temperature Transformation TTT diagram. The study showed that cooling rates above 400 °C/s are required for obtaining a fully martensitic microstructure. The schematic representation of TTT diagram is shown in Figure 1-11.
Figure 1-11 Time Temperature Transformation (TTT) diagram (Ahmed and Rack 1998).

1.2.3.3 Diffusional Transformation

When titanium alloys are cooled at relatively slow rates from the single $\beta$ phase field, the $\alpha$ phase nucleates preferentially at the prior $\beta$ grain boundaries forming a continuous layer of grain boundary $\alpha$ (GB$\alpha$). The orientation relationship of the grain boundary $\alpha$ with the $\beta$ grain at $\beta$ grain boundaries was reported by Stanford and Bate (Stanford and Bate 2004). They found that grain boundary $\alpha$ was quite inconsistent regarding how they relate with the $\beta$ grains at the $\beta$ grain boundary, i.e. in some cases, the GB$\alpha$ exhibit a Burgers orientation relationship with one of the $\beta$ grains at the $\beta$ grain boundary whereas in other cases such orientation relationship is not observed.

Similarly, during continuous cooling, $\alpha$ plates nucleate either on the interfaces of grain boundary $\alpha$ or on the $\beta$ grain boundary itself and grows into the $\beta$ grains as parallel plates until they are met by other $\alpha$ plates that nucleate from the other side of the boundary and which may belong to different variants of Burgers relation.
Variant selection and its effect on texture in Ti-6Al-4V (Lütjering, G 2003, and Ahmed and Rack 1998). The $\alpha$ plates that nucleate from the GB$\alpha$ tends to have low misorientations and are in Burgers orientation relationship with the $\beta$ phase, (i.e. $<1\overline{2}0>_{\alpha}||<111>_{\beta}$). Furthermore, with increasing cooling rates, the width of individual $\alpha$ plates become thinner and a smaller number $\alpha$ plates within the colony are observed. Thus, the number of $\alpha$ plates nucleating from either side of the $\beta$ grain boundaries or grain boundary $\alpha$ can no longer fill the grain interior. This can lead to nucleation of $\alpha$ not only on grain boundaries but also from the existing $\alpha$ plates in the grain interior. This microstructural morphology consisting of thinner $\alpha$ plates within the colonies is designated as “basket weave” or Widmanstätten structure (Lütjering, G 2003, Teixeira, J et al 2008). However, two- dimension (2D) micrographs may not provide enough information on the morphology and the shape of the $\alpha$ and as to reveal the location of nucleation and development of growth. This complexity has led to three- dimension (3D) microstructural analysis (Sharma H, et al., 2010).

A slower cooling rate would result in a lamellar microstructure, which consists of parallel plates of $\alpha$ separated by the retained $\beta$ matrix between them. Lamellar microstructure and the sequence of diffusional transformation are exemplified in Figure 1-12. One of the drawbacks of this schematic (Figure 1-12) is that it does not show the formation of grain boundary $\alpha$. It has been shown recently that the early $\alpha$ that develop from the grain boundary $\alpha$ precipitates in Ti-6Al-4V on both sides of the grain boundary have similar crystallographic orientation (Obasi G. C et al., 2011). The $\alpha$ plates develop and grow in opposite direction into the $\beta$ grain matrix. The grain boundary length-scale of these plates is dependent on the extent of the grain growth.
Large grain size has large grain boundary length-scale compared to small grain size (Obasi G. C et al., 2011).

Figure 1-12 Microtextural evolution during diffusional transformation (Donachie M. J. Jr., 2004).
Chapter 2  **Texture and Variant Selection**

2.1  **Texture**

Metallic materials, like titanium and its alloys, are composed generally of crystalline grains. In perfectly recrystallised condition each grain has a single crystallographic orientation. The orientation of the crystal relative to a reference frame defines the crystallographic orientation of that crystal. Grain orientation distributions within the polycrystal are rarely random due to the processing history to which the material has been subjected, such as casting and solidification from melt as well as thermomechanical processes. The alignment of these crystals during material processing is termed texture.

Texture has significant influence on the mechanical and physical properties of materials because of the anisotropic properties of individual grains. According to Bunge (Bunge H. J, 1982), about 20-50% of the physical and mechanical properties, such as Young’s modulus, Poisson’s ratio, strength, ductility, toughness, magnetic permeability, electrical conductivity and thermal expansion, are affected by the crystallographic texture of a material.

In the following sections, the basic concept of texture is described which form background knowledge applied to study the texture evolution and variant selection studies in the subsequent chapter.
2.1.1 Texture representation and analysis

This section focuses on the description of texture representation, orientations and the basic descriptor of crystal orientations and their applications. In order to effectively describe the crystal orientation, two coordinate systems are required: the specimen coordinate system, for example (RD, TD, ND) representing the rolling, transverse and normal directions respectively) for rolled products and the crystal coordinate system, ([100], [010] [001]) directions for bcc and [0002], [10 10], [11 20] for hcp (Randle and Engler, 2000).

2.1.1.1 Coordinate system

The specimen coordinate system is specified by the direction of the external shape of the specimen; for a specimen deformed by rolling, the directions associated with the external shape of specimen are the rolling direction (RD), the direction normal to the plane (ND) and the transverse direction (TD). These directions are Cartesian i.e. at right angles to each other. The three (RD, ND, and TD) axes notation is only applicable for rolled products; for deformed specimen with uniaxial symmetry only one axis is specified in the specimen coordinate system while the other two are arbitrarily chosen (Randle and Engler, 2000). Figure 2-1 illustrates the direction of rolled specimen.
Texture and Variant Selection

The crystal coordinate system is specified by directions in the crystal. The choice of the coordinate system depends on the crystal symmetry. For orthogonal symmetry (cubic, tetragonal and orthorhombic) the axes [100], [010], [001] coordinate system is chosen because it forms an orthogonal frame (Randle and Engler, 2000). This coordinate system is used for β titanium due its body centre cubic crystal structure. For hexagonal close packed crystal symmetry, the α titanium, four index crystal coordinate system is used. The axes are [1010], [1210], [0001], and are normalised (orthonormal) to achieved the same length. This is done by multiplying the zone axis, reference to the crystal coordinate system, with the transformation matrix, \( M \). The transformation matrix according to (Randle and Engler, 2000) is given in equation 2.1.

\[
M = \begin{pmatrix}
a & -a/2 & 0 \\
0 & (a\sqrt{3})/2 & 0 \\
0 & 0 & c
\end{pmatrix}
\]

Figure 2-1 Sample coordinate systems for rolled products
Where \( a \) and \( c \) are lattice parameters. Reverting back to the crystal reference system is by simply reversing the initial transformation of the coordinate i.e. by multiplying by the inverse matrix \( (M^{-1}) \). The orthonormalised crystal coordinate systems of body centered cubic and hexagonal close packed systems are depicted in Figure 2-2.

![Figure 2-2 Orthonormalised crystal coordinate systems (a) cubic, (b) hexagonal](image)

### 2.1.1.2 Descriptors of orientation

The crystallographic orientation is defined as position of crystal coordinate system with reference to the specimen coordinate system. The orientations are expressed in three dimensional orientation space and for complete description of orientations, three independent variables are required. The variables can be expressed in different ways resulting in different orientation descriptor (Randle and Engler, 2000). Orientation descriptors are discussed in detail in this section.
2.1.1.3 Miller and Bravais Indices Notation

The Miller and Bravais indices are commonly used to represent crystallographic directions and planes of a crystal with respect to the reference frame. For instance, the crystallographic orientation in rolled sheets of material with a body centre cubic or hexagonal close packed crystal structure is represented by \{hkl\}<uvw> and \{hkil\}<uvtw> for body centre cubic and hexagonal close pack materials respectively. For hexagonal metals this indicates that \{hkil\} of these grains lie parallel to the sheet plane (ND), whereas their <uvtw> point parallel to the rolling direction (RD) (Wang and Huang 2003).

2.1.1.4 Euler angles

The Euler angles describe the sequence of rotations of a crystal with respect to a coordinate system. The rotation converts the specimen coordinate system onto the crystal coordinate system, which identifies the orientation. There have been two most commonly used notations in literatures to describe Euler angles, Roe and Bunge notations (Roe R. J. 1965 and Bunge, 1982). In this study, the convention formulated by Bunge (Bunge, 1982) is used. The sequence of rotations is illustrated in Figure 2-3 and shows the rotation between the specimen and crystal axes.

1. A rotation of $\phi_1$ about the $Z_1$ axis (ND) transforms the $X_1$ axis (TD) to $X_1'$ (TD') and $Y_1$ (RD) to $Y_1'$ (RD')

2. A rotation of $\Phi$ about the $X_1'$ axis, will transform the $Z_1$ axis into $Z_1''$ and $Y_1$ axis into $Y_1''$. 
(3) A rotation of $\phi_2$ about the rotated axis $Z_1''$, converts $Y_1$ into $Y_1''$ and $X_1'$ into $X_1''$

Where $\phi_1$, $\Phi$, $\phi_2$, are the Euler angles (Bunge notation). The three Euler angles can be expressed as points in three dimensional coordinate systems called the Euler space. Euler space is often used to present orientation distribution function (ODFs), which are frequently used to describe texture (Randle and Engler, 2000).

![Figure 2-3 Schematic representation of the three Euler angles rotation ($\phi1$, $\Phi$, $\phi2$) using Bunge notation](image)

### 2.1.1.5 Rotation matrix

Rotation matrix is another way in which orientation can be described. The matrix is expressed as a 3x3 square matrix that describes the rotation of orientation ($g$). The first row of the matrix is given by the cosines of the angles $\alpha_1$, $\beta_1$ and $\gamma_1$ between the first crystal axis $C_c[100]$, and each of the sample axes $X_1$, $Y_1$, and $Z_1$ as shown in Figure 2‐4. Similarly, the second row of the matrix is given by cosines of the angles $\alpha_2$, $\beta_2$ and $\gamma_2$ between the second crystal axes $C_c[010]$, and each of the three sample axes. $X_1$, $Y_1$, and $Z_1$. The third row of the matrix comprises of the cosines of the angles $\alpha_3$, $\beta_3$ and $\gamma_3$ between the third crystal axes $C_c[001]$, and each of the three sample axes $X_1$, $Y_1$, and $Z_1$ (Randle and Engler, 2000). The orientation matrix is illustrated in equation 2.2.
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Figure 2-4 Schematic representation of the relationship between crystal and specimen coordinate systems

\[
g = \begin{pmatrix} g_{11} & g_{12} & g_{13} \\ g_{21} & g_{22} & g_{23} \\ g_{31} & g_{32} & g_{33} \end{pmatrix} = \begin{pmatrix} \cos \alpha_1 & \cos \beta_1 & \cos \gamma_1 \\ \cos \alpha_2 & \cos \beta_2 & \cos \gamma_2 \\ \cos \alpha_3 & \cos \beta_3 & \cos \gamma_3 \end{pmatrix}
\]  

(2.2)

Where \( g \) is the orientation and \( \alpha, \beta \) and \( \gamma \) are the angles between crystal axes respectively.

The equation expresses the orientation matrix in terms of direction cosines. The rotation matrix has unique mathematical applications; the matrix is orthonormal i.e. the row and column of the matrix are unit vectors. The 3x3 matrix has nine numbers but only three independent variables are required to represent orientations, which show that there are non-independent variables in the matrix. The cross product of any two rows or columns gives the third and for any row or columns the sum of the squares of the three elements of the matrix is equal to one. One of the significant properties of orientation matrix is that it can be used to calculate all other descriptors of orientation.
The elements of the orientation matrix, as expressed in the equation (2.2), can be expressed in terms of Euler angles (Randle and Engler, 2000).

\[
\begin{align*}
    g_{11} &= \cos \phi_1 \cos \phi_2 - \sin \phi_1 \sin \phi_2 \cos \phi \\
    g_{12} &= \sin \phi_1 \cos \phi_2 + \cos \phi_1 \sin \phi_2 \cos \phi \\
    g_{13} &= \sin \phi_2 \sin \phi \\
    g_{21} &= -\cos \phi_1 \sin \phi_2 - \sin \phi_1 \cos \phi_2 \cos \phi \\
    g_{22} &= -\sin \phi_1 \sin \phi_2 + \cos \phi_1 \cos \phi_2 \cos \phi \\
    g_{23} &= \cos \phi_2 \sin \phi \\
    g_{31} &= \sin \phi_1 \sin \phi \\
    g_{32} &= -\cos \phi_1 \sin \phi \\
    g_{33} &= \cos \phi
\end{align*}
\] (2.3)

### 2.1.1.6 Axis/Angle

Orientation can be represented as a rotation of a crystal about specific axis. This is called axis/angle pair, this descriptor utilises the orientation of a neighbour crystal or grain as the reference orientation rather than the specimen coordinate system. In that case the misorientation between the two crystals can be represented easily by a common axis and an angle (Heinz and Neumann 1991).

### 2.1.2 Macrotexture representation

The discussions covered so far have been on mathematical representation of single orientation such as Euler angles and orientation matrix. These mathematical expressions are often presented in graphical form and projections representing statistical orientation distribution obtained from many different grains in terms of pole figures (PF), inverse pole figures (IPF) and orientation distribution functions (ODFs).
In this section, these three most widely used methods of representing the average crystallographic orientation are presented.

### 2.1.2.1 Pole Figures

Pole figure is one of the commonly used methods of texture representation. A pole figure is a two-dimensional stereographic projection of a unit sphere on the equatorial plane, which shows the variation of pole density with pole orientation for a selected set of crystal planes with respect to a reference direction. In constructing a pole figure, the poles from the lattice plane are projected in an imaginary reference sphere, referred to as the unit sphere, residing around the crystal. A point on the surface represents the crystallographic orientation of the crystal with respect to reference direction; it represents plane normal direction. This is illustrated for (0001) planes in a hexagonal crystal in Figure 2-5.
The position of a pole in the stereographic projection is given by two angles $\alpha$ and $\beta$. The $\alpha$ is the azimuth and $\beta$ describes the rotation of the pole around the polar axis with reference to the directions (Randle and Engler 20001). To characterise the crystallographic orientation of the crystal in Figure 2-5, the distribution of the corresponding poles in terms of the angles $\alpha$ and $\beta$ relate to the external reference direction. For instance in the case of a rolling symmetry, the RD is usually the north pole of the sphere, so that $\alpha = 0^\circ$, while the rotation angle $\beta = 0^\circ$ is associated with the ND.
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Figure 2-6 Stereographic projection (a) two-dimensional, (b) equal area (Wenk and Knocks, 1987).

The equal area projection is another type of projection other than two-dimensional stereographic projection. The advantage of equal area projection over two-dimensional projection is that the distribution of a set of random points on the surface of the sphere appears uniform in equal area projection, whereas in the case of two-dimensional projection it is concentrated at the centre and at the periphery as illustrated in Figure 2-6. One of the drawbacks of pole figures is that it is a semi quantitative representation of texture. However, when the texture is less complex, a series of pole figures can be used as quantitative representation of texture (Wang and Huang 2003). The (0002) and (110) pole figures in Figure 2-7 describes a typical recrystallisation textures in Ti-6Al-4V generated by uni-axial rolling the material at 950 ºC followed by heat treating the material at the same temperature. In order to compare pole figures of different samples, the orientation densities must be standardised to units that are non-independent on the experimental parameters (Wenk and Knocks, 1987). The intensity of pole distributions is visualised in term of contours and colour and is expressed as multiples of random distributions (mrd).
2.1.2.2 *Inverse pole figures (IPF)*

The deformation textures of materials deformed by uniaxial deformation modes are usually fibre textures and the results are mostly represented as inverse pole figures. The inverse pole figures are projection of pole density in a stereographic triangle. They are most useful for axisymmetric samples which require a specification of only a single axis (Humphreys, F. Y 1996). Inverse pole figures can also describe the rolling texture, but in this case, each of the specimen principal strain axes (RD, TD and ND) will be required. The inverse pole figures show the density variation of three planes in the stereographic triangle, in contrast to PF, which shows only the density of the specific crystallographic plane. Three inverse pole figures (for RD, ND, and TD) based on the same data as presented in Figure 2-7, are shown in Figure 2-8.
2.1.2.3 **Orientation Distribution Function (ODF)**

As discussed earlier in (section 2.5.1), the information contained in a single pole figure is incomplete. This is due to the fact that the entire pole figure range is not covered during measurements using x-ray diffraction. Secondly, the display of three-
dimensional information on two-dimensional projection plane further reduces texture information available in pole figures (Wang and Huang 2003). For quantitative description of texture, a three-dimensional projection i.e. orientation distribution function (ODFs) can overcome these shortcomings of pole figure representation. Orientation distribution functions (ODFs) describes the frequency of occurrence of particular orientations in three dimensional (Euler) orientation space (Randle and Engler, 2000). This space is defined by the three Euler angles ($\varphi_1$, $\Phi$, $\varphi_2$), which represent a set of three successive rotations that bring the crystal axes into coincidence with the specimen axes (RD, TD and ND) as shown in Figure 2-3.

The same $\alpha$ texture presented in Figure 2-7 is quantified within the framework of three dimensional space by imposing orthotropic sample symmetry as shown in $\varphi_2$ sections of ODFs in Figure 2-9. It can be seen that in Figure 2-9 the orientation density in the ODF ($\varphi_2$ sections) do not vary within the range of 5 °. The orientation density at 0 ° and 60 ° are similar due to crystal symmetry of hexagonal materials, i.e. for every 60 ° rotation the same section of the ODF is obtain, which in principle should have the same texture components. In this study the $\varphi_2$= 0° section of the ODF was used to represent the $\alpha$ texture evolution during thermomechanical processing of Ti-6Al-4V. The reason why $\varphi_2$= 0° section was used to represent the $\alpha$ textures is due to fact that the orientations with relatively high density were included in this section. Therefore, it can be used to sufficiently describe texture evolution in this material.
Figure 2-9 ODF sections of α texture of Ti-6Al-4V recrystallised at 950 °C for 8 h and cooled at 1 °C/min

Figure 2-10 shows the schematic representation of the hexagonal crystal in different part of $\varphi_2= 0$ section of the ODF and how they related to the (0002) pole figure.
In the case of $\beta$ phase, an example of ODF sections is shown in Figure 2-11. Again, orientation density is found to be concentrated in a specific section of the ODF. It can be seen from the figures that relatively high orientations density were included in $\varphi_2 = 45^\circ$. This suggests that $\varphi_2 = 45^\circ$ section of ODF can sufficiently represent the $\beta$ texture. This justifies the reason why $\varphi_2 = 45^\circ$ was used in $\beta$ texture representation in this study.

Figure 2-10 Schematic representation of orientation of the hexagonal crystals in different part of the ODF ($\varphi_2=0$) section and (0002) pole figure
Determination of Orientation distribution function from pole figures

In the conventional X-ray texture measurements, the direct measurement of ODFs is not possible because the method is only capable of determining pole figures. However, mathematical methods have been developed that allow calculation of ODFs from several numerical pole figure data obtained from the conventional texture measurement. Such calculation is referred to as pole figure inversion (Kallend, J. S., 1998). It is important to note that the number of pole figures required for ODF calculations depends on crystal symmetry of the sample. Several approaches have been developed to calculate ODFs, but the most commonly used are those proposed independently by Bunge and Roe, the harmonic and direct methods. The harmonic method computation performed using Fourier space will be discussed here. A
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detailed mathematical analysis of the direct method can be found in Bunge, (Bunge, 1982).

Harmonic method

The principle behind the harmonic method is based on the fitting of a series expansion to pole figure data and ODF. The most appropriate series expansion function for the computation of ODF is the spherical harmonic function for a spherical coordinate system, which is the case of the orientation data obtained from the pole figure. The basic principles of series expansion to compute ODF as proposed by Bunge (Bunge, 1982) are outline below. According (Bunge, 1982), an ODF can be expressed as a series of generalised spherical harmonic functions,

$$ f(\varphi_1 \Phi \varphi_2) = \sum_{l=0}^{\infty} \sum_{m=1}^{l(l+1)} \sum_{m'=1}^{l(l+1)} c_{l m}^m T_l^m(\varphi_1 \Phi \varphi_2) $$

2.4

Where \((\varphi_1 \Phi \varphi_2)\) is the crystal orientation, \(c_{l m}^m\) are the series expansion coefficients that describe the ODF and \(T_l^m(\varphi_1 \Phi \varphi_2)\) are spherical harmonic functions.

The two angles \(\alpha\) and \(\beta\), in a given two-dimensional pole figure \(p(\alpha, \beta)\) which correspond to the region in the three-dimensional ODF \(f(\varphi_1 \Phi \varphi_2)\) can be expanded in series expansion in form of equation 2.5,

$$ P(\alpha \beta) = \sum_{l=0}^{\infty} \sum_{n=1}^{l(l+1)} F_l^n K_l^n(\alpha, \beta) $$

2.5

Where \(F_l^n\) are the pole figure coefficients, while \(K_l^n(\alpha, \beta)\) are the harmonic functions.

The pole figure coefficients \((F_l^n)\) and the series expansion coefficients that describe the ODF \((c_{l m}^m)\) are related by equation 2.6.
\[ F_1^n = \frac{4\pi}{2l+1} \sum_0^{l(l)} C_l^{\mu\nu} K_l^{\mu\nu} (\lambda, \chi) \]

\((\lambda, \chi)\) is the polar coordinate of a pole in the crystal coordinate system and the asterix indicates complex-conjugate quantity. This equation forms a system of linear equations, which can be used to obtain the coefficient \(C_l^{\mu\nu}\). The number of pole figures required for this calculation depends on the sample symmetry being measured. For example, hcp materials as in case of titanium require five pole figures to be measured whereas three to four are sufficient for materials with a cubic crystal structure (Kallend, J. S 1998). The problem associated with this method is the so-called ghost error, the false maxima and minima created due to lack of odd-order series expansion coefficients. Additional calculations are required to correct the ghost error. These methods have been developed into software tools to calculate ODFs from pole figures. An example of such software is POPLA (Kallend J. S. et al., 1991).
2.2 **Texture evolution during thermomechanical processing**

In this section, texture evolution (*i.e.* deformation, recrystallisation and transformation texture), during thermomechanical processing of hexagonal metals and their alloys, is presented with particular emphasis on titanium alloys. The evolution of texture strongly depends on deformation modes (*e.g.* rolling, extrusion), deformation temperature, percentage thickness reduction, solute content, second phases and grain size. Additional contributory factors include phase volume fraction, strain rate, annealing condition, heating and cooling rates, preheat temperature and inter-pass reheating.

### 2.2.1 Deformation texture (Rolling texture)

Deformation of polycrystalline materials is accompanied by dislocation movement along slip systems, changes in grain shape and orientation, grain rotation and internally generated stress. The combinations of these factors make the understanding of texture development rather complex. The evolution of deformation texture in hcp materials during processing has been reviewed by Humphreys and Wang (Humphreys, F. Y 1996 and Wang and Hua ng 2003). During cold rolling of hcp metals, the orientation of the basal pole (0002) has been ascribed to combination of active slip and twinning. However, it should be pointed out that the (0002) orientation pole figure is not consistent in all hcp metals due to difference in c/a ratios. As a result of this variation in the (0002) orientation pole figure, the deformation textures in hcp metals have been classified into three groups based on their c/a ratio and active deformation systems. This is illustrated in Figure 2-12.
showing different texture obtained during rolling. As reviewed by Humphreys, in metals with c/a ratios greater than 1.633 (e.g., Zn and Cd), the position of the basal poles is tilted 20-30° away from the normal direction (ND) towards the rolling direction (RD). This texture is developed as a result of the combination of basal slip and twinning (Humphreys, F. Y 1996).

For metals with c/a ratios close to the ideal value of 1.633, such as Mg, the rolling texture tends to form a strong texture component concentrated at the normal direction (\{0002\} \langle1100\rangle), which is due to twinning and basal slip activity (Wang and Huang 2003). Metals with c/a ratios less than the ideal, 1.633, such as titanium and zirconium, the basal plane is rotated by 30-40° away from the ND towards the transverse direction (TD). The formation of this type of texture is ascribed to twinning and slip on prismatic planes (Wang and Huang 2003).
Figure 2-12 Schematic representations of pole figures of hexagonal metals according to their c/a ratios (Wang and Huang, 2003)

The simulation of rolling texture carried out by Wang et al. (Wang and Huang, 2003) is in agreement with the work by Humphreys (Humphreys, F. Y 1996). The list of commonly observed operating deformation systems in hcp metals is shown in Table 2-1.
The table clearly indicates the predominant active slip systems during deformation, which led to evolution of deformation texture in hcp metals. The primary deformation mode in titanium is prismatic \( <11 \bar{2}0> \) slip, which means the critical resolve shear stress is lowest in the prismatic plane \( \{10\bar{1}0\} \). The deformation textures appear slightly different for different material compositions. The effect of the alloying element and dominant deformation mechanism according to Tenckhoff (Tenckhoff, E., 1980) is illustrated in Figure 2-13; three types of textures components were proposed c-type, r-type and t-type. The c-type is a fibre texture component with their \( (0002) \) poles parallel to the normal direction. The r-type has their \( (0002) \) poles titled about 10º away from normal towards the rolling direction. In t-type, the \( (0002) \) poles are tilted towards the transverse direction and the \( (10\bar{1}0) \) lying parallel to rolling.
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direction. Zaefferer (Zaefferer, S., 2003) related the observed texture based on the solute content, active slip and twinning systems. Three titanium alloys, two CP-Ti with different oxygen contents (1000 ppm and 2000 ppm) and Ti-6Al-4V, were used for this investigation. Zaefferer (Zaefferer, S., 2003) suggested that the activities of different deformation mechanisms are reflected in the different positions of (0002) poles. He reported that considerable activity of basal glide and absence of both $<c + a>$ glide and twinning are responsible for the formation of the c-type and r-type deformation texture. This finding contradicts the findings of Humphreys and Wang (Humphreys, F. Y 1996 and Wang and Huang 2003).

Zaefferer (Zaefferer, S., 2003) also demonstrated that alloying elements such as oxygen has significant influence on the deformation texture formation and slip and twinning activities in hcp metals. In titanium with low oxygen content of 1000 ppm, the observed t-texture is well developed. Formation of the t-type was ascribed to high activity of twinning due to low oxygen content and $<c + a>$ glide. On the other hand, in the alloy with 2000 ppm oxygen (i.e. higher oxygen content), twinning was completely suppressed. Therefore, the t-type deformation texture formed is less developed. This is mainly due to the absence of twinning and $<c + a>$ glide and increased activity of the basal glide (Zaefferer, S., 2003).
The influence of volume fraction of constituent phases on deformation texture development in two phase alloys (Ti-6Al-4V) has been reported by several workers (Dunst, D 1994, Morii, K 1986, Inagaki, H, 1990, and Peters, M 1980). Since volume fraction of the constituent phases depend on the temperature and the chemical composition of the alloy, Inagaki (Inagaki, H., 1990) illustrated the volume fraction effect on the deformation texture evolution by investigating Ti alloys deformed by hot rolling at four different temperature regimes within the $\alpha + \beta$ phase and above the $\beta$
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transus temperature. The result is exemplified in the form of ODFs ($\Phi=30^\circ$) and (0002) pole figures depicted in Figure 2-14 and Figure 2-15.

Figure 2-14 ODF representation showing the effect of deformation at *viz.* the volume fraction of $\beta$ phase (Inagaki, H., 1990)
The same type of textures were observed for materials deformed at temperature below 900 °C; the volume fraction of the β phase at temperatures below 900 °C is estimated to be less than 40 vol. % and therefore it was postulated that the deformation of material is governed by α phase. On the other hand, at temperature above 900 °C when the volume fraction of β exceeds the α volume fraction, the deformation texture changes very significantly. With large β phase volume fraction, the deformation at this temperature is expected to be controlled by the β phase and hence the contribution of the β phase to the deformation texture evolution is expected to be dominant (Inagaki, H. 1990).
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It is difficult to compare the (0002) pole figure observed by Inagaki H (Inagaki, H. 1990) with the typical (0002) pole figures formed during uni-directional hot rolling at temperature below 900 °C and above as reported in other studies (Morii, K 1986 and Peters, M 1980). Due to no clear description of the nature of the rolling processes in their study. Figure 2-16 exemplified a typical (0002) pole figure observed during uni-directional rolling of Ti-6Al-4V at temperatures below 900 °C and above obtained in this study, which is in agreement with the work of Morii, K and Peters, M (Morii, K 1986 and Peters, M 1980). From Figure 2-16 the uni-directional rolling at temperature below 900 °C (800 °C) produced slightly stronger deformation texture than the texture above 900 °C. This observation is not completely understood.

Figure 2-16 Pole figure representation of samples rolled at 800 °C and 950 °C.

Morii and Peters (Peters, M., 1980 and Morii, K., 1986) have also reported similar texture evolution at different temperatures. Morii suggested that due to a small volume fraction of the β phase below 900 °C, the α grains are in contact with each other and could impose constraints as in single phase materials during deformation. Thus, slip of these grains occur by combined slip systems of <a> and < c + a >.
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However, during deformation above 900 °C the volume fraction of β exceeds the α phase; as expected the α grains are completely separated from each other. This tends to remove the constraint imposed in the material deformed below 900 °C. Therefore, slip might occur on <a> -systems only.

The argument presented here by Morii (Morii, K 1986) is supported by the work of Dunst (Dunst, D 1994) who reported a similar influence of increasing β phase on deformation texture evolution, even though the deformation temperature was kept constant at 800 °C. Dunst (Dunst, D 1994) investigated four different specimens of Ti-Al-V, with different volume fractions of the β phase (4 %, 22 %, 64 % and 82 %) uni-directional rolled at 800 °C. The variation in the β phase volume fractions were obtained by varying the Al and V composition of the alloy. Their findings showed that the major texture components in specimens with 4 % β phase are two fibre textures with (0002) parallel to ND and (0002) scattered 20° around ND with an additional weak orientation component of \{1\overline{2}14\}<\overline{1}010>. This is a typical single phase α-titanium alloy deformation texture, which is a product of the basal deformation mode (Morii, K 1986, Humphreys, F. Y 1996 and Wang and Huang, 2003). Increasing β phase volume fraction (22 % β), resulted in the formation of new texture components: \(<\overline{1}010>/>\text{RD fibre and orientation tubes were found, i.e. the } \{1\overline{2}10\}<\overline{1}010>, \{1\overline{2}13\}<\overline{1}010> \text{ texture components and a minor component } \{1\overline{2}17\}<\overline{1}010> \text{ fibre. The } \{1\overline{2}10\}<\overline{1}010> \text{ component is sharper compared to the } \{1\overline{2}13\}<\overline{1}010> \text{ texture component. Simulation of the } \langle\overline{1}010>/>\text{RD fibre texture revealed that slip on all }<\text{a}> \text{ modes is responsible for this texture development. Again this result corroborates the results of Morii obtained during deformation at a higher
temperature (900 °C); he reported increased volume fraction of β increased slip activity in all the <a> modes. The presence of increased β phase means that deformation of the rather anisotropic α phase no longer requires five independent slip systems, since incompatibilities can be accommodated easily by strains in the more isotropic β. This mechanism justifies the difference between α textures in two phase alloys and those present in single phase alloys.

The effect of thickness reduction and cooling rate after deformation on the development of deformation texture in both α and α + β alloys was demonstrated by (Inagaki, H, 1990, Tanabe, A 1980 and Wagner, F., 2002). It was suggested that the peak position of the main texture components changes as reduction in thickness increases; at lower thickness reduction (73 %), the texture is characterised by texture peaks located 20 ° away from the ND towards the RD in (0002) pole figures. At reduction above 86 %, the main texture is located at 30 ° from the ND towards the RD in (0002) (Tanabe, A., 1980). The justification given is that recrystallisation seems more pronounced in higher thickness reduction. A more accurate effect of the thickness reduction on deformation texture in both α and α + β alloys were presented in the form of ODFs by (Inagaki, H, 1990); the argument is that the texture components located 20 ° and 30 ° away from ND towards the RD and TD direction in (0002) pole figure might include other texture components which are not part of the deformation textures. The ODF precisely identifies orientation changes at different thickness reductions; for instance, the (2111)[2211], corresponding to texture component 20 ° away from ND to RD at 73 % reduction, is replaced by (2115)[0110] which is similar to texture component 30 ° away from ND to TD at 86 % reduction. At 50 % thickness reduction, the texture intensity is quite weak. There was no significant change in deformation texture due to different cooling rates except that
fast cooling rates of specimens, deformed at temperature above 900 °C, degrade the intensity of deformation texture. However, the specimens deformed at lower temperature are insensitive to cooling rates (Inagaki, H, 1990).

The β deformation texture has been studied by number of authors, (Dunst, D 1994, Morii, K 1986, Inagaki, H, 1990, Wenk, H-R 2004 and Inoue, H 1992). The development in β phase is greatly affected by rolling reduction in terms of the orientation density (Inoue, H 1992). In general, the main deformation texture components observed for the β phase are \{001\}<110>, \{112\}<110> and \{111\}<211> texture components, which belong to the so-called α-fibre (<110>//RD) and γ fibre texture (<111>//ND) usually observed in rolled bcc metals. The γ fibre texture is strengthened with increasing volume fraction of the β phase (Dunst, D 1994). The most dominant slip mode in bcc metals is slip by, \{110\} <111> (Wenk, H-R 2004). The deformation and annealing texture of bcc metals are shown in the \(\varphi_2\) section of the ODF in Table 2-2 and Figure 2-17 highlights the α-fibre (<110>//RD) and γ fibre texture (<111>//ND) texture.
Table 2-2 Deformation texture components in bcc metals (β titanium) (Wenk, H-R 2004).

<table>
<thead>
<tr>
<th>Texture component</th>
<th>Miller indices {hkl}&lt;uvw&gt;</th>
<th>Code</th>
<th>Euler angles (º) (Bunge notation)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube</td>
<td>{001}&lt;100&gt;</td>
<td>B</td>
<td>φ₁  45  Φ  0  φ₂  45</td>
</tr>
<tr>
<td>Rotated cube</td>
<td>{001}&lt;110&gt;</td>
<td>A</td>
<td>φ₁  0   Φ  0   φ₂  45</td>
</tr>
<tr>
<td></td>
<td>{111}&lt;110&gt;</td>
<td>Z₁</td>
<td></td>
</tr>
<tr>
<td></td>
<td>{111}&lt;110&gt;</td>
<td>Z₂</td>
<td></td>
</tr>
<tr>
<td></td>
<td>{111}&lt;11₂&gt;</td>
<td>L₁</td>
<td></td>
</tr>
<tr>
<td></td>
<td>{111}&lt;11₂&gt;</td>
<td>L₂</td>
<td></td>
</tr>
<tr>
<td></td>
<td>{112}&lt;11₀&gt;</td>
<td>I</td>
<td></td>
</tr>
<tr>
<td>Goss</td>
<td>{110}&lt;001&gt;</td>
<td>D</td>
<td>φ₁  90  Φ  90  φ₂  45</td>
</tr>
</tbody>
</table>

Figure 2-17 Schematic representation of the deformation texture in (β titanium) bcc metals.
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The texture in this study is presented using orthotropic sample symmetry; this is justified by comparing the ODF plotted in $0^\circ \leq \varphi_1 \leq 90^\circ$ section (orthotropic) and $0^\circ \leq \varphi_1 \leq 360^\circ$ (without symmetry). The Figure clearly demonstrated that the orientations found in this $0^\circ \leq \varphi_1 \leq 90^\circ$ section of the ODF is replicated in the remaining sections of the $0^\circ \leq \varphi_1 \leq 360^\circ$. Therefore, orthotropic sample symmetry can be applied without excluding any orientation in the Euler space.

The review so far has only centered on deformation texture evolution due to uni-directional rolling. Deformation modes other than uni-directional rolling (e.g. cross rolling and extrusion) produce a different kind of deformation texture from those presented earlier. Figure 2-19, illustrates the discrepancies observed in the deformation texture due to cross rolling. It can be seen that the transverse part of the mixed basal/transverse texture observed in Figure 2-19a in the case of rolling at 800
°C was completely suppressed during cross rolling at 800 °C. However, cross rolling at 980 °C appears to re-introduce the basal component that was absent in the (0002) pole figure during uni-directional rolling above 900 °C. Peters M., et al., (Peters, M 1980) suggested that by changing the rolling direction by 90 ° the basal planes responsible for transverse texture might be in an unfavourable positions. This argument is not completely convincing, since one has to consider the temperature gradient during the rolling process. Keeping in mind that if the temperature falls much below the processing temperature can lead to such observed effect.
Figure 2-19 Pole figure representation of uni-directional rolling (800 and 960 °C) and cross rolled samples at 800 °C and 980 °C (Peters, M 1980).

Furthermore, a different deformation texture is observed during uniaxial extrusion of hexagonal metals. The deformation process results in a formation of the fibre texture i.e. the so-called “cylindrical texture” (U. F. Kocks and Wang), the basal plane normals \{0001\} of the fibre are perpendicular to the extrusion axis. The fibre textures are commonly represented as inverse pole figures. Similarly, in β titanium, the effect of processing methods was also observed. Cross rolled β titanium showed some kind discrepancies compared to uni-directionally rolled specimen (Singh, A. K 1999).
2.2.2 Recrystallisation texture

Recrystallisation is the nucleation and growth of new grain structure from the deformed grain structure, which can lead to dramatic changes in texture. In bcc and face centred cubic (fcc) metals, there is an established relationship between the deformed and recrystallised texture; for example a~25° rotation around the <110> direction for bcc and 30-40° rotation around <111> direction for fcc is observed (Bunge, 1982). However, there have been few reports of crystallographic relationships between deformed and recrystallisation texture in hexagonal metals, e.g. a rotation of ~30° around <0001> and ~90° around <10 0> (Engler, O 1999). Earlier reports suggested no significant changes in rolling texture during recrystallisation of hexagonal metals (Barrett, C. S. and Massalski, T. B. 1980). This was reinforced to a large extent by Wagner (Wagner, F., 2002) who studied the evolution of texture in low alloyed titanium. This study revealed that the rolling texture was the dominant texture after recrystallisation with additional components. The observed texture change was due to continuous recrystallisation or extended recovery, which was mainly influenced by the formation of subgrains via dislocation rearrangement and coarsening of cells and subgrains. Unlike hcp materials, recovery in bcc usually initiates nucleation of new grains, which subsequently grow with large misorientation within the matrix, resulting in a significant texture change. However, the slight texture variation can be attributed to selective grain growth, which is influenced, by texture inheritance and microstructural development during the recrystallisation stage (Wagner, F. 2002). Similarly, in two phase titanium alloys, the rolling texture is similar to the texture after recrystallisation (Shang, S 2002). Although the texture
strengthened during recrystallisation, the overall texture is similar as exemplified in the rolled and recrystallisation texture in Figure 2-20 obtained in the present study.

![Figure 2-20 The ODF of the deformation and recrystallisation texture](image)

Finally, from the findings so far in the literature, the deformation texture evolution in titanium alloys is a combination of intrinsic and extrinsic factors, which is determined by the active deformation systems. The detail literature review demonstrates that the mechanisms of texture evolution in Ti alloys are still poorly understood.

### 2.2.3 Phase transformation and variant selection

This section addresses the impact of phase transformation occurring in hexagonal metallic materials with specific reference to the phenomenon of “variant selection” occurring during phase transformation. Some metallic materials undergo phase transformation when subjected to certain temperatures and pressure. In most cases, the product phase has an orientation relationship with the parent phase. Depending on the chemical composition and cooling rate from elevated temperature, the transformation of titanium and its alloys may follow martensitic or diffusional-
Texture and Variant Selection

controlled nucleation and growth as discussed earlier in section 1.2.3.2 and 1.2.3.3 respectively. In most cases, the phase transformation proceeds with a definite orientation relationship between the mother and daughter phase, *i.e.* the so-called Burgers orientation relationship (Burgers, W. G., 1934). The Burgers orientation relationship is illustrated in Figure 2-21.

![Burgers orientation relationship](image)

**Figure 2-21 The Burgers orientation relationship**

There are six β orientations that can transform from single α and twelve possible α variants that can transform from a single parent β grain. Thus, theoretically there are total of fifty seven possible variants during $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation (Shang, S et al. 2002).
The $\alpha \rightarrow \beta$ phase transformation is accompanied by a lattice strain transformation under unconstrained conditions, lattice strains in the range of 10% expansion of $\sim 10\%$ along $[2 \overline{1} \overline{1} 0]_\alpha$ to become $[1 0 0]_\beta$, $\sim 10\%$ contraction along $[0 1 10]_\alpha$ to become $[0 1 \overline{1}]_\beta$ and $\sim 1\%$ contraction along the $[0 0 0 2]_\alpha$ to become $[0 1 \overline{1}]_\beta$ (Bieler, T. R et al. 2005). On the other hand, in the case of $\beta \rightarrow \alpha$ contraction is expected along a $<110>_{\beta}$ direction and contraction of 2% and expansion of 10% along the two perpendicular $<110>_{\beta}$ directions (Holt, R. A., et al., 1985). If all variants are present, the $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation should produce perfectly random $\alpha$ texture in the end. However, the observed texture is never random, due to variant selection (Preuss, M et al. 2007). Details of the phenomenon and mechanisms are present in the following section.

### 2.2.4 Variant selection

During $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation, it is expected that all variants of an orientation would occur with equal statistical probability. However non-multiplication of variants and preference of certain variants can be attributed to variant selection. This phenomenon has been well studied in bcc metals (e.g., steel) (Brückner, G et al., 2001, Hutchinson, B et al., 2005, Butrón-Guillén M. P et al., 1997). In titanium and zirconium alloys, variant selection has been reported both during displacive and diffusional transformation (Jourdan, C et al. 1991, Bhattacharyya, D et al. 2003, Lonardelli, I et al. 2007, Bohemen, Van S.M.C et al. 2008, Romero, J et al. 2009). Variant selection occurrence has been reported in zirconium alloys during simple $\beta$ annealing (Humbert, M et al. 2003); the effect may be intensified by application of stress during transformation (Gey, N et al. 1996, Gey, N et al. 1997). It has been
reported that hot rolling above $\beta$ transus and heavy cold rolling before $\beta$ heat treatment leads to variant selection occurrence in titanium alloys during $\beta \rightarrow \alpha$ phase transformation (Zhu, Z. S et al. 1996, Gey, N et al. 1996). The transformation texture during $\alpha \rightarrow \beta$ and $\beta \rightarrow \alpha$ transformation is dependent on the degree of variant selection occurrence, which has been linked to several mechanisms. Understanding these variant selection mechanisms is essential in order to develop physically based predictive models for transformation textures.

2.2.4.1 Variant selection mechanisms

The proposed variant selection mechanisms are numerous; however, they can be classified based on the following principles.

(1) Residual stresses from the deformation process and externally applied stresses might lead to selection of certain variants. Gautier, et al. and Heung and co-workers (Gautier, E et al. 1995, Heung, H. N et al. 2003) have shown evidence for preferential selection of variants in steel under tensile residual stress and applied load. Variant selection intensified as the stress level was increased. However, the high temperature titanium phase is extremely soft above $\beta$ transus (~ about 4.3MPa yield strength for Ti-6Al-4V) (Rosen R. S, et al., 1999), which makes it very difficult to apply stresses to the material at that stage without forcing the material to deform plastically. Therefore, establishing the role of stress induced variant selection in titanium and for the same reason in zirconium alloys is far more difficult than in steel.
(2) The metallurgical state of deformed high-temperature titanium phase (e.g. residual dislocations and other crystal defects) may assist the nucleation of certain variants (Furuhara and Maki 2001, Zhu, Z. S et al. 1996, Gey, N et al. 1996, Gey, N et al. 1997). This principle is practicable if the β phase annealing is avoided prior to transformation. The regions in the material with high densities of dislocations created during deformation are known to be unstable and could cause easy formation of α nuclei of the new phase (Butrón-Guillén M. P et al. 1997). These highly strained regions perturb the crystallographic stable structure and sometimes lead to preferential formation of pre-lattice of the transformed product. Crystal defects on specific planes and directions in the β phase most often have a significant influence on the formation of the α hexagonal lattice, thus favouring certain variants during transformation. Based on this selection rule, Gey et al. (Gey, N et al., 1997) proposed a transformation texture model to study variant selection occurring during β→α transformation. The preferentially selected variants were found to have a strong link with the shear on the \{112\}<111> slip systems of the mother β phase prior to transformation (Gey, N et al., 1997).

(3) Neighbouring adjacent β grain pairs with common (110) planes are created during thermomechanical processing having characteristics that favour nucleation of certain orientation variants (Bohemen, Van S.M.C et al. 2008, Cayron, C. 2008). A β/β grain boundary between two adjacent parent grains, with features such that α nucleation or growth is favourable into both, will strengthen that specific orientation variant (Bhattacharyya et al., 2003, Stanford and Bate 2004, Bhattacharyya et al., 2007). The β→α phase
transformation is accompanied by nucleation and growth of the $\alpha$ plates; the nucleation can occur from the prior $\beta$ grain boundaries or within the $\beta$ grains. In titanium and zirconium, the nucleation of the $\alpha$ plates are preceded by the formation of grain boundary $\alpha$ (GB$\alpha$) (Lutjering and Williams, 2003, Ahamed and Rack, 1998). Once GB$\alpha$ is formed, the nucleation of the plates into grain interior occurs, thus one can suggest that the selection of the variant takes place at the prior $\beta$ grain boundaries. It has been suggested by Bhattacharyya et. al. (Bhattacharyya et. al. 2003) that the interfacial energy of the grain boundary is minimized if the $\alpha$ variants on both sides of the boundaries select a common (0002)$\alpha$ plane related to a common $<110>\beta$ plane in both $\beta$ grains. The similarity between d-spacing of the (0002)$\alpha$ and (110)$\beta$ planes would then decrease the critical nucleus size for $\alpha$ precipitation and consequently reduce the activation energy for $\alpha$ nucleation. This nucleation advantage can lead to early growth of $\alpha$ laths on both sides of the grain boundaries until the colony encounters other lamellae growing from different directions. Humbert, M. et al (Humbert, M. et. al 2006) attributed it to strain elastic energy induced by transformation strain at the boundaries of $\beta$/\$\beta$. The maximum angular misalignment between $<110>\beta$ at which this mechanism occurs is $\sim$10° and once this angle is exceeded no variant selection was observed (Stanford and Bate 2004).

Even though there seems to be a consensus on this mechanism leading to variant selection, the origin of these special grain pairs is not fully understood. Two main concepts have been proposed, according to Bhattacharyya et. al. (Bhattacharyya et al. 2007), rotation in adjacent $\beta$ grains by $\sim$10.5° about a common $\{110\}_\beta$ axis and
special boundaries which conforms to the specific ~ 60° rotation about a <111> direction (twin boundary) could promote the same α orientation being nucleated on either side of the β boundary. This is in agreement with the work of Germain et al (2007), the specific misorientations that favours these β/β boundaries with similar α variants as reported by Germain et. al are summarised in Table 2-5 (Germain et al. 2007).

The second concept, where β grains have common (110) plane but the α variants that preferentially form on each sides of the β grain boundaries have close c-axes orientation but not necessarily similar crystallographic orientation (Stanford and Bate 2004). However, Cayron (Cayron 2008) offered an alternative explanation to the high incidence of these special β grain pairs. He found that β grains that share a common (110)β pole are actually the β variants created previously from the α-β transition. He argued that after β forging, which involves the first transition, α becomes the parent grains of the β variants produced by transition to β during the thermomechanical treatment and based on the fact that in Ti-6Al-4V the β grains at high temperature are thermodynamically more favourable to grow from the pre-existing β nuclei. Therefore, the α variants produced by β transition to α can be in Burgers orientation relationship coincidently with two adjacent β grains if those β grains are products of the transformation of α to β phase.

(4) Remnant precipitates of the low temperature phase at high temperature act as special nucleation sites or inhibits grain coarsening if it is not completely transformed into the new phase. Defects in the grain matrix act as nucleation sites for new phases during transformation; these regions of inhomogeneities could be either grain boundaries or remnant phases. As the nucleation site
may control the orientation of the new phase, the final orientation would depend on the presence of these sites. In this case, the remnant phase becomes a source of variant selection; in titanium, the transformation texture is modified by pre-existing $\alpha$ phase when cooled from $\alpha + \beta$. At very low cooling rates, if the remnant primary $\alpha$ ($\alpha_p$) followed the orientation relationship with the $\beta$ phase, the secondary $\alpha$ ($\alpha_s$) would grow from the remnant $\alpha_p$. This has been illustrated by Semiatin et al. (Semiatin et al. 2003), with $\sim 27\%$ remnant $\alpha$ phase. In the case of slow cooling (<0.5°C/s), the $\alpha_s$ grows from the pre-existing $\alpha_p$ and at higher cooling rate (>3.5°C/s), the nucleation seems to occur on the $\beta$ grain boundary. This means that at very low cooling rates, the $\alpha_p$ controls the transformation texture, which could indeed be the source of texture memory often reported in these alloys (Wenk, H-R et al., 2004, Romeo, J et al., 2009).

The mechanism was explored by Humbert et al. (Humbert et al. 2006). They found large macrozones (regions of close crystallographic orientation) after annealing titanium within the $\alpha + \beta$ phase field. It was observed that about 10% of $\alpha_s$ that nucleated at the $\alpha_p/\beta$ boundaries had misorientation corresponding to the Burgers orientation relationship. In 80% of these cases, the $\alpha_s$ occurs with crystallographic orientation closest to that of the $\alpha_p$. This suggests that the macrozone resulted from the crystallographic similarity of the $\alpha_p$, and which was strengthened by the $\alpha_s$ orientation.

(5) The effect of grain shape on variant selection has been investigated by number of authors (Humber, M et al., 1987, Butrón-Guillén M. P et al. 1997 and
Hutchinson et al. 2005). For instance, in the case of flattened and elongated grain shapes of the high temperature phase, resulting from the forged process. Certain variants might show preferential precipitation (Hutchinson et al. 2005). The directionality of the grain might dictate the transformed orientation (Butrón-Guillén M. P et al., 1997). For example, the growth direction of lath-like product of Widmanstätten or bainitic structure in steel is more favourable along the rolling direction (RD) than in the normal (ND) or transverse directions (TD) for heavily deformed grains. Similar observations have been made by Humber et. al. (Humber, M et al., 1987). In titanium, the geometry of the sample plays an active role in favouring certain orientation variants (Jourdan, C et al., 1991). The favoured orientations can be deduced from the orientation relationships. The variant selection rule due to elongated grain shape can be considered relevant for anisotropic grains shape, however, for titanium alloys the grain shape of high temperature phase is isotropic, therefore the role of grain shape might be difficult to establish.

(6) Transformation-induced stresses lead to volume contraction and expansion between grains, leading to selection of certain variants at the expense of the other variants during phase transformation. The effect of displacive transformation is accounted for in this group. The shape change strains of variants in the individual β grains during displacive transformation (Wang, S.C et al., 2003) and texture modification due to stress field in the neighbouring grains (Stanford and Bate, 2005) leads to preferential selection of variants. The effect of stress state on the volume fraction orientation variants in each β grain is illustrated in Figure 2-22 (Stanford and Bate, 2005).
The number of variants observed in each grain was consistent with the Burgers orientation relationship with twelve $\alpha$ variants produced in each $\beta$ grain, but with different volume fractions. This suggests that some variants are favoured compared to others. It is important to point out that the orientation observed is not the entire orientation of the grains, considering that the observed grain are two dimensional slice and not the entire grain. Therefore, such conclusion will be more reasonable if the three dimensional slice covering the orientation of the entire grain is investigated.

Hutchison et al. has demonstrated that the biaxial compression that accompanies the volume expansion during phase transformation in steel will preferentially activate certain variants (Hutchison et al. 2005). This observation is similar to work by Humbert et. al. (Humbert et al. 1987), however, the origin is different. According to Hutchison et al (Hutchison et al. 2005) the biaxial compression is
due to micro segregation of alloying elements, which results in different layers of steel with different transformation temperatures. A variety of models have been proposed based on this variant selection rule. Humbert et al. (Humbert et al., 2003) developed a model based on elastic anisotropy, which was applied on zirconium alloys whilst biaxial compression (Hutchinson et al. 2005) and elastic interaction between transforming region during displacive transformation (Bate and Hutchinson 2000) have been applied in steel and titanium.

2.2.4.2 Reconstruction of high temperature $\beta$ phase

The phase transformation occurring in titanium and zirconium alloys is such that the retained volume fraction of the high temperature $\beta$ phase is statistically very small at ambient temperature and cannot be representative of the $\beta$ texture. Texture measurements using neutron time-of-flight (TOF) diffraction (Jourdan, C et al. 1991, Lonardelli, I et al. 2007) can overcome the aforementioned experimental difficulty by capturing statistically reliable data of the high temperature $\beta$ phase. Although they can provide excellent information of the global texture, they do not provide information on the microstructural scale, i.e. information on neighbouring $\beta$ grains. EBSD provides the ability to combine macroscopic texture data with information obtained on the microstructural scale. The information regarding the high temperature $\beta$ texture can be obtained from EBSD data sets recorded during $\beta \rightarrow \alpha$ phase transformation, which often occurs with specific orientation relationships (Burgers). In order to exploit this, methods of deducing high temperature $\beta$ texture from $\alpha$ EBSD orientation maps obtained at ambient temperature are of particular interest.
Humbert et al. (Humbert, M et al. 1994, Humbert, M et al. 1995) proposed a method to determine the $\beta$ orientation from inherited daughter variants in cases where there are close adherence to the Burgers orientation relationship. The number of $\alpha$ variants considered to obtain the required unique solution for the mother $\beta$ grain was between three and two, three for less favourable cases and two for the most favourable case. The $\beta$ reconstruction method established by Humbert et al. was applied to analysed orientation data recorded by EBSD. Early applications of this method were cumbersome due to the fact that identification of $\alpha$ variants was dependent on the judgment of the operator since it was manually chosen (Moustahfid et al., 1997). A more robust approach was later proposed to improve the existing method (Humbert et al., 2002). In this case, the number of variants required to calculate the $\beta$ orientation was no longer restricted to three and more deviation from the Burgers orientation relationship was allowed. The basis of this method is to obtain the six potential mother $\beta$ orientations from each inherited daughter $\alpha$ variant. Thus the true mother orientation is deduced by identifying a cluster of $\beta$ orientations between these different sets of orientation. The cluster of orientation is evaluated by averaging a group of closest orientation from each set of solutions (Humbert et al., 2002). The method is exemplified in Figure 2-23. It is important to note that at this stage the reconstruction method is confined to a few $\beta$ grains.
The automation of the method was carried out by Humbert et al. (Humbert et al., 2003) to achieve automatic reconstruction of larger orientation maps containing many prior β grains. Steps were taken to systematically identify the α variants belonging to the same mother β grain by considering specific misorientation angles and axes between the adjacent measurements. The α variants inherited from the same mother β grain are assumed to have specific misorientation related to the Burgers orientation relation. According to the Burgers orientation relationship, five characteristic misorientation types between α variant inherited from the same mother β grain are possible, as represented in Table 2-3. By plotting these misorientation angles and axes on the recorded α EBSD map, the prior β grain boundaries can be identified.
Texture and Variant Selection

Table 2-3 Specific misorientation angles of $\alpha$ variants belong to the same mother $\beta$ grain

<table>
<thead>
<tr>
<th>Angles (º)</th>
<th>Axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.529</td>
<td>$\hat{c}=$[0001]</td>
</tr>
<tr>
<td>60</td>
<td>$\hat{d}_2=$[1210]</td>
</tr>
<tr>
<td>60.832</td>
<td>$\hat{d}_1$ at 80.97 º from $\hat{c}$ in ($\hat{d}_3$, $\hat{c}$) plane</td>
</tr>
<tr>
<td>63.262</td>
<td>$\hat{d}_2$ at 72.73 º from $\hat{c}$ in ($\hat{d}_2$, $\hat{c}$) plane</td>
</tr>
<tr>
<td>90</td>
<td>$\hat{d}_3$ at 5.26 º from $\hat{d}_2$, in basal plane</td>
</tr>
</tbody>
</table>

The developed $\beta$ reconstruction algorithm based on these specific misorientations was used on the EBSD orientation map. The identification of $\alpha$ variants belonging to the same $\beta$ grains was based on the following assumptions: if the misorientation between two neighbour points are above a certain value (usually 3 º), the points belong to different variants. Secondly, if the deviation of misorientation angle between the neighbouring variants is within a certain tolerance (3 to 5 º) to any of the theoretical misorientation listed in Table 2-3, there is very strong likelihood that the different variants belong to the same $\beta$ grain, whereas if the deviation is above the mentioned value, the variants are deemed to belong to a different $\beta$ grain. One of the pitfalls of the $\beta$ reconstruction algorithm is the incorrect unification of two prior $\beta$ grains. This is due to two neighbouring $\beta$ grains having sometimes variants with specific misorientation that coincides with one of the theoretical misorientation angles mentioned in Table 2-3. Therefore, as a consequence, the grain reconstruction would fail (Gey et al., 2003). However, by varying the tolerance angle, this problem could be minimized but not completely overcome. Manual procedures are therefore incorporated, which makes the procedure rather cumbersome as identification of the inherited $\alpha$ depends on the judgment of the operator.

The uncertainty found in the $\beta$ reconstruction was addressed by Cayron and co-workers (Cayron 2006 and Cayron et al. 2006). They suggested that, rather than the
Texture and Variant Selection

three maxima of α variants used to determine the mother β orientation in the work of Humbert et al., four α variants are actually needed. This is due to the fact that there are special cases where the three α variants give rise to two possible mother β solutions, i.e. one is the twin of the other through the (111) mirror plane. The problem of incorrect unification of two neighbouring grains during neighbour-to-neighbour grain β reconstruction (Gey et al., 2003) was examined. Cayron et al. (Cayron et al. 2006) concluded that this reconstruction method works satisfactorily for titanium alloys, but would be unsuitable for martensitically transformed steels due to a large number of possible variants and high stresses generated during such transformation, leading to larger deviation in the misorientation angles based on the crystallographic relationship. Thus, they proposed an alternative method, which they claim would incorporate both steels and titanium alloys that have undergone martensitic transformation. This approach checks all possible triplets between variants to deduce those variants inherited from the mother grain.

Due to the shortcoming of the β reconstruction as identified by Cayron and co-workers (Cayron 2006 and Cayron et al., 2006), a modified β reconstruction has been proposed by Germain et. al. (Germain et. al 2007). In this modified approach, the justification is that they calculated probability that a unique β solution could be found from a set of randomly selected variants, i.e. assuming all twelve variants occurred during the transformation. The result of this procedure is exemplified in Table 2-4. The result suggests that 4 and not 3 variants, as suggested by Humbert et. al. (Humbert et. al 1995), are required to deduce a unique solution. However, they considered the β reconstruction approach by Cayron et. al. (Cayron et. al., 2006), which checks possible triplets between variants to determine variants belonging to the
same grains, as time consuming. Alternatively, a faster approach was introduced to identify α variants inherited from the same mother β grains; in this approach a reference α variant was first chosen from the same mother β grains.

<table>
<thead>
<tr>
<th>Number of variants (α)</th>
<th>Probability (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>72.72</td>
</tr>
<tr>
<td>3</td>
<td>98.18</td>
</tr>
<tr>
<td>4 or more</td>
<td>100</td>
</tr>
</tbody>
</table>

If during the reconstruction, the adjacent α variant has a misorientation with the reference variant within the prescribed tolerance of one of the specific misorientations, it is assumed to belong to the same grain. The neighbour-to-neighbour reconstruction is illustrated in Figure 2-24.
Figure 2-24 Calculation of $\alpha$ variants using misorientation criterion in different situation of mother $\beta$ grains and inherited $\alpha$ variants (a) $\alpha/\alpha$ misorientation at prior $\beta/\beta$ boundaries are different from misorientation according to Burgers ($\Delta^b$): the prior $\beta/\beta$ boundary is detected by considering neighbouring variants only. (b) An $\alpha/\alpha$ misorientation at prior $\beta/\beta$ boundary with slight deviation from ($\Delta^b$) : the prior $\beta/\beta$ is not detected by considering the misorientation between the neighbouring orientation variants only.(c) Same as in (b) but the variants are gathered starting from the reference variant. (d) Same as in (c) but starting from a different reference variant (Germain et al. 2007)

Even though there have been improvements, the misorientation criterion could not resolve two $\alpha$ variants with close orientation, which nucleated from either side of the $\beta/\beta$ boundaries. This is one of the variant selection mechanisms, often reported in the literature (Stanford and Bate 2004). Germain et. al. (Germain et. al 2007) established that these $\beta/\beta$ boundaries with similar $\alpha$ variants nucleating on both sides occur with specific misorientations between them, by calculating every possible misorientation between all the six potential mother grains of an $\alpha$ variant. The specific misorientations for that favours these $\beta/\beta$ boundaries with similar $\alpha$ variants as reported by Germain et. al (Germain et al. 2007) are summarised in Table 2-5.
Table 2-5 Specific misorientation of β grain pairs with common α variants on both sides of the boundary.

<table>
<thead>
<tr>
<th>Angles</th>
<th>Axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.5º</td>
<td>[110]_β</td>
</tr>
<tr>
<td>49.5º</td>
<td>[110]_β</td>
</tr>
<tr>
<td>60º</td>
<td>[110]_β</td>
</tr>
<tr>
<td>60º</td>
<td>[111]_β</td>
</tr>
</tbody>
</table>

A fully automated reconstruction method was proposed by Glavicic *et al.* (Glavicic *et al.*, 2003a, 2003b and 2004); this approach uses the Montte-Carlo β misorientation minimisation technique, which avoids manual selection of the α laths. The approach to determine the mother β orientation involves identifying α variants and deducing their likelihood of being part of the same β grain by examining neighbour to neighbour misorientations. Once the misorientation of neighbour points is less than a certain threshold, the points are deemed to belong to the same variant. The Monte-Carlo technique is used to select random pairs of points from the adjacent α variants and from these points, six possible mother β solutions for each point are deduced. The six possible β solutions are evaluated and the pair of β solutions with minimum misorientation angle deviation between them is assigned a probability number based on the likelihood that the two variants are inherited from the same mother β grain. Although this technique has been validated (Glavicic et al 2004) by correctly determining the texture and the β phase grain size using X-ray methods, there have been cases where errors occurred during reconstruction of EBSD orientation maps. A variant-based reconstruction technique has been developed by Davies *et al.* (Davies *et al.* 2007). In this approach the work of Humber and co-workers formed the background work (Humbert, M et al. 1994, Humbert, M et al. 1995), which was
utilised by Davies et al in their reconstruction approach. As discussed above, Humbert et. al. proposed that six potential mother $\beta$ orientations can be precipitated from a single $\alpha$ variant and by evaluating the misorientation between two $\alpha$ variants, a unique solution can be found. However, in the variant-based $\beta$ reconstruction technique, the inherited $\alpha$ variants do not have to be grouped into a cluster but rather each $\alpha$ variant is evaluated independently by comparing the misorientation with its neighbours. In order to choose the correct $\beta$ solution from the six potential solutions for each of the $\alpha$ variant, the misorientation angles between the points at the borders of a variant and adjacent points in adjacent variants are analysed. Consequently, by compiling the results of analysed misorientation, the correct solution should be the one, which is obtained most often. It is found that in most cases the approach leads to a single solution. The step to step reconstruction process is depicted in Figure 2-25.
Figure 2-25 The variant-based $\beta$ reconstruction method (a) $\alpha$ variants are identified by considering a minimum misorientation angle, each variant is separately analysed. (b) the potential $G^\beta_p$ solution are derived from misorientation analysis across boundary between the variants and its neighbours. (c) Primary reconstruction where most frequent solution is selected (d) secondary reconstruction where missing solution is identified.

The variant-based $\beta$ reconstruction shows significant improvement over the other reconstruction methods discussed earlier based on its ability to resolve the unification of two $\beta$ grains when $\beta$ grains with close orientation. Any possible substructure within the $\beta$ grains can also be detected, since this approach is not based on reconstruction of the boundaries, but on finding mother orientation of each $\alpha$ variant independently. However, there have been few errors exhibited by this reconstruction method: (1) Once the prior $\beta$ grain has no adjacent variants, which can result from pseudo-symmetry or misindexing, the variant-based reconstruction may not provide solutions. (2) When two adjacent $\alpha$ variants from different mother $\beta$ grains and
having misorientation lower than that used to detect the variants during reconstruction (3°). This leads to error in the reconstruction, the two variants are erroneously detected as single variants and misorientations are then analysed for the two variants combined. This drawback of the variant based reconstruction technique to resolve two adjacent α variants with similar crystallographic orientation has also been reported in the grain based approach by Germain et al. (2007). Each of the method enumerated above has recorded significant success in specific area. The variant based approach can be used to reconstruct deformed and as well as restored β microstructures. Also in variant based approach any reconstruction errors are always restricted to single variants and will appear as inconsistencies in the originally correctly reconstructed β grains. Whereas in grain based approach, any error resulting from reconstruction may appear in large areas in the correctly reconstructed map. This makes variant based approach crucial tool in the present project. The variant-based β reconstruction method was chosen to determine the β textures and former β grain boundaries in the present study.
Chapter 3 **Experimental methods**

In this section the detail sample preparation procedures and characterisation methods are presented. This includes microstructural analysis by optical microscopy (OM), texture measurement and analysis using different diffraction techniques: laboratory X-ray diffraction, EBSD and neutron diffraction. Laboratory X-ray and Electron Backscatter Diffraction (EBSD) are used for ex-situ texture measurements, while neutron diffraction is used for in situ texture measurement. One of the fundamental challenges when measuring textures of $\beta$ transformed Ti- alloys is the large $\beta$ grain size. When using a surface technique, the measurement of 1000 grains with a grain size of 500 $\mu$m requires an illuminated area of approximately 200 mm$^2$. While EBSD provides the ability to combine macroscopic texture data with information obtained on the microstructural scale, the requirement of producing large EBSD maps to capture a sufficient number of $\beta$ grains and the fact that a small step size needs to be employed in order to capture relevant microstructural information renders this methodology very time consuming. Furthermore, EBSD is mainly an ex-situ characterisation technique. While one might consider using EBSD at high temperatures, the high affinity of oxygen to titanium makes such studies rather difficult. In contrast, neutron time-of-flight diffraction can overcome some of these issues, as it is not compromised by surface effects due to high penetration depth of the probe. More importantly, neutron diffraction enables one to probe large volumes of material in order of hundreds of mm$^3$ in minutes and therefore can provide excellent information of the global texture, even in large grain size material.
3.1 **Materials**

The material used in this study, Ti-6Al-4V, was provided by TIMET, UK and produced by conventional ingot metallurgy and uni-axially rolling. Some of the materials was given to the School of Engineering, University of Birmingham for alloying with yttrium. The material was placed in an induction skull melting furnace under vacuum of $4 \times 10^2$ mbar. The vessel was backed filled with high purity argon to a partial pressure of 200 mbar in order to reduce the evaporation of volatile alloying elements. Power was applied to the melting coil in 50 kW steps up to maximum power input of 350kW. Once fully molten, yttrium (0.4 wt. %) was added from an additional device and the melt was held for 60 s to allow full dissolution and homogenization. The melt was poured under gravity into a 100 mm diameter cast iron ingot mould and left to solidify and cooled to room temperature. The chemical compositions of both alloys are shown in Table 3-1 and Table 3-2. The analysis was provided by TIMET as shown in Table 3-1. No further analysis was carried out in the material containing yttrium. This due to the re-melting was done under high vacuum and the furnace back filled with high purity argon to ensure no significant loss of material or oxygen pickup and the resulting ingot shows no significant change in weight when compared to weight of the charge. Both batches of alloys were cut into blocks of 40 x 40 x 20 mm$^3$, β heat treated and quenched before subjecting to uni-directionally hot rolling.
### Experimental methods

#### 3.1.1 DSC measurement

Due to chemical compositional difference between the two variants of Ti-6Al-4V (Ti-6Al-4V and Ti-6Al-4V-0.4wt %Y), it was important to determine the $\beta$ transus temperature of each variant. This was considered to be essential in order identify if the processing temperature would have to be adjusted for each alloy to maintain similar conditions.

Differential Scanning Calorimeter (DSC) was used to determined $\beta$ transus temperatures of two variants of Ti-6Al-4V. The DSC is an analytical technique used to determine thermal conductivity of a material by monitoring the heat capacity change that occurs as a specimen is heated. The heat capacity change is recorded as a function of increasing temperature. The measurement is normally carried out in air, inert gases such as helium or argon and vacuum atmosphere.

### Table 3-1 Elemental composition of the convectional ingot metallurgy (IM) processed material

<table>
<thead>
<tr>
<th>Elements</th>
<th>Fe</th>
<th>V</th>
<th>Al</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>B</th>
<th>Y</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%(min)</td>
<td></td>
<td>3.8</td>
<td>6</td>
<td>-</td>
<td>.17</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Bal</td>
</tr>
<tr>
<td>Wt%(max)</td>
<td>.3</td>
<td>4.5</td>
<td>6.7</td>
<td>.1</td>
<td>.23</td>
<td>.03</td>
<td>.005</td>
<td>.001</td>
<td>Bal</td>
</tr>
</tbody>
</table>

### Table 3-2 Elemental composition of Ti-6Al-4V 0.4Y

<table>
<thead>
<tr>
<th>Elements</th>
<th>Fe</th>
<th>V</th>
<th>Al</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>B</th>
<th>Y</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%(min)</td>
<td></td>
<td>3.8</td>
<td>6</td>
<td>-</td>
<td>.17</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Bal</td>
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<tr>
<td>Wt%(max)</td>
<td>.3</td>
<td>4.5</td>
<td>6.7</td>
<td>.1</td>
<td>.23</td>
<td>.03</td>
<td>.005</td>
<td>.4</td>
<td>Bal</td>
</tr>
</tbody>
</table>
Experimental methods

Prior to the DSC measurement, the calorimeter was first calibrated for temperature and sensitivity. The measurement was performed with samples weighing about 120-170 mg placed in Al₂O₃ crucible and heated at 5°C/min to 1150°C and cooled at 5°C/min. Due to a high affinity of Ti-alloys for oxygen at elevated temperature, the measurements were carried out under a dynamic flow of argon (50ml/min) to avoid oxidation of the alloy. The transformation temperature (β transus) is marked as A and B for Ti-6Al-4V and Ti-6Al-4V-0.4Y respectively in the heating curves shown in Figure 3-1.

![DSC measured transformation temperature of the two variants of Ti-6Al-4V](image)

The DSC measurements show that the β transus temperatures of both alloys were approximately 990 °C. Therefore, there was no need to adjust the rolling and solution heat treatment temperatures.
3.1.2 Hot rolling

Prior to hot rolling process all batches of the alloys were first $\beta$ heat treated and quenched in order to achieve a fairly random texture. The rolling process was carried out to introduce particular texture components prior to studying variant selection during $\beta$ heat treatment. As described earlier, it is well established that the rolling temperature has a very pronounced effect on the $\alpha$ deformation and recrystallization texture. In the present case the aim was to generate a strong transverse texture by rolling the material at 950 ºC while rolling at 800 ºC should provided an additional strong basal texture component. The aim was to understand the influence of the initial $\alpha$ and $\beta$ textures on final texture and variant selection during the $\alpha\rightarrow\beta\rightarrow\alpha$ phase transformation.

The rolling was conducted using a two-high, non-reversing mill at the Materials Science Centre, University of Manchester, with a pair of rolls of diameter 250 mm rotating in one direction. The rolls rotate with the surface velocity exceeding the speed of the incoming material; friction along the contact interface acts to move the metal forward. The deformation achieved for a single pass between the pair of rolls depends on the frictional condition along the interface. For uni-directional rolling, the reduction in thickness is accompanied by an increase in length and width of the test piece. The material is elongated along the rolling direction and is squeezed uniformly in the thickness direction. Blocks of dimensions 40 x 40 x 20 mm$^3$ of Ti-6Al-4V and Ti-6Al-4V-0.4Y were uni-directionally rolled at 15% reduction per pass. Prior to rolling, the samples were placed at the centre of a preheated electrical resistance furnace set to a temperature of either 800 ºC or 950 ºC within the $\alpha + \beta$ phase field. The samples were heated for at least 1 hour and once the desired temperature had
Experimental methods

been achieved, the samples were passed in between the pair of rollers. After the initial temperature was achieved, the furnace temperature was maintained at chosen temperature throughout the duration of the process. Due to the fact that the rolls were cold which leads to temperature gradient between the rolls and the material surface, the samples were reheated for 2 min in the furnace in-between each pass. To ensure that the chosen temperature for each specimen was maintained in-between each pass a K-thermocouple was inserted into the furnace such that it lied underneath the specimen. After the last pass, a final reduction in thickness of 75% was achieved corresponding to a final thickness of 4.9 mm. The rolling schedule and schematic representation of the rolling and annealing processes are shown in Table 3-3 and Figure 3-11. It is important to note that, α-case formation was not observed even though the rolling process was carried out in an uncontrolled atmosphere.

<table>
<thead>
<tr>
<th>No of passes</th>
<th>Reduction rate (%)</th>
<th>Reduction (mm)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15</td>
<td>3</td>
<td>17</td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>2.6</td>
<td>14.5</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>2.1</td>
<td>12.3</td>
</tr>
<tr>
<td>4</td>
<td>15</td>
<td>1.8</td>
<td>10.5</td>
</tr>
<tr>
<td>5</td>
<td>15</td>
<td>1.6</td>
<td>8.9</td>
</tr>
<tr>
<td>6</td>
<td>15</td>
<td>1.3</td>
<td>7.6</td>
</tr>
<tr>
<td>7</td>
<td>15</td>
<td>1.1</td>
<td>6.4</td>
</tr>
<tr>
<td>8</td>
<td>15</td>
<td>1.0</td>
<td>5.5</td>
</tr>
<tr>
<td>9</td>
<td>15</td>
<td>0.8</td>
<td>4.7</td>
</tr>
</tbody>
</table>
Experimental methods

3.1.3 Recrystallisation heat treatment

After hot rolling test pieces were subjected to a recrystallisation heat treatment with the aim to generate similar starting microstructure morphology. This was achieved by heat treating all materials at 950 °C for 8 hours followed by slow cooling (1 °C/min) to room temperature before sample were taking out of the furnace. As it will be demonstrated latter, such heat treatment is known to produce an equiaxed $\alpha$ microstructure with $\beta$ phase mainly sitting in the triple junctions between the $\alpha$ grains.

The heat treatment was carried out in a horizontal solid alumina tube furnace with a tube diameter of 380 mm. This furnace offers precise temperature control and excellent temperature uniformity across the tube due to a small tube diameter. To
Experimental methods

achieve uniform temperature, both ends of the tube are fitted with tapered insulating plugs. In order to maintain the furnace with an inert atmosphere throughout the duration of the heat treatment, argon is introduced through the insulating plug. An outer mesh is constructed and fitted around the furnace to promote natural air cooling and to protect the operator from the hot surface. The furnace is calibrated with a K-type thermocouple inserted through the insulating plug; the calibration procedure was done for each heat treatment process. The calibrated tube furnace was preheated to a temperature of 950°C before the specimens were inserted (half of the rolled slabs).

3.1.4 β heat treatment

The aim of the β heat treatment was to study variant selection particularly during β→α phase transformation. Since in industrial process cooling rates are very slow, the cooling rate applied in the present case was 1 °C/min. It is known from previous record that such cooling rate promotes α growth from the former β grain boundaries and colonies rather than Widmanstätten microstructure (Lütjering, G 2003). In order to study the potential effect of β grain growth, material was subjected to either 1050 °C (about 55 °C above β transus) or 1150 °C (155 °C above β transus). In addition, the addition of yttrium to Ti-6Al-4V was expected to provide a way to limit β grain growth. In all cases, small cuboids with dimension 10 x 10 x 5 mm³ were heated at these temperatures (1050 and 115 °C) for 30 minutes. The heat treatment profile is shown in Figure 3-3.
Experimental methods

In order to capture $\alpha$ products that formed during the early stage of cooling, interrupted cooling experiments were carried out. In this case, the material was $\beta$ heat treated to 1150 °C for 30 min and allowed to cool at 1 °C/min until 10 °C below the $\beta$ transus temperature. Thereafter, the material was water quenched to stop further $\alpha$ growth by diffusional phase transformation. Martensitic phase transformation took place during quenching, which provided a distinctively different morphology than the $\alpha$ formed during the early stage of slow cooling. The schematic representation of the interrupted heat treatment is shown in Figure 3-4.

Figure 3-3 Schematic representation of heat treatment profile carried at 1050 °C and 1150 °C
Experimental methods

Figure 3-4 The schematic representation of the interrupted heat treatment profile carried at 1150 ºC, cooled at 1 ºC to 980 ºC and water quenched

3.2 Metallographic characterisation methods

3.2.1 Metallographic sample preparation

For metallographic inspection, samples were mid-sectioned into two halves and hot mounted in Bakelite using a Struers Labopress-3. The mounting was carried out using a force of 20 kN, with 7 min heating at 180 ºC, followed by 3 minute cooling. The mounted specimens were ground using rotating discs of abrasive silicon carbide paper at speed of 300 rpm. The grinding was performed in steps, starting with coarser paper 400, 600, 800, 1000, 1200, and final grinding with of 2500 grit paper. Each grinding step took about 10-20 min. After the final grinding with 2500 paper, the samples were thoroughly washed followed by rinsing with alcohol and dried. This is
Experimental methods

sufficient preparation for laboratory X-ray examinations. However, further sample preparation is required for optical microscopy and EBSD.

The next stage in the sample preparation is the polishing steps. This is done on the polishing discs (Buehler Metaserv) covered with a soft cloth impregnated with abrasive diamond particles and rotating at 300 rpm. One squirt of oil based polishing lubricant was applied for every 2 min to reduce friction. The rough polishing was divided into three steps: The first polishing was carried out using coarser abrasive particles of 6µm for 10 min followed by the second and third steps with 3µm and 0.25µm abrasive particles for 10 min each. In order to avoid contamination, the polishing steps were followed by washing and drying of the samples. Fine polishing was carried out using oxide polishing solution (OPS) on the polishing wheel at a speed of 5 rpm for 1 h. After polishing the samples were etched at room temperature. This was done to reveal the microstructure of the alloy through selective chemical attack using Kroll's reagent comprising 2 % HF, 8 % HNO₃ and 90 % H₂O (by volume) for 10s. At this stage, the sample is prepared sufficiently for optical microscopy examination. For EBSD analysis three additional sequences of polishing and etching steps are required. Firstly, the samples were OPS polished for 1 h to remove the etched surfaces thereby reducing the strain induced during grinding. The alternating process of OPS polishing and etching was repeated two times prior to final OPS polishing which was done for 2 h. This procedure resulted in the largest indexing rate of crystallographic phases and the best quality of the Kikuchi patterns obtained during the EBSD measurement.
3.2.2 Optical microscopy (OM)

The microstructural characterisation was performed using optical microscope (Olympus TH3). The samples were examined under bright field. The $\beta$ grain size was determined using the mean linear intercept method (ASTM E112). This method is well established for measuring average grain size of processed optical images. Parallel horizontal and vertical lines are drawn across the optical micrographs and the point where the lines intercept a grain boundary are noted. The average linear intercept is calculated by adding all these segments together and dividing by the number of complete grains the lines passed across. The accuracy of this method to obtain grain size of an alloy measured using optical microscopy depends on the judgment of the operator to determine the grain boundaries. Generally optical micrographs of $\beta$ heat treated Ti-6Al-4V reveal the former $\beta$ grain structure as grain boundary $\alpha$ and can be easily identified. Consequently, optical micrograph was used to determine $\beta$ grain size in addition to measurement carried out by EBSD and $\beta$ reconstruction.

3.3 Texture measurement

Most measurement techniques used for texture analysis are based on the diffraction of electrons, neutrons or X-rays on the crystal to provide information on their crystallographic arrangement. The wavelength of the incident radiation source determines the suitability of each measurement technique, hence the penetration depth
required. The method of generation of radiation and its transfer to the material under
study determines the size of the probe for detecting sample orientation (Randle and
Engler, 2000, Escher and Gottstein 1998). The texture measurement could be
classified into either macrotexture or microtexture with reference to the irradiated
volume of material, which provides the orientation information. The microtexture
refers to orientation of individual grains while average values obtained from many
different grains are called macrotexture. Laboratory X-ray diffraction, Electron
Backscatter Diffraction (EBSD) and neutron diffraction are the techniques most
widely used to measure texture. Although other method exists, such as transmission
electron microscopy (TEM), acoustic and magnetic methods, in this study laboratory
X-ray, EBSD and neutron diffraction were utilized for texture measurement and
would be discussed in details in the following section.

3.3.1 Orientation imaging by EBSD

Electron Back-Scattered Diffraction (EBSD) is a technique which measures
orientation of the individual grains in a given sample via electron diffraction in the
scanning electron microscope (SEM) as shown in Figure 3-5 In EBSD, the diffracted
electrons form a pattern on the fluorescent screen and this pattern has features of the
crystal structure and orientation of sample area from which the information is
acquired. The diffraction pattern can be used to determine the crystal orientation,
grain boundary misorientation and provide information about local crystalline
perfection. This is quite unique since not only the orientation but also the spatial
location is determined, which gives good correlation between the microstructure and
the crystallographic orientation (Randle and Engler, 2000).
Experimental methods

The major advantage of this technique is that it can execute an automated diffraction analysis, to give orientation data with spatial resolution, in most systems better than 100 nm and in addition, the rate at which the diffraction is captured, indexed and stored is very high (Spence, J. C. H., 1999).

There are two possible scanning modes used during EBSD measurement; the stage control and beam scan. In stage scan, the electron beam is held stationary while the sample stage is moved. For beam scan, the sample stage is held stationary whereas the beam moves. The major advantages of stage scan over beam scan are: (1) elimination of defocusing issues associated with moving the beam over the tilted sample which results in change of the working distance. This can lead to the image being out of focus. Secondly, the calibration distance between the pattern centre and the distance between the sample and phosphor screen will alter due to movement of the beam (Oxford instruments, 2004). This could be remedied by calibrating at two working distances and interpolating for intermediate working distance values. It is necessary to understand the range of working distances for which EBSD will remain accurately calibrated in order to obtain good pattern quality. The disadvantage of stage scanning is that it is much slower than beam scan.
Figure 3-5 Typical Electron BackScatter Diffraction (EBSD) set up in Scanning Electron Microscope (SEM) chamber (Oxford instrument, 2004).

The EBSD system comprises the electron gun, detector, video camera interfaced to the phosphor screen, a camera-controlled diffraction pattern processor unit, a computer to control data processing and pattern indexing, analysis and output. The sample for the EBSD analysis is tilted 70 ° from the horizontal position in the SEM sample chamber. This is to optimize both the contrast in the diffraction pattern and also the fraction of electrons scattered from the sample (Oxford instruments, 2004).

The beam of electrons through the electron gun is directed to the area of interest on the sample surface. The atoms in the material inelastically scatter a fraction of the electrons, with small loss in energy, to form a divergent source of electrons close to the sample surface. Some of the scattered electrons are incident on the atomic planes having an angle equal to two theta which is equal to the Bragg’s angle. According to Bragg’s law, for a given lattice spacing and wavelength, diffraction of an incident radiation can only occur if the lattice planes are at a specific angle with respect to the incident beam (Bragg, W. H, 1913).
Experimental methods

The diffracted electrons form sets of pairs of large angle cones matching each of the diffracting planes. The phosphor screen on the front of the detector is fluoresced by the electrons from the sample to form the electron backscatter diffraction pattern (EBSP). The acquired diffraction pattern can be viewed with the help of a charged couple device (CCD) video camera (Schwartz A. J et al., 2000). The EBSP consist of a set of Kikuchi bands which are characteristic of the sample crystal structure and its orientation with respect to the reference frame. The resultant diffraction pattern can be interrogated and computer algorithms allow the crystal structure and orientation to be obtained and stored.

A modern EBSD system can capture diffraction pattern, index them and store the result at rates of 1000 frames per second (Schwartz A. J et al., 2000). The spatial resolution is related to the electron beam diameter and this is dependent on the type of electron source and probe current used. The angular resolution for modern EBSD at present is 0.25-1 ° and a spatial resolution down to grain diameter of 10 to 20 nm beam (Oxford instruments, analytical 2007).

Furthermore, the quality of the EBSP image can be improved by both frame averaging and background correction. A single image captured by CCD may appear noisy and to improve the clarity of the image, the setting of the software should be selected in such way that more than one frame is captured and averaged to reduce the noise. For background correction, the initial image obtained shows weak Kikuchi bands and to correct this effect, a background image is acquired while the microscope is in TV mode and at a low magnification. During this operating condition, there are no Kikuchi bands observed due to the beam scanning very quickly over a large area of the sample. The background image acquired is subtracted automatically from the
initial image, hence clearer and more evenly illuminated Kikuchi bands are obtained. (Randle and Engler, 2000).

3.3.1.1 Microscope/EBSD operating condition

The understanding of the operating conditions of the microscope is very crucial towards acquiring good quality diffraction patterns and final EBSD maps. The effects of variation of some of the microscope operating conditions such as the probe current, accelerating voltage, working distance and magnification are briefly discussed.

1. **Probe current**

Increasing the probe current will increase the fraction of the electrons contributing to the diffraction pattern, which leads to high indexing rates and high quality of EBSP maps. However, there should be a compromise in increasing the probe current, since the spatial resolution decreases with large beam sizes due to large spread of the beam on the sample (Humphreys, 2001). In this study probe current was kept at 4.5 nA.

2. **Accelerating voltage**

The electron wavelength and Kikuchi band width are reduced by increasing accelerating voltage. In addition, it increases pattern contrast brightness and indexing rates, but decreases spatial resolution. Poor pattern quality might be obtained at lower accelerating voltages. An accelerating voltage of 20kV is commonly used in EBSD analysis.

3. **Spot size**

Spot size has significant influence on the spatial resolution, and the pattern quality. For weak patterns, increasing the spot size will improve pattern quality. The spot size used in this work was in the range 6-6.5 nm.

4. **Working distance**
Experimental methods

The working distance was always adjusted to achieve a focused beam, which depends on the microscope. For example, in this work, using a working distance in the range 13-15 mm in a FEI Sirion FEG SEM and working distances of 23-26 mm in the CamScam FEG SEM resulted in good quality EBSP patterns.

(5) Magnification

The magnification governs the area covered during the experiment, it also improves indexing rates. For a low indexing rate, increasing the magnification can improve the indexing rate.

(6) Timing per frame

This is the time used to acquire a frame. It is usually in milliseconds; lower times result in higher acquisition rates. However, there should be a compromise in choosing time per frame, since it affects the brightness of the EBSP. In this work, timing per frame used was 30 ms, which gave clear EBSPs.

(7) Frame averaging

Increasing number of frames will increase indexing rates and lower the acquisition rates. Frame averaging leads to clearer EBSPs. An average of three frames was used in this work.

(8) Maximum and minimum number of bands

Increasing maximum and minimum number of bands increases the reliability of the indexing but reduces the indexing and acquisition rates. The maximum and minimum number of bands used in this work for indexing was 6/4.
3.3.1.2 Interpreting Kikuchi patterns

The point of intersection of diffracting planes with the phosphor screen of an EBSD system forms the centre line of the Kikuchi bands. Hence, the indexed Kikuchi bands correspond to the Miller indices of diffracting crystal planes that form them. Unlike the intersection of the Kikuchi bands corresponds to the zone axis in the crystal. The diffracted electrons form a cone having a semi angle of about \((90^\circ-\theta^\circ)\). For EBSD, this is regarded as large angles, thus the Kikuchi bands are assumed to be straight lines (Oxford instruments, 2004). The width \((h)\) of a Kikuchi band can be estimated by using the following equation,

\[
h \approx 2s\theta \approx \frac{n\lambda}{d}
\]  

(3.1)

Where \(s\) is the distance from the sample to the screen, \(n\) is an integer, \(\lambda\) is the wavelength of the electrons, \(d\) is spacing of the diffracting plane, and \(\theta\) is the angle between the incidence of the electrons on the diffracting plane. As it can be seen from equation (3.1) the width of the kikuchi bands is affected by the d-spacing. Wider d-spacing will result in thinner Kikuchi bands.

3.3.1.3 Orientation measurement

Based on the Kikuchi band positions, the crystal orientation can be determined by computer processing of the digitised diffraction pattern collected by the CCD camera. The positions of the bands can be converted to linear features of single points in Hough space using the Hough transform (Escher and Gottstein 1998).

\[
\rho = a \cos \theta + b \sin \theta
\]  

(3.2)
Experimental methods

Applying Hough transform, the coordinate \((a, b)\) of the diffraction pattern transforms to the coordinate \((\rho, \theta)\) in Hough space. A single point represents a straight line in Hough space, \(\rho\), which is the perpendicular distance from the origin and \(\theta\), the angle it makes with the \(a\)-axis. The Kikuchi bands appear as bright regions in Hough space; this region can be used to calculate the original position of the band (Oxford instruments, 2004).

In addition, if the EBSD calibration is done correctly, the angle between the planes that produce the Kikuchi bands can be matched with the known list of angles between planes for the crystal system being analysed. Thus, the correct Miller indices are assigned to each band. Finally, the orientation of the lattice with respect to sample coordinate system is determined. The sequence of EBSD data acquisition and processing are shown in Figure 3-6.
Figure 3-6 The capture, processing and analysis of EBSPs
3.3.1.4 Orientation maps

Large sample areas can be scanned by the combination of beam and stage scan to obtain multi-maps, which can be stitched together using HKL Channel 5 software. The measured crystal orientation data can be displayed in the form of orientation maps. These maps are processed to give a variety of information about the microstructural features of the sample. Figure 3-7 shows an example of an inverse pole figure (IPF) map where different colours represent different crystallographic orientation with respect to the sample coordinate system. The colours indicate which crystallographic plane is parallel to the sample direction. For example, the red colour indicates the <0002> plane normal parallel to the rolling direction (RD). The IPF does not give complete information about the orientation because the rotation about a plane normal to the map/sample surface is not indicated (Oxford instruments, 2004).

![Inverse pole figure map showing orientation of α colonies](image)

Figure 3-7 Inverse pole figure map showing orientation of α colonies
3.3.1.5 Pseudosymmetry and misindexing

Pseudosymmetry in a hcp crystal occurs mostly when Kikuchi patterns of a crystal are misoriented by 30 ° about the $\bar{c}$ axis resulting in wrongly indexed points in an EBSD map. These problems are ascribed to poor pattern quality due to crystal imperfections around the grain boundaries as well as overlapping of two patterns. Consequently, the software might not average the Kikuchi patterns obtained correctly; the patterns are either assigned with a wrong orientation value or it is identified as a wrong phase. Although the problem of misindexing is rare in bcc metals, they can be easily identified as highly misoriented points with reference to the adjacent point. The misindexing can be resolved by improving sample preparation or adjusting the microscope and EBSD acquisition parameters such as probe current, working distance and number of bands for obtaining pattern solutions. Due to crystal imperfections, particularly around areas close to grain boundaries, misindexing is rarely avoided, but it can be systematically removed and extrapolated during post EBSD processing (Humphreys 2001).

3.3.1.6 Texture analysis of Ti-6Al-4V by EBSD

For a combined orientation map and texture analysis, a CamScan Field Emission Gun Scanning Electron Microscope (FEGSEM), equipped with HKL Technology Channel 5 software was used. EBSD maps were recorded to characterise the recrystallised texture and the inherited texture from the $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation. Orientation images of mid-thickness sections were recorded using a combination of stage and beam scans. Due to the large grain size and thick lamellar structures of the
transformed microstructure, a step size of 10 µm was used and large areas of about 5 x 5 mm² to 7 x 7 mm² were analysed. Ideally, the EBSD scan of the transformed microstructure would have been about 10 times the size in order to characterise more than 1000 β grains. However, the chosen area size had to be a compromise between measurement time and the number of β grains. The individual α colonies on the orientation map could be easily identified, since they have the same crystallographic orientation. Due to the low volume fraction of β phase at room temperature in Ti-6Al-4V the number of β measurement points was insufficient to represent high temperature β phase texture. Therefore, β orientation was calculated from the α orientation based on the Burgers orientation relationship (Burgers, W. G. 1934) using the β reconstruction software developed by Davies et al. (Davies, P. S. 2007). Details about the β reconstruction software are found in section 2.2.4.2. The orientation data obtained from these maps were used to calculate the Orientation Distribution Functions (ODFs) using harmonic methods as implemented in TEXTAN III (Bate 1990) and the pole figures were recalculated from the ODF. The EBSD acquisition parameters that provided satisfactory indexing rates and the size of maps collected are listed in Table 3-4.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Map size (mm²)</th>
<th>Step size (µm)</th>
<th>Spot size (µm)</th>
<th>Magnification (µm)</th>
<th>Working distance (mm)</th>
<th>Beam current (kV)</th>
<th>Indexing rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RX-Ti-64</td>
<td>5 x 5</td>
<td>0.5</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>80</td>
</tr>
<tr>
<td>RX-Ti-64-0.4Y</td>
<td>5 x 5</td>
<td>0.5</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>78</td>
</tr>
<tr>
<td>β HT-Ti-64-1050°C</td>
<td>7 x 7</td>
<td>10</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>85.6</td>
</tr>
<tr>
<td>β HT-Ti-64-1150°C</td>
<td>7 x 7</td>
<td>10</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>82.4</td>
</tr>
<tr>
<td>β HT-Ti-64-0.4Y 1050°C</td>
<td>7 x 7</td>
<td>10</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>91.5</td>
</tr>
<tr>
<td>β HT-Ti-64-0.4Y 1150°C</td>
<td>7 x 7</td>
<td>10</td>
<td>6.5</td>
<td>200</td>
<td>27</td>
<td>20</td>
<td>92.9</td>
</tr>
</tbody>
</table>

Variant selection and its effect on texture in Ti-6Al-4V
Experimental methods

The reconstructed $\beta$ orientation map was further utilized to determine the average grain size so as to compliment the grain size measured from processed optical images using the line intercept method (ASTM E112). This method has also been implemented in channel 5 software, which uses the crystallographic orientation data to calculate the mean grain size rather than the processed image. It utilizes a misorientation angles between neighbouring grains to determine the grain boundary, in which case, crystallographic orientation variants within each grains has very little deviation ($<5^\circ$), whereas neighbouring grain orientation has large misorientation. The boundary between two neighbouring grains is referred to as high angle boundary and the threshold of misorientation angle for which a boundary is classified as high angle boundary is in the range of 5 to 15°.

3.3.2 Texture measurement by X-ray Diffraction

3.3.2.1 General Description of X-ray

For polycrystalline materials, the laboratory X-ray technique is an established method used to measure macrotextures. It gives an average texture for the specimen volume. Although laboratory X-ray diffraction is traditionally used to measure macrotextures, it can also be used for microtexture measurements depending on the size of the laboratory X-ray beam compared to the grain size. Small X-ray beam sizes have been utilized in synchrotron radiation, which provides higher intensities compared to the conventional X-ray tube (Wenk, H-R 2004). There are two modes used in X-ray measurements: transmission and reflection modes. For this study, the reflection method, developed by Schulz, L. G (Schulz 1949), was used, which employs a texture
goniometer. The principle behind X-ray diffraction is based on Bragg’s law (equation (3.3)).

\[ n\lambda = 2d \sin \theta \] (3.3)

Where \( n \) is an integer, \( \lambda \) is the wavelength of the electrons, \( d \) is the lattice spacing and \( \theta \) is angle of incidence. In reflection geometry only incomplete pole figures are measured i.e. pole distance of 80° from the sample surface normal (Wenk, H-R 2004).

The principal components of the laboratory X-ray system are the goniometer with the sample stage, X-ray generator, laboratory X-ray tube, detection system and computer system to record the intensities and analyse the data. Laboratory X-rays are produced when accelerated electrons with high velocity collide with the target materials, usually Cu, Co or Mo. The interaction between the electrons and the target leads to emission of energy with a range of wavelengths called continuous radiation. In addition, the electrons interact with the atoms of the target metal producing radiation with wavelengths characteristic of the target metal.

The atomic shells (K, L, M, etc) radiate different characteristic lines with specific wavelengths during interaction with electrons. These lines form characteristic spectrums of the target metal (Cullity, B. D., 1978). In X-ray diffraction, only the K lines with short wavelength are used. This is due to the fact that the short wavelength has lower absorption compared to long wavelengths (Noyan, I. C. and Cohen J. B 1987). The K-\( \alpha \) and K-\( \beta \) are the two strongest lines among the various K lines produced. Various K lines and the continuous spectrum that are not utilized during the X-ray analysis are filtered out using a monochromator.
Experimental methods

Figure 3-8 shows a schematic representation of the X-ray measurement technique. To obtain the crystallographic orientation of a given crystal, the setting of the goniometer with respect to the external reference frame has to be determined. During the measurement, the sample is exposed to the monochromatic beam at the correct diffraction angle for reflection at a particular lattice plane; the detector is set at the corresponding Bragg angle (2 theta) with respect to the incident beam. The reflected intensity is recorded only if the corresponding lattice plane is in the reflection condition. In most cases, when no reflection is obtained the sample is tilted to bring the crystal planes in reflecting condition. (Randle and Engler 2001).

Figure 3-8 Schematic representation of laboratory X-ray diffraction (wenk H-R, 2004)
Experimental methods

One of the limitations of X-ray is the defocusing effect (decrease in intensity) which causes deviation of the irradiated surface from the focusing sphere surface as the sample is tilted, especially at high angles. Other sources of defocusing are: misalignment of the goniometer, incorrect adjustment of the specimen and diffraction from different depths within the sample depending on the absorption coefficient. The defocusing effect can be determined using randomly oriented powder specimens; the correction can be achieved by interpolating with the known data of the existing bulk powder sample. It is important to mention that X-ray angular resolution is dependent on the size of the receiving slits. Narrow slit will result to good resolution and reduced X-ray counts and wide receiving slit, leads to lots of X-ray counts and poor resolution. Therefore, there have to be a compromise, typically 0.2 mm receiving slit size for maximum X-ray counts and resolution (~ 5°).

3.3.2.2 Texture measurement of Ti-6-Al-4V by X-ray diffraction

The texture measurements were performed using a laboratory X-ray diffractometer Philips ATC-3, equipped with a cobalt X-ray tube (wavelength 1.79285 Å) and texture goniometer. The goniometer with the sample was mounted in such a way that the rolling direction of the sample pointed towards the X-ray source. The machine voltage and current setting during the measurements was 40 kV and 45 mA, respectively. Three incomplete pole figure (0002), (10 10) and (11 20) and their background intensities were obtained during the experiment, which satisfies the minimum number of pole figures required to represent texture in hcp materials. The pole figures were measured for ten seconds at each location in 5° tilt steps from 0 to 85° and azimuthal step of 5° over the entire 360°. The typical peak position and diffraction spectrum of Ti alloys are shown in Figure 3-9.
Experimental methods

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Figure 3-9 Typical lab x-ray spectrum of β heat treated Ti-6Al-4V alloy

The raw pole figures data were fitted, background intensities and defocusing corrected using the software X”Pert Texture (PANalytical 2005). The background correction was done by subtracting the measured background intensities from raw pole figure data. The ODFs were calculated from corrected pole figures using direct method as implemented in the software X”Pert Texture (PANalytical 2005). The full width half-maximum (FWHM) from each peak of the three measured pole figure reflections were used to calculate the defocusing correction factor. The FWHM from the pole figure reflections and the corresponding 2θ values are listed in Table 3-5.

Table 3-5 X-ray parameters used for defocusing correction

<table>
<thead>
<tr>
<th>Hkl</th>
<th>2θ (°)</th>
<th>FWHM</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>41.2</td>
<td>0.6</td>
</tr>
<tr>
<td>002</td>
<td>44.9</td>
<td>0.7</td>
</tr>
<tr>
<td>110</td>
<td>75.3</td>
<td>0.6</td>
</tr>
</tbody>
</table>
Experimental methods

Texture measurements using Laboratory X-ray was restricted to as-rolled and recrystallised materials due to small X-ray beam size (~100 µm), small beam divergence (Eriko A, et al. 2008) and large grain size of the transformed materials (~500 µm). Due to small beam divergence, the incident beam might be hitting a single grain, which could lead to large variation of intensity when utilised for texture measurement of large grains. Although this problem could be minimised by simply oscillating the sample, but for the size of the sample (10 x10 x5 mm³) it was difficult to oscillate the sample. Also, unlike EBSD, X-ray does not provide additional β grain boundary information; which is critical factor in this present study. Therefore, for these reasons no X-ray measurement was carried out on the transformed materials.

3.3.3 Texture measurement by neutron diffraction

Neutron diffraction is an established tool for quantitative crystallographic analysis in polycrystalline materials. Neutron diffractometers using time-of-flight (TOF) principle operates in a different mode compare to convectional neutron diffractometers. Unlike convectional nuclear reactors, which has continuous output, at spallation sources focused bunches of high powered protons are made to hit the target periodically, producing a pulse of polychromatic neutrons (Sivia D. S, 2011). In neutron time-of-flight (TOF) the initial position and velocity of the pulse are fixed, while the final position and time after the neutron is detected are measured.

The Low absorption of neutrons is advantageous for studying texture in bulk materials with coarse grains due to deeper penetration depth and larger spot sizes of neutron beam compared to laboratory X-ray and electron diffraction. Neutron diffraction also eliminates the problem of defocusing observed in X-ray diffraction.
Experimental methods

Texture is measured by recording diffraction patterns as a function of scattering angle, either by rotating the sample on a goniometer or by using a multi-detector, which surrounds the sample.

The degree of texture information obtained during measurement is given by the product of the number of pole figures (hkl) times the number of sample orientations (Wenk H.-R and Van Houtte, P., 2004). Consequently, the use of polychromatic beam time-of-flight (TOF) neutron diffraction and many detectors at fixed scattering angles have significantly reduced the time of large volume texture measurements as both reciprocal space and orientation space are covered simultaneously in one measurement. The extraction of the orientation distribution function (ODF) from TOF neutron diffraction data is well documented in literature. MAUND (Von Dreele, R.B, 1997) and GSAS (Lutterotti, L. et al., 1997) has been developed to determine the orientation coefficient from TOF neutron diffraction data.

In this study, the General Material Diffractometer (GEM) beam line based at the neutron spallation source ISIS, United Kingdom was used. GEM is a high-count rate materials diffractometer designed to study both crystalline and amorphous materials. It has the ability to acquire texture information at a fast rate without rotating the sample. At ISIS neutrons are produced from liquid methane moderators at temperatures between -175 °C and -163 °C (Hannon, A. C., 2005). The relatively long flight path (length of the primary flight path) of GEM and time-of-flight leads to high resolution in reciprocal space, which is beneficial for powder diffraction. Consequently, good separation of Bragg peaks in powder diffraction patterns is achieved (Hannon, A. C., 2005). The primary flight path on GEM is devoid of air to prevent the attenuation of the beam by air scattering and there are a series of slits.
Experimental methods

positioned at different locations along the primary flight path to accurately maintain beam dimension. Other components found along the primary flight path are two disc choppers placed at different positions from the moderator and these are used to solve frame overlap problems. The sample on GEM is housed within an evacuated sample chamber, which is about 16.550 m from the moderator. The sample chamber contains an oscillating radial collimator, which adjusts the secondary flight path, hence reducing the background. The dimension of the incident beam on the sample is controlled by the beam-scrapper located before the sample.

The detectors are located outside the sample chamber. The detectors are composed of ZnS/6Li scintillator detectors with 7000 individual detector elements arranged in modules of about 100 parallel elements (Hannon, A. C., 2005). The modules are arranged such that each element is tangential to a Debye-Scherrer cone of diffraction. The modules are grouped into 6 detector banks with considerable coverage of approximately 4sr (4 sterad=unit of solid angle) (Kockelmann, W., 2006). The maximum solid angle possible is 4*pi =12.5 sr, which means that the detectors on GEM cover one quarter of the possible angles. This high coverage makes it possible to quantitatively analyse bulk texture without sample rotation. The detectors are extremely stable with 0.1 % variation in efficiency over a 24 h period (Kockelmann, W., 2006). The basic instrument parameters on GEM are shown in Table 3-6. It can be seen from this table that the angular resolution on GEM is 10 º. The schematic layout of 6 detector banks of the GEM array and the pole figure coverage for a single sample orientation are shown in Figure 3-10 and Figure 3-11. The various symbols in pole figures represent the angular location of the group of detectors coverage (Kockelmann, W., 2006).
### Experimental methods

#### Table 3-6 Basic instrument parameters on GEM

**Basic instrument parameters:**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>L1: Primary flight path</td>
<td>17 m</td>
</tr>
<tr>
<td>L2: Secondary flight path</td>
<td>1-2.5 m</td>
</tr>
<tr>
<td>Wavelength bandwidth</td>
<td>3.5 nm</td>
</tr>
<tr>
<td>d-spacing range</td>
<td>0.2-40 nm</td>
</tr>
<tr>
<td>Best resolution</td>
<td>0.34 %</td>
</tr>
</tbody>
</table>

**Angular coverage:**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 theta -coverage</td>
<td>1.2-171.4 °</td>
</tr>
<tr>
<td>1 element (typical of bank 5)</td>
<td>0.2 x 10 deg² (0.004 stetrad)</td>
</tr>
<tr>
<td>Total GEM</td>
<td>4 sterad</td>
</tr>
</tbody>
</table>
Figure 3-10 Schematic representation of the detector banks of the GEM detector arrays showing angular locations of the 6 detector group (Kockelmann, W 2006).

Figure 3-11 Pole figure coverage for single sample orientation.
3.3.3.1 Texture measurement of Ti-6Al-4V by Neutron diffraction

In order determine the degree of variant selection occurrence during $\alpha \rightarrow \beta$ (heating) and $\beta \rightarrow \alpha$ (cooling) it was essential to carry out in situ texture measurements on samples that had the same starting condition as the ones investigated by ex-situ studies.

Due to the poor scattering properties of Ti-6Al-4V, relatively long counting times of 30 min were necessary to measure 6 reflections with sufficient accuracy. Cuboids of $20 \times 15 \times 5 \text{ mm}^3$ were cut from the recrystallised samples and in situ neutron diffraction measurements were performed up to 1250 ºC. The samples were fitted into either a vanadium sample holder (room temperature experiment) or niobium sample holder (high temperature experiment) that was subsequently inserted into a vacuum furnace with niobium heating elements. The incident beam was $30 \times 20 \text{ mm}^2$ and the sample was orientated to have the rolling direction parallel to the incident beam. In this way, the entire sample volume of 1500 mm$^3$ was illuminated. Texture measurements were carried out at room temperature, 800, 950, 1050, 1250 ºC and cooled to 210 ºC at 10 ºC/min. The samples were allowed to stabilise for 10 min before a texture measurement was undertaken, bringing the total measure time per point to 40 min. The processing routes for the material before undertaking the GEM experiment are again shown in Figure 3-12a. The temperature profile applied during the in situ texture measurement is displayed in Figure 3-12b.
Experimental methods

An assembly of 164 separate detector groups was used during the experiment with each group having an angular coverage of ~10° x 10°. The data were normalized to the incident neutron flux distribution and corrected for detector efficiencies. The 164 detector groups were converted into 164 d-spacing patterns. The 164 diffraction patterns were simultaneously Rietveld refined using the MAUD software (Von Dreele R. B., 1997, Lutterotti, L. et al., 1999). The diffraction spectra, together with the

Figure 3-12 The schematic representation of β quenching (H), uni-directional rolling (UD), recrystallisation (RX), (b) solution heat treatment (β-HT) profile of Ti-6Al-4V and Ti-6Al-4V-0.4Y processes.
Experimental methods

fits of measured data using the vanadium and niobium sample holders are shown in Figure 3-13

![Image](image.png)

Figure 3-13 Measured spectra at room temperature of sample in the vanadium holder, (b) Fitting of measured spectra at room temperature of sample in the vanadium holder, (c) measured spectra at 950°C of sample in the niobium holder, (d) Fitting of measured spectra

As a result of texture, the peak intensities of each of the measured spectra do not conform to those predicted by structure factor. Therefore, each cell of the orientation distribution function was refined in order to best fit the intensities of the Bragg peaks in the calculated rietveld spectra to each of the 164 measured spectra. This analysis was performed using the extended Williams-Imhof-Matthies-Vinel (E-WIMV) algorithm as implemented in MAUD and plotted using TEXTAN III (Bate, P 1990). Individual pole figures \{0002\}, \{10\overline{1}0\}, \{1\overline{1}20\} were recalculated from the ODF and plotted using the Beartex software (Wenk H-R, et al. 1998).
Chapter 4  **Data Processing**

In this chapter the detailed EBSD data post-processing procedures were outlined. The additional analysis tools such as the $\beta$ reconstruction software were necessary in order to gain understanding of the microstructure and texture of the high temperature $\beta$ phase, which is critical in understanding variant selection. The code developed to determine the common (110) neighbours was very useful to explain the effect of processing temperature on the variant selection mechanism related to neighbouring $\beta$ grain orientations. The detailed procedure for fitting and refinement of the TOF raw data was covered. Finally, this part of the thesis gives a clear understanding of procedures used to accomplish the results presented in subsequent chapters.

### 4.1  **EBSD Post processing**

The post EBSD processing using the Channel 5 software is aimed at obtaining high quality $\alpha$ inverse pole figure (IPF) maps (for reconstruction of high temperature $\beta$ phase) by reducing the effects of pseudosymmetry and misindexing points as previously discussed in section (3.3.1.5). The non-indexed points are identified as white patches on the map; the systematic mis-indexing of the hexagonal phase corresponding to a 30º rotation about the $c$ axis appear as dark patches on the map. This mis-indexing could result from two possible sources: poor pattern quality due to imperfection of crystals close to the grain boundaries and when the Kikuchi patterns obtained cannot be resolved by the software due to the overlap of two patterns. The systematic post-processing steps are itemized below and exemplified in the Figure 4-1(a-d),
(1) Removal of wild spikes: These are isolated points on the map.

(2) The second step is extrapolation of the non-indexed points from neighbouring index points using HKL Channel 5 software; this is done in steps, aimed at reducing the percentages of non-indexed points. This is a necessary step to prepare α maps used for β reconstruction because during β reconstruction from the α variant, the adjacent neighbouring variant is required. However, the process of removal of non-indexed points is used with caution, since large portions of non-indexed points can indicate the presence of unknown phases or contamination. After noise reduction, the β phase is removed to obtain a complete α phase. This file is loaded in the β reconstruction software.
developed by Davies et al. (Davies. 2007) (described in detail in section 2.2.4.2). The reconstructed file is reloaded in Channel 5 software to plot the β phase IPF map illustrated in Figure 4-2(a-d)

![Figure 4-2 EBSD data post processing](image)

Figure 4-2 EBSD data post processing (a) raw β orientation IPF map after β reconstruction, (b) after wide spike correction, (c) after mis-indexing correction showing the β grain boundaries, (d) band contrast map.

(3) Grain boundary determination: Two types of β/β grain boundaries were determined by superimposing the β IPF map on the α IPF map. The analysis of β grain boundaries showed evidence of α variants with similar orientations on both sides of the prior β grain as previously reported in (Bhattacharyya, D 2003, Stanford, N 2004). The β grain boundaries of the 150 grains studied are illustrated in Figure 4-3. The β Boundaries with the specific misorientations that favours these β/β boundaries with similar α variants as reported by Germain (Germain et al., 2007), are indicate with white circle on the map and black circle representing grain boundaries with different α orientations variants nucleating on both sides of the grain boundaries.
4.1.1 Determination of number common <110>β poles

The aim of this calculation is to determine the number β grain pairs with closely aligned (110) poles by determining the misorientation angles between them, which in this case should be <10°. A statistical analysis was carried out on the reconstructed β texture. The reconstructed β orientation data was used to plot β texture ODF using TEXTAN III (Bate, P 1990). Due to large orientation data, the β texture was recreated with randomly outputted 1000 Euler angles from the ODFs of the β phase generated during rolling at 800 °C and 950 °C as shown in Figure 4-4 a and c respectively.
Comparing the generated \( \beta \) texture ODF with the recreated \( \beta \) texture ODFs (b and d) confirms that 1000 Euler angles were sufficient to accurately represent measured \( \beta \) texture. The present approach to calculate the likelihood of close (110) pole alignment consists of 3 steps: (1) determination of the components of the basis vector, (2) calculation of the translation vector of the reciprocal lattice and hence the reciprocal lattice vector of the 6 \{110\} (normals) and finally (3) determination of angles between their normals when pairing all combinations of 1000 Euler angles. A crystal of any orientation can be described using the three sets of Euler angles \{\phi_1, \Phi, \ldots\}.
Variant selection and its effect on texture in Ti-6Al-4V

\[ C_c = g^\beta C_s \] (4.1)

Where \( C_c \) and \( C_s \) are the crystal and sample coordinate systems. The basis vector \((a, b, c)\) is calculated by first determining the orientation matrix, \( g^\beta \), for each of the 1000 Euler angles using equation (4.2); both columns and rows of the matrix are unit vectors.

\[
g^\beta = \begin{bmatrix}
\cos\phi_\beta \cos\varphi_\beta & -\sin\phi_\beta \sin\varphi_\beta \cos\beta & \sin\phi_\beta \cos\varphi_\beta \\
-\cos\phi_\beta \sin\varphi_\beta & -\sin\phi_\beta \cos\varphi_\beta \cos\beta & \sin\phi_\beta \sin\varphi_\beta \\
\sin\phi_\beta & \cos\phi_\beta & 0
\end{bmatrix}
\] (4.2)

The three cell unit vectors \((a, b, c)\) are converted into Cartesian co-ordinates by multiplying the transpose of this \( g^\beta \) matrix with the three specific crystal directions \([100], [010] \) and \([001]\), using the following equation (4.3)

\[
a = g^\beta^\top \begin{bmatrix} 1 \\ 0 \\ 0 \end{bmatrix}, \quad b = g^\beta^\top \begin{bmatrix} 0 \\ 1 \\ 0 \end{bmatrix}, \quad c = g^\beta^\top \begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix}
\] (4.3)

We can obtain the reciprocal lattice base vectors \((a_1, a_2, a_3)\) and hence the reciprocal lattice vector in Cartesian space \((u^*(hkl))\) of any of the 6 \(\{110\}\) in crystal space using equation (4.4) and (4.5)

\[
a_1 = \frac{b + c}{a(b + c)}, \quad a_2 = \frac{c + a}{a(b + c)}, \quad a_3 = \frac{a + b}{a(b + c)}
\] (4.4)

\[ u^*(hkl) = ha_1 + ka_2 + la_3 \] (4.5)

Where \((hkl)\) are the Miller indices of the \(\{110\}\) plane. Finally, the angles, \(\theta\) between six reciprocal lattice plane normals of one set of Euler angle and the reciprocal lattice plane normals of the second set of Euler angles \(([hkl]_1 \) and \([hkl]_2 \) normals) were
calculated using equation (4.6). The calculation is done for the 1000 sets of Euler angles, which generated thirty six million angles.

\[
\theta = \cos^{-1} \left( \frac{u^*(h_1k_1l_1)u^*(h_2k_2l_2)}{[u^*(h_1k_1l_1)][u^*(h_2k_2l_2)]} \right) \tag{4.6}
\]

Due to large volume of data generated by this calculation, a Matlab code was developed which does this calculation automatically and plots frequency of occurrence of each angle. It was possible to identify angles that are <10°, which have likelihood of having neighbouring β grains with closely aligned (110) β planes. The details of this code are found in appendix 3.

### 4.2 Neutron diffraction data processing

The analysis of the TOF data was carried out using the Rietveld (Rietveld 1969) method as implemented in the software MAUD (Lutterotti, L 1997). The MAUD program has many functions, which include texture and residual stress calculation. The raw data files obtained from TOF texture measurement was converted into a *par template which was produced by Ariel data processing program on GEM. Ariel normalizes the data to incoming neutron flux distribution, makes corrections for detector efficiencies, and separate the 7000 GEM detector elements into 164 groups. Another important file is CIF file which contain the name of data file and number of detector groups. The CIF files are generated for 6 GEM detector banks.

The CIF and the *par file should be in the same working directory; the *par file is loaded into MAUD and both crystal and instrument parameters are refined until the calculated diffraction spectra match the experimental data. The refinement procedures are as follows:
(1) Refine scale and background parameters

(2) Refine instrument and structure parameters (no texture)
   (a) Refine lattice parameters for two phase alloy, the lattice parameter to
       refine depends on the phase that is dominant
   (b) Sample displacement (y, z, only)
   (c) 2theta/zero Shifts (pattern) displacements
   (d) Refine B factor (iso)
   (e) Refine phase volume fractions

After 1 and 2 refining steps there was a good fit between the experimental data and
background, peak position and peak profile. Step 1 and 2 is followed by step 3,
texture refinement procedure.

(3) The texture refined using E-WIMV incorporated in MAUD and setting the
    ODF resolution to 10°

An incomplete pole figure was extracted from each diffraction peak and the E-WIMV
algorithm was used to calculate ODFs from these pole figures. The \{0002\}, \{10\bar{1}0\},
\{11\bar{2}0\} pole figures were recalculated from the ODFs from the MAUD processed
data using Beartex software (Wenk H-R, et al. 1998). The pole figures data was
converted the orientation files and plotted as ODFs and pole figures using TEXTAN
III (Bate, P 1990).

4.3 **ODF and pole figure plots using TEXTAN III**

The transformations and the representations of texture (ODFs and pole figures) were
carried out using TEXTAN-III, an in-house software developed by Bate, P (Bate P,
1990). In TEXTAN-III, It was developed based on the symmetric spherical harmonic
functions proposed by Bunge, H. J (Bunge, H. J, 1982). This is an infinite series
expansion, but it is clear that a truncation at a finite value of \( l = L_{\text{max}} \) is necessary in practice, which depends on the number of data points measured. For single orientation a smaller \( L_{\text{max}} \) value (8 to 10) will result in a smoother curve, which represents the ‘distribution density’ of the orientation. In general for robust calculation of large data, the value of \( L_{\text{max}} \) is chosen to be sufficiently large (22 to 34) to permit reproducing the essential features of the orientation distribution. The Gaussian half width controls the orientation spread and does not add to the intensity. In this software a modest value of 5° Gaussian half widths was maintained in all the ODFs and pole figures plots. The software was also utilised to predict α textures from high temperature β phase texture assuming no variant selection. The Burgers orientation relationship is applied as a rotation of Euler angles taking into account the crystal symmetry of the parent and daughter crystals. The α textures were predicted from individual β texture, as shown in the result section of this thesis. It is important to state that due to both crystal and sample symmetrical effect was taken into accounted in this software the symmetrical equivalent of each individual orientation component is included in the ODF.
Chapter 5

Effect of β Grain Growth on Variant Selection and Texture Memory Effect during α → β → α Phase Transformation in Ti-6Al-4V
Effect of β Grain Growth on Variant Selection and Texture

Memory Effect during α → β → α Phase Transformation in Ti-6Al-4V

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Manchester M1 7HS, UK

Abstract

In the present study, the texture evolution and the role of β grain growth on variant selection during β → α phase transformation have been investigated in Ti-6Al-4V with and without 0.4wt% yttrium addition. The aim of adding yttrium was to control β grain growth above the β transus by pinning grain boundaries with yttria. Both materials were first thermomechanically processed to generate similar starting microstructures and crystallographic textures. Subsequently, both materials were solution heat treated above the β transus followed by slow cooling to promote growth of the α lath structure from grain boundary α. Additional interrupted slow cooling experiments were carried out to identify the α lamellae that nucleate first from β grain boundaries. Detailed EBSD analysis was carried out and it was found that the β heat treatment did not generate new texture components although the intensities of the individual components changed dramatically depending on the alloy/β grain size. Variant selection was assessed by comparing measured α texture components with predicted α texture components based on the high temperature β texture assuming equal variant selection. It was found that with increasing β grain size variant selection intensified favouring the \( \{\varphi_1, \phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} \) texture component. Interrupted cooling experiments revealed that α nucleates first on β grain boundaries that are formed by
two β grains having a common (1 1 0) normal and that these α lamellae display almost exclusively a {φ1, Φ, φ2} {90°, 30°, 0°} orientation. Consequently, the dominance of this variant with increasing β grain size can be related to the relative free growth of this particular α texture component into an “empty” β grain.

**Keywords**: Ti-6Al-4V, Grain growth, Texture evolution, Variant selection and Texture memory

1. Introduction

Microstructures in most engineering alloys are controlled by thermomechanical processing. Titanium alloys are no exception and due to their low temperature hexagonal close packed crystal structure, they are also particularly prone to develop strong crystallographic deformation textures that can significantly affect the mechanical properties of the final product [1]. Titanium, as well as Zirconium, exhibits an α hexagonal close packed (hcp) phase at low temperature, which transforms to a β body centre cubic (bcc) phase at high temperature. By adding β stabilising alloying elements it is possible to design two-phase titanium alloys, which generally display superior mechanical properties compared to a single phase alloy. Titanium alloys are usually processed from ingot by using vacuum melting and subsequently subjected to a series of thermomechanical processing stages in the single β phase and the α + β phase field [2]. β heat treatment is part of the processing route in order to chemically homogenise the material. Due to the cleanliness of typical titanium alloys, and hence absence of carbides or other high temperature non-metallic precipitates, heat treatment in the single-phase β region leads to very rapid grain growth. The phase transformation during heating (α → β) and cooling (β → α) is governed by the so–called Burgers orientation relationship {0 0 0 2}_α || {1 1 0}_β and <1 1 -20>_α || <1 1 1>_β) with 6 possible β orientation variants during the α→β phase transformation and 12 possible α orientations that can
transform from a single parent β grain during β→α phase transformation [3]. The two parallel planes are both closest packed planes while the two parallel directions correspond to the nearest neighbour direction [4]. The crystallographic orientation relationship is represented schematically in Figure 1. If all 72 variants occur with equal probability during an α → β→α phase transformation the final α texture should be essentially randomised regardless of the initial α texture. However, it has been demonstrated by a number of researchers [5-6, 7-8] that during both displacive and diffusional phase transformation of titanium and zirconium alloys, the initial α texture is to a certain extent retained after a full heat treatment cycle. This “texture memory” effect has been reported widely for single crystal titanium [7] and polycrystalline titanium and zirconium alloys [8, 9] and has often been attributed to variant selection. For example, for zirconium and titanium, Romero et al [5] and Lonardelli et al [6] demonstrated that perfect texture memory is a result of incomplete α to β phase transformation. Although it is clear that any retained α during heat treatment can act as a nucleation site during cooling, it was also suggested that the absence of β grain growth due to the pinning of retained α might contribute to the perfect memory effect [5].

Variant selection mechanisms in both bcc and hexagonal materials during phase transformation have often been linked to transformation strain and elastic anisotropy [10, 11]. In the case of β grains transforming to α under unconstrained conditions, contraction in the range of 10% is expected along a <1 0 0>β direction and contraction of 2% and expansion of 10% along the two perpendicular <1 1 0>β direction [12]. Bhattacharyya, et al. [13] reported that in the case of neighbouring β grains with a common (1 1 0)β pole, (0 0 0 2)α variants are selected on both sides of the prior β grain boundary with almost the same crystallographic orientation. The two (0 0 0 2)α variants are closely alignment with the common <1 1 0>β pole in preference to the other five <1 1 0>β poles. Even though numerous studies on the crystallography of these “special β grain pairs” [14-16] confirmed the suggestion made by
Bhattacharyya, the fundamental concept responsible for this variant selection mechanism is poorly understood. This observation is of fundamental importance from a practical point of view since these large regions of practically identical crystallographic orientation are suspected to be the root source for the formation of macrozones [17]. More recently, Cayron [18] suggested that the β grains with the common (1 1 0)β planes are β variants inherited from the α parents that transform to β during the α-β phase transformation.

The aim of the present work has been to shed further light on the α variant selection mechanism described by Bhattacharyya, et al. [13], investigate the effect of β grain growth and also study its practical impact on texture formation. Since β grain growth is very difficult to control in an alloy such as Ti-6Al-4V alloy, a second variant was produced that includes 0.4 wt% yttrium in order to pin β grain boundaries in the high temperature single phase region [19]. In-house thermomechanical processing was carried out to achieve the best comparable microstructure and texture of the two alloys before undertaking β heat treatments to study variant selection.

2. Experimental Methods

2.1 Material

The material used in the present study was Ti-6Al-4V produced by conventional ingot metallurgy and uni-axially rolling and provided by TIMET, UK. Some of this material was remelted at the University of Birmingham to add 0.4 wt.% of yttrium. The nominal chemical compositions of both alloys are shown in Table 1. DSC measurements of both alloys determined a β transus of 990 °C, confirming indirectly, that the remelting procedure for the yttrium containing alloy had not changed the alloy chemistry apart from the yttrium addition (no significant oxygen pick up). Blocks of 40 × 40 × 20 mm were cut from both alloy variants and heat treated at 1050 °C followed by water quenching. These blocks were subsequently
uni-directional rolled using a calibrated furnace set to 950 ºC. The soaking time of the samples was 1 hour before starting the rolling process. A total reduction of 75% was achieved by having a 15% reduction per pass with 2 min reheating between each pass. The rolling and heat treatment processes are shown schematically in Fig. 2.

The rolled material was subsequently heat treated for 8 hours at 950 ºC followed by 1 ºC min\(^{-1}\) cooling in a tube furnace under argon atmosphere. Samples with dimensions of 10 × 10 × 5 mm\(^3\) were cut from the centre of the small slabs and subjected to β heat treatments in an argon atmosphere at 1050ºC and 1150 ºC for 30 min and again subsequently cooled at 1 ºC min\(^{-1}\) to room temperature.

Mid sections of the β heat treated samples were prepared following standard grinding/polishing procedures and etched in 2% HF, 8% HNO\(_3\) and 90 % H\(_2\)O (Kroll’s reagent) for 10 s for optical inspection. Electron backscatter diffraction (EBSD) was used to characterize the texture inherited from the \(\alpha \rightarrow \beta \rightarrow \alpha\) transformation. To improve Kikuchi pattern quality, the samples were OPS polished (1 hour) and etched repeatedly with the last polishing step taking 2 hours.

A CamScan Field Emission Gun Scanning Electron Microscope (FEGSEM), equipped with HKL Channel 5 software was used for EBSD data acquisition and analysis. Due to the coarse β grain size and large α colony structure, a step size of 10µm was used, which allowed covering an area of 7 × 7 mm\(^2\) during each scan in order to measure a reasonable number of β grains for texture analysis. The EBSD orientation data was used to plot pole figures and the orientation distribution functions (ODFs). One of the experimental difficulties when studying variant selection is the requirement to determine the high temperature β texture. The most reliable in-situ techniques, which are not compromised by surface effects, are neutron and high energy synchrotron x-ray diffraction. Although they can provide excellent information of the global texture, they do not provide information on the microstructural scale, i.e.
information on neighbouring $\beta$ grains. In order to determine the high temperature $\beta$ texture, the $\alpha$ orientation maps, recorded by EBSD, were used to reconstruct the $\beta$ grain structure based on the Burger’s orientation relation between the parent and daughter phases. The software used for this $\beta$ reconstruction has been developed at the University of Sheffield within IMMPETUS (Institute for Microstructural and Mechanical Process Engineering). The software checks each $\alpha$ lath and identifies the neighbouring laths that are most likely to have the same parent $\beta$ orientation. In order to obtain a successful reconstruction a minimum allowable misorientation angle between $\alpha$ variants of a common $\beta$ grain is defined, which in the present case was $3^\circ$. Further details on the methodology are described in [20-23] and details regarding the software can be found in [20]. The information from the reconstruction was subsequently reloaded into Channel 5 to plot the $\beta$ orientation maps. These maps were also used to measure $\beta$ grains size distributions based on the linear intercept method (ASTM E112) [24] and identify neighbouring $\beta$ grains with common $\langle 1 1 0 \rangle$ poles. The total number of measured $\beta$ grains was about 150 for each temperature condition (1050 ºC and 1150 ºC). In order to assess the level of variant selection the expected $\alpha$ texture, assuming no occurrence of variant selection during the $\beta \rightarrow \alpha$ transformation, was calculated from the reconstructed $\beta$ texture. The transformations and the representations of texture (ODFs and pole figures) were carried out using TEXTAN-III, an in-house software developed by P. Bate [25]. In TEXTAN-III, the Burgers orientation relationship is applied as a rotation of Euler angles taking into account the crystal symmetries of the parent and the daughter crystals. It applies the transformation to experimental data by using the spherical harmonic method proposed by H. Bunge [26].
3. Results

3.1 Microstructure and Texture Evolution

Optical micrographs of Ti-6Al-4V with and without yttrium are shown in Figures 3a and b after recrystallising the material at 950ºC for 8 hours. The figures reveal in both cases a fully equiaxed primary α microstructure with a mean grain size of 18 μm and retained β at the triple junctions of primary α (α_p). The α textures of the two alloys at this stage are presented in form of ODF sections and representative quarter pole figures, in Figure 4. The ODF section presented here are the \( \varphi_2 = 0^\circ \) for α phase texture and \( \varphi_2 = 45^\circ \) for β phase texture [27].

The ODF sections at \( \varphi_2 = 0^\circ \) of each alloy have a strong transverse texture component at Euler angles \( \{\varphi_1, \Phi, \varphi_2\} \{0^\circ, 90^\circ, 0^\circ\} \) with maximum intensity of 16 times random intensity. This orientation corresponds to the basal pole components rotated towards TD (transverse direction during rolling) in the \( (0 0 0 2) \) pole figure as shown in Figure 4c and d. This is the so-called transverse texture often found in Ti-6Al-4V deformed at temperatures above 900 ºC. The second component is a weak orientation component at Euler angles \( \{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} \) with maximum intensity of 6 times random intensity. This orientation corresponds to basal poles rotated about 30º away from ND (= normal direction to the rolling plane) towards RD (rolling direction) in the \( (0 0 0 2)\alpha \) pole figure. The similarity of the grain morphology and the α textures in both alloys demonstrates that yttrium additions did not have any significant effect on the microstructure produced during thermomechanical processing. Hence, an almost identical starting microstructure was achieved in the two alloys before undertaking any β heat treatment for variant selection studies.

Optical micrographs of the fully transformed lamellar microstructures are presented in Figure 5 for Ti-6Al-4V and Ti-6Al-4V-0.4Y heat treated at 1050 ºC and 1150 ºC. The microstructures are populated by large lamellar colonies of α. In the case of conventional Ti-6Al-4V the prior β grains are easily identified by continuous film of grain boundary α (α_{GB})
in Figure 5a and b, whereas in the yttrium containing alloy, Figure 5c and d, grain boundary $\alpha$ was less distinct. However, in both cases it was possible to confirm that $\alpha$ colonies had mainly grown from grain boundary $\alpha$. There was no indication that $\alpha$ had predominantly nucleated from yttria particles. In order to reduce uncertainty in determining the $\beta$ grain size in Ti-6Al-4V-0.4Y, all $\beta$ grain size measurements were based on reconstructed EBSD maps of the $\beta$ phase. The grain size distribution and mean grain size was determined by defining high angle boundaries to have a misorientation of at least 5º. Figure 6 compares the grain size distribution of the two alloys when heat treated at 1050 ºC (Figure 6a) and 1150 ºC (Figure 6b). It can be seen that conventional Ti-6Al-4V displays a wider grain size distribution than the yttrium variant for both heat treatment temperatures with a significant tail towards grain sizes exceeding 1 mm. Table 2 summarises the mean grain size values for the 4 conditions. It shows that the mean $\beta$ grain size of conventional Ti-6Al-4V is about twice as large as for Ti-6Al-4V-0.4Y when heat treated at the same temperature. It can also be seen that increasing the temperature to 1150 ºC further coarsens the $\beta$ grains in both alloys although the temperature effect is small compared to the effect of yttria pinning $\beta$ grain boundaries.

The $\alpha$ transformation texture of both alloys after $\beta$ heat treatment at 1050 ºC and 1150 ºC are shown in the ODF sections at $\varphi_2 = 0^\circ$ in Figure 7. The two alloys show similar texture components but significant differences in the texture intensity. The $\alpha$ textures of the conventional Ti-6Al-4V presented in Figure 7a and b are composed of a comparatively weak <0001> fibre texture component with maximum intensity of 6 times random while the second component with Euler angles \{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} is very strong with maximum intensities of 12 (T = 1050 ºC) and 14 (T = 1150 ºC) times random. In the case of the yttrium containing alloy it is obvious from Figure 7c and d that the same texture components are found but that the intensities of the <0 0 0 1> fibre texture and the second component at the Euler angles \{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} are very similar. If one compares the strength of the \{90^\circ, 30^\circ, 0^\circ\} texture component between the four samples it can be seen that the

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Variant selection and its effect on texture in Ti-6Al-4V
conventional alloy heat treated at 1150 °C shows the strongest intensity, followed by the conventional alloys heat treated at 1050 °C and the yttrium variant heat treated at 1150 °C and at 1050 °C. Therefore, there seems to be a clear link between texture intensity and β grain size. When comparing the ODF sections at φ2 = 0° in Figure 4 and 7 it also becomes clear that the main texture components are the same even though their intensities are different. This suggests that the transformation texture is affected by texture memory as previously reported for titanium alloys by a number of researchers (for example [7, 8]).

3.2 Variant selection

As mentioned before, in order to judge the level of variant selection, one has to compare the measured α texture after β to α phase transformation with the predicted α texture based on the β texture assuming no variant selection. This was carried out for the four different conditions in the present study, Ti-6Al-4V with and without yttrium and heat treated at 1050 and 1150 °C. The texture results are presented in Figure 8 for Ti-6Al-4V and Figure 9 for Ti-6Al-4V-0.4Y. In all cases, the β texture displayed comparatively strong α-fibre and weak γ-fibre texture components, which is a characteristic of hot rolled bcc materials. The strong α texture at the Euler angles {90°, 30°, 0°} was not the product of the strong α-fibre component as would be expected but was the product of one of the weak γ-fibre texture components rotated 10° away from ND at the Euler angles {φ1, Φ, φ2} ( {90°, 66°, 0°} ±5°) as shown with dotted line in Figure 8a. The discrepancy between the modelled and measured α texture for Ti-6Al-4V heat treated at 1050 and 1150 °C is clearly visible when comparing Figure 8c and d with Figure 8e and f. The intensities of the two main texture components from the modelled α texture are about 6 times random intensity while the measured α texture showed 12 times random intensity when heat treated at 1050 °C and 14 times random intensity when heat
treated at 1150 °C. In the case of Ti-6Al-4V-0.4Y a very different picture is seen. Here, the modelled (Figure 9c and d) and measured (Figure 9e and f) α textures look far more similar although the intensities of the measured texture components are still slightly higher than what would be expected from the predictions assuming no variant selection. The intensities of the modelled α texture components is about 6 times random intensity whereas the measured α texture components show intensities of 7 times random intensity when heat treated at 1050 °C and 8 times random intensity when heat treated at 1150 °C. Both alloys also indicate that the discrepancy between predicted and measured α texture increases with increasing heat treatment temperature.

In summary, the results of the texture measurements clearly indicate that there is a strong correlation between β grain growth and level of variant selection when heat treating Ti-6Al-4V and the yttrium containing variant above the β transus. In the following section, the possible route causes are discussed for such correlation.

4. Discussion

It is clear from the Burger’s relationship that the transformation from α → β and back to α assuming no variant selection will result in multiplying the α orientation components since a single α grain can theoretically give rise to six β orientations and a single β grain should transform to twelve α orientations [27]. Consequently, even an exceptionally strong and sharp α texture should be changed dramatically when such the material is heat treated above the β transus. In the present investigation, it was observed in Figure 7 that the β heat treatment did not produce new α texture components compared to the starting recrystallisation texture shown in Figure 4. Instead, only the intensities of the already present components changed with the intensity of the weak {φ1, Φ φ2} {90°, 30°, 0°} texture components being strengthened whereas the intensity of the <0 0 0 1> fibre texture was reduced. The retained texture components are an indication of some texture memory in Ti-6Al-4V, which is not
related to any retained α grains, as previously reported in [6], since the material was heat treated well above the β transus (+50 °C and +150 °C for Ti-6Al-4V).

In addition, the present study has demonstrated that with increasing β grain size the \{90°, 30°, 0°\} texture component is strengthened significantly. An important factor of the present work is that the samples were cooled down slowly from above the β transus promoting α lath growth from grain boundary α (which sits on the prior β grain boundaries). Figure 10a displays the orientation of the α lath structure using IPF colouring in combination with the β grain boundaries (black lines, at least 5° misorientation criterion) that were determined from the β reconstruction. The analysis of these α colonies in combination with β grain boundaries showed evidence of α variants with similar orientations on both sides of the prior β grain as previously reported in [13, 14]. The β grain boundaries of the 150 grains studied here can be divided into two groups: Group 1 represents boundaries with α variants of similar orientations nucleating on both sides of the prior β grain boundaries and Group 2 represents grain boundaries with α variants of different orientations nucleating on both sides of the grain boundaries, see Figure 10b. As expected, a common \(<1 1 0>\)β pole of the two neighbouring grains was identified if the grain boundary belonged to Group 1, as demonstrated in Figure 10c. It was also found that in this case the misorientation between the two common \(<1 1 0>\)β poles did not exceed 10°. For better visualization, the three-dimensional crystal orientation of the α products and β grains are shown Fig 10 d and e. Although, the two β grain orientations in Figure 10e are not completely similar, they both have one (1 1 0) plane with similar poles and consequently α products nucleated on both sides of the β grain boundary with the same (0 0 0 2) crystallographic orientation. Importantly, these α products correspond in most cases to the texture component with Euler angles \(\{\varphi 1, \Phi, \varphi 2\}\) \{90°, 30°, 0°\}, which is the texture component that has become dominant after transformation.

Figure 11a and b highlights the regions in the orientation map that represent the \{90°, 30°, 0°\} texture component. Since these maps also include the β grain boundaries it can be seen
clearly that most of the \{90^\circ, 30^\circ, 0^\circ\} texture component regions are across \(\beta\) grain boundaries providing evidence that the development of the strong \{90^\circ, 30^\circ, 0^\circ\} texture component is indeed related to the variant selection mechanism of common \(\langle 1 \ 1 \ 0 \rangle \beta\) poles. Figure 11 also demonstrates that the \(\{90^\circ, 30^\circ, 0^\circ\}\) texture component regions have grown substantially larger in coarse \(\beta\) grains (Figure 11a) compared to small \(\beta\) grains (Figure 11b) providing the link between \(\beta\) grain size and dominance of the \(\{90^\circ, 30^\circ, 0^\circ\}\) texture component.

Due to the Burgers relationship, \(\alpha\) variants within the same \(\beta\) grain can only produce 5 different misorientation angles and axes, which are \(60^\circ/\langle 11-20 \rangle\), \(60.83^\circ/\langle 1.377,-1,2.377,0.359 \rangle\), \(63.26^\circ/\langle 10,5.5,-3 \rangle\), \(90^\circ/\langle 1-2.38,1.38,0 \rangle\), and \(10^\circ/\langle 0001 \rangle\). The ratio between the 5 is 2:3:2:2:1 in the absence of any variant selection [28]. Since in the present case different levels of variant selections have been observed, the relative misorientation distributions have been plotted and compared to the case of no variant selection in Figures 12. Since the \(60^\circ\), \(60.83^\circ\), \(63.26^\circ\) misorientation angles were indistinguishable; they were merged in Figure 12 to a single misorientation angle labelled \(60^\circ\). Figure 12 shows that the measured misorientation angle distributions differ from the expected one. The difference is relatively small for Ti-6Al-4V-0.4Y heat treated at 1050 °C, which displays the smallest \(\beta\) grain growth, and largest in the case of conventional Ti-6Al-4V, which also has the largest \(\beta\) grains. This further confirms the increasing level of variant selection with increasing \(\beta\) grain size. It can also be seen that the frequency of the \(10^\circ\) misorientation angle goes up with increasing \(\beta\) grain size. This can be interpreted as an indication of the increasing length scale of Group 1 \(\beta\) grain boundaries resulting in low misorientation \(\alpha\) colonies across the former \(\beta\) grain boundary, which is also evident when comparing Figure 12a and b.

In order to further understand the significant growth of the \(\{90^\circ, 30^\circ, 0^\circ\}\) texture component with increasing \(\beta\) grain size, early growth of the \(\alpha\) lamellae from \(\beta\) grain boundaries was studied by applying interrupted cooling experiments. A Ti-6Al-4V sample was heat treated at 1150 °C for 30 min and cooled at 1 °C min\(^{-1}\) to 975 °C followed by water quenching. Even
though fast quenching does not enable one to retain the \( \beta \) phase the different lath morphology, generated during quenching compared to slow cooling, made it easy to identify the \( \alpha \) lamellae that had formed during the early stage of cooling. Figure 13a and b show SEM micrographs that were recorded from such samples. A large number of \( \beta \) grain boundaries were studied in this way and it was found that in the early stage of diffusional transformation the nucleation of \( \alpha \) products on both sides of \( \beta \) grain boundaries were largely favoured (Figure 13a) compared to \( \alpha \) lamellae nucleating on only one side of the \( \beta \) grain boundaries (Figure 13b). It has been suggested in [13] that the interfacial energy of the grain boundary is minimized if the \( \alpha \) variants on both sides of the boundaries select one specific \( (1 1 0)\beta \) plane common to both \( \beta \) grains. The similar d-spacing values of the \( (0 0 0 2)\alpha \) and \( (1 1 0)\beta \) planes would then decrease the critical nucleus size for \( \alpha \) precipitation and consequently reduce the activation energy for \( \alpha \) nucleation. The crystallographic orientation of early grown \( \alpha \) variants was investigated by EBSD. Figure 13c shows \( \alpha \) lamellae grown on both sides of a \( \beta \) grain boundary during the early stage of cooling (between the dotted lines). The ODF section of \( \phi^2=0^\circ \) and quarter pole figure calculated from this region are shown in Figure 13d and e. It can be seen that these early \( \alpha \) lamellae again correspond to the \{\( \phi_1, \Phi, \phi_2 \)\} \{90\( ^\circ \), 30\( ^\circ \), 0\( ^\circ \)\} texture component, which is the dominant texture component after \( \beta \rightarrow \alpha \) phase transformation shown in Figure 8 and 9. In contrast, Figure 13e shows the texture component of an early grown \( \alpha \) lamellae region only grown on one side of the prior \( \beta \) grain boundary (rare situation). This \( \alpha \) product provides a different texture component contributing to the weak \(<0 0 0 1>\) fibre texture component seen in Figures 8 and 9. The nucleation advantage shown here will lead to early growth of \( \alpha \) laths on both sides of the grain boundaries until the colony encounters other lamellae growing from different direction. Since at this stage, other \( \alpha \) variants are hardly present in the \( \beta \) grain, the growth of this particular \( \alpha \) variant is mainly limited by the \( \beta \) grain size. In other words, if the \( \beta \) grain is very large, the \( \alpha \) variant nucleating early can grow very far and become a far more
dominant texture component (in case this variant is indeed of the same texture component, see Figure 12a) compared to early $\alpha$ variants growing in a smaller $\beta$ grain (Figure 12b). It should be noted that this work did not find any indication that yttria particles themselves acted as early nucleation sites. However, they might have played a minor role and further investigations are continued in this field.

Conclusions:

Two variants of Ti-6Al-4V were studied $\beta$ heat treated at two different temperatures in order to shed light on the role of $\beta$ grain size on $\alpha$ variant selection and texture memory. Grain growth was controlled in one variant by having yttria pinning the $\beta$ grain boundaries during high temperature heat treatment. Care was taken to generate similar starting microstructures and textures before $\beta$ heat treatment to single out $\beta$ grain size as a variable and study its effect. The ability to reconstruct $\beta$ grains of EBSD maps enabled a detailed study, which led to the following conclusions:

- The grain boundary pinning effect due to precipitation of $\text{Y}_2\text{O}_3$ at prior $\beta$ grain boundaries was evident at 1050 °C and 1150 °C. This led to half the $\beta$ grain size observed in Ti-6Al-4V-0.4Y compared to conventional Ti-6Al-4V when $\beta$ heat-treated at the same temperature. A similar but less pronounced effect of grain growth was observed when comparing heat treatment temperature for the same alloy.

- The $\beta$ heat treatment did not change the main texture components, although the individual texture component intensities had changed dramatically after $\beta$ heat treatment. When comparing the two alloys, significant differences were observed in terms of texture intensities although the starting textures were almost identical.
Consequently, these texture variations are an indication of texture memory and variant selection.

- It was shown that $\beta$ heat treatment of both Ti-6Al-4V variants, with a recrystallisation texture dominated by Euler angles $\{\varphi_1, \Phi, \varphi_2\} \{0^\circ, 90^\circ, 0^\circ\}$, results in variant selection based on neighbouring $\beta$ grains with nearly parallel $<110>$, which leads to strengthening of the $\{90^\circ, 30^\circ, 0^\circ\}$ texture component.

- Variant selection during the $\beta$ to $\alpha$ phase transformation is significantly stronger in conventional Ti-6Al-4V, which develops a larger $\beta$ grain size, compared to Ti-6Al-4V-0.4Y. It was found that nucleation of the $\alpha$ lath on both sides of prior $\beta$ boundaries is preferred to nucleation on only one side and that such early nucleation is associated with $\beta$ grain pairs having a common (<10° deviation) $<110>$ pole. Such early $\alpha$ nucleation promotes large regions of $\alpha$ texture component with Euler angles $\{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\}$, which corresponds to the strong texture component that was observed after transformation.

- It is proposed that the growth of the $\{90^\circ, 30^\circ, 0^\circ\}$ texture component is mainly limited by the $\beta$ grain size and consequently, this texture component grows in dominance with increasing $\beta$ grain size.

Finally, it is important to note that the observed effect of $\beta$ grain size on variant selection and texture strengthening is highly relevant from an application point of view since the $\beta$ grain size does increase from the outer surface to the core of a billet. As demonstrated here, large regions with a particular texture component might therefore be expected deep inside a billet.
Such regions might be difficult to break up from a crystallographic point of view with the potential of resulting in macrozone formation during further thermomechanical processing.

Acknowledgements

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References


**Table captions:**

Table 1: Elemental compositions of the two variants of Ti-6Al-4V alloy.

Table 2: Mean grain size of Ti-6Al-4V and Ti-6Al-4V-0.4Y showing β grain size variation at 1050°C and 1150°C.

**Figure captions:**

Figure 1: Schematic representation of the orientation relationship between the α phase (hcp) and β phase (bcc) showing the Burger Relationship (BR).

Figure 2: The schematic representation of β quenching (H), uni-directional rolling (UD), recrystallisation (RX), solution heat treatment (β-HT) of Ti-6Al-4V and Ti-6Al-4V-0.4Y processes.

Figure 3: Optical micrographs of samples recrystallised (RX) at 950°C for 8hr (a) Ti-6Al-4V and (b) Ti-6Al-4V-0.4Y.

Figure 4: EBSD measured ODF (φ2=0) section and pole figure of α RX texture (a) Ti-6Al-4V and (b) Ti-6Al-4V-0.4Y (c) Ti-6Al-4V (d) Ti-6Al-4V-0.4Y.

Figure 5: Optical micrographs of β heat treated samples (a) Ti-6Al-4V at 1050°C, (b) Ti-6Al-4V at 1150°C, (c) Ti-6Al-4V-0.4Y at 1050°C and (d) Ti-6Al-4V-0.4Y at 1150°C.

Figure 6: The β Grain size distributions of samples after β heat treatment (a) Ti-6Al-4V and Ti-6Al-4V-0.4Y at 1050°C (b) Ti-6Al-4V and Ti-6Al-4V-0.4Y at 1150°C.
Figure 7: EBSD measured ODF (φ2=0) section of α transformation texture (a) Ti-6Al-4V β HT at 1050°C, (b) Ti-6Al-4V β HT at 1150°C, (c) Ti-6Al-4V-0.4Y β HT at 1050°C and (d) Ti-6Al-4V-0.4Y β HT at 1150°C.

Figure 8: Ti-6Al-4V EBSD reconstructed β texture heat treated at 1050°C (a) and at 1150°C (b). EBSD calculated α texture from β texture according to BR (1050°C) and (d) at 1150°C. EBSD measured α texture at 1050°C (e) and 1150°C (f).

Figure 9: Ti-6Al-4V-0.4Y EBSD reconstructed β texture heat treated at 1050°C (a) and at 1150°C (b). EBSD calculated α texture from β texture according to BR (1050°C) and (d) at 1150°C. EBSD measured α texture at 1050°C (e) and 1150°C (f).

Figure 10: (a) α IPF map superimposed on reconstructed β high angle grain boundary map showing Group 1 (close crystallographic relationship) and Group 2 (without close crystallographic relationship), (b) Pole figures of Group 2 without close alignment of (0002) pole with the (110) pole in adjacent β grains, (c) Pole figures of Group 1 showing close alignment of (0002) pole with the (110) pole of adjacent β grains, (d) The three-dimensional crystal orientation of group 2 GB α variants and adjacent β grains, (e) The three-dimensional grain orientation of Group 1 GB α variants and the adjacent β grains.

Figure 11: Regions in the IPF orientation Map corresponding to the texture component (90º, 30º, 0º) (a) Ti-6Al-4V and (b) Ti-6Al-4V-0.4Y.

Figure 12: Relative misorientation angle distribution of inherited α phase showing the misorientations of reference alloy without variant selection and Ti-6Al-4V and Ti-6Al-4V-0.4Y HT at 1050°C and 1150°C.
Figure 13: SEM micrographs of Ti-6Al-4V β heat treated at 1150°C for 30min and cooled at 1°C/min to 975°C followed by water quench showing (a) α lath growing on both sides of prior β grain boundary (b) α lath on one side of prior β grain boundary, (c) EBSD map of α lath on both sides of prior β grain boundary (d) EBSD measured ODF and Pole figure corresponding to α lath on both sides of prior β grain boundary (e) EBSD measured ODF and Pole figure corresponding to α lath on one side of prior β grain boundary.
Table 1

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<th>Elements</th>
<th>Fe</th>
<th>V</th>
<th>Al</th>
<th>C</th>
<th>O</th>
<th>N</th>
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Table 2

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Figure 1

Variant selection and its effect on texture in Ti-6Al-4V
Figure 2

Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V

Figure 3
Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V

Figure 5
Figure 6

(a) 1050 °C

Frequency (normalised)

Grain Size (µm)

(b) 1150 °C

Frequency (normalised)

Grain Size (µm)
Figure 7

Variant selection and its effect on texture in Ti-6Al-4V
Figure 8

Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V

Figure 9

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Variant selection and its effect on texture in Ti-6Al-4V
Figure 11

Variant selection and its effect on texture in Ti-6Al-4V

(a) Ti-6Al-4V
(b) Ti-6Al-4V-0.4Y

(90°, 30°, 0°) Texture component
Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V
Chapter 6

The influence of rolling temperature on texture evolution and variant selection during $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation in Ti-6Al-4V
The influence of rolling temperature on texture evolution and variant selection during $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation in Ti-6Al-4V

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Abstract

The role of starting texture on variant selection has been studied during $\alpha \rightarrow \beta \rightarrow \alpha$ transformation in Ti-6Al-4V. By rolling at different temperatures followed by recrystallization, material with either a strong basal texture or a strong transverse texture was generated. Subsequently, both conditions were heat treated above the $\beta$ transus followed by slow cooling. The degree of variant selection was assessed by comparing the strength of the measured and predicted $\alpha$ texture from high temperature $\beta$ texture assuming equal occurrence of all possible variants during $\beta \rightarrow \alpha$ transformation. It was found that even though the material rolled originally at 800 $^\circ$C displayed a stronger $\alpha$ texture after $\beta$ heat treatment it was the material rolled originally at 950 $^\circ$C that showed greater variant selection. Variant selection mechanism has been discussed in terms of the generated $\beta$ texture and common $<1 \ 1 \ 0>$ poles in neighbouring $\beta$ grains selecting similar $\alpha$ variant on both sides of the prior $\beta$ grain boundary. Predictions of possible $<1 \ 1 \ 0>$ pole misorientation distributions for the two investigated $\beta$ textures showed that the combination of texture components generated during rolling Ti-6Al-4V at 950 $^\circ$C increases the likelihood of having $\beta$ grain pairs with closely
aligned (1 1 0) planes compared to rolling at 800 °C. Therefore, it can be proposed that avoiding the generation of certain combinations of β texture components during thermomechanical processing has the potential of reducing variant selection during subsequent β heat treatment.

Keywords: Ti-6Al-4V, Rolling Temperature, Texture evolution and Variant selection

1. Introduction

The thermomechanical processing route of an engineering alloy determines the microstructure and crystallographic texture and must be controlled for optimum mechanical properties. Titanium alloys are no exception because of their hexagonal close packed (hcp) crystal structure at low temperature, they are particularly prone to develop strong crystallographic textures that can significantly affect the mechanical properties of the final product [1]. Therefore, it is important to understand the through process microstructure evolution and the effect of varying thermomechanical process parameters. Titanium alloys are usually subjected to a series of thermomechanical processing stages in the α + β phase and single β phase field [2]. Since the volume fraction of β (bcc crystal structure) increases with temperature, the behaviour of the alloy during deformation will partly depend on the amount of β phase at a given temperature. At 800 °C the volume fraction of β in Ti-6Al-4V is ~ 15% [3] and one might expect that α phase dominates the deformation behaviour. In contrast, at 950°C the volume fraction of β is about 70% [4], and hence the contribution of the β phase to the deformation behaviour is expected to be important [3]. The crystallographic texture that develops during unidirectional rolling at these two temperatures usually gives rise to two major types of textures: Firstly, rolling at 800 °C develops a basal/transverse (B/T) texture in
which the basal poles are located along the plate normal and along the transverse direction in the (0 0 0 2) pole figure of the $\alpha$ phase. Secondly, at 950 °C, a transverse (T) and in some cases axial (R) texture develops. In this case the basal poles are located along the transverse direction and rolling direction in the (0 0 0 2) pole figure [5,6].

Heat treating titanium alloys above the $\beta$-transus is commonly performed during the processing. In the case of Ti-6Al-4V with a typical oxygen content, this means that the material needs to be heat treated above 995 °C [7]. Such heat treatment is either carried out at an early stage of the process to homogenise the materials or at a late stage to generate a damage tolerant microstructure [8]. The phase transformation during heating ($\alpha \rightarrow \beta$) and cooling ($\beta \rightarrow \alpha$) is governed by the so called Burgers orientation relationship $\{0 0 0 2\}_{\alpha} \parallel \{1 1 0\}_{\beta}$ and $<1 1 2>_{\alpha} \parallel <1 1 1>_{\beta}$, with 12 possible $\alpha$ orientations that can transform from a single parent $\beta$ grain during $\beta \rightarrow \alpha$ and 6 possible $\beta$ orientation variants during the $\alpha \rightarrow \beta$ phase transformation [9]. The relationship between the two planes corresponds to the closest packed planes while the two directions correspond to the nearest neighbour direction [10]. If all the variants occur with equal volume fractions during an $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation, the final $\alpha$ texture should be significantly randomized regardless of the initial $\alpha$ texture. However, it has been demonstrated by a number of researchers [11-13] that the measured $\alpha$ texture does not coincide quantitatively with the one expected after both displacive and diffusional phase transformation in titanium alloys. This disagreement between the predicted and measured $\alpha$ texture has been ascribed to variant selection.

Variant selection mechanisms in materials that undergo an allotropic phase transformation have been linked to transformation strain and elastic anisotropy [13-17]. The elastic stress generated during unconstrained transformation of $\beta$ to $\alpha$ grains in titanium, can lead to 10% contraction along one of the $<1 1 0>$ $\beta$ direction and 2% contraction and 10% expansion along
the two perpendicular $<1 \ 1 \ 0>$ β direction [18]. While these strains are very significant, it needs to be kept in mind that Ti-6Al-4V is extremely soft close to the β transus making it practically impossible to load the material elastically without significant plastic deformation. Therefore, establishing the role of transformation strain on variant selection is difficult.

Bhattacharyya, et al., [19] reported that in the case of neighbouring β grains with a common $<1 \ 1 \ 0>$ β pole, α variants are selected on both sides of the prior β grain boundary, which have their $<0 \ 0 \ 0 \ 2>$ α pole nearly parallel to the common $<1 \ 1 \ 0>$ β pole. Hence, special pairs of β grains can promote variant selection when α nucleates first on β grain boundaries (most common case during normal cooling conditions of Ti-6Al-4V), i.e. variant selection. This observation is of fundamental importance from a practical point of view since these large regions of practically identical crystallographic orientation are suspected to be the root source for the formation of macrozones [20]. More recently, S. C. Vogel, et al., [21] reported that in two phase Ti-alloy, it is thermodynamically favourable for β phase to grow from pre-existing residual β during α → β phase transformation rather than to nucleate within the α grain and Cayron [22] suggested that the β grains with the common (1 1 0) β planes are β variants inherited from the α parents that transform to β during the α → β phase transformation. Obasi G. C., et al., [23] demonstrated that the degree of variant dominance during β → α phase transformation is related to β grain growth. They showed that nucleation of α first occurs at grain boundaries with β grain pairs having a common (1 1 0) normal. Consequently, these α variants become dominant. Since these α variants have a common orientation, they also strengthen the α texture of the material. It has also been reported that the final texture and preferential selection of variants is strongly dependent on the processing parameters such as deformation temperature and heat treatment [13, 24]. Inoue, et al., [13] suggested that the preferential formation of an α transformation texture can be explained by taking into account
the active slip system with respect to β orientation during deformation. Even though numerous studies have confirmed a number of different possible variant selection mechanisms in titanium alloys [14, 15, 23-26], their importance and dominance in respect to texture evolution has not been clarified. In the present work the aim has been to study the effect of deformation temperatures (800°C and 950°C) and their different resultant microstructure on α variant selection during subsequent β heat treatment.

In-house thermomechanically-processing was carried out to obtain two different starting textures but the same microstructural morphology before undertaking β heat treatments. Detailed texture and microstructure analysis allowed the quantification of β grain neighbours with common (1 1 0) poles and made it possible to relate the likelihood of observing such pairs to specific combinations of β texture components.

2. Experimental procedures

The material used in the present study was Ti-6Al-4V produced by conventional ingot metallurgy and provided by TIMET, UK. The nominal chemical composition of the alloy is shown in Table 1. Blocks of 40 × 40 × 20 mm were cut from the alloy, heat treated at 1050 °C and water quenched. The texture generated during this process can be seen in Figure 1(a). These blocks were subsequently uni-directionally hot rolled using the facilities at the University of Manchester. The specimens were placed in a standard calibrated furnace set to 800 °C and 950 °C, respectively. The soaking time was 1h for each specimen before starting the rolling process. A total sample thickness reduction of 75% was achieved through 5 passes at a reduction rate of 15% per pass and 2 min reheating in-between. The schematic representation of the rolling and heat treatment processes are shown in Figure 1(b).
Laboratory X-ray diffraction was used to determine the rolling texture of the processed material. The rolled material was subsequently heat treated for 8 hours at 950 °C in a preheated tube furnace under argon atmosphere and cooled to room temperature at a rate of 1 °C min⁻¹. Samples with dimensions of 10 × 10 × 5 mm³ were cut from the centre of the small slabs and subjected to β heat treatments at 1150 °C for 30 minutes and cooled at 1 °C min⁻¹ under an argon atmosphere. It is important to note that slow cooling rates are typical during industrial processing of this alloy. Mid sections of the β heat-treated samples were prepared following standard grinding/polishing procedures and etched in 2% HF, 8% HNO₃ and 90% H₂O (Kroll’s reagent) for 10 s prior to optical microscopy examination. In order to undertake detailed grain orientation and micro/macrotexure studies, Electron Backscatter Diffraction (EBSD) was carried out. For best Kikuchi pattern acquisition, the samples were polished with OPS for about 2 hours and etched repeatedly during polishing. A CamScan Field Emission Gun Scanning Electron Microscope (FEG-SEM), equipped with HKL Channel 5 software was used for EBSD data acquisition and analysis. Due to the coarse β grain size and large lamellar colonies after β heat treatment a step size of 10μm was chosen, which allowed covering sufficiently large areas (7 × 7 mm²) to measure a reasonable number of β grains for texture analysis while still capturing enough details of the columnar microstructure.

Determining the high temperature β texture at ambient temperature in Ti-6Al-4V is one of the experimental difficulties when studying variant selection. The most reliable in-situ techniques, which are not compromised by surface effects, are neutron and high-energy synchrotron X-ray diffraction. Although they can provide excellent information of the global texture, they do not provide information at the microstructural scale, i.e. information about neighbouring β grains. In order to determine the high temperature β texture, the α orientation maps recorded by EBSD were used to reconstruct the β grain structure based on the Burger’s orientation relation between the parent and daughter phases. The software used for the β
reconstruction has been developed at the University of Sheffield within IMMPETUS (Institute for Microstructural and Mechanical Process Engineering). The software compares the orientation of individual α lamellae and identifies the most likely parent β orientation between appropriate neighbours. In order to obtain a successful reconstruction, a maximum allowable misorientation angle between α variants of a common β grain is defined, which in the present case was a misorientation angle of 3°. Further details on the β orientation reconstruction methodology are described in [27-30]. Particular details with regards to the reconstruction software applications can be found in [27]. The information from the reconstruction was subsequently reloaded into Channel 5 to plot β orientation maps. TEXTAN III [31] was used to plot ODFs of the β texture from the reconstructed values and to predict the α texture using a transformation model assuming no preferred variant selection. In this way it was possible to describe the disagreement between the predicted and measured α texture and relate the level of variant selection to the initial β texture resulting from the deformation temperatures. The reference systems for the Euler angles, are such that the specimen coordinate systems; the rolling direction (RD), transverse direction (TD) and normal direction (ND) are Cartesian and are parallel to crystal coordinates [10̅10], [11̅20] and [0002] respectively. This convection was used in all the figures in this work.

3. Results

3.1 Microstructure and texture evolution

Optical micrographs of Ti-6Al-4V rolled at 800 °C and 950 °C followed by recrystallisation heat treatment (RX) for 8 hours at 950 °C (α + β phase region) and slow cooling (1°C min⁻¹) are shown in Figure 2(a) and (b). It can be seen that a fully equiaxed primary α microstructure
was obtained for both conditions with a mean grain size of 18\(\mu\)m (determined by linear intercept method) and retained \(\beta\) at the triple junctions of primary \(\alpha\). Figures 2(c) and (d) show the fully transformed microstructures obtained after \(\beta\) heat treatment at 1150 °C and cooled at a rate of 1°C min\(^{-1}\). The microstructures are populated by large \(\alpha\) lamellar colonies that nucleated from grain boundary \(\alpha\) (GB\(\alpha\) - formed on \(\beta\) grain boundaries). Figure 3 represented in form of a histogram with bin width of 100 \(\mu\)m compares the \(\beta\) grain size distribution after \(\beta\) heat treatment for both conditions displaying no significant difference. Here the \(\beta\) grain size was in fact determined from \(\beta\) orientation maps reconstructed from EBSD \(\alpha\) orientation maps (see Figure 8). Additional analysis of the \(\beta\) grain size from optical micrographs confirmed the \(\beta\) grain size distribution determined by EBSD. Since it was found in a previous study [23] that \(\beta\) grain growth has a significant influence on the \(\alpha\) transformation texture, achieving the same \(\beta\) grain size in the present study was critical in order to identify the effect of different starting textures on variant selection during subsequent \(\beta\) heat treatment. The mean \(\beta\) grain size of both materials was \(\sim\)500\(\mu\)m.

The textures of the materials rolled at 800 °C and 950 °C are presented in form of representative quarter pole figures (Figure 4) and ODF’s (Figure 5). In the case of the as-rolled condition, pole figures and ODFs were plotted using two different scales since the texture strength in this condition was significantly weaker than for the other two conditions. The ODF sections presented here are the \(\varphi_2 = 0°\) for \(\alpha\) phase texture and \(\varphi_2 = 45°\) for \(\beta\) phase texture. The \(\alpha\) texture components formed are represented with specific names U, V, W, X and Y as shown in Table 2. Figures 4(a) and 5(a) show the 800 °C rolling \(\alpha\) texture, the main texture components are U \(((\bar{1}2 \bar{1} 0) \{1 0 \bar{1} 0\})\) and V \(((\bar{1} \bar{2} \bar{1} 5)\{\bar{5} \bar{1} 0 \ 5 \bar{6}\})\) located at the Euler angles \(\{\varphi_1, \Phi, \varphi_2\} \{3.9°, 86°, 0°\}\) and \(\{\varphi_1, \Phi, \varphi_2\} \{88.4°, 43°, 0°\}\) of \(\varphi_2 = 0°\) sections of the ODF. The B/T texture observed in the present study is consistent with results previously reported in [5-6, 32] when Ti-6Al-4V was rolled at comparative temperature. The weak U
texture component corresponds to the basal pole aligned in the transverse direction (TD) while the V texture component corresponds to the basal poles aligned about 30° away from the normal direction (ND) towards the RD as shown in Figure 4a. In contrast, the material rolled at 950 °C displayed a relatively strong transverse (T) basal texture while the basal poles aligned about 30° off ND were weak, Figure 4(b) and 5(b) The observed textures are in good agreement with textures for Ti-6Al-4V rolled uniaxially at similar temperatures [5-6, 32]. As expected, comparing the as-rolled texture with the texture after the recrystallisation heat treatment revealed for both rolling conditions that no new texture components were generated during recrystallisation [5, 33] However, recrystallisation strengthened the texture components significantly without changing the trends between them, Figure 4(c),(d) and 5(c),(d).

The corresponding \( \alpha \) transformation textures are presented in Figures 4(e), (f) and 5(e), (f). Both specimens show similar texture components with different texture intensities. This is evident both in the pole figures and ODFs. The transformed \( \alpha \) textures of Ti-6Al-4V, originally rolled at 800 ºC, Figure 5(e), is composed of weak U, X ((\( \overline{1} \ 2 \ \overline{1} \ 0 \))\( \{1 \ 0 \ \overline{1} \ 2 \}) \) and W ((\( \overline{1} \ 2 \ \overline{1} \ 5 \))\( \{9 \ 5 \ 4 \ 3 \}) \) texture components with maximum intensity of 6 times above random texture distribution while the texture component V at the Euler angles \{\( \varphi_1, \Phi, \varphi_2 \} \{88.4^\circ, 43^\circ, 0^\circ \}\) is very strong with maximum intensities of 18 times above random. In the case of the specimen rolled at 950°C it is obvious from Figure 5f that the same texture components are found but the intensity of the texture component U at the Euler angles \{\( \varphi_1, \Phi, \varphi_2 \} \{88.4^\circ, 43^\circ, 0^\circ \}\) is 16 times random which is slightly lower than that of the specimen rolled at 800°C. Comparing Figure 5e and f it becomes apparent that the \( \beta \) heat treatment of the material rolled originally at 800 °C has resulted in a stronger texture compared to the \( \beta \) heat treatment of the material rolled originally at 950 °C.
3.2 Variant selection

The effect of the initial rolling texture on variant selection during $\beta \rightarrow \alpha$ phase transformation was investigated by comparing texture intensities of the measured and predicted $\alpha$ texture after both materials had been heat treated at 1150 °C. In order to predict the $\alpha$ texture in the absence of any variant selection, it was first necessary to determine the high temperature $\beta$ texture. This was achieved, as described in the experimental procedures, by reconstructing the $\beta$ microstructure from large $\alpha$ maps recorded by EBSD. The information from the $\beta$ texture determined from the reconstructed microstructure was subsequently used to predict the $\alpha$ texture based on the Burgers orientation relationship. During the calculation no preferential selection of any of the 12 possible variants was assumed during the $\beta \rightarrow \alpha$ transformation, taking into account the crystal symmetries of the parent and the daughter crystals during the rotation. The transformation was applied to the reconstructed $\beta$ phase data using the spherical harmonic method developed by Bunge [34] as implemented in TEXTAN-III [31]. The measured $\alpha$ textures as well as the $\beta$ textures and predicted $\alpha$ textures are presented in Figure 6. The $\beta$ textures display both $<1\ 1\ 0>$/RD $\alpha$ fibre and $<1\ 1\ 1>$/ND $\gamma$ fibres texture components rotated 10 ° away from ND, which is very similar to the texture found in rolled and recrystallised bcc material. It was found that the $(113)[5\ 81]$ component of the $<1\ 1\ 0>$/RD $\alpha$ fibre (marked as A) and $<1\ 1\ 1>$/ND $\gamma$ fibre in $\beta$ phase (marked as B, C, D, E) transformed to V, U, W, and X texture components in the $\alpha$ phase with significant difference in texture intensities. Figure 6 shows that the measured $\alpha$ transformation texture of the material originally rolled at 800 °C and 950 °C do not agree quantitatively with the predicted $\alpha$ transformation textures. This is evident when comparing Figures 6(c) and (d) with Figures 6(e) and (f). The intensity of the main texture component, V in the predicted $\alpha$ textures of the material originally rolled at 800 °C is 13 while the measurements show a value of 18 times
random texture distribution. In the case of the material originally rolled at 950°C, the predicted main texture component is 6 times random distribution while the measurements displays a value of 16. Hence, even though the material rolled at 800 °C results in a stronger α transformation texture, variant selection is more prominent in the material that was rolled at 950 °C.

4. Discussion

The observed B/T texture of specimen hot rolled at 800 °C in Figure 4 (a) and the T texture in Figure 4(b) of the specimen hot rolled at 950 °C are in agreement with previous studies [5,6] and the root cause for these rolling textures as well as the texture strengthening during recrystallisation shall not be further discussed here as this aspect is not central to the present work. Instead, the discussion will focus on the effect of different starting textures on the α transformation texture and on the main observation that the material rolled originally at 950 °C displayed significantly more variant selection than the material rolled originally at 800 °C.

In materials with a bcc crystal structure the α fibre texture is associated with a rolling/recovery texture while the γ fibre texture indicates recrystallisation. The material originally rolled at 800 °C displays a strong <110>//RD α texture component while the <111>//ND γ fibres texture components rotated 10 ° about ND, clearly observable in the material originally rolled at 950 °C, are hardly visible. Therefore, the results in Figure 6(a) and (b) suggest that rolling at 800 °C followed by a heat treatment in the two-phase region resulted in significantly less β recrystallisation in the β phase compared to rolling and heat treating the material at 950 °C. Work by [21] has shown that the original β texture obtained from rolling and recrystallization does not change during β heat treatment, since it is thermodynamically favourable for β to grow from pre-existing β phase rather than nucleating
from $\alpha$ grains. It has also been demonstrated [22] that the $\beta$ texture components developed during the first thermomechanical route, remain in place during subsequent $\beta$ heat treatment.

Previous studies on variant selection mechanisms during allotropic phase transformation in materials with a bcc and hcp crystal structure have been linked to transformation strain and elastic anisotropy. At high temperature, the $\beta$ titanium is extremely soft with yield strength of about 4.3 MPa [35], the material cannot withstand significant loading without plastic deformation. Therefore, establishing the role of transformation strain on variant selection is difficult. On the other hand, the role of neighbouring $\beta$ grains with a common $<1 1 0>$ $\beta$ pole promoting variant selection through nucleation of grain boundary $\alpha$ that fulfils the Burgers relationship for both $\beta$ grains [19] is well established. In the present case, the extensive orientation maps recorded for the texture analysis were used to determine the number of $\beta$ grain pairs that fulfil such criterion for variant selection. Figure 7a demonstrates that the material rolled originally at 950$^\circ$C shows a higher number of $\beta$ grain pairs with a common $<110>$ pole that can promote variant selection when $\alpha$ nucleates first on $\beta$ grain boundaries compared to the material rolled originally at 800$^\circ$C. These $\beta$ grain pairs with a common $<110>$ pole (special $\beta$ grain pairs) displayed $\alpha$ variants with closed orientation on either side of the prior $\beta/\beta$. Figure 7b and c gives examples of “special” and “non-special” $\beta$ grain pairs observed by EBSD mapping. A quantitative analysis of these special $\beta$ grain pairs revealed that the length scale of the $\alpha$ variant with close orientation in the material rolled originally at 800$^\circ$C was about 6% while the material rolled originally at 950$^\circ$C had 10%, as demonstrated in Figure 7d. It was found that the orientation of the common $<110>$ pole was almost exclusively in the direction that provides the required (0 0 0 2) normal to explain the over-proportional representation of the V component. The angular tolerance for this behaviour
was ~10 between the <110> poles in the adjacent β grains, whereas there was no variant selection when this angle is exceeded [25].

The reconstructed β crystal orientation map superimposed on the α crystal orientation map in Figure 8(a) and (b) highlights the V component (grey) in both transformed microstructure together with the β grain boundaries (white). It should be pointed out that white lines do not draw the entire contour of the β grains. Similar observation has been made by N. Gey [36], according to Gey, this occurs when two neighbouring β display specific orientation such that α colonies inherited from two β grains during β → α phase transformation present specific misorientation corresponding to Burgers orientation relationship.

Further evidence of high incidence of variant selection in the material originally rolled at 950°C is demonstrated in Figure 9. The high level of similar orientated α colonies in the case of material originally rolled at 950°C increased the deviation of the misorientation distribution of the inherited α within the β grains when compared to that of the theoretical distribution corresponding to the Burgers orientation. Figure (9) shows plots of the α misorientation distribution for both materials together with the theoretical distribution that corresponding to the Burgers orientation relationship [37]. Note that the three near 60° misorientation angles have been plotted as one. Increased levels of 10° misorientation indicate more than expected α neighbours with a low misorientation, which is most clearly the case for the material rolled originally at 950°C.

In order to understand the difference in β grain pairs with a common <110> pole between the two conditions, the individual β texture components and fibres, together with the resulting α texture components from the transformation, were calculated and are plotted as ODFs in
Figure 10. The most dominant $\beta$ texture component, \{6.7°, 21.4°, 45°\} is plotted in Figure 10(a). It can be seen that the $\beta$ transformation results in a number of $\alpha$ texture components including the experimentally dominating V component, Figure 10(b). Figure 10(c) plots texture components of the \langle111\rangle||\ ND $\gamma$ fibre rotated 10° away from ND in $\beta$ and Figure 10(d) plots the resulting $\alpha$ texture components. It can again be seen that among the different $\alpha$ texture components the V is again observed while the other $\alpha$ texture components appear at different positions compared to Figure 10b. In other words, when transforming the $\alpha$ fibre and $\gamma$ fibre components of the $\beta$ phase there is one common $\alpha$ phase texture component, which happens to be exactly the texture component that was strengthened by variant selection.

In order to provide further evidence that the $\beta$ texture generated by rolling the material at 950°C is more likely to have $\beta$ grain pairs with closely aligned \langle110\rangle poles, a statistical analysis was carried out. First, both $\beta$ textures were recreated by using TEXTAN-III to output randomly 1000 Euler angles from the ODFs of the $\beta$ phase shown in Figure 6(a) and (b). Figure 11 compares the measured with the recreated ODFs confirming that 1000 Euler angles were sufficient to accurately represent the two different $\beta$ textures.

Figure 12 plots the relative frequency of misorientation between \{110\} in the range of 0 to 20° using a bin size of 1°. Details of this calculation are found in the appendix. Previous work has shown that a misalignment between \langle110\rangle poles of less than 10° can lead to $\alpha$ variant selection [16]. Figure 12 shows that rolling and heat treating the material at 950°C has generated a $\beta$ texture with a greater likelihood of having neighbouring $\beta$ grains with closely aligned \langle110\rangle $\beta$ planes than rolling the material at 800°C. Particularly in the critical range below 10° the difference in frequency is very noticeable. Hence, the increased degree of variant selection of the material rolled originally at 950°C compared to the material originally rolled at 800°C seems to be clearly related to the combination of specific $\beta$ texture
components making it more likely to obtain pairs of β grains with closely aligned (110) planes. These aligned planes subsequently result in early grain boundary α formation due to the reduced activation energy for α nucleation from which α colonies with a common (0002) normal grow on both sides of the boundary [19 and 23]. This work demonstrates that, in order to reduce the tendency of large α colonies growing with a very similar crystallographic orientation on both sides of a former β grain boundary, the choice of prior rolling temperature can be critical. This might be of particular importance if one considers that these regions of similar orientation might be the route source for macrozone formation during subsequent thermomechanical processing as described in [20].

Finally, both rolling conditions produced the combination of <110>//RD α and <111>//ND γ fibre texture components rotated 10° away from ND although the material rolled at 800°C showed very little of the γ fibre component and therefore less variant selection (but not no variant selection). Still, the material rolled originally at 800°C displays a stronger texture after β heat treatment than the material rolled at the higher temperature. This is related to the very strong β texture that was generated during α + β heat treatment carried out prior to β heat treatment.

5 Conclusions

A detailed study of the effect of starting microstructure on variant section during β heat treatment has been carried out in an effort to enhance our understanding of variant selection in Ti alloys. Two different starting textures were generated by rolling material at 800°C and 950°C, both conditions were subsequently heat treated at 950°C followed by slow cooling in order to obtain the same equiaxed grain morphology. Both conditions were β heat treated
followed by slow cooling to promote nucleation of $\alpha$ from grain boundaries. The main findings of the work can be summarised as follows:

1. Detailed texture analysis of the $\alpha$ phase and reconstructed $\beta$ phase after $\beta$ heat treatment revealed that even though the overall $\alpha$ texture is stronger in the material rolled originally at 800°C, variant selection is more dominant in the material first rolled at 950°C.

2. Variant selection has been discussed in terms of $\beta$ grain pairs with common $<110>$ poles. In the present case it was found that the number of grain boundaries with these special $\beta$ grain pairs was larger in the material rolled originally at 950°C than 800°C. Consequently, more aligned $\alpha$ colonies on both sides of a former $\beta$ grain boundary were found in the material rolled at 950°C. The $\alpha$ variants chosen by this mechanism were responsible for $\alpha$ textures after $\beta$ heat treatment that was stronger than one would expect.

3. Studying the $\beta$ texture components enabled to identify the root course for the increased number of special $\beta$ grain pairs in the material rolled at 950°C. It was found that the combination of a $<110>$/ND $\alpha$ fibre component with $<111>$/ND $\gamma$ fibre components rotated 10° away from ND increases the likelihood of having $\beta$ grain pairs with closely aligned (110) planes. Therefore, it can be proposed that avoiding such combinations of $\beta$ texture components by controlling the $\alpha + \beta$ rolling temperature has the potential of reducing variant selection.

4. Even though variant selection was most dominant in the material rolled at 950°C, the texture strength after $\beta$ heat treatment was still stronger in the material rolled at 800°C. This was explained by a difference in prior $\beta$ texture. Therefore, variant
selection does not only affect macrotextures but is also likely to play an important role in the development of microtexture and macrozone.

Acknowledgments

The authors are grateful for financial support from the EPSRC under EP/E048455/1, Rolls-Royce plc. and material provision from TIMET UK. The authors are also particularly indebted to Brad Wynne and his research group at the University in Sheffield for developing the $\beta$ reconstruction software, which was key for undertaking the research work presented here.
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Appendix

The present approach to calculate the likelihood of close <110> pole alignment consists of 3 steps: (1) determination of the components of the basis vector, (2) calculation of the translation vector of the reciprocal lattice and hence the reciprocal lattice vector of the 6 \( \{110\} \) (normals) and finally (3) determination of angles between their normal when pairing all combinations of 1000 Euler angles. A crystal of any orientation can be described using the three sets of Euler angles \( \{\phi_1, \Phi, \phi_2\} \) with reference to the specimen and crystal coordinate system as shown in equation (1).

\[
C_c = g^\beta C_s
\]  

(1)

Where \( C_c \) and \( C_s \) are the crystal and sample coordinate systems. The basis vector \((a, b, c)\) is calculated by first determining the orientation matrix \( g^\beta \), for each of the 1000 Euler angles using equation (2) both columns and rows of the matrix are unit vectors.

\[
g^\beta = \begin{bmatrix}
\cos \phi_1 \cos \phi_2 \cos \Phi - \sin \phi_1 \sin \phi_2 \cos \Phi & \sin \phi_1 \cos \phi_2 + \cos \phi_1 \sin \phi_2 \cos \phi_3 & \sin \phi_1 \sin \phi_2 \\
-\cos \phi_1 \sin \phi_2 \cos \Phi - \sin \phi_1 \cos \phi_2 \sin \phi_2 \cos \phi_3 & -\sin \phi_1 \cos \phi_2 - \cos \phi_1 \sin \phi_2 \cos \phi_3 \cos \phi_2 & \cos \phi_1 \sin \phi_2 \\
\sin \phi_1 \sin \phi_2 \sin \phi_3 & -\cos \phi_1 \sin \phi_2 \sin \phi_3 & \cos \phi_2
\end{bmatrix}
\]  

(2)

The three cell unit vectors \((a, b, c)\) are converted into Cartesian co-ordinates by multiplying the transpose of this \( g^\beta \) matrix with the three specific crystal directions \([100], [010] \) and \([001]\), using the following equation (3)

\[
a = g^\beta * \begin{bmatrix}
1 \\
0 \\
0
\end{bmatrix}, \quad b = g^\beta * \begin{bmatrix}
0 \\
1 \\
0
\end{bmatrix}, \quad c = g^\beta * \begin{bmatrix}
0 \\
0 \\
1
\end{bmatrix}
\]  

(3)

We can obtain the reciprocal lattice base vectors \((a_1, a_2, a_3)\) and hence the reciprocal lattice vector in Cartesian space \((u_{(hkl)})\) of any of the 6 \( \{110\} \) in crystal space using equation (4) and (5)
\[ a_1 = \frac{b + c}{a(b + c)}, \quad a_2 = \frac{c + a}{a(b + c)}, \quad a_3 = \frac{a + b}{a(b + c)} \]  
\[ u_{(hkl)} = h a_1 + k a_2 + l a_3 \]  
(4)  
(5)

Where \( (hkl) \) are the Miller indices of the \( \{110\} \) plane. Finally, the angles, \( \theta \) between the various normals \([hkl]\) were calculated using equation (6) generating thirty six million combinations.

\[
\theta = \cos^{-1} \left( \frac{u^* (h_1 k_1 l_1) u^* (h_2 k_2 l_2) \langle h_1 k_1 l_1 \rangle |u^* (h_2 k_2 l_2)\rangle}{|u^* (h_1 k_1 l_1)\rangle |u^* (h_2 k_2 l_2)\rangle} \right) 
\]  
(6)
Table captions

Table 1: Elemental compositions of Ti-6Al-4V

Table 2 Alpha texture components

Figure captions

Figure 1: Texture product and schematic representation of thermomechanical processes (a) Randomised (0002) and (10\,0) pole figures prior to hot rolling process (b) β quenching (H), uni-directional rolling (UD) at two temperature regimes, recrystallization at 950°C for 8h and cooled at 1°C/min (RX), solution heat treatment at 1150°C for 30min and cooled at 1°C/min (β-HT)

Figure 2: Optical micrographs of samples at different thermomechanical processing routes (a) rolled at 800°C and RX at 950°C, (b) rolled at 950°C and RX at 950°C, (c) rolled at 800°C, RX and β HT at 1150°C (d) rolled at 950°C, RX and β HT at 1150°C

Figure 3: The β Grain size distributions of materials rolled at 800 and 950°C and β heat-treated at 1150°C.

Figure 4: Figure 4: (0002) pole figures for (a) as rolled at 800°C condition, (b) as rolled at 950°C condition, (c) rolled at 800°C and RX condition (d) rolled at 950°C and RX condition (e) rolled at 800°C, RX and β HT condition and (f) rolled at 950°C, RX and β HT condition. Note that a) and b) were obtained by x-ray diffraction while the other pole figures were obtained by EBSD.
Figure 5: ODF ($\varphi_2=0^\circ$) section of $\alpha$ textures (a) as rolled at 800°C condition, (b) as rolled at 950°C condition, (c) rolled at 800°C and RX condition, (d) rolled at 950°C and RX condition, (e) rolled at 800°C, RX and $\beta$ HT condition and (f) rolled at 950°C, RX and $\beta$ HT condition. Note that a) and b) were obtained by x-ray diffraction while the other ODF’s were obtained by EBSD.

Figure 6: ODF ($\varphi_2=45^\circ$) section of $\beta$ textures (a) rolled at 800°C, RX and $\beta$ HT condition, (b) rolled at 950°C, RX and $\beta$ HT condition, calculated $\alpha$ transformation texture from $\beta$ texture according to BR (c) material originally rolled at 800°C (d) material originally rolled at 950°C and measured $\alpha$ transformation texture (e) material originally rolled at 800°C and (f) material originally rolled at 950°C. The $\alpha$ texture component is indicated as A and the $\gamma$ fibre texture components are indicated as B, C, D, E.

Figure 7: Quantitative representation of (a) number of $\beta$ grains that have a neighbouring $\beta$ grain with a closely aligned (110) plane, (b) example of such special $\beta$ grain pair resulting in aligned $\alpha$ colony growth on both sides of the former $\beta$ grain boundary and (c) example of non-special $\beta$ grain pair resulting in no variant selection, (d) $\alpha$ colonies length scale for $\alpha$ variants with similar crystallographic orientation on both sides of the $\beta$ Grain boundaries and $\alpha$ variants with dissimilar crystallographic orientation on both sides of the $\beta$ Grain boundaries.
Figure 8: Regions with similar α colonies (\overline{1} \ 2 \ \overline{1} \ 5)\{5 \ \overline{1}0 \ 5 \ 6\} texture component) on both sides of the β Grain boundaries are shown in (grey) and β Grain boundaries (white) for (a) material rolled originally at 800°C and (b) material rolled originally at 950°C.
Figure 9: Quantitative representation relative misorientation angle distribution of inherited $\alpha$ of materials rolled at 800 and 950 ºC and theoretical misorientation according Burgers orientation relationship during $\beta \rightarrow \alpha$ phase transformation.

Figure 10: ODFs of individual $\beta$ texture components with Gaussian distribution ((a) and (c)) and their calculated $\alpha$ transformation texture components based on $\beta \rightarrow \alpha$ Burgers orientation relationship ((b) and (d)).

Figure 11: ODFs of reconstructed $\beta$ texture and representative $\beta$ texture of 1000 Euler angles from the ODFs of the $\beta$ phase of Ti-6Al-4V rolled at 800 and 950 ºC (a) reconstructed $\beta$ texture of Ti-6Al-4V rolled at 800 ºC (b) reconstructed $\beta$ texture of Ti-6Al-4V rolled at 950 ºC (c) representative $\beta$ texture of Ti-6Al-4V rolled at 800 ºC and (d) representative $\beta$ texture of Ti-6Al-4V rolled at 950 ºC

Figure 12: Plots of relative frequency of misorientation between $<110>$ in the range of 0 to 20 º using bin size of 1 º for the two materials.
Table 1

<table>
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<tr>
<th>Elements</th>
<th>Fe</th>
<th>V</th>
<th>Al</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>B</th>
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Table 2

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<tr>
<td>V</td>
<td>(1215)[51056]</td>
<td>{88.4, 43, 0}</td>
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<tr>
<td>W</td>
<td>(\overline{1}2\overline{1}5)[5\overline{9}4\overline{3}]</td>
<td>{74.3, 88.4, 0}</td>
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<tr>
<td>X</td>
<td>(12\overline{1}0)[101\overline{2}]</td>
<td>{52, 35, 0}</td>
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<tr>
<td>Y</td>
<td>(12\overline{1}0)[0001]</td>
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Figure 2

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<td>After β HT</td>
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Figure 3

HT at 1150 °C

Frequency (normalised)

Grain Size (µm)

- Red: Ti-64 Rolled at 800 °C
- Blue: Ti-64 Rolled at 950 °C
Figure 4

<table>
<thead>
<tr>
<th>Ti-64 Rolled at 800°C pole figures</th>
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<tr>
<td>(a)</td>
<td>(b)</td>
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<td>(c)</td>
<td>(d)</td>
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<td>(e)</td>
<td>(f)</td>
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Figure 5

<table>
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<td>(b)</td>
<td>$\Phi = 90^\circ$</td>
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<td>(c)</td>
<td>(d)</td>
<td>Section $\varphi_2 = 0^\circ$</td>
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<td>(e)</td>
<td>(f)</td>
<td></td>
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</tbody>
</table>

- $\{\varphi_1, \Phi, \varphi_2\}$
  - {3.9°, 86°, 0°}
  - {52°, 35°, 0°}
  - {88.4°, 43°, 0°}
  - {88.4°, 88.4°, 0°}
  - {74.3°, 88.4°, 0°}
Figure 6

Rolled at 800°C HT 1150°C

(a) $\varphi_2 = 45^\circ$  
(b) $\varphi_2 = 45^\circ$

Rolled at 950°C HT 1150°C

(c) $\varphi_2 = 0^\circ$  
(d) $\varphi_2 = 0^\circ$

Measured $\beta$ Texture  
Calculated $\alpha$ Texture  
Measured $\alpha$ Texture
Figure 7

(a) Graph showing the number of β grains in special and non-special grains.

(b) Image showing a 200 µm scale.

(c) Image showing a map of the material.

(d) Graph showing the percentage of α colonies length scale in similar and non-similar α orientations for Ti-64 rolled at 800°C HT 1150°C and Ti-64 rolled at 950°C HT 1150°C.
Figure 8

(a) 2000 μm

(b) 2000 μm

“Butterfly α colonies”
Figure 9
Figure 10

- $\phi = 90^\circ$
- $\phi_2 = 45^\circ$
- $\phi_2 = 0^\circ$

$\alpha$ Texture components:
- $\{\phi_1, \phi, \phi_2\}$
- $\{3.9^\circ, 86^\circ, 0^\circ\}$
- $\{88.4^\circ, 43^\circ, 0^\circ\}$
- $\{74.3^\circ, 88.4^\circ, 0^\circ\}$
- $\{52^\circ, 35^\circ, 0^\circ\}$

$\beta$ Texture components:
- $\{6.7^\circ, 21.4^\circ, 45^\circ\}$
- $\langle 111 \rangle // ND$ rotated $10^\circ$

\[\begin{array}{c}
\text{Phase} \\
\phi = 90^\circ \\
\phi_2 = 45^\circ \\
\phi_2 = 0^\circ
\end{array}\]
Figure 12

![Graph showing the frequency of occurrence of angles for Ti-64 rolled at different temperatures. Legend: Red line represents Ti-64 rolled at 800°C HT at 1150°C, blue dotted line represents Ti-64 rolled at 950°C HT at 1150°C. The x-axis represents angles in degrees, and the y-axis represents frequency of occurrence in percent.]
Chapter 7

In situ studies on the influence of $\beta$ Grain growth on Texture Evolution and Variant Selection during Phase Transformation in Ti-6Al-4V
In situ studies on the influence of β Grain growth on Texture evolution and Variant Selection during Phase Transformation in Ti-6Al-4V

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Abstract

In the present study, in situ phase transformation experiments have been carried out using neutron diffraction to monitor the texture evolution during the α→β→α phase transformation in Ti-6Al-4V with and without 0.4% yttrium additions. The aim of adding yttrium was to control β grain growth above the β transus by pinning grain boundaries with yttria. First, both alloys were thermomechanically processed to generate a similar starting α texture and grain morphology. Subsequently, both materials were heat treated above the β-transus up to 1250°C followed by furnace cooling to 210°C to promote diffusional phase transformation starting from β grain boundaries. In-situ texture measurements were taken during α → β → α phase transformation starting at room temperature, 800°C, 950°C, above β transus (1050°C
and 1250°C), and back to near room temperature. The degree of variant selection was determined by comparing the predicted transformation texture during heating and cooling based on the Burgers relationship and the assumption of no variant selection with the measured textures. It was found that during heating \( \beta \) grows from pre-existing \( \beta \) and that the \( \beta \) texture evolved even before the \( \beta \) transus was exceeded. The \( \beta \) texture strengthened noticeably above the \( \beta \) transus, forming a \(<1 1 1> / \text{ND} \) \( \gamma \) fibre texture rotated 10° away from ND, in the case of conventional Ti-6Al-4V but not Ti-6Al-4V-0.4Y, which was related to \( \beta \) grain coarsening. Significant variant selection was found during \( \beta \rightarrow \alpha \) transformation in the case of the former that could be related to the formation of certain \( \beta \) texture components that contribute to the \(<1 1 1> / \text{ND} \) \( \gamma \) fibre texture rotated 10° away from ND.

Keywords: Titanium alloys, Texture evolution, Neutron diffraction, Variant selection

1. Introduction

Understanding the microstructural evolution during processing engineering alloys is critical in order to optimise processing parameters. In materials such as titanium alloys, an important microstructural feature is the crystallographic texture, which is affected greatly by heat treatment and thermomechanical processing. Titanium, as well as zirconium, exhibits an hexagonal close packed (hcp) crystal structure (\( \alpha \) phase) at low temperature, which transforms to body centre cubic (bcc) \( \beta \) phase at high temperature. A crystallographic orientation relationship exists between the daughter phase and
mother phase during such transformation, which is the so-called Burgers orientation relationship. The Burgers orientation relationship is described by \{0 0 0 2\}_a \parallel \{1 1 0\}_\beta and \<1 1 2 0\>_a \parallel \<1 1 1\>_\beta. The relationship between the two planes corresponds to the closest packed planes while the two directions correspond to the nearest neighbour direction [1]. As a consequence of this relationship, a single \(\alpha\) grain has 6 possible \(\beta\) orientation variants during the \(\alpha \rightarrow \beta\) phase transformation and 12 possible \(\alpha\) orientations from a single parent \(\beta\) grain during cooling, i.e. reverse phase transformation \(\beta \rightarrow \alpha\) [2]. If all 6 variants during \(\alpha \rightarrow \beta\) and all 12 variants during \(\beta \rightarrow \alpha\) form with equal probability the final \(\alpha\) texture should be nearly randomized regardless of the initial \(\alpha\) texture. However, a number of researchers [3-5] have demonstrated that such \(\beta\) heat treatment does not produce the expected randomized \(\alpha\) phase texture. Instead, the texture components found in the material before \(\beta\) heat treatment are still observed in the transformed \(\alpha\) texture although the texture strength has usually decreased. This disagreement between the predicted and measured \(\alpha\) texture has been ascribed to variant selection. In literature, variant selection mechanisms during phase transformation have often been linked to transformation strain and elastic anisotropy [3, 4]. In the case of unconstrained transformation condition, the \(\beta \rightarrow \alpha\) transformation is accompanied by contraction in the range of 10% along a \(\langle 110\rangle\)_\(\beta\) direction and contraction of 2% and expansion of 10% along the two perpendicular \(\langle 110\rangle\)_\(\beta\) direction [5]. However, it needs to be kept in mind that Ti-6Al-4V is extremely soft close to the \(\beta\) transus and it is practically impossible to load the material elastically without significant plastic deformation. The effect of neighbouring \(\beta\) grain orientations has been reported by [6,7]. In the case of neighbouring \(\beta\) grains with a common \(\langle 1 1 0\rangle\) \(\beta\)
pole, α variants are selected on both sides of the prior β grain boundary, which have their \( <0 0 0 2 > \) α pole nearly parallel to the common \( <1 1 0 > \) β pole. Hence, special pairs of β grains can promote variant selection when α nucleates first on β grain boundaries (most common case during normal cooling conditions of Ti-6Al-4V). More recently, G. Obasi, et al., [8] has demonstrated that variant selection is related to the nucleation of α variants and here the orientation of neighbouring β grains plays a role. It seems that as the β grains grow a β texture evolves that increases the likelihood of nucleating particular variants and then they can also grow substantially into the open grain. This observation was explained by studying early α nucleation during cooling and identifying β grain boundaries of “special β grain pairs” as preferred sites for α formation. Consequently, these α variants can grow freely into unoccupied β grains becoming the dominant variant. The β texture in this work was determined by reconstructing the β microstructure from EBSD orientation maps recorded at room temperature, which limits the number of β grains that can be studied. Since β reconstruction of EBSD requires a fulfilled Burgers relationship, i.e. lamellar microstructures, this methodology also excludes any variant selection studies during \( \alpha \rightarrow \beta \) phase transformation.

To date, most variant selection studies in titanium alloys have been carried out using EBSD and x-ray diffraction [6, 7, 8, 9, and 10]. One of the major difficulties when analysing textures of β heat treated \( \alpha + \beta \) titanium alloys alloys is the large β grain size that is generated during heat treating the material above the β transus. In contrast to steel, conventional titanium alloys do not contain any carbides that restrict grain coarsening when holding the material in the high temperature single phase region. While EBSD provides the ability to combine macroscopic texture information with
information on the microstructural scale, the requirement of very large EBSD maps to capture a sufficient number of \( \beta \) grains in combination with a small step size in order to capture microstructure information, makes this methodology very time consuming. Furthermore, EBSD is mainly an ex-situ characterisation technique. While one might consider using EBSD at high temperatures, the high affinity of titanium to oxygen makes such studies rather unreliable.

In contrast, neutron time-of-flight diffraction (TOF) and high energy synchrotron x-ray diffraction [10-13] can overcome some of these issues as they are not compromised by surface effects. Particularly, neutron diffraction enables one to probe large volumes of material (many hundreds of \( \text{mm}^3 \)) and therefore can provide excellent information of the global texture [14]. The capability to acquire quantitative bulk crystallographic texture from a single measurement in matter of minutes without sample reorientation has been demonstrated recently using the General Materials Diffractometer (GEM) at ISIS pulse neutron source at UK Rutherford Appleton Laboratory [13, 15]. In addition, the high temperature furnace facility on GEM enables to carry out in situ texture measurements as a function of temperature to monitor and quantify texture variation during phase transformation. Previous in-situ texture measurements during thermal cycling using neutron diffraction have documented evidence of texture memory and variant selection during the \( \alpha \rightarrow \beta \rightarrow \alpha \) phase transformation in Ti-6Al-4V alloy [11]. However, the data presented in [11] suggests strongly that the \( \beta \) transus was not completely exceeded during the thermal cycle.

In the present study, GEM was used to measure in-situ the texture evolution during phase transformation. The aim was to study the evolution of the \( \beta \) texture during
heating in the two phase region and above the β transus and its affect on variant selection. Considering the difficulty of constraining β grain growth in Ti-6Al-4V, yttrium was added to a second variant in order to pin β grain boundaries in the high temperature single-phase region [16]. Thermomechanically-processing was carried out to achieve the best comparable microstructures and textures between the two alloys before undertaking variant selection studies.

2. Experimental Methods

2.1 Material

Ti-6Al-4V produced by conventional ingot metallurgy and uni-axially rolling was provided by TIMET, UK. Some of this material was remelted at the University of Birmingham to add 0.4 wt.% of yttrium. The nominal chemical compositions of both alloys are shown in Table 1. Both alloys were first thermomechanically processed in order to create a recrystallised, equiaxed microstructure of the same grain size (about 18 µm) and with a very similar α texture. Details about the process and microstructural images can be found in [8].

2.2 Texture Measurements by Neutron Diffraction

Cuboids of 20 x 15 x 5 mm³ were cut from the processed material for in situ neutron diffraction measurements up to 1250°C. The samples were fixed into either a vanadium sample holder (room temperature experiment) or niobium sample holder (high temperature experiment) that was subsequently inserted into a vacuum furnace with niobium heating elements. The in-situ texture measurements were performed on the GEM diffractometer at the ISIS pulsed neutron source, Rutherford Appleton laboratory, Chilton, UK. GEM has considerable coverage in orientation space with six
detector banks equipped with 7000 individual detector elements yielding coverage of
~4sr [15]. Due to this high detector coverage, quantitative texture measurements can
be performed with single specimen orientation. Detailed information about GEM can
be found in [15, 17]. One detector element is typically about 5 x 200 mm² in size and
aligned in such way that its scintillator is well matched to Debye Scherrer cones. As a
result, a detector element at 90° scattering angle has a solid angle of 0.2° x 10⁹. Due
to the poor neutron scattering properties of Ti-6Al-4V relatively long counting times
of 30 min were necessary for measuring several reflections with a sufficient count
rate. Texture measurements were carried out at room temperature, 800, 950, 1050,
1250°C and at 210°C. The β transus of Ti-6Al-4V is at approximately 995°C [18].
The incident beam was 30 x 20 mm² and the sample was orientated to have the rolling
direction parallel to the incident beam. In this way, the entire sample volume of 1500
mm³ was illuminated. An assembly of 164 separate detector groups was used during
the experiment with each group having an angular coverage of ~ 10° x 10°. The data
was normalized to the incident neutron flux distribution and corrected for detector
efficiencies. The 164 detector groups were converted into 164 d-spacing patterns,
which correspond to texture detector grouping. The 164 diffraction patterns were
simultaneously Rietveld refined using the MAUD software [19, 20]. Experimental
diffraction spectra together with their fits are shown in Figure 1 when using the
vanadium and niobium sample holders. It can be seen that only the titanium
reflections were observed. As a result of texture, the peak intensities of each of the
measured spectra do not conform to those predicted by structure factor. Therefore,
each cell of the orientation distribution function was refined in order to best-fit the
intensities of the Bragg peaks in the calculated rietveld spectra to each of the 164
measured spectra. This analysis was performed using the extended WIMV (E-WIMV)
algorithm as implemented in MAUD. The textures were plotted in form of ODF and pole figures using TEXTAN III developed by Bate [21]. TEXTAN-III was also used to predict transformation textures assuming no variant selection. This was done by applying the Burgers orientation relationship as a rotation of Euler angles taking into account the crystal symmetries of the parent and the daughter crystals. TEXTAN III applies the transformation to experimental data by using the spherical harmonic method proposed by H. Bunge [22]. In this way it was possible to describe the differences between the predicted and measured $\beta$ and $\alpha$ texture and to identify the level of variant selection during heating and cooling. The reference systems for the Euler angles, are such that the specimen coordinate systems; the rolling direction (RD), transverse direction (TD) and normal direction (ND) are Cartesian and are parallel to crystal coordinates $[10\bar{1}0]$, $[1\overline{1}20]$ and $[0002]$ respectively. This convection was used in all the figures in this work.

### 2.3 $\beta$ grain size analysis

For the purpose of determining the accurate $\beta$ grain size of the two variants of Ti-6Al-4V after the $\beta$ heat treatment, mid sections of the samples were prepared following standard grinding/polishing procedures and etched in 2% HF, 8% HNO$_3$ and 90% H$_2$O (Kroll’s reagent) for 10s for optical inspection. The grain size analysis was carried out on reconstructed $\beta$ grains from EBSD analysis. For this purpose, the samples were further OPS polished (1hour) and etched repeatedly with the last polishing step taking 2 hours. A CamScan Field Emission Gun Scanning Electron Microscope (FEGSEM), equipped with HKL Channel 5 software was used for EBSD data acquisition and analysis. In order to reconstruct the $\beta$ microstructure it was
necessary to use a step size sufficiently small (10 μm) to capture the transformed α microstructure (lamellar microstructure with large α colonies). The software applied to reconstruct β grains has been developed at the University of Sheffield within IMMPETUS (Institute for Microstructural and Mechanical Process Engineering). Details regarding the β reconstruction methodology can be found in [23].

3 Results

3.1 Microstructure and Texture Evolution

Figure 2 shows the starting microstructure and α textures of the two alloys of Ti-6Al-4V after annealing treatment at 950ºC for 8hours. The textures are presented in \( \varphi_2 = 0 \) section of the ODF. Six α texture components presented in form Euler angles \( \{ \varphi_1, \Phi, \varphi_2 \} \) (°) were generated and most noticeable is a strong transverse texture component at Euler angles \( \{ \varphi_1, \Phi, \varphi_2 \} \{3.6°, 86°, 0°\} \), which is texture typical component of Ti-6Al-4V rolled above 900ºC. The second significant component is the \( \{ \varphi_1, \Phi, \varphi_2 \} \{88.4°, 43°, 0°\} \) orientation of basal poles rotated 30° away from the normal direction (ND) towards the RD direction and four other orientations variants \( \{74.3°, 88.4°, 0°\}, \), the \( \{60.3°, 2.3°, 0°\}, \{25°, 21.1°, 0°\} \)and \( \{2.3°, 2.3°, 0°\} \). The similarity of the α textures in both alloys, together with the same grain size, demonstrates that yttrium additions did not have any significant effect on the grain morphology and α textures produced during thermomechanical processing. Hence, an almost identical starting condition was achieved before undertaking any β heat treatment for in-situ texture measurements and variant selection studies. The optical micrographs of Ti-6Al-4V
and Ti-6Al-4V-0.4Y after β heat treatment at 1250°C and furnace cooling are shown in Figure 3a and b. The microstructures are populated by large α lamellar colonies. The prior β grains are easily identified by continuous films of grain boundary α (α_{GB}) in Figure 3a and b. The continuous films of grain boundary α (α_{GB}) in the material containing yttrium confirms that nucleation of α colonies were indeed from the β grain boundary α and not from Y_2O_3 particles precipitated at the prior β grain boundaries. Figure 3c compares the β grain size distribution of both alloys determined from the reconstructed β map recorded by EBSD. It can be seen that conventional Ti-6Al-4V displays a wider grain size distribution than the yttrium variant with a significant tail towards grain sizes exceeding 3 mm. The mean β grain size of conventional Ti-6Al-4V is twice as large as for Ti-6Al-4V-0.4Y when heat treated at the same temperature.

The evolution of the α and β textures during thermal cycling of both alloys to temperatures well above the β-transus (α→β) and back to 210°C (β→α) are shown in Figure 4 – 7. At 800°C the measured ODF α texture (Figure 4a and b) appears very similar to the α texture recorded at room temperature, Figure 2c and d. At 950°C, still no significant difference was observed for the α texture when comparing the two alloys, Figure 4c and d. However, the intensity of the strong \{φ_1,Φ, φ_2\} \{3.9°, 86°, 0°\} component at room temperature or at 800°C is significantly reduced at 950° for the two alloys. Due to the low β volume fraction at room temperature it was only possible to acquire β reflections of sufficient quality at 800°C. At this temperature the β phase fraction was determined to be 30 vol. % from the neutron diffraction analysis. The high temperature β texture of the two alloys at 800°C is shown in Figure 5a and b. In both alloys the β texture exhibits rotated cube texture and
Variant selection and its effect on texture in Ti-6Al-4V

<111>//ND γ fibre texture components rotated 10 ° away from ND, which very similar to the deformation and recrystallised texture often observed in bcc materials. While the rotated cube texture intensity is very significant at 800°C compared to the <111>//ND γ rotated 10 ° away from ND in Ti-6Al-4V, the Ti-6Al-4V-0.4Y shows a far less pronounced cube texture and only a slight indication of a <111>//ND γ texture rotated 10 ° away from ND. The β textures at 950°C are represented in Figure 5c and d. The β volume fraction at 950°C was about 80 % according to the neutron diffraction analysis. While the β texture of the yttrium containing alloy shows little difference to the texture recorded at 800°C, the rotated cube texture seen in conventional Ti-6Al-4V has weakened. Upon further heating to 1050°C, the samples are fully transformed to β phase. The β texture of conventional Ti-6Al-4V has now lost the rotated cube texture component and has strengthened the γ fibre texture rotated 10 ° away from ND, Figure 6a. In contrast, the yttrium containing alloy has developed an almost fully randomised texture, Figure 6b. Further heating both alloys to 1250°C continued this trend with Ti-6Al-4V displaying texture sharpening (Figure 6c) while Ti-6Al-4V-0.4Y retained a very weak texture (Figure 6d). The next step in the thermal cycle was to furnace cool the material to 210°C (average cooling rate was 10 °C/min) and study the β→α phase transformation. It is fair to assume that no noticeable microstructural changes take place between 210°C and room temperature. As shown in Figure 3a and b, the microstructure is now fully lamellar with α laths having nucleated on the prior β grain boundary. The α textures are represented by ODFs for both alloys at 210°C in Figure 7a and b. Two very different pictures are seen for the two alloys. The relatively strong β texture observed in conventional Ti-6Al-4V at high temperature has resulted in two very strong α texture components in a
slightly rotated position in the ODF. The \{74.3°, 88.4°, 0°\} basal texture component in the original rolling direction in (0 0 0 2) pole figure has strengthened significantly. In addition, the \{88.4°, 43°, 0°\} corresponding to basal poles located at 30° away from the ND towards the RD has been strengthened. On the other hand, the strong \{3.9°, 86°, 0°\} transverse basal texture component that was observed before heating, Figure 2c, has now completely disappeared, Figure 7a. The α texture of the yttrium containing alloy is drastically different after cooling. Figure 7b demonstrates that the intensities are extremely low, indicating an almost random texture.

### 3.2 Predictions of β and α transformation textures

Figure 8 displays the predicted β transformation textures in the absence of α→β variant selection while Figure 9 shows the predicted α transformation textures after cooling the materials from 1250°C in the absence of β→α variant selection. Figure 8a and b demonstrate that the predicted textures using the transformation model do not agree both in quantity and in intensity with the measured β textures before significant β grain growth at 950°C (Figure 8c and d) or after the β-transus had been exceeded (Figure 9a and b). The predicted α transformation texture of the conventional Ti-6Al-4V shows weak texture components, Figure 9a. While the measured texture displays some of the predicted components (Figure 9c), they are twice as strong as the predicted ones. In contrast, the yttrium containing alloy shows similar texture strength between predictions (Figure 9b) and measurements (Figure 9d) although not necessarily the same components.
4. Discussion

4.1 Texture Evolution during $\alpha \rightarrow \beta$ phase transformation

As demonstrated above, neutron diffraction provides the possibility to measure both $\alpha$ and $\beta$ phase texture evolution in situ during $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation. The results show that some $\alpha$ texture weakening was observed in both alloys during heating but without any fundamental change of texture components. Figure 10 plots the calculated pole figures for the measurements recorded at room temperature, 800°C and 950°C, i.e. below the $\beta$-transus. As one would expect, the (0002) pole figures do not change significantly when the material is heated to 950°C. However, the measurement at 950°C shows that $(1 \bar{1} 2 \theta \ 0)$ pole density towards the rolling direction is significantly reduced. This observation has been made previously in CP titanium and was attributed to preferential transformation of recrystallisation texture components [11]. It can also be noted that the $(0 \ 0 \ 0 \ 2)$ $\alpha$ pole figures recorded at room temperature, 800°C and 950°C (Figure 10a-c) show no resemblance to the $(110) \beta$ pole figures recorded at 800°C or 950°C (Figure 10d-e). This indicates that $\beta$ which forms during heating only grows from the pre-existing $\beta$ phase. In contrast, when comparing the $\alpha$ phase pole figures recorded after cooling with the $\beta$ phase pole figures recorded at 1250°C, Figure 11a and b, one can see a clear resemblance of the $(1 \ 1 \ 0) \beta$ and $(0 \ 0 \ 0 \ 2) \alpha$ pole figures as expected from the Burger’s orientation relationship $\{0 \ 0 \ 0 \ 2\}_\alpha/\{1 \ 1 \ 0\}_\beta$ and $\{1 \ 1 \ 2 \ \theta\}_\alpha/\{1 \ 1 \ 1\}_\beta$ [13, 24].

An important observation of the in-situ heating studies is the differences in $\beta$ texture evolution for the two alloys. Even though the $\alpha$ textures after rolling and recrystallisation were practically identical for the two alloys, Figure 2c and d, at
800°C conventional Ti-6Al-4V displayed a markedly stronger β texture than the yttrium containing alloy, Figure 5a and b. It is worth noting that the conventional Ti-6Al-4V displayed a weak γ fibre texture at this stage, which is hardly visible in Ti-6Al-4V-0.4Y. When conventional Ti-6Al-4V exceeds the β transus very significant β grain growth takes place and it is clear from the texture analysis that grain coarsening is associated with a preferred growth of grains associated with the γ fibre, Figure 6a and c. In the case of the yttrium containing alloy, the β texture that develops above the β transus is very weak, Figure 6b, although at 1250°C a γ fibre becomes again visible, Figure 6d. Therefore, the absence of a γ fibre rotated 10° away from ND in the two-phase region in combination with more limited β grain growth due to the presence of yttria particles restricts the development of a strong β texture in the single phase region of Ti-6Al-4V-0.4Y. It seems plausible that minimising grain boundary energy drives preferential growth of grains associated with the γ fibre. This is in line with the formation of a γ fibre texture in IF steel during recrystallisation, which is also driven by minimising grain boundary energies [25].

4.2 Texture evolution during β→α phase transformation

Following an α→β→α phase transformation in the absence of variant selection, there are 72 possible orientation variants a single α grain can assume once it has transformed back [26]. In the absence of variant selection one would expect a nearly perfectly random texture even if the material had a sharp starting texture. The paragraph above has already demonstrated that in the case of an alloy that retains the high temperature β phase to room temperature, the first step towards randomisation during α → β phase transformation does not take place since new β grows from pre-
existing β. However, the β texture changes very significantly during β grain growth with the tendency of increasing γ fibre strength in the β phase with increasing β grain size. After cooling, the comparison of the measured α texture with the calculated α texture assuming no variant selection shows very significant differences for conventional Ti-6Al-4V but very little differences for the yttrium containing variant. It is also notable that the measured α texture is significantly stronger in Ti-6Al-4V compared to Ti-6Al-4V-0.4Y. The discrepancy between the predicted and measured α textures can be attributed to variant selection as previously demonstrated by EBSD studies in [8]. The present work further confirms these findings as neutron diffraction enabled to study a much larger volume of material than EBSD.

At this point it is necessary to summarize the main mechanisms of variant selection, which have been presented in the literature as precursor to texture memory. Variant selection has been discussed for both bcc and hexagonal materials including titanium, zirconium and steels [6-8, 27]. According to Hutchinson et al., [3] in their recent review, three main possible source of variant selection are possible: (a) stress generated during thermal cycle due to volume change; (b) untransformed remnant thin film of the initial phase or second phase at boundary of the high temperature phase, acting as nuclei for growth during cooling, (c) formation of grain boundaries in the high temperature phase with optimum characteristics for nucleation of certain orientations of the low temperature phase. The first mechanism is unlikely in Ti and Zr as the high temperature phase at temperatures above the β transus is very soft as mentioned before. In the present case, the material was heated to a temperature about 250°C above the β-transus ensuring that no remnant α phase is present. This leaves the third mechanism, which has been verified in Ti alloys when the material was cooled slowly from the β transus resulting in preferred nucleation and growth of
similar α orientations across two neighbouring β grains with a common <110> pole [6-8]. The potential effect of β grain coarsening on variant dominance, considering this mechanism, has been studied in [8] showing that the early nucleation of certain variants results in a more effective growth of them when the β grains are very coarse. The present work shows in addition to this that the actual β texture also evolves with coarsening β grain size. In order to evaluate the different β texture components, in respect of their potential to enhance variant selection, Figure 12 plots the individual β texture components that make up the γ fibre texture rotated 10° away from ND (a, c and d) and the associated α texture components after phase transformation following Burgers relationship (b, d and f). It is noticeable that even though each β texture component develops a number of different α texture components, there are two resulting α texture components, at Euler angles {80.1°, 30.7°, 0°} and {48.5°, 88°, 0°}, Figure 12b and f, that result from two different β texture components, Figure 12a and e. Coincidently, it is these two α texture components that become dominant during variant selection, Figure 9c. Therefore it seems that the combination of texture components can enhance variant selection. In the present case, these combinations develop when significant β grain coarsening takes place.

**Conclusions:**

The α and β texture evolution was studied in-situ using neutron diffraction during heating Ti-6Al-4V and a variant containing 0.4 wt.% Y above the β transus and cooling it back to near room temperature. Both alloys were heated to a peak temperature of 1250°C, which is about 250°C above the β transus of Ti-6Al-4V. Grain growth was restricted in the alloy containing yttrium addition, which
precipitated as yttria and effectively pinned the β grain boundaries during high temperature heat treatment. Similar starting microstructures and α textures were generated before β heat treatment in an attempt to single out β grain size as a variable and study its effect. The ability to measure texture in situ enabled capturing the high temperature β texture for detailed variant selection study, which led to the following conclusions:

(1) The grain boundary pinning effect due to precipitation of Y₂O₃ at β grain boundaries was evident up to 1250°C. It was found that Ti-6Al-4V displays a wider grain size distribution than the yttrium containing alloy with a significant tail towards grain sizes exceeding 3 mm.

(2) Continuous grain boundary α was found in both alloys confirming that α nucleated from the grain boundary in both alloys and not from Y₂O₃ precipitates in the case of Ti-6Al-4V-0.4Y.

(3) A comparison of the predicted β textures from the room temperature α texture measurements and measured β textures before the material exceeded the β transus confirmed that β phase grows from the pre-existing β phase.

(4) In-situ high temperature texture measurements highlighted that each alloy displayed significantly different β textures before they exceeded the β transus with the conventional alloy showing some early sign of a γ fibre texture rotated 10° away from ND. Once both alloys had exceeded the β transus, conventional Ti-6Al-4V developed a significantly stronger β texture (γ fibre texture) than the yttrium containing alloy. This suggests that significant β grain growth might favour strengthening of the γ fibre texture rotated 10° away from ND.
(5) After cooling to near room temperature, conventional Ti-6Al-4V displayed a significantly stronger texture than predicted from the measured $\beta$ texture assuming no variant selection. In contrast, Ti-6Al-4V-0.4Y showed very little signs of variant selection.

(6) The analysis of individual $\beta$ texture components and their transformation to $\alpha$ texture components has demonstrated the potential that a combination of $\beta$ texture components might promote variant selection as they have the potential in resulting in coinciding $\alpha$ texture components. As a consequence, a strong $\gamma$ fibre texture rotated 10° away from ND, which in the present case was related to significant $\beta$ grain coarsening, seems to have the potential of enhancing variant selection when $\alpha$ nucleates and grows from $\beta$ grain boundaries.

Finally, it is important to note that the observed effect of $\beta$ grain size on variant selection and texture strengthening is highly relevant from an application point of view since the $\beta$ grain size does increase from the outer surface to the core of a billet.

**Acknowledgements**

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**Table captions**

Table 1: Elemental compositions of Ti-6Al-4V and Ti-6Al-4V-0.4Y

**Figure captions**

Figure 1: Measured and fitted spectra at room temperature of (a) sample in the vanadium holder, (b) Fitting of measured spectra at room temperature of sample in the vanadium holder, (c) measured spectra at 950°C of sample in the niobium holder, (d) Fitting of measured spectra at 950°C of sample in the niobium holder.

Figure 2: Optical micrographs of samples recrystallised at 950°C for 8hr (a) Ti-6Al-4V and (b) Ti-6Al-4V with 0.4wt% Yttrium addition (Ti-6Al-4V -0.4Y). Neutron diffraction room temperature measured α texture ODF of recrystallised (a) Ti-6Al-4V (b) Ti-6Al-4V-0.4Y.

Figure 3: Optical micrographs of β heat treated samples at 1250°C (a) Ti-6Al-4V and (b) Ti-6Al-4V-0.4Y, (c) the β Grain size distributions of Ti-6Al-4V and Ti-6Al-4V-0.4Y after β heat treatment at 1250°C.

Figure 4: In situ measured α textures ODF during α→β phase transformation (heating) (a) Ti-6Al-4V at 800°C (b) Ti-6Al-4V-0.4Y at 800°C (c) Ti-6Al-4V at 950°C (d) Ti-6Al-4V-0.4Y at 950°C.

Figure 5: In situ β texture ODF recorded during α→β phase transformation (heating) (a) Ti-6Al-4V at 800°C (b) Ti-6Al-4V-0.4Y at 800°C (c) Ti-6Al-4V at 950°C (d) Ti-6Al-4V-0.4Y at 950°C.
Figure 6: In situ $\beta$ texture ODF recorded during heating in the $\beta$ phase field (a) Ti-6Al-4V at 1050°C (b) Ti-6Al-4V-0.4Y at 1050°C (c) Ti-6Al-4V at 1250°C (d) Ti-6Al-4V-0.4Y at 1250°C.

Figure 7: In situ $\alpha$ texture ODF recorded during $\beta \rightarrow \alpha$ phase transformation (cooling) (a) Ti-6Al-4V at 210°C (b) Ti-6Al-4V-0.4Y at 210°C.

Figure 8: Calculated $\beta$ textures from measured $\alpha$ texture according to Burgers orientation relationship (a) calculated $\beta$ texture ODF of Ti-6Al-4V (b) calculated $\beta$ texture ODF of Ti-6Al-4V-0.4Y (c) measured $\beta$ texture ODF of Ti-6Al-4V at 950°C and (d) measured $\beta$ texture ODF of Ti-6Al-4V-0.4Y at 950°C.

Figure 9: Calculated $\alpha$ textures from measured $\beta$ texture according to Burgers orientation relationship (a) calculated $\alpha$ texture ODF of Ti-6Al-4V (b) calculated $\alpha$ texture ODF of Ti-6Al-4V-0.4Y (c) measured $\alpha$ texture ODF of Ti-6Al-4V at 210°C and (d) measured $\alpha$ texture ODF of Ti-6Al-4V-0.4Y at 210°C.

Figure 10: In situ $\alpha$ and $\beta$ textures pole figures recorded during $\alpha \rightarrow \beta$ phase transformation of Ti-6Al-4V showing no orientation relationship between the (0002)$\alpha$ and (110)$\beta$ during heating (a) (0002) and (1120) pole figure recorded at room temperature (b) (0002) and (1120) pole figure recorded at 800°C (0 0 0 2) and (1 120)
pole figure recorded at 950ºC (d) (1 1 0) and (1 1 1) pole figures recorded at 800ºC (e) (110) and (111) pole figures recorded at 950ºC.

Figure 11: In situ $\alpha$ and $\beta$ textures pole figures recorded at 210ºC for $\alpha$ texture and 1250ºC for $\beta$ texture of Ti-6Al-4V showing orientation relationship between the (0002)$\alpha$ and (110)$\beta$ during $\beta \rightarrow \alpha$ phase transformation (a) (0002) and (1120) pole figure recorded at 210ºC (b) (110) and (111) pole figures recorded at 1250ºC.

Figure 12: ODFs of individual $\beta$ texture components plotted using Gaussian distribution ((a), (c) and (e)) and their calculated $\alpha$ transformation texture components based on $\beta \rightarrow \alpha$ Burgers orientation relationship ((b), (d) and (f)).
### Table 1

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Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V
Figure 3

Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V
Figure 5

Variant selection and its effect on texture in Ti-6Al-4V
Figure 6

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<td>(b) $\phi=45^\circ$</td>
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<td>(c) $\phi=45^\circ$</td>
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Figure 7

Variant selection and its effect on texture in Ti-6Al-4V
Variant selection and its effect on texture in Ti-6Al-4V
### Figure 9

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<th>Measured α texture</th>
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| **Ti-6Al-4V HT 1250°C**

- **φ = 90°**
- **φ = 0°**

| **Ti-6Al-4V-0.4Y HT 1250°C**
|----------------------|--------------------|
| **φ = 90°**
| **φ = 0°**

**Variant selection and its effect on texture in Ti-6Al-4V**
Figure 10

<table>
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Variant selection and its effect on texture in Ti-6Al-4V
Figure 11

Variant selection and its effect on texture in Ti-6Al-4V
## Figure 12

### β individual texture components

- (a) \( \varphi = 45° \)
- (b) \( \varphi = 58.3°, \varphi_1 = 59.6°, \varphi_2 = 45° \) ± 5°
- (c) \( \varphi = 78.6°, \varphi_1 = 60.7°, \varphi_2 = 45° \) ± 5°

### Transformed α texture components

- (a) \( \Phi = 90° \)
- (b) \( \varphi = 90° \)
- (c) \( \Phi = 90° \)
- (d) \( \Phi = 90° \)
- (e) \( \Phi = 90° \)
- (f) \( \Phi = 90° \)

### α texture components

- (a) \( \{ \varphi, \Phi, \varphi_1 \} \pm 5° \)
  - \( \{ 80.1°, 30.7°, 0° \} \)
  - \( \{ 44.5°, 39.6°, 0° \} \)
  - \( \{ 9.8°, 88°, 0° \} \)
  - \( \{ 48.6°, 88°, 0° \} \)
  - \( \{ 9.1°, 47.5°, 0° \} \)

- (b) \( \{ \varphi, \Phi, \varphi_1 \} \pm 5° \)
  - \( \{ 5°, 37.6°, 0° \} \)
  - \( \{ 59.3°, 34.6°, 0° \} \)
  - \( \{ 33.6°, 388°, 0° \} \)
  - \( \{ 88°, 88°, 0° \} \)

- (c) \( \{ \varphi, \Phi, \varphi_1 \} \pm 5° \)
  - \( \{ 80.1°, 30.7°, 0° \} \)
  - \( \{ 20.8°, 39.6°, 0° \} \)
  - \( \{ 44.5°, 39.6°, 0° \} \)
  - \( \{ 9.8°, 88°, 0° \} \)
  - \( \{ 48.6°, 88°, 0° \} \)
  - \( \{ 73.2°, 88°, 0° \} \)
7.1 *Comparison of neutron diffraction, EBSD, X-ray diffraction measured α texture*

The texture measurement and analysis using different diffraction techniques: laboratory X-ray diffraction, EBSD and neutron diffraction. Laboratory X-ray and Electron Backscatter Diffraction (EBSD) were used for ex-situ texture measurements, while neutron diffraction is used for in situ texture measurement. In the order to effectively compare the textures obtained from these three techniques it was essential to carry out the textures measurements on samples that had the same starting condition. Therefore, the specimen rolled at 950 °C and heat treated at 950 °C for 8 h and cooled at 1 °C/min was used for this measurement. The measured recrystallization α textures using the three techniques are presented in form of pole figures in Figure 7-1. It can be seen that there is a good agreement between the results obtained with the three techniques. The sets of pole figures obtained with neutron, EBSD and lab x-ray diffractions show practically the same features, with excellent match both shape and intensity. The observed difference in intensity in basal (0002) planes and prism planes (1010) and (1120) in the neutron and x-ray measured pole figures compared to EBSD were in the neighbourhood of 1 mrd, this might be attributed to spatial resolution. It is interesting to note that the agreement in the measured pole figures features using the three techniques clearly demonstrated their capabilities in accurate texture measurements. It also successfully demonstrated that crystallographic texture data can be reliably obtain in GEM in a ‘single shot’ without sample rotation and that the EBSD and X-ray diffractions dataset obtained over the measured area are sufficiently enough to statistically represent the texture of the material.
Figure 7-1 Recrystallisation α texture pole figures measured using different techniques (a) EBSD (b) X-ray diffraction (c) Neutron diffraction
Chapter 8  **Conclusions**

In the present study, the effect of thermomechanical processing steps on texture evolution and variant selection during phase transformation in Ti-6Al-4V has been studied. Attempts were made to establish the dominant mechanisms involved in texture evolution and variant selection during processing and $\alpha\rightarrow\beta\rightarrow\alpha$ phase transformation. Moreover, thermomechanical routes that could reduce the degree of variant selection occurrence during processing and phase transformation have been explored, which may reduced the formation of macrozones in Ti-6Al-4V. The results obtained here are reproducible and applicable to other hexagonal metals like zirconium.

The investigations were conducted using three characterisation techniques: EBSD, laboratory X-ray and neutron diffraction. Due to the metallurgical state of the material after deformation, the laboratory X-ray method was used to measure the texture of the as-rolled materials and in order to capture relevant microstructural information (i.e. information about the $\beta/\beta$) for variant selection studies, electron backscattered diffraction (EBSD) was used to measure the recrystallised and transformed materials at ambient temperatures. *In-situ* texture measurements were carried out using neutron time-of-flight diffraction to study variant selection occurring during heating $\alpha\rightarrow\beta$ and cooling $\beta\rightarrow\alpha$ phase transformations. A detailed literature review at the beginning of this thesis summarises the fundamental principles needed to understand this work. Finally, in the following section, important conclusions drawn from this study are summarised and future work presented.
β Grain growth

The effect of β grain size on variant selection and the formation of large α colonies across β grain boundaries was established in convectional Ti-6Al-4V and Ti-6Al-4V-0.4Y alloys. It was demonstrated that with increasing β grain size variant selection intensified favouring a specific α texture components resulting in ‘butterfly’ type of α colonies. Such large regions of the same crystallographic region are suspected to cause macrozones during subsequent thermomechanical processing.

The fundamental concept responsible for this variant selection mechanism was demonstrated in this study. Interrupted cooling experiments revealed that α nucleates first on β grain boundaries that are formed by two β grains having a common (1 1 0) normal and that these α colonies display almost exclusively a similar crystallographic orientation. In case of convectional Ti-6Al-4V, the dominance of these variants with increasing β grain size can be related to the relative free growth of these specific α texture components into an “unoccupied” β grain. However, in Ti-6Al-4V-0.4Y it was proposed that the growth of these specific α colonies is mainly limited by the β grain size and consequently, these texture components grow in dominance with increasing β grain size.

The grain boundary pinning effect due to precipitation of Y₂O₃ at prior β grain boundaries was evident at 1050°C and 1150°C. This led to half the size of β grains observed in Ti-6Al-4V-0.4Y compared to conventional Ti-6Al-4V when β heat-
treated at the same temperature. A similar but less pronounced effect of grain growth was observed when comparing heat treatment temperatures for the same alloy.

In this work, a certain degree of texture memory was observed. The $\beta$ heat treatment did not change the main texture components, although the individual texture component intensities had changed dramatically after $\beta$ heat treatment. When comparing the two alloys, significant differences were observed in terms of texture intensities although the starting $\alpha$ textures were almost identical. The similarity of the starting $\alpha$ texture in this study was critical in order to identify the effect of $\beta$ grain growth on variant selection and texture memory.

**Effect of deformation temperature**

In this study, it was possible to generate two different starting crystallographic orientations by deforming Ti-6Al-4V at two different temperatures (800 °C and 950 °C). It was demonstrated that uniaxial rolling at 800 °C produced B/T texture, whereas the rolling at 950 °C produced T texture. The effect of these textures on variant section during $\beta$ heat treatment was established in this work. It was found that even though the material rolled originally at 800 °C displayed a stronger $\alpha$ texture and relatively small regions of macrozones after $\beta$ heat treatment it was the material rolled originally at 950 °C with large regions of macrozones that showed greater variant selection.

It was established in this study that the generated $\beta$ texture played a significant role in the formation of these macrozones. The combination of certain $\beta$ texture components belonging to $\gamma$-fibre texture rotated 10 ° away from ND generated during rolling and $\beta$
heat treatment of Ti-6Al-4V at 950 °C increases the likelihood of having β grain pairs with closely aligned (1 1 0) planes compared to rolling at 800 °C. Predictions of possible <1 1 0> pole misorientation distributions for the two investigated β textures show an increase in the misorientation angles <=10 ° in material originally rolled at 950 °C compared to material rolled at 800 °C. Therefore, it was proposed that avoiding the generation of certain combinations of β texture components during thermomechanical processing had the potential of reducing variant selection during subsequent β heat treatment.

In this study, it was found that the number of prior β grain boundaries with these ‘special β grain pairs’ was larger in the material rolled originally at 950 °C than 800 °C. Consequently, more aligned α colonies on both sides of a former β grain boundary were found in the material rolled at 950 °C, which could potentially lead to formation of macrozones. The α variant chosen by this mechanism was responsible for α textures after β heat treatment and was stronger than one would expect.

It was also established that even though variant selection was most dominant in the material rolled at 950°C, the texture strength after β heat treatment was still stronger in the material rolled at 800°C. This was explained by a difference in prior β texture. Therefore, variant selection does not only affect macrotextures but seems to contribute significantly to the development of microtexture and macrozones.
In situ texture measurement

The ability to measure texture in situ enabled capturing the high temperature $\beta$ texture for detailed variant selection study in two Ti-6Al-4V alloys, which led to the following.

It was found that during heating $(1\ 1\ 0)\beta$ and $(0\ 0\ 0\ 2)\alpha$ pole figures show no resemblance, which suggested that $\beta$ grows from pre-existing $\beta$ and not from the $\alpha$ phase even though $\alpha$ is the dominant phase at low temperature. The dominant $\beta$ textures were the cube texture and significantly weak $<1\ 1\ 1>/\text{ND}$ $\gamma$ fibre rotated 10° from ND $\beta$ texture evolved even before the $\beta$ transus was exceeded. The $\beta$ texture strengthened noticeably above the $\beta$ transus, forming a $<1\ 1\ 1>/\text{ND}$ $\gamma$ fibre texture rotated 10° from ND, in the case of conventional Ti-6Al-4V but not Ti-6Al-4V-0.4Y, which was related to $\beta$ grain coarsening. Significant variant selection was found during $\beta \rightarrow \alpha$ transformation in the case of the former that could be related to the formation of certain $\beta$ texture components that contribute to the $<1\ 1\ 1>/\text{ND}$ $\gamma$ fibre texture.

It was found that variant selection during heating i.e. $\alpha \rightarrow \beta$ phase transformation is significantly lower compared to variant selection observed during cooling $\beta \rightarrow \alpha$ phase transformation. This means early stage $\alpha$ nucleation favours the variant selection based on neighbouring $\beta$ grains with nearly parallel $<110>$ leading to variant selection.
Conclusions

The continuous films of grain boundary α (αGB) in the material containing yttrium confirms that nucleation of α colonies were indeed nucleated from the β grain boundary α and not from Y₂O₃ precipitates at the prior β grain boundaries.

Finally, it is important to note that the observed effect of β grain size on variant selection and texture strengthening is highly relevant from an application point of view since the β grain size does increase from the outer surface to the core of a billet. As demonstrated here, large regions with a particular texture component might therefore be expected deep inside a billet and further thermomechanical processing might not completely break these α colonies. One might suggest that this is the source of macrozones often found in these alloys after thermomechanical processing. The texture variation and variant selection observed by deforming at different temperatures can be used as reference point in designing more robust thermomechanical processing routes for both titanium and zirconium alloys.
Future work

Based on the results obtained in this study, it is clear that deformation temperature has significant influence on texture evolution and variant selection. Therefore, it is recommended that further work be done in this area.

(1) Deformation at different temperatures would provide more insight on which rolling temperature would give less variant selection. Rolling at different temperatures has been found to influence the development of the so-called “butterfly α colonies” which are the potential source for macrozones during subsequent further thermomechanical processing. The formation of the so-called “butterfly α colonies” have been linked to variant selection and dominant growth of the selected variants. Therefore, by changing the rolling temperatures at 20 ° intervals starting at 800 °C even above the β transus, the degree of occurrence of these so-called “butterfly α colonies” could be determined. This study would be very relevant to both titanium and zirconium industries because it could be used to design an appropriate thermomechanical processing route that would result in insignificant amounts of the so-called “butterfly α colonies”.

(2) Variation of inter-pass holding times is another important aspect that was not investigated during the course of this study. It would be important to see how the inter-pass holding times affect the formation of these so-called “butterfly α colonies”. These two identified areas would shed further light in order to limit the evolution of “butterfly α colonies”, and optimize the mechanical properties.
(3) Understanding the microstructural evolution in the $\beta$ phase of conventional $\alpha + \beta$ alloys is hindered by the extremely fast grain growth of the $\beta$ phase when the material is exceeded the $\beta$ transus. In conventional Ti-6Al-4V, $\beta$ grains grow by many hundreds of microns within seconds making time resolved studies very difficult. In addition, the high temperature phase cannot be frozen when the material is quenched. One of the interesting finding of the present work was that $\beta$ grain growth resulted in the formation of a strong $\gamma$ fibre texture rotated 10 $^\circ$ from ND, which is very similar to strong $\gamma$ fibre texture that is usually associated with recrystallisation in bcc steel, which is clearly a different phenomenon. Based on this fact, there is no clear understanding of the formation of these $\gamma$-fibre texture in Ti- alloys. Therefore, to gain more understanding of the $\beta$ microstructure it would be necessary to study more heavily $\beta$-stabilized alloys (Ti-21S) that shows very slow $\beta$ grain growth and in which the $\beta$ phase is completely retained at relatively slow cooling (air cooling) at room temperature. While the addition of Yttrium was a first step to control $\beta$ grain growth, one might argue that the particles have an additional affect. Choosing an alloy such as Ti-21S would allow the following studies:

- Detailed $\beta$ grain growth studies and the possible effect on the development of and strengthening of $\gamma$ fibre texture rotated 10 $^\circ$ from ND, which has been identified as potential root cause of formation of macrozones can be studied with this alloy.

- It will provide an insight on the $\beta$ texture during and after $\beta$ heat treatment, which would verify how correct the measured $\beta$ texture in the two phase $\alpha + \beta$ alloy.
Future work

- One could avoid the potential effect of yttria during $\beta \rightarrow \alpha$ transformation in Ti-21S.

The preliminary result of grain size distribution of Ti-21S as function of temperature and holding time is presented Figure 8-1 and the measured $\beta$ texture and representative $\beta$ texture of 1000 Euler angles in Figure 8-2. Also presented here is the relative frequency misorientation distribution between the $<110>$ in the rage of 0-20º using bin size of 1º. The result shows that the misorientation angles increased as the intensity of the $\gamma$-fibre texture increases.

![Graph showing grain size distributions](image)

**Figure 8-1** The $\beta$ Grain size distributions of the as received Ti-21S, HT at 900°C for 15h and WQ and HT at 1000°C for 15h and WQ.
Future work

<table>
<thead>
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<th>Measured β Texture</th>
<th>Representative of β Texture of 1000 Euler angles</th>
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<td>(c) $\phi_2 = 45^\circ$</td>
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<td>(e) $\phi_2 = 90^\circ$</td>
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Figure 8-2 Measured β textures and representative of β textures ODF ($\phi_2 = 45^\circ$) of 1000 Euler angles (a) as received Ti-21S measured β texture, (b) Representative of 1000 Euler angles of the as received Ti-21S β texture, (c) as received Ti-21S measured β texture (smaller scale) (d) Representative of 1000 Euler angles of the as received Ti-21S β texture (smaller scale), (e) Ti-21S HT at 900°C for 15h and WQ measured β texture (f) Ti-21S HT at 900°C for 15h and WQ Representative of 1000 Euler angles β texture, (g) Ti-21S HT at 1000°C for 15h and WQ measured β texture, (h) Ti-21S HT at 1000°C for 15h and WQ Representative of 1000 Euler angles β texture.
Future work

Variant selection and its effect on texture in Ti-6Al-4V

Figure 8-3 Figure 3: Relative frequency misorientation distribution between the <110> in the rage of 0-20° using bin size of 1°.
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Appendix 1

The effect of Processing Temperature on Texture Evolution and Variant Selection during Phase Transformation in Ti-6Al-4V
The effect of Processing Temperature on Texture Evolution and Variant Selection during Phase Transformation in Ti-6Al-4V

G. C. Obasi, S. Birosca, D. G. Leo Prakash, J. Quinta da Fonseca and M. Preuss

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In the present study, the role of starting texture on texture evolution and variant selection during \( \alpha \rightarrow \beta \rightarrow \alpha \) transformation has been studied. Firstly, the samples were thermomechanically processed at different temperatures in order to generate significantly different \( \alpha \) texture but similar recrystallized grain morphology in both cases. Subsequently, both materials were recrystallized and then heat-treated above the \( \beta \) transus followed by \( 1^\circ \)C/min cooling rate to promote diffusional phase transformation starting from \( \beta \) grain boundary. EBSD texture analysis in both specimens showed that \( \beta \) heat treatment produced \( \alpha \) texture components that were similar to the recrystallized \( \alpha \) texture but with different intensities. The degree of variant selection was assessed by comparing the strength of the measured and predicted \( \alpha \) texture from high temperature \( \beta \) texture assuming equal occurrence of all the possible 12 variants during \( \beta \rightarrow \alpha \) transformation. It was found that the material rolled at 950°C showed a greater tendency to variant selection than the material rolled at 800°C but the latter shows an overall stronger texture after \( \beta \) heat treatment. The findings are discussed in terms of \( \alpha \) nucleation energies and combination of \( \beta \) texture components promoting the occurrence of \( \beta \) grain pairs with a common (110) pole.

**Keyword:** Ti-6Al-4V, Rolling Temperature, Texture evolution and Variant selection

1. Introduction

The thermomechanical processing route of an engineering alloy is a critical step to generate the desired microstructure and crystallographic texture for optimum mechanical properties. Titanium alloys are no exception and due to their low temperature hexagonal close packed crystal structure, they are particularly prone to develop strong crystallographic textures that can significantly affect the mechanical properties of the final product [1]. Titanium alloys are usually subjected to series of thermomechanical processing stages in the single alloy is a critical step to generate the desired microstructure [1].

**Table 1:** Chemical composition of the alloy used.

<table>
<thead>
<tr>
<th>Elem.</th>
<th>Fe</th>
<th>V</th>
<th>Al</th>
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<th>N</th>
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<th>Y</th>
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<tbody>
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<td>6</td>
<td>-</td>
<td>.17</td>
<td>-</td>
<td>-</td>
<td>.001</td>
<td>Bal</td>
</tr>
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</table>

Variant selection and its effect on texture in Ti-6Al-4V
Two blocks of 40×40×20mm were cut from the alloy, heat treated at 1050°C and water quenched in order to create a fairly random texture. These blocks were subsequently unidirectionally hot rolled at either 800°C and 950°C. The total sample thickness reduction of 75% was achieved through 5 passes at a reduction rate of 15% with 2 min reheating between each pass. The rolled specimens were subsequently heat treated for 8 hours at 950°C in a preheated tube furnace under argon atmosphere and cooled to room temperature at a rate of 1°C/min. Samples with dimensions of 10×10×5 mm³ were cut from the centre of the small slabs, subjected to β heat treatments at 1150°C for 30 minutes and cooled at 1°C/min under an argon atmosphere. Mid sections of the β heat-treated samples were prepared following standard grinding/polishing procedures and etched in Kroll’s reagent prior to optical microscopy examination. For better understanding of microstructure evolution and microtexture as well as crystallographic texture analysis Electron Backscatter Diffraction (EBSD) was used in this study. Due to the coarse β grain size after β heat treatment, relatively large 7×7mm² EBSD maps were recorded. The EBSD orientation data was used to plot pole figures and the Orientation Distribution Functions (ODFs). In order to determine the high temperature β texture, the α orientation maps were used to reconstruct the β grain structure based on the Burger’s orientation relation between the parent and daughter phases. The software used for the β reconstruction has been developed at the University of Sheffield within IMMPETUS (Institute for Microstructural and Mechanical Process Engineering). In order to obtain a successful reconstruction, a maximum allowable misorientation angle between α variants of a common β grain is defined, which in the present case was a misorientation angle of 3°. Further details on the β orientation reconstruction methodology are described in [12-15]. The information from the reconstruction was subsequently reloaded into Channel 5 to plot the β orientation map. TEXTAN III was used to plot ODFs of the β texture from the reconstructed values and to predict the α texture using a transformation model assuming no preferred variant selection [16]. In this way it was possible to describe the disagreement between the predicted and measured α texture and relate the level of variant selection as function of the initial β texture resulting from the deformation temperatures.

3. Results and discussion

3.1 Microstructure and texture before β heat treatment

The anneal heat treatment at 950°C of the material rolled at 800°C and 950°C resulted in comparable grain morphology with a mean grain size of 18µm and retained β at the triple joints of primary α. The textures of the materials rolled at 800°C and 950°C followed by the heat treatment are presented in form of ODF’s in Figure 1. The ODF sections presented here are the φ2 = 0° for α phase texture and φ2 = 45° for β phase texture.

Both conditions are dominated by the α texture component at (-12-10)[10-10] located at the Euler angles {φ1, Φ, φ2} {0°, 90°, 0°} of φ2 = 0° section of the ODF, which represents the transverse basal texture component. In both conditions an additional α texture component is visible at (-12-15)[5-1056] { {φ1, Φ, φ2} {90°, 30°, 0°} of φ2 = 0° section of the ODF}, which represents basal poles aligned about 30° off ND towards RD. This second component is comparatively strong in the material rolled at 800°C and weak in the material rolled at 950°C.

3.2 Microstructure and texture after β heat treatment

After the β heat treatment at 1150°C, the β grain size distribution in the two conditions displayed no significant differences. The mean β grain size of both materials was ~500µm according to EBSD and optical micrograph measurements. The slow cooling rate resulted in a columnar lamellar microstructure with lamellae having grown from grain boundary α. The α transformation textures of the material originally rolled at 800°C and 950°C are presented in Figures 2.

It appears that both specimens showed similar texture components with different texture intensities. The transformed α textures of the Ti-6Al-4V originally rolled at 800°C is composed of weak (-12-10)[10-10], (-12-10)[10-12] and (-1215)[9-5-43] texture components with maximum intensity of 6 times random texture distribution while the

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texture component (-12-15)[5-1056] at the Euler angles \{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} is very strong with maximum intensities of 18 times random distribution. In the case of the specimen rolled at 950°C the intensity of the texture component (-12-15)[5-1056] at the Euler angles \{\varphi_1, \Phi, \varphi_2\} \{90^\circ, 30^\circ, 0^\circ\} is slightly lower than that of the specimen rolled at 800°C (16 times random distribution). The other two texture components are also lower in intensity in the material originally rolled at 950°C. Therefore the specimen rolled at 800°C shows a stronger α texture than the material originally rolled at 950°C after β neat treatment.

### 3.3 Variant selection

The effect of the initial rolling texture on variant selection during \(\beta \rightarrow \alpha\) phase transformation was investigated by comparing texture intensities of the measured and predicted α texture after both materials had been heat treated at 1150°C. In order to predict the α texture in the absence of any variant selection, it was first necessary to determine the high temperature β texture by reconstructing the β microstructure from large EBSD maps (about 100 β grains). The reconstructed microstructure was subsequently used to calculate the α texture based on the Burgers orientation relationship assuming no variant selection. The measured α textures as well as the reconstructed β textures and predicted α textures are presented in Figure 3.

![Figure 3: EBSD reconstructed ODF (φ2=45) section of β textures](image)

**Figure 3.** EBSD reconstructed ODF (φ2=45) section of β textures (a) rolled at 800°C + β HT (b) rolled at 950°C + β HT. EBSD calculated α texture from β texture according to Burger’s orientation relationship, (c) rolled at 800°C + β HT (d) rolled at 950°C + β HT. EBSD measured α textures (e) rolled at 800°C + β HT (f) rolled at 950°C + β HT.

The β texture displayed both α (A) and γ (B-E) fibre components rotated 10° away from ND, which are similar textures found in rolled and recrystallised bcc material. When comparing the predicted α textures with the measured α textures after β heat treatment it can be seen that all expected texture components can indeed be observed in the measurement. However, the intensities of the individual texture components varied between predictions and measurements. In the material originally rolled at 800°C the predicted and measured intensity of the main texture component at (-12-15)[5-1056] was 13 versus 18 times random distribution. In the case of the material rolled at 950°C, the difference was far more significant with a prediction of 6 times random distribution while in fact 16 times random distribution was measured. Hence, even though the material rolled at 800°C results in a stronger transformation α texture, variant selection seems to be more prominent in the material that was rolled at 950°C.

### 4. Discussion

As described earlier, it has been previously observed that neighbouring β grains with a common (110) β pole promote the same (0002) α variant on both sides of the prior β grain boundary that is in close alignment with the common (110) β pole in preference to the other five (110) β poles. In the present case, the extensive orientation maps were used to determine the number of β grain pairs that fulfil such criterion for variant selection. Figure 4. Examples are also given of “special” and “non-special” β grain pairs. It was observed that the orientation of the common (110) pole was almost exclusively in the direction that provides the required (0002) normal to explain the over-proportional representation of the (-12-15)[5-1056] component.

![Figure 4: Quantitative representation of α variants nucleating from the prior β grain boundaries](image)

**Figure 4:** Quantitative representation of α variants nucleating from the prior β grain boundaries (a) number of special β grains and non-special β grains of the material rolled at 800°C and 950°C + β HT, (b) special β grain pairs, (c) non special β grain pairs

It can be clearly seen that the material rolled at 950°C shows a higher number of grain pairs with a common (110) pole compared to the material rolled at 800°C. When considering grain boundaries, it was found that in the material rolled at 800°C about 6% of the grain boundaries were between special β grain pairs compared to 10% in the material rolled at 950°C. This observation raises the question of why the material originally rolled at 950°C might be more prone to pairs of β grains with a common (110) pole. To shed more light on this, the individual β texture components and fibres and the resulting α texture components from the transformation have been calculated.
and plotted in Figure 5. The most dominant β texture component \((113)\) \([5-81]\) is plotted in Figure 5a. It can be seen that the β transformation results in a number of α texture components including the experimentally dominating \((-12-15)\)[5-1056] component, Figure 5b. Figure 5c plots texture components of the \(<111>||\) ND γ fibre in β and Figure 5d plots the resulting α texture components. It can again be seen that among the different α texture components the \((-12-15)\)[5-1056] is observed again while the other α texture components appear at different positions, compare Figure 5b and d.

Figures 5a-d show that the α texture component, which seems to be strengthened by variant selection, coincides with one of the (110) poles observed for the (113) \([5-81]\) and \(<111>||\) ND γ fibre in β. Further calculations confirmed that these two β texture components indeed provide the required (110) pole that coincides with the selected (0002) pole. Hence, the observations suggest that it is specific combinations of β texture components that enhance variant selection during β to α phase transformation in Ti alloys during slow cooling conditions.

5. Summary
A detailed study of the effect of starting microstructure on variant section during β heat treatment has been carried out in an effort to enhance our understanding of variant selection in Ti alloys. The main findings can be summarised as follows:

1. It was possible to generate same grain morphologies but different starting α textures in Ti-6Al-4V by rolling the material at either 800°C or 950°C followed by annealing the material at 950°C.
2. Subsequent β heat treatment and detailed texture analysis of the α phase and reconstructed β phase revealed that even though the overall α texture is stronger in the material originally rolled at 800°C, variant selection is more dominant in the material first rolled at 950°C.
3. Variant selection has been discussed in terms of β grain pairs with common (110) poles. After β heat treatment, the material originally rolled at 950°C displayed a significant γ fibre texture in the β phase (typical sign for recrystallisation in bcc material), which was far less pronounced in the material rolled at 800°C.

4. It was also found that the combination of a dominant β texture component, which is part of the α fibre in bcc material, and the γ fibre particularly observed in the material rolled at 950°C provide common (110) poles that seem be well aligned with the (0002) poles that are strengthened by variant selection. Therefore, variant selection in Ti alloys slowly cooled from the β transus seems to be a result of specific combinations of β texture components.

6.0 REFERENCES
The authors are grateful for financial support from the EPSRC under EP/E048455/1, Rolls-Royce plc. and material provision from TIMET UK. The authors are also particularly indebted to Brad Wynne and his research group at the University in Sheffield for developing the β reconstruction software, which was key for undertaking this work.

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Appendix 2

In situ observation on the influence of $\beta$ Grain growth on Texture Evolution during Phase Transformation in Ti-6A-4V
In situ observation on the influence of $\beta$ Grain growth on Texture Evolution during Phase Transformation in Ti-6A-4V

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Keywords: Ti-6Al-4V, $\beta$ Grain Growth, Texture Evolution

Abstract.
In the present study, in situ phase transformation experiments have been carried out using neutron diffraction to monitor the texture evolution during the $\alpha \rightarrow \beta \rightarrow \alpha$ phase transformation in Ti-6Al-4V with and without 0.4% yttrium additions. The aim of adding yttrium was to control $\beta$ grain growth above the $\beta$ transus by grain boundary pinning. In the present case, strengthening of the $\beta$ texture, occurring during $\beta$ grain coarsening resulted in strengthening of particular $\beta$ texture components, which increases the likelihood of $\alpha$ texture modification by selective growth of $\alpha$ variants on the common (110) $\beta$ grain boundaries into unoccupied large $\beta$ grains.

1. Introduction

Material performance of high performance alloys such as titanium alloys is strongly dependent on the microstructural and crystallographic texture evolution during thermomechanical processing. To date, most texture studies in titanium and zirconium alloys have been carried out using EBSD and x-ray diffraction [1, 2]. One of the major difficulties when analysing textures of $\beta$ heat treated $\alpha + \beta$ titanium alloys or zirconium alloys is the large $\beta$ grain size that is generated during heat treating the material above the $\beta$ transus. In contrast to steel, conventional titanium and zirconium alloys do not contain carbides that restrict grain coarsening when holding the material in the high temperature single phase region. While EBSD provides the ability to combine macroscopic texture information with information on the microstructural scale, the requirement of very large EBSD maps to capture a sufficient number of $\beta$ grains in combination with a small step size in order to capture microstructure information, makes this methodology very time consuming. Furthermore, EBSD is mainly an ex-situ characterisation technique. In contrast, neutron time-of-flight diffraction (TOF) and high energy synchrotron x-ray diffraction [3-5] can overcome some of these issues as they are not compromised by surface effects. Particularly neutron diffraction enables one to probe large volumes of material (many hundreds of mm$^3$) and therefore can provide excellent information of the global texture. The capability to acquire quantitative bulk crystallographic texture from a single measurement in matter of minutes without sample reorientation has been demonstrated recently using the general Materials (GEM) diffractometer at ISIS pulse neutron source at UK Rutherford Appleton Laboratory [6]. Together with the high temperature furnace facility on GEM, it is possible to carry out in situ texture measurements as a function of temperature to monitor and quantify texture variation during phase transformation. In the present study, the general material diffractometer (GEM) at the ISIS pulsed neutron source at the UK Rutherford Appleton laboratory was used to measure in-situ the texture evolution during phase transformation. The aim was to study the evolution of the $\beta$ texture during heating in the two-phase region and above the $\beta$ transus. Considering that it is difficult to control the $\beta$ grain growth in an alloy such as Ti-6Al-4V alloy, a
second variant was produced by adding about 0.4wt% yttrium to pin β grain boundaries in the high temperature single-phase region [7].

2. Experimental Methods

The material used in the present study was Ti-6Al-4V produced by TIMET, UK. Some of this material was remelted at the University of Birmingham to add 0.4 wt. % of yttrium. The chemical compositions of both alloys are shown in Table 1.

Table: 1 Elemental compositions of the two variants of Ti-6Al-4V alloy

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</tbody>
</table>

Details of the rolling and recrystallisation processes can be found in [11]. For both alloys, the microstructure prior to the β heat treatment was a recrystallised equiaxed microstructure of the same grain size and with a very similar α texture. Cuboids of 20 x 15 x 5 mm³ were cut from the recrystallized samples for in situ neutron diffraction measurements up to 1250°C. The samples were fixed into either a vanadium sample holder (room temperature experiment) or niobium sample holder (high temperature experiment) that was subsequently inserted into a vacuum furnace with niobium heating elements. The in-situ texture measurements were performed on the GEM diffractometer at ISIS pulsed neutron source. GEM has considerable coverage in orientation space with six detector banks equipped with 7000 individual detector elements yielding coverage of ~4Sr [6]. Due to this high detector coverage, quantitative texture measurement can be performed with single specimen orientation. Detailed information about GEM can be found in [6]. Texture measurements were carried out at room temperature, 800, 950, 1050, 1250°C and at 210°C. The β transus of Ti-6Al-4V is at approximately 995°C [8]. The incident beam was 30 x 20 mm² and the sample was orientated to have the rolling direction parallel to the incident beam. In this way, the entire sample volume of 1500 mm³ was illuminated. An assembly of 164 separate detector groups was used during the experiment with each group having an angular coverage of ~ 10⁰ x 10⁰. The data were normalized to the incident neutron flux distribution and corrected for detector efficiencies. The 164 detector groups were converted into 164 d-spacing patterns, which correspond to texture detector grouping. The 164 diffraction patterns were simultaneously Rietveld refined using the MAUD software [9, 10]. The values of the orientation distribution function (ODF) cells were then extracted using the extended WIMV (E-WIMV) algorithm as implemented in MAUD. Individual pole figures {0002}, {10-10}, {11-20} were recalculated from the ODF and plotted using the Beartex software.

3. Results.

3.1 Microstructure and Texture Evolution

When heating both alloys to 800°C and 950°C, the α textures appear very similar compared to the α textures observed at room temperature, Figure 1a and b. Due to the low β volume fraction at room temperature it was only possible to acquire β reflections of sufficient quality at 800°C. At this temperature the β phase fraction was determined to be about 30
vol. %. The high temperature β texture of the two alloys at 800ºC is shown in Figure 2a and b.

No significant difference in terms of β texture was detected between the two alloys at this stage. The β textures at 950ºC are represented in Figure 3a and b. While the β texture of the yttrium containing alloy shows no difference to the one recorded at 800ºC, the β texture of conventional Ti-6Al-4V has developed an additional cube texture component and texture sharpening. Upon further heating to 1050ºC, the materials are fully transformed to β phase. The β texture of conventional Ti-6Al-4V continues to change and sharpen while the texture of the yttrium containing alloy becomes essentially fully randomised. Further heating of both alloys to 1250ºC continued this trend with Ti-6Al-4V displaying texture sharpening, Figure 4a, while Ti-6Al-4V-0.4Y exhibited an almost perfectly random texture, Figure 4b.

The next step in the thermal cycle was to furnace cool the material to 210ºC (average cooling rate was 10ºC/min) and study the β→α phase transformation. It is fair to assume that no noticeable microstructural changes take place between 210ºC and room temperature. The microstructure is now fully lamellar with α laths having nucleated on the prior β grain boundary. The α textures are represented by pole figures for both alloys at 210ºC in Figure 5a and b. Two very different pictures are seen for the two alloys. The relatively strong β texture observed in conventional Ti-6Al-4V at high temperature has resulted in some α texture components that are now stronger than before β heat treatment while the yttrium containing alloy displays an almost random α texture.

4. Discussion

The data presented here demonstrate that neutron diffraction enables measuring both α and β phase texture evolution in situ during α→β→α phase transformation. The results show that there was no significant change of the α texture {0002} pole figures during heating to 950ºC. The evolution of the β texture differed significantly between the two different alloys. While the β texture was effectively randomised in Ti-6Al-4V with yttrium addition at 1050ºC and 1250ºC (Figure 4b) the β texture was strengthened in conventional Ti-6Al-4V (Figure 4a). The fundamental difference between the two alloys is the β grain size distribution that was generated during the β heat treatment. In the case of conventional Ti-6Al-4V, the mean β grain size at the end of the heat
treatment cycle was 800 µm compared to 400 µm in the yttrium doped version. It seems that the extensive β grain growth observed in conventional Ti-6Al-4V has resulted in preferential growth of favourably oriented β grains belonging to potentially the original cast texture. The (0002) pole figure of conventional Ti-6Al-4V recorded at 210ºC (Figure 5a) has some features that were already observed at room temperature prior to β heat treatment. The pole densities at RD and at 30º away from ND towards RD were strengthened and additional poles appeared compared (0002) α pole figures recorded at room temperature. Comparing the yttrium doped alloy and conventional Ti-6Al-4V clearly shows high levels of variant selection. As discussed in [1], large β grains allow relatively free growth of α variants from β grain boundaries with two adjacent β grains having a common (110) normal. Since the d-spacing of the (0002)α and (110)β planes is very similar, the critical nucleus size for α precipitation is decreased resulting in a reduced nucleation energy. It can also be noted that the (0002) α pole figures recorded at room temperature, 800ºC and 950ºC show no resemblance to the (110) β pole figures recorded at 800ºC or 950ºC. This indicates that β that forms during heating only grows from the pre-existing β phase. In contrast, during β→α phase transformation, one can see a clear resemblance of the (110) β and (0002) α pole figures as expected from the Burger’s orientation relationship \{0002\}α/{110}\β and {11-20}α/{111}\β.

5. Conclusions:
The evolution of the β texture and its effect on α variant selection has been studied in two variants of Ti-6Al-4V in-situ using neutron diffraction. It was found that restricting β grain growth during β heat treatment by yttrium additions has a very pronounced effect of the evolution of the β texture and subsequent α transformation texture. It was also found that the β texture that develops during heating is related to the pre-existing β phase and is not governed by the Burgers relationship during α to β phase transformation.

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References
Appendix 3

Step 1

function
[] = Step1D(LineNumber, Angle1, Angle2, Angle3, Question, RootDirectory)

GBA = [Angle1, Angle2, Angle3];

x = [1; 0; 0];
y = [0; 1; 0];
z = [0; 0; 1];

a1 = [1; 1; 0];
a2 = [1; 0; 1];
a3 = [0; 1; 1];
a4 = [1; -1; 0];
a5 = [1; 0; -1];
a6 = [0; 1; -1];

ag(1,:) = a1;
ag(2,:) = a2;
ag(3,:) = a3;
ag(4,:) = a4;
ag(5,:) = a5;
ag(6,:) = a6;

a = GBA(1,1);
b = GBA(1,2);
c = GBA(1,3);

GA1 = [cosd(a) * cosd(c) - sind(a) * sind(c) * cosd(b),
      sind(a) * cosd(c) + cosd(a) * sind(c) * cosd(b),
      sind(c) * sind(b);
      -cosd(a) * sind(c) - sind(a) * cosd(c) * cosd(b),
      -sind(a) * sind(c) + cosd(a) * cosd(c) * cosd(b),
      cosd(c) * sind(b);
      sind(a) * sind(b),
      -cosd(a) * sind(b),
      cosd(b)];

xa = GA1' * x;
ya = GA1' * y;
za = GA1' * z;
AA = [xa, ya, za];

A = [
    for gf = 1:6
        Temp = ag(gf,:);
        A(:,gf) = AA * Temp;
    end
    AFileName = ['A - Line ' int2str(LineNumber) '.txt'];
dlmwrite([RootDirectory ' [110] Files/' AFileName], A, 't')
    if Question == 'y' || Question == 'Y'
        AAb = (cross(ya, za)) / (dot(xa, (cross(ya, za))));
        AAb = (cross(za, xa)) / (dot(xa, (cross(ya, za))));
    end
    end
]

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\[ AAc = \frac{\text{cross}(xa, ya)}{\text{dot}(xa, (\text{cross}(ya, za)))}; \]
\[ AAR = [AAa, AAb, AAc]; \]
\[ \text{clear } AAa \ AAb \ AAc \]

\[ \text{AR} = []; \]
\[ \text{for } gf = 1:6 \]
\[ \text{Temp} = ag(gf, :)'; \]
\[ \text{AR}(:, gf) = AAR \times \text{Temp}; \]
\[ \text{assignin}('\text{base}', ['\text{AR} ' int2str(gf)], A) \]
\[ \text{end} \]

\[ \text{ARFileName} = ['\text{AR - Line ' int2str(LineNumber) '}.txt']; \]
\[ \text{dlmwrite([RootDirectory} ['[1 1 0] Files/' ARFileName]), AR, '\t'); \]
\[ \text{end} \]

\[ \text{end} \]
Appendix 3

Step 2

function []=Step2(CurrentLine, NumberofLines,RootDirectory)

StaticA=dlmread([RootDirectory ' [1 1 0] Files\ ' 'A - Line '
int2str(CurrentLine) '.txt']);
A1=StaticA(:,1);
A2=StaticA(:,2);
A3=StaticA(:,3);
A4=StaticA(:,4);
A5=StaticA(:,5);
A6=StaticA(:,6);

LinestoMultiplyWith=1:NumberofLines;
Loc=find(LinestoMultiplyWith==CurrentLine);
LinestoMultiplyWith=LinestoMultiplyWith(Loc+1:end);

mkdir([RootDirectory 'Line ' int2str(CurrentLine) ' Multiples\'])

for i=1:length(LinestoMultiplyWith)

    CurrentMultipleLine=LinestoMultiplyWith(i);
    CurrentMultipleArray=dlmread([RootDirectory ' [1 1 0] Files\ ' 'A - Line '
int2str(CurrentMultipleLine) '.txt']);
    disp(['Current Multiple: ' int2str(CurrentLine) ' x ' 
int2str(CurrentMultipleLine) ' Out Of ' int2str(NumberofLines)])
    B1=CurrentMultipleArray(:,1);
    B2=CurrentMultipleArray(:,2);
    B3=CurrentMultipleArray(:,3);
    B4=CurrentMultipleArray(:,4);
    B5=CurrentMultipleArray(:,5);
    B6=CurrentMultipleArray(:,6);

    c1=acosd(dot(A1,B1)/(norm(A1)*norm(B1)));
    c2=acosd(dot(A1,B2)/(norm(A1)*norm(B2)));
    c3=acosd(dot(A1,B3)/(norm(A1)*norm(B3)));
    c4=acosd(dot(A1,B4)/(norm(A1)*norm(B4)));
    c5=acosd(dot(A1,B5)/(norm(A1)*norm(B5)));
    c6=acosd(dot(A1,B6)/(norm(A1)*norm(B6)));

    c7=acosd(dot(A2,B1)/(norm(A2)*norm(B1)));
    c8=acosd(dot(A2,B2)/(norm(A2)*norm(B2)));
    c9=acosd(dot(A2,B3)/(norm(A2)*norm(B3)));
    c10=acosd(dot(A2,B4)/(norm(A2)*norm(B4)));
    c11=acosd(dot(A2,B5)/(norm(A2)*norm(B5)));
    c12=acosd(dot(A2,B6)/(norm(A2)*norm(B6)));

    c13=acosd(dot(A3,B1)/(norm(A3)*norm(B1)));
    c14=acosd(dot(A3,B2)/(norm(A3)*norm(B2)));
    c15=acosd(dot(A3,B3)/(norm(A3)*norm(B3)));
    c16=acosd(dot(A3,B4)/(norm(A3)*norm(B4)));
    c17=acosd(dot(A3,B5)/(norm(A3)*norm(B5)));
    c18=acosd(dot(A3,B6)/(norm(A3)*norm(B6)));

    c19=acosd(dot(A4,B1)/(norm(A4)*norm(B1)));
    c20=acosd(dot(A4,B2)/(norm(A4)*norm(B2)));
c21=acosd(dot(A4,B3)/(norm(A4)*norm(B3)));  
c22=acosd(dot(A4,B4)/(norm(A4)*norm(B4)));  
c23=acosd(dot(A4,B5)/(norm(A4)*norm(B5)));  
c24=acosd(dot(A4,B6)/(norm(A4)*norm(B6)));  

c25=acosd(dot(A5,B1)/(norm(A5)*norm(B1)));  
c26=acosd(dot(A5,B2)/(norm(A5)*norm(B2)));  
c27=acosd(dot(A5,B3)/(norm(A5)*norm(B3)));  
c28=acosd(dot(A5,B4)/(norm(A5)*norm(B4)));  
c29=acosd(dot(A5,B5)/(norm(A5)*norm(B5)));  
c30=acosd(dot(A5,B6)/(norm(A5)*norm(B6)));  

c31=acosd(dot(A6,B1)/(norm(A6)*norm(B1)));  
c32=acosd(dot(A6,B2)/(norm(A6)*norm(B2)));  
c33=acosd(dot(A6,B3)/(norm(A6)*norm(B3)));  
c34=acosd(dot(A6,B4)/(norm(A6)*norm(B4)));  
c35=acosd(dot(A6,B5)/(norm(A6)*norm(B5)));  
c36=acosd(dot(A6,B6)/(norm(A6)*norm(B6)));  

CMatrix=[c1 c2 c3 c4 c5 c6 c7 c8 c9 c10 c11 c12 c13 c14 c15 c16  
c17 c18 c19 c20 c21 c22 c23 c24 c25 c26 c27 c28 c29 c30 c31 c32 c33  
c34 c35 c36];  

CMatrix=round(CMatrix);  

for ii=1:36  
dlmwrite([RootDirectory '\Line ' int2str(CurrentLine) 'Multiples\Line ' int2str(CurrentLine) ' x Line 'int2str(CurrentMultipleLine) '.txt'],CMatrix,'\t');  
end

end  
end
Step 3

function []=Step3(CurrentLine, NumberofLines, RootDirectory)

StaticA=dlmread([RootDirectory '\1 \0] Files\' 'AR - Line ' int2str(CurrentLine) '.txt']);
A1=StaticA(:,1);
A2=StaticA(:,2);
A3=StaticA(:,3);
A4=StaticA(:,4);
A5=StaticA(:,5);
A6=StaticA(:,6);

LinestoMultiplyWith=1:NumberofLines;
Loc=find(LinestoMultiplyWith==CurrentLine);
%LinestoMultiplyWith(Loc)=[];
LinestoMultiplyWith=LinestoMultiplyWith(Loc+1:end);

mkdir([RootDirectory '\Line ' int2str(CurrentLine) '\ Multiples\'])

for i=1:length(LinestoMultiplyWith)

    CurrentMultipleLine=LinestoMultiplyWith(i);
    CurrentMultipleArray=dlmread([RootDirectory '\1 \0] Files\' 'AR - Line ' int2str(CurrentMultipleLine) '.txt']);
    B1=CurrentMultipleArray(:,1);
    B2=CurrentMultipleArray(:,2);
    B3=CurrentMultipleArray(:,3);
    B4=CurrentMultipleArray(:,4);
    B5=CurrentMultipleArray(:,5);
    B6=CurrentMultipleArray(:,6);

    disp(['Currently Processing File ' int2str(CurrentMultipleLine) '... Please Wait...'])

    c1=acosd(dot(A1,B1)/(norm(A1)*norm(B1)));  
c2=acosd(dot(A1,B2)/(norm(A1)*norm(B2)));  
c3=acosd(dot(A1,B3)/(norm(A1)*norm(B3)));  
c4=acosd(dot(A1,B4)/(norm(A1)*norm(B4)));  
c5=acosd(dot(A1,B5)/(norm(A1)*norm(B5)));  
c6=acosd(dot(A1,B6)/(norm(A1)*norm(B6)));

    c7=acosd(dot(A2,B1)/(norm(A2)*norm(B1)));  
c8=acosd(dot(A2,B2)/(norm(A2)*norm(B2)));  
c9=acosd(dot(A2,B3)/(norm(A2)*norm(B3)));  
c10=acosd(dot(A2,B4)/(norm(A2)*norm(B4))); 
c11=acosd(dot(A2,B5)/(norm(A2)*norm(B5))); 
c12=acosd(dot(A2,B6)/(norm(A2)*norm(B6)));

    c13=acosd(dot(A3,B1)/(norm(A3)*norm(B1)));
    c14=acosd(dot(A3,B2)/(norm(A3)*norm(B2)));
    c15=acosd(dot(A3,B3)/(norm(A3)*norm(B3)));
    c16=acosd(dot(A3,B4)/(norm(A3)*norm(B4)));
    c17=acosd(dot(A3,B5)/(norm(A3)*norm(B5)));
    c18=acosd(dot(A3,B6)/(norm(A3)*norm(B6)));
    c19=acosd(dot(A4,B1)/(norm(A4)*norm(B1)));

c20 = acosd(dot(A4, B2) / (norm(A4) * norm(B2)));  

CMatrix = [c1 c2 c3 c4 c5 c6 c7 c8 c9 c10 c11 c12 c13 c14 c15 c16  
c17 c18 c19 c20 c21 c22 c23 c24 c25 c26 c27 c28 c29 c30 c31 c32 c33  
c34 c35 c36];  

CMatrix = round(CMatrix);  

for ii = 1:36  
    dlmwrite([RootDirectory 'Line ' int2str(CurrentLine) '  
     Multiples\Reciprocal Line ' int2str(CurrentLine) ' x Line '  
     int2str(CurrentMultipleLine) '.txt'], CMatrix, '	');  
    end  
end
Step 4 Master process

```matlab
function []=MasterProcess()
%--------------------------------------------------------------------
%---------------------------- Options Block -------------------------
%--------------------------------------------------------------------
NumberofLines=10;
NameofFile='GBA.txt';
Data=dlmread(NameofFile);
Question2=input('Do you wish to output the Regular Angles? (y/n): ',
's');
Question=input('Do you wish to output the reciprocals? (y/n): ',
's');
RootDirectory='C:\MATLAB701\work\Euler Codes\';
Degrees=0;   %This must be 1 for Degrees and 0 for Radians
%--------------------------------------------------------------------
%--------------------------------------------------------------------
%--------------------------------------------------------------------

disp(' ')
disp('Currently Processing Step 1... Please Wait...')
disp(' ')
dir(RootDirectory, '[1 1 0] Files')
for i=1:NumberofLines
    Angle1=Data(i,1);
    Angle2=Data(i,2);
    Angle3=Data(i,3);
    if Degrees==1
        Step1D(i,Angle1,Angle2,Angle3,Question,RootDirectory)
    end
    if Degrees==0
        Step1R(i,Angle1,Angle2,Angle3,Question,RootDirectory)
    end
end

if Question2=='Y'|Question2=='y'
disp(' ')
disp('Currently Processing Step 2... Please Wait...')
disp(' ')
for i=1:NumberofLines
    Step2(i,NumberofLines,RootDirectory)
    disp(' ')
disp('Processing Next Set of Multiplications... Please Wait...')
disp(['Current Working Line: ' int2str(i)])
disp(' ')
end
end
```

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if Question=='y'|Question=='Y'
    disp(' ')
    disp('Currently Processing Step 3... Please Wait...')
    disp(' ')
    for i=1:NumberofLines
        disp(' ')
        disp('Processing Next Set of Multiplications... Please Wait...')
        disp(['Current Working Line: ' int2str(i)])
        disp(' ')
        Step3(i,NumberofLines,RootDirectory)
    end
end

disp('Function Processing Complete!')
end
Step 5 Plotting process

```matlab
function []=PlottingProcess()

%------------------------------------------------------------------
%------
%---------------------------- Options Block -------------------------
%------
%------------------------------------------------------------------

RootDirectory='C:\MATLAB701\work\Euler Codes\';
LineNumber=[1:10];
OriginalNumberofLines=10;
PlotReciprocals=1; %Enter 1 to plot the reciprocals or 0 to plot the normal
CriticalAngle=20; %Enter the Critical Angle (Multiples of 10 please)

tic
NBINS=180/CriticalAngle;
NBINS=round(NBINS);
MasterMatrix=[];
disp('Function is Processing... Please Wait...')
for i=1:length(LineNumber)
    CurrentLineNumber=LineNumber(i);
    LinestoMultiplyWith=1:OriginalNumberofLines;
    Loc=find(LinestoMultiplyWith==CurrentLineNumber);
    LinestoMultiplyWith(Loc)=[];
    folderName=['Line ' int2str(CurrentLineNumber) ' Multiples\'];
    NumberofFiles=length(LinestoMultiplyWith);
    for ii=1:NumberofFiles
        WorkingLine=LinestoMultiplyWith(ii);
        disp(['Currently Assembling File ' int2str(i) ' - ' int2str(WorkingLine)]);
        if PlotReciprocals==0
            CurrentFile=[RootDirectory folderName 'Line ' int2str(CurrentLineNumber) ' x Line ' int2str(WorkingLine) '.txt' ];
        end
        if PlotReciprocals==1
            CurrentFile=[RootDirectory folderName 'Reciprocal Line ' int2str(CurrentLineNumber) ' x Line ' int2str(WorkingLine) '.txt' ];
        end
        CurrentFile=dlmread(CurrentFile);
        MasterMatrix=[MasterMatrix CurrentFile];
    end
end
figure()
hist(MasterMatrix,NBINS)
titl=['Lines ' int2str(LineNumber(1)) ' to ' int2str(LineNumber(end)) ' Histogram'];
title(titl)
xlabel('Angle (Degrees)')
```

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ylabel('Frequency of Occurrence')
dlmwrite('MasterMatrix.txt', MasterMatrix, '\t')

CriticalMatrix=find(MasterMatrix<=CriticalAngle);
CriticalMatrix=MasterMatrix(CriticalMatrix)';
dlmwrite('CriticalMatrix.txt',CriticalMatrix, '\t')
bins=0:1:CriticalAngle;
FrequencyMatrix=[];

for i=1:length(bins)
    FindValue=bins(i);
    StatsValue=find(CriticalMatrix==FindValue);
    StatsValue=length(StatsValue);
    FrequencyMatrix(i,1)=StatsValue;
end

FrequencyMatrix=[bins' FrequencyMatrix];
dlmwrite('FrequencyMatrix.txt',FrequencyMatrix, '\t')
figure()
XAxis=0:1:CriticalAngle
plot(XAxis,FrequencyMatrix)
titl=['Lines ' int2str(LineNumber(1)) ' to ' int2str(LineNumber(end)) ' Critical Angle Frequency'];
title(titl)
xlabel('Angle (Degrees)')
ylabel('Frequency of Occurrence')
toc

end