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Recrystallisation in a rapidly annealed low cost β-titanium alloy

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University of Wollongong
Recrystallisation in a Rapidly Annealed Low Cost

\(\beta\)-Titanium Alloy

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<th>Meaning</th>
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<tbody>
<tr>
<td>β-Ti</td>
<td>Beta Titanium</td>
</tr>
<tr>
<td>AVG</td>
<td>Average</td>
</tr>
<tr>
<td>CD80</td>
<td>Cold-drawn at room temperature to 80% reduction in cross section area</td>
</tr>
<tr>
<td>CD90</td>
<td>Cold-drawn at room temperature to 90% reduction in cross section area</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron back-scattering diffraction</td>
</tr>
<tr>
<td>ECD</td>
<td>Equivalent circle diameter</td>
</tr>
<tr>
<td>GNB</td>
<td>Geometrically necessary boundary</td>
</tr>
<tr>
<td>GOS</td>
<td>Grain orientation spread</td>
</tr>
<tr>
<td>HAGB</td>
<td>High angle grain boundary</td>
</tr>
<tr>
<td>IDB</td>
<td>Incidental dislocation boundary</td>
</tr>
<tr>
<td>IM</td>
<td>Internal misorientation</td>
</tr>
<tr>
<td>LAGB</td>
<td>Low angle grain boundary</td>
</tr>
<tr>
<td>LCB</td>
<td>Low cost beta</td>
</tr>
<tr>
<td>LCB-Ti</td>
<td>Low cost beta Titanium</td>
</tr>
<tr>
<td>ODF</td>
<td>Orientation distribution function</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>TMP</td>
<td>Thermo mechanical processing</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray diffraction</td>
</tr>
<tr>
<td>VHN</td>
<td>Vickers hardness number</td>
</tr>
</tbody>
</table>
Abstract

This study investigates the recrystallisation behaviour of cold drawn low cost beta (LCB) Ti-1.5Al-6.8Mo-4.5Fe-0.2O (wt.%) by tracking the evolution of its microstructure and microtexture during rapid resistance annealing. The studied material was solution treated at 850 °C for 1800 s, followed by water quenching, then cold-drawn at room temperature to 80% (CD80) and 90% (CD90) reduction in diameter and consequently rapid resistance heating to select peak temperatures at rates of 10 and 20 °C.s⁻¹. All aspects of the present study have been characterised using advanced Electron Back-Scattering Diffraction (EBSD) and X-ray diffraction techniques. The EBSD maps were deconstructed into deformed, recovered, newly nucleated and growing grain subsets based on average internal misorientation, morphology and size of the microstructural features. XRD was used to obtain the bulk texture after accounting for background, irradiated volume and defocusing errors. The microhardness of LCB-Ti alloys after each annealing condition was measured by Vickers hardness testing.

Recovery was found to play an important role during rapid annealing. The partially recrystallised microstructures of CD80 and CD90 LCB-Ti were found to be heterogeneous such that the unrecrystallised grains had high aspect ratios with some deformed grains containing in-grain shear bands. A fewer number of shear bands were noted in the CD80 samples as compared to the CD90 samples. Discontinuous recrystallisation was manifested as newly nucleated (sub)grains formed mostly at the grain boundaries of deformed grains; with fewer nucleation events occurring at the in-grain shear bands. The density of the newly nucleated (sub)grains in the CD90 samples is approximately double that of the newly nucleated (sub)grains in the CD80 samples. In the nearly full recrystallised condition, the average equivalent grain size of the CD80 sample is nearly 1.5 times larger than that of the CD90 sample. An increase
of heating rate from 10 to 20 °C.s⁻¹ resulted in 1.7 times smaller final recrystallised grain size of the CD90 samples.

Bulk and micro-textures textures of the cold drawn and partially recrystallised conditions in CD80 and CD90 LCB-Ti were characterised by the (110) partial fiber parallel to the drawing direction. During the rapid annealing of CD80 and CD90 samples, the texture of the newly nucleated subset comprised a combination of orientation components that inherently possess high ((110)[110] and (111)[110]) and low ({001}[110] and (110)[001]) stored energy carried over from the cold drawing stage. At the end of the rapid annealing, texture of the approximately fully recrystallised conditions for CD80 and CD90 showed the presence of weak {001}[110] and (110)[001] orientation components.

The values of yield strength estimated from the Hall-Petch relationship is in general agreement with the values determined experimentally from Vickers hardness. The contribution from LAGBs to the true stress was found such to decrease with the progress of recrystallisation.
Acknowledgements

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I would like to express my special thanks to my supervisor, Dr. Azdihar Gazder who trained and guided me throughout my research work.

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I highly appreciate the help given to me by Mr. Ahmed Saleh for setting up my experiments and for sharing his knowledge during the analytical process.

Finally, I wish to thank my beloved family, friends and colleagues in Thai Nguyen University of Technology (TNUT) who supported me throughout my studies away from home.
Chapter 1

Literature review

1.1 Introduction

Titanium is a chemical element denoted as Ti with an atomic number of 22 and a low density of 4.54 g/cm³. It is widely used for aerospace and non-aerospace applications due to its inherently high strength and superior corrosion resistance. One of the most noticeable features of Ti is that it can exist in more than one crystallographic form. Commercially pure titanium (CP Ti) experiences an allotropic phase transformation at 882°C from hexagonal close packed (hcp) α-phase at lower temperature to the body centered cubic (bcc) β-phase [1]. The temperature of this phase transformation can be changed through the addition of alloying elements with the aim of improving on the balance between strength and ductility. The influence of the various alloying elements is presented in Fig. 1.1 [2] as follows:

- α-stabilising elements include Al, O, N, Ga, and C. These elements raise the phase transformation temperature between the α and β phases and help to further stabilise the α-phase at room temperature (Fig. 1.1a)
- β-stabilising elements comprise V, Cr, Mn, Fe, Co, Ni, Cu, W, Mo, Nb. These elements reduce the phase transformation temperature and help stabilise the β-phase at room temperature. The β-stabilisers can be divided into two groups. The first group comprises V, Mo, W and Ta and is β-isomorphous (Fig. 1.1b). The second group comprising Cu, Cr, Mn, and Fe is β-eutectoid with eutectoid temperatures up to 335°C (Fig. 1.1c).
Fig. 1.1: Basic phase diagrams for Ti alloys. The alloying elements affecting the various phase diagrams are (a): Al, O, N, C, Ga; (b) Mo, W, V, Ta; (c) Cu, Mn, Cr, Ni, Fe. The dotted lines in (b) and (c) show the martensite start ($M_s$) temperatures [3].

1.2 Titanium alloy classification

1.2.1 α and near α-alloys

Alloys in this group have the primary addition of α-stabilising elements and only trace amounts of β-stabilisers (< 2%) like V or Fe (Table 1.1) [4]. α and near α alloys provide good corrosion resistance due to the automatic creation of a stable oxide layer on the surface of the material. These alloys also offer good weldability due to their lack of response to heat treatment. The main disadvantage of these alloys is insufficient mechanical strength due to their lack of solid solution strengtheners and their resistance to heat treatment.

1.2.2 α+β alloys

These alloys possess a balance between α and β stabilising elements and result in a mix of α and β phases at room temperature (Table 1.2) [4]. Due to the appearance of the two phases, various thermo-mechanical processing (TMP) routes are employed in order to control the microstructures and mechanical properties of these alloys. Usually, the thermo-mechanical processing consists of four steps namely homogenisation, deformation, annealing and ageing. In these steps, parameters such as composition, temperature, time, heating and cooling rates affect the microstructure and the end crystallographic texture of the material.
Table 1.1: A glossary of some $\alpha$-Ti alloys with their compositions denoted in wt.%. The balance is Ti [4].

<table>
<thead>
<tr>
<th>$\alpha$-Ti Alloy and Grade</th>
<th>Pd</th>
<th>Mo</th>
<th>Al</th>
<th>Ni</th>
<th>V</th>
<th>Sn</th>
<th>Ru</th>
<th>Fe</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-Ti</td>
<td></td>
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<td>Grade 4</td>
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<tr>
<td>Ti-0.2Pd</td>
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<td></td>
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<tr>
<td>Grade 7</td>
<td>0.12-0.25</td>
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<td>Grade 11</td>
<td>0.12-0.25</td>
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<td>Ti-0.05Pd</td>
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<tr>
<td>Grade 16</td>
<td>0.04-0.08</td>
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<tr>
<td>Ti-0.3Mo-0.9Ni</td>
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<tr>
<td>Grade 12</td>
<td>0.20-0.40</td>
<td>0.6-0.9</td>
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<tr>
<td>Ti-3Al-2.5V</td>
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<tr>
<td>Grade 9</td>
<td>2.5-3.5</td>
<td>2.0-3.0</td>
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<tr>
<td>Ti-3Al-2.5V-0.05Pd</td>
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<td></td>
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</tr>
<tr>
<td>Grade 18</td>
<td>0.05</td>
<td>2.5-3.5</td>
<td>2.0-3.0</td>
<td></td>
<td></td>
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<tr>
<td>Ti-3Al-2.5V-0.1Ru</td>
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<tr>
<td>Grade 28</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Grade 6</td>
<td>2.0-3.0</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn ELI</td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>
**Table 1.2:** A glossary of some (α+β)-Ti alloys with their compositions denoted in wt.%. The balance is Ti [4].

<table>
<thead>
<tr>
<th>Commercial name</th>
<th>Alloy Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-811</td>
<td>Ti-8Al-1V-1Mo</td>
</tr>
<tr>
<td>IMI 685</td>
<td>Ti-6Al-5Zr-0.5Mo-0.25Si</td>
</tr>
<tr>
<td>IMI 834</td>
<td>Ti-5.8Al-4Sn-3.5Zr-0.5Mo-0.7Nb-0.35Si-0.06C</td>
</tr>
<tr>
<td>Ti-6242</td>
<td>Ti-6Al-2Sn-4Zr-2Mo-0.1Si</td>
</tr>
<tr>
<td>Ti-6-4</td>
<td>Ti-6Al-4V (0.200)</td>
</tr>
<tr>
<td>Ti-6-4 ELI</td>
<td>Ti-6Al-4V (0.130)</td>
</tr>
<tr>
<td>Ti-662</td>
<td>Ti-6Al-6V-2Sn</td>
</tr>
<tr>
<td>IMI 550</td>
<td>Ti-4Al-2Sn-4Mo-0.5Si</td>
</tr>
</tbody>
</table>

### 1.2.3 β alloys

These alloys contain sufficient amounts of β-stabilising elements that enable the β-phase to be retained in metastable or stable condition at room temperature (Table 1.3) [3]. Molybdenum equivalence ([Mo]eq) is used to describe the relative influence of beta stabilisers in multi-component titanium alloys and is given by [4]:

\[
[\text{Mo}]_{eq} = [\text{Mo}] + 0.2 [\text{Ta}] + 0.28 [\text{Nb}] + 0.4 [\text{W}] + 0.67 [\text{V}] + 1.25 [\text{Cr}] + 1.25 [\text{Ni}] + 1.7 [\text{Mn}] + 1.7[\text{Co}] + 2.5 [\text{Fe}]
\]

**1.2.3(a) Metastable β alloys**

One of the differences between metastable and stable β alloys is that metastable β alloys have ability to respond to heat treatment. Metastable β alloys can exhibit a 100% β-phase fraction following quenching. However, metastable β phase will precipitate an α-phase upon ageing below the β-transus temperature [3] (Fig. 1.2). Since these alloys provide a very high strength-to-weight ratio, excellent hardenability, their microstructures and textures are optimised to improve strength and ductility [5-8]. Currently, these alloys are predominantly used in aerospace and biomedical applications.
Table 1.3: Composition, category and $\beta$-transus temperature of several $\beta$-Ti alloys [9].

<table>
<thead>
<tr>
<th>Commercial name</th>
<th>Alloy composition</th>
<th>Category (Mo equivalent)</th>
<th>$\beta$-transus temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy C</td>
<td>Ti-35V-15Cr</td>
<td>Beta (47)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ti-40Mo</td>
<td>Beta (40)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ti-30Mo</td>
<td>Beta (30)</td>
<td></td>
</tr>
<tr>
<td>TIMETAL 125</td>
<td>Ti-6V-6Mo-5.7Fe-2.7Al</td>
<td>Metastable (24)</td>
<td>704</td>
</tr>
<tr>
<td>B120 VCA</td>
<td>Ti-13V-11Cr-3Al</td>
<td>Metastable (23)</td>
<td>650</td>
</tr>
<tr>
<td>1-8-5</td>
<td>Ti-1Al-8V-5Fe</td>
<td>Metastable (19)</td>
<td>825</td>
</tr>
<tr>
<td>TMZF</td>
<td>Ti-12Mo-6Zr-2Fe</td>
<td>Metastable (18)</td>
<td>743</td>
</tr>
<tr>
<td>Ti-LCB</td>
<td>Ti-4.5Fe-6.8Mo-1.5Al</td>
<td>Metastable (18)</td>
<td>800</td>
</tr>
<tr>
<td>VT35</td>
<td>Ti-15V-3Cr-1Mo-5Nb-3Al-3Sn-5Zr</td>
<td>Metastable (16)</td>
<td></td>
</tr>
<tr>
<td>Beta-C</td>
<td>Ti-3Al-8V-6Cr-4Mo-4Zr</td>
<td>Metastable (16)</td>
<td>795</td>
</tr>
<tr>
<td>IMI 205</td>
<td>Ti-15Mo</td>
<td>Metastable (15)</td>
<td>727</td>
</tr>
<tr>
<td>8-8-2-3</td>
<td>Ti-8V-8Mo-2Fe-3Al</td>
<td>Metastable (15)</td>
<td>775</td>
</tr>
<tr>
<td>Beta 21S</td>
<td>Ti-15Mo-2.6Nb-3Al-0.2Si</td>
<td>Metastable (13)</td>
<td>807</td>
</tr>
<tr>
<td>15-3</td>
<td>Ti-15V-3Cr-3Sn-3Al</td>
<td>Metastable (12)</td>
<td>760</td>
</tr>
<tr>
<td>Beta III</td>
<td>Ti-11.5Mo-6Zr-4.5Sn</td>
<td>Metastable (12)</td>
<td>745</td>
</tr>
<tr>
<td>VT22</td>
<td>Ti-5V-5Mo-5Al-2Cr-1Fe</td>
<td>Metastable (8)</td>
<td>850</td>
</tr>
</tbody>
</table>
Low cost LCB-Ti alloys with the typical composition Ti-6.8Mo-4.5Fe-1.5Al was developed over 15 years. It is also one of the most attractive β-Ti alloys due to its affordable ferro-molybdenum alloying additions. Typically, this alloy has a combination of high strength (yield strength at room temperature is 1350 N.mm\(^{-2}\)), low density (4.79 g.cm\(^{-3}\)) and low modulus (tensile and shear modulus are 11.10\(^4\) and 43 N.mm\(^{-2}\), respectively). It is most commonly used for springs and provides weight and space savings of up to 65% and 25%, respectively compared to its nearest steel equivalent [10]. Like other metastable β-Ti alloys, the microstructure and texture control of LCB-Ti can be achieved through thermo-mechanical processing.

1.3. Thermo-mechanical processing of β-Ti alloys

It is known that solid solution treatment in β-field followed by ageing (β-STA treatment) can be used to improve the mechanical properties of β-Ti alloys. Microstructures obtained after this treatment comprise α precipitates distributed in the β-phase matrix. While β phase-grain size strongly impacts ductility, the volume fraction and morphology of the α precipitates influences the overall mechanical strength. The drawback of this treatment is that at higher strength levels, the ductility of the β-Ti alloys decreases due to the large β grain size. This disadvantage can be resolved by employing thermo-mechanical processing.

Fig. 1.2: Pseudo-binary β-isomophous phase diagram showing the areas of metastable and stable beta titanium alloys [3].

1.2.3(b) Low cost LCB-Ti alloys
treatment including β solid solution treatment, cold deformation, annealing and ageing.

1.3.1 β Solid solution treatment

Solid solution heat treatments comprise heating the β-Ti alloys above the β-transus temperature followed by quenching in order to obtain a 100% metastable β-phase microstructure due to the dissolution of the primary α-phase.

Ivasishin et al. [11] compared the microstructures of LCB-Ti alloy after β solid solution treatment followed by water quenching using a conventional furnace (for 0.5 hr at 850°C) and continuous heating (with a heating rate of 20 °Cs⁻¹ to 970°C) (Fig. 1.3).

![Fig. 1.3: Microstructures of LCB-Ti after solid solution treatment via (a) conventional furnace and (b) continuous heating technique and water quenching [11].](image)

Microstructures after both of the above heat treatments exhibited a single β-phase with equiaxed β grains. The β grain size obtained by heating in a conventional furnace is in the range of 150 to 200 μm while the β grain size after continuous heating treatment is significantly smaller with an average grain size of 50 μm. Similar microstructures comprising 50 μm average β grain sizes were obtained in LCB-Ti after solid solution treatment by rapid heating at a rate of 20 °Cs⁻¹ to a peak temperature of 930°C (Fig. 1.4) [12].
In both experiments, the peak heating temperatures were above $\beta$-transus (800°C) in order to dissolve the large primary $\alpha$-grains. The very high peak heating temperature in [11] (970°C, Fig. 1.3b) was chosen due to the high volume fraction of primary $\alpha$-phase associated with its large grain sizes. The fact that the same average $\beta$-grain size was obtained in the two experiments for different peak heating temperatures (970°C and 930°C) can be attributed to the difference in the initial microstructures before solid solution treatment.

1.3.2 Cold deformation

1.3.2.1 Effect of cold deformation on microstructure

Cold deformation plays an important role in that it provides the crystalline defects and the stored energy to control the final $\beta$ grain size during subsequent recrystallisation. During annealing, the defects act as a source for nucleation while the stored energy in the material is the driving force for recrystallisation. It should also be noted here that cold deformation can also be used to modify the precipitation behavior in metastable $\beta$-Ti alloys in order to achieve high densities of fine $\alpha$ precipitates during ageing [13-15].

Cold deformation has a severe effect on the microstructure of metals. Firstly, the changes in grain morphology result in an increase in the total grain boundary area. Secondly, the appearance of an internal substructure comprising micro bands,
shear bands and subgrains within the grains is the result of the net of accumulation of dislocations.

Ohyama et al. [16] investigated the deformation microstructures of three β-Ti alloys including Ti-15V-3Cr-3Sn-3Al, Ti-16V-10Sn and Ti-16V-4Sn after solution treatment at 1000°C and cold rolling to either 5% or 60% thickness reduction. The microstructures of the three alloys after 5% thickness reduction are shown in Fig. 1.5.

![Microstructures of the 5% cold rolled materials](image)

**Fig. 1.5:** Microstructures of the 5% cold rolled materials: (a) Ti-15V-3Cr-3Sn-3Al, slip; (b) Ti-16V-10Sn, twinning; (c) Ti-16V-4Sn, martensitic transformation [16].

Fig. 1.5a shows the microstructure of Ti-15V-3Cr-3Sn-3Al that deformed via \{112\}\{111\} slip. Fig. 1.5b illustrates deformation twins formed in Ti-16V-10Sn such that the appearance of secondary twinning is associated with primary twinning. This behavior is characteristic of β-Ti alloys and has been reported previously in Ti-11.6 Mo with \(332\)[11\(\bar{3}\)] primary twins and \(112\)[1\(\bar{1}\)\(\bar{1}\)] secondary twins [17] and in Ti-11.5Mo-6Zr-4.5Sn with both primary and secondary twinning on \(332\)[11\(\bar{3}\)] [18]. Fig. 1.5c demonstrates banded structures with deformed grains aligned parallel to the macroscopic cold-rolling direction.

The microstructures of the three alloys after 60% cold rolling reduction are presented in Fig. 1.6. These microstructures comprise elongated grains (Fig. 1.6(a-c)), distorted twins (Fig. 1.6(b)) and deformation bands (Fig. 1.6(a-c)).
Singh et al. [19] studied the microstructural changes in LCB Ti-10V-4.5Fe-1.5Al after hot rolling at 750°C, solution solutioning at 825°C for 8h and water quenching (in order to obtain a single β-phase microstructure). The material was subsequently cold rolled to thickness reductions between 20 and 60%. From the microstructures of the specimens cold rolled to 20, 40 and 60% reduction (Fig. 1.7), it can be seen that with the greater amount of cold working, the grains are more elongated along the rolling direction. While twinning was not observed in the starting material, its activity increases with greater cold rolling reduction.

Ivasishin et al. [11] researched the precipitation and recrystallisation behavior of β-Ti alloys during continuous heat treatment. In that study, the microstructure of Ti-15-3 was revealed after β solid solutioning for 0.5 hr at 900°C and water quenching. The obtained β phase microstructure has an average grain size of ~200 µm. Thereafter, cold rolling was undertaken to thickness reductions of 20, 50, 70 and 90% (Fig. 1.8). The microstructure showed mostly β-phase grains with small
amounts of \( \omega \)-phase present at low reductions. In addition, the grains were continuously elongated along the rolling direction while their widths along the normal direction decreased with greater cold rolling thickness reduction.

\[ \text{Fig. 1.8: Microstructure of Ti-15-3 alloy after } \beta \text{ solution treatment and cold rolling to thickness reductions of (a) 20\%, (b) 50\%, (c) 70\% and (d) 90\% [11].} \]

The deformation mechanism in LCB-Ti alloys at room temperature, that is characterised by dislocation glide and the absence of twinning, and the deformation microstructures of LCB-Ti alloys cold worked at room temperature at strain rates from 0.01s\(^{-1}\) to 1s\(^{-1}\) were also revealed in [20]. In that study, the as-received specimen was annealed above the \( \beta \) transus temperature at 815\(^{\circ}\)C for 30 min then cooled at the rate of 5 \(^{\circ}\)Cs\(^{-1}\) to room temperature. The microstructure of the solution heat treated specimen exhibits equiaxed \( \beta \) grains with the appearance of some \( \omega \)-phase particles.

It should be noted to mention about the condition for the non-equilibrium \( \omega \)-phase can be formed either: (i) athermally during quenching via a diffusionless process or, (ii) isothermally via diffusional processes during ageing at temperatures below 400\(^{\circ}\)C. The appearance of the \( \omega \)-phase affects phase stability, microstructure and mechanical properties of the material during thermo-mechanical treatments [20].
The microstructure of LCB-Ti after cold rolling at a strain rate of 0.01 s$^{-1}$ to plastic strains of 0.06, 0.18, and 1.15 is shown in Fig. 1.9.

![Fig. 1.9: Microstructures of LCB-Ti after cold rolling at a strain rate of 0.01 s$^{-1}$ to the strains of (a) 0.06, (b) 0.18 and (c) 1.15 [20].](image)

Up to a strain of 0.06, the microstructure was characterised by single slip. At a strain of 0.18, most of the grains exhibited multiple slip. After the high strain to 1.15, the microstructure showed a non-uniform deformation with wavy patterned grains. The $\omega$-phase was revealed by TEM via diffraction for the various conditions. At a strain rate of 0.1 s$^{-1}$ and for plastic strain up to 0.16, the microstructure shows similar features to that after deformation at a strain rate of 0.01 s$^{-1}$ to 0.18 plastic strain with the appearance of multiple slip (Fig. 1.10).

![Fig. 1.10: Microstructures of LCB-Ti after cold rolling at a strain rate of 0.1 s$^{-1}$ to a strain of 0.16 [20].](image)

At a very high strain rate of 1.0 s$^{-1}$, the occurrence of inhomogeneous deformation associated with shear bands was shown (Fig. 1.11). A high degree of heterogeneous deformation was observable as some elongated grains contained in-grain shear bands while other grains without shear bands were equiaxed. TEM
examinations indicated that deformation accommodation was dominated by multiple slip inside the shear bands, whereas deformation in grains without shear bands was accommodated by planar and in some cases, double slip.

![Microstructures of LCB-Ti](image)

**Fig. 1.11:** Microstructures of LCB-Ti after cold rolling at a strain rate of 1s\(^{-1}\) to a strain of 0.26 in (a) elongated grains inside shear bands and (b) equiaxed grains outside the shear bands [20].

1.3.2.2 Texture of cold deformed β-Ti

Texture is defined as the distribution of preferred crystallographic orientations of a polycrystalline sample. If orientations within a sample are random, the sample does not have any preferred orientation or texture. It is known that the texture affects the level of anisotropy in mechanical properties; and especially the superelastic properties of a material.

To date, only limited research into the texture of β-Ti alloys has been undertaken. The investigations are summarised as follows.

Singh *et al.* [19] studied the texture changes in LCB-Ti Ti-10V-4.5Fe-1.5Al. This study found the orientation components shown in Table 1.4 in the material after β solution treatment at 825°C for 8h, water quenching and cold rolling to thickness reductions from 20% to 60%.
Table 1.4: Cold rolled texture components and their intensities for LCB-Ti Ti-10V-4.5Fe-1.5Al [19].

<table>
<thead>
<tr>
<th>Texture component</th>
<th>Euler angles</th>
<th>f(g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>φ₁</td>
<td>φ₂</td>
</tr>
<tr>
<td>(015)&lt;100&gt;</td>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td>(001)&lt;210&gt;</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>(112)&lt;110&gt;</td>
<td>0</td>
<td>30</td>
</tr>
<tr>
<td>(011)&lt;100&gt;</td>
<td>0</td>
<td>45</td>
</tr>
<tr>
<td>(001)&lt;130&gt;</td>
<td>45</td>
<td>0</td>
</tr>
<tr>
<td>(113)&lt;332&gt;</td>
<td>90</td>
<td>30</td>
</tr>
</tbody>
</table>

In the Table 1.4, the orientation component {001}(110) with its relatively high intensity \( f(g) = 15.5 \) has been overlooked. The authors also have concluded that the absence of the α and γ-fibers is probably due to the insufficient rolling reduction (up to only 60% of the original thickness) to develop these fibers (Fig. 1.13).

\[ \phi_2 = 0^\circ \text{ and } 45^\circ \text{ ODF section of Ti-10V-4.5Fe-1.5Al after } \beta \text{ solution treatment and cold rolling to } 60\% \text{ thickness reduction [19].} \]

Suwas et al. [22] investigated the evolution of the transformation texture in the same alloy Ti-10V-4.5Fe-1.5Al after processing via the following steps: casting and forging at 900°C in the β phase field to obtain an average grain size of 175 µm, further heat treatment at 900°C to achieve larger average grain sizes of 250 µm, followed by cold rolling to 80% thickness reduction through conventional and cross-rolling. The study revealed that a strong γ-fiber was observed in the large grain samples after both conventional and cross-rolling. At smaller grain sizes, the
\( \gamma \)-fiber disappeared in the conventionally rolled sample while the cross-rolled sample exhibited a weak \( \gamma \)-fiber (Fig. 1.14).

![Fig. 1.14: \( \phi_2 = 45^\circ \) ODF sections of Ti-10V-4.5Fe-1.5Al after \( \beta \) solution treatment and cold rolling to 80% thickness reduction for (a) conventional rolling, (b) cross-rolling with larger grain sizes and smaller grain sizes in order [22].](image)

Gurao \textit{et al.} [23] also conducted research on LCB Ti-10V-4.5Fe-1.5Al following a thermo-mechanical processing schedule similar to that described in [22]. Their results are approximately the same with the absence of the \( \alpha \)-fiber and the presence of a weak \( \gamma \)-fiber in the conventionally rolled sample and a strong \( \gamma \)-fiber in the cross-rolled sample.

Recently, shape memory alloys (SMAs) based on the \( \beta \)-Ti system have attracted much attention for biomedical applications due to their shape memory effect and superelasticity. For example, Wang \textit{et al.} [24] investigated the texture and superelastic behavior of cold-rolled TiNbTaZr alloy (Fig. 1.15), Kim \textit{et al.} [25] studied the texture and shape memory behavior of Ti–22Nb–6Ta alloy (Fig. 1.16), Inamura [26] conducted research into the effect of \{001\}(110) texture on the
superelastic strain of Ti–Nb–Al, Hosoda et al. [27] studied the effects of short heat treatments on the superelastic properties of Ti–Nb–Al. In all these studies [24-27], the ingot samples were homogenised at 1000°C for 7200 s, then severely cold rolled between 90% and 99% thickness reduction. The texture components obtained in these specimens comprised a well developed \{001\}\{110\} orientations and fairly weak \{112\}\{111\} orientation components; although the latter was not seen in [24].

**Fig. 1.15:** $\phi_2 = 45^\circ$ ODF sections of Ti-Nb-Ta-Zr alloy after homogenisation treatment and cold rolling to thickness reductions of 90% [24].

**Fig. 1.16:** $\phi_2 = 45^\circ$ ODF section of Ti–22Nb–6Ta alloy after homogenisation treatment and cold rolling to thickness reductions of (a) 90%, (b) 95%, (c) 99% [25].
1.3.3 Annealing

1.3.3.1 Background

During the annealing of cold deformed materials, the process of recovery, recrystallisation and grain growth are taking place concurrently.

- Recovery can happen either during deformation at high temperatures (dynamic recovery) or during annealing (static recovery). It is defined as the process by which deformed grains can reduce their stored energy via the removal and rearrangement of dislocations that accumulate during. The changes in microstructure during recovery are relatively inhomogeneous and do not influence the high-angle grain boundaries between deformed grains. The changes in mechanical properties are characterised by a decrease in strength (or softening) and an increase in ductility due to the reduction of dislocation densities through recovery.

- Discontinuous recrystallisation is described as the process by which new grains nucleate and grow to consume the originally deformed grains. It is different from recovery in that the microstructural changes are associated with the formation and migration of high angle grain boundaries and that these processes are driven by the stored energy of deformation and the temperature of annealing. A significant decrease in strength and an increase in ductility occur during recrystallisation; such that it is a generally more prominent effect in comparison to recovery.

- On the other hand, continuous recrystallisation occurs uniformly with a discernable nucleation stage. Recrystallisation may also occur either during deformation at high temperatures (dynamically) or during annealing after deformation (statically).

- Grain growth is known as the process in which an increase in grain size occurs via a curvature driven migration of high angle grain boundaries resulting in an overall decrease in boundary area. The difference between the recrystallisation and grain growth phenomena is the driving force. Whereas, the driving force for the recrystallisation process is the stored energy of dislocations introduced during deformation, that for grain growth is the energy of the high angle grain boundaries around their immediate vicinity. Abnormal grain growth is defined as that process
of secondary recrystallisation under which grain coarsening is inhomogeneous and a few isolated grains grow at the expense of the average recrystallised matrix.

![Image](image_url1)

**Fig. 1.17:** Microstructure schematics after: (a) deformation, (b) recovery, (c) partially recrystallisation, (d) full recrystallisation, (e) grain growth and (f) abnormal grain growth [28].

### 1.3.3.2 Microstructure after annealing

The microstructures of LCB Ti-10V-4.5Fe-1.5Al after β solution treatment at 825°C for 8h, water quenching and cold rolling to 60% thickness reductions and annealing for 2 hrs at 820°C and 860°C are given in Fig. 1.18 [19].

![Image](image_url2)

**Fig. 1.18:** Microstructures of Ti-10V-4.5Fe-1.5Al alloy after β solution treatment, cold rolling to 60% and annealing at (a) 820°C, (b) 860°C [19].
The fully recrystallised microstructure containing equiaxed grains with average sizes of 40 µm (820°C) and 75 µm (860°C), respectively, were found to have replaced the twinned and elongated grains after deformation. It was also to be noted that the higher annealing temperature resulted in a coarser microstructure. The microstructure of β-Ti VT22 after solid solutioning for 0.5 hr at 950°C, water quenching, cold rolling to 40% thickness reduction and continuous heating at the rates of 5 °C/s and 50 °C/s to peak temperatures ranging between 875°C and 1000°C was investigated in [11] (Fig. 1.19). The number of nuclei located at the boundaries of primary β grains, slip bands or martensite laths (arrows in Figs. 1.19(b-c)) was found to increase with greater cold rolling reduction and heating rate. Due to the small magnitude of cold rolling and the resultant non-uniformity of strain, some areas do not show evidence of the nucleation of new grains. Thus, in these regions, it was assumed that (sub)grain coalescence and continuous recrystallisation had occurred [29-31] (Fig. 1.19(f)).

The microstructures of β-Ti Ti-15-3 after β solution treatment for 0.5 hr at 900°C, cold rolling to a reduction of 70% and continuous heating at 5 °C/s to peak temperatures of 750°C and 850°C [11] are seen in Fig. 1.20. Nucleation events were shown to take place predominantly at the grain boundaries. Compared to VT22 which underwent 40% cold rolling thickness reduction, the annealed microstructure of Ti-15-3 after 70% cold rolling was finer and more uniform as a result of greater and more homogenous deformation.
Fig. 1.19: Microstructures of VT22 after β solution treatment, cold rolling to 40% thickness reduction (a) and continuous heating at the rates of (b, d, f) 5 °Cs⁻¹ or (c, e, g) 50 °Cs⁻¹ to peak temperatures of (b) 875°C, (c, d) 900°C, (e, f) 950°C and (g) 1000°C [11].

Fig. 1.20: Microstructures of Ti-15-3 after β solution treatment, cold rolling to 70% thickness reduction and continuous heating at the rate of 5 °Cs⁻¹ to peak temperatures of (a) 750°C and (b) 850°C [11].
Ivasishin et al. [12] worked on improving the microstructure of Ti-LCB by rapid heat treatment. The material was solution treated either in a conventional furnace at 820°C for 0.5 hr (to obtain a β grain size of 90 µm) or by rapid heating (to achieve a β grain size of 20 µm), followed by water quenching. Thereafter, the material was cold drawn to 50 and 70% thickness reduction. The cold drawn materials were then rapidly heated at 20 °Cs⁻¹ in order to produce a fully recrystallised microstructure. The resulting size of the fully recrystallised grains was found to be significantly dependent on the β grain size in the solid solution condition. Finer grain sizes were obtained after the recrystallisation of the rapid heated samples (Fig. 1.21). In these microstructures, the ω and α phase precipitates are absent due to the relatively fast heating rate. During continuous heating, the first nuclei were formed at the boundaries of the deformed β grains. The final grain size also depends on the amount of cold drawing. For example, with the same starting β grain size, cold drawing to 70% thickness reduction and then continuous heating at 20 °Cs⁻¹ resulted in a 8 µm average recrystallised grain size compared to the 14 µm grain size obtained after cold drawing to 50% thickness reduction and annealing at the same heating rate. The finer grain size in the 70% cold deformed sample was explained by the higher dislocation density resulting in the presence of more defect sites for preferential nucleation.

**Fig. 1.21:** Microstructures of Ti-LCB after rapid heating at the rate of 20 °Cs⁻¹ of the same samples after (a) furnace solid solutioning, 50% reduction, (b) solid solutioning by rapid heating, 50% reduction and (c) solid solutioning by rapid heating, 70% reduction [12].
Ivasishin et al. [11] also revealed the microstructures of LCB-Ti after β solid solution treatment 0.5 hr at 850°C, water quenching, cold drawing to 50% thickness reduction and rapid heating at the rate of 20 °Cs⁻¹ to peak temperatures of 800°C, 840°C and 870°C (Fig. 1.22). At 800°C, the material was partially recrystallised, whereas fully recrystallised microstructures were observed at 840°C and 870°C.

**Fig. 1.22:** Microstructures of Ti-LCB after β solution treatment, cold drawing to 50% thickness reduction and continuous heating at the rate of 20 °Cs⁻¹ to peak temperatures of (a) 800°C, (b) 840°C and 870°C [11].

**1.3.3.3 Effect of cold deformation on recrystallisation**

In β-Ti alloys, an increase in the cold working prior to annealing results in a decrease in the starting and finishing temperatures for recrystallisation (Figs. 1.23 and 1.24). This is usually explained by the higher stored energy accrued at higher cold deformation resulting in greater driving force and a lower activation energy for recrystallisation.
Recrystallisation start (closed symbols) and finish (opened symbols) temperature of LCB-Ti at heating rates of 5 °Cs⁻¹ and 20 °Cs⁻¹ [11].

Cold deformation also influences microstructure after annealing. First, a higher cold working results in a smaller β grain size after annealing (see Section 1.3.3 for details) (Fig. 1.25).
Fig. 1.25: The influence of cold (or hot) reduction on β grain size in recrystallised condition for three β-Ti alloys heated at the rate of 5 °Cs⁻¹ [11].

Secondly, the higher cold working produces a more uniform β grain size microstructure after annealing [12]. Alternatively, smaller cold rolling reductions result in the non-uniformity of strain such that the recrystallised microstructure is characterised by a distribution of coarse and fine grains. For example, β-Ti alloy VT22 after β solution treatment, cold rolling to 40% thickness reduction and continuous heating at the rate of 5 °Cs⁻¹ showed very coarse β grains distributed among finer β grains (Fig. 1.19 (f)).

1.3.3.4 Effect of initial β grain size after solid solution treatment on the final recrystallised microstructure

During the thermo-mechanical processing of β-Ti alloys, the β grain size after solid solution treatment significantly affects the final recrystallised grain size after cold deformation and heat treatment such that a smaller β grain size after solid solution treatment results in a smaller β grain size after annealing [12]. This can be attributed as follows. When deformed grain sizes are small, the distances between nucleated grains are also small. Thus, in the case of the finer deformed grain microstructure, the grain growth is restricted when the nucleated grains impinge against each other leading to smaller final β grain sizes. The above is highlighted in the later Table 1.5 such that the initial β grains after solid solution treatment with
grain sizes of 20 µm and 90 µm, cold rolling to 50% thickness reduction and continuous heating at the rate of 20 °C s⁻¹ resulted in a recrystallised β-phase microstructure with grain sizes of 12 µm and 30 µm, respectively. As mentioned previously, in order to obtain a small β grain size after solid solution treatment, rapid heating is the recommended treatment instead of heating in conventional furnaces [32-34].

1.3.3.5 Effect of heating rate in annealing on recrystallisation

In β-Ti alloys, annealing in conventional furnaces usually results in either inhomogeneous microstructures or large β grain sizes. For example, heating below the β-transus temperature (in order to obtain an α+β phase microstructure) causes anisotropy in the mechanical properties because of the residual α phase. On the other hand, heating above the β-transus temperature in order to receive a uniform β-phase microstructure results in the significant growth of β grains; thus leading to a drop in ductility [34]. Alternatively, rapid heating by resistance or induction heating can be employed in order to obtain a fine, uniform β microstructure after recrystallisation. When choosing the heating rate, it is important to understand its effect on the subsequent recrystallisation behavior of β-Ti alloys. For example, high heating rates during annealing restrict the phase transformation of β to ω and α phases [11]. At a heating rate of 5 °C s⁻¹, this transformation is totally eliminated in LCB-Ti and Ti-15-3 (Fig. 1.26).
Fig. 1.26: Precipitation of $\alpha$-phase in Ti-15-3 during heating at various rates for the undeformed condition (solid C curve) and after a thickness reduction of 70% (dashed C curve) [11].

In addition, high heating rates also increase the temperature for the start and finish of recrystallisation (Fig. 1.23).

Moreover, the heating rate also affects the $\beta$-transus temperature at which the transformation of $\alpha+\beta\rightarrow\beta$ occurs via the dissolution of primary $\alpha$ into the $\beta$ phase (Fig. 1.27). As seen from Fig. 1.27, an increase in the heating rate results in an increase of the $\beta$-transus temperature.

Fig. 1.27: Effect of heating rate on $\beta$-transus temperature [34].
In addition, the heating rate during annealing has a strong impact on the β grain size. Ivasishin et al. [34] compared the recrystallised average β grain size as a function of temperature in LCB-Ti at heating rates of 20 °Cs⁻¹ and 50 °Cs⁻¹ (Fig. 1.28).

![Fig. 1.28: Effect of heating rate on β grain size in relation with temperature [34].](image)

It was observed that below the β-transus temperature, the growth of the β grain size in both cases is similar due to the inhibition by the α-phase. However, at the temperatures above the β-transus temperature, due to the dissolution of the α-phase, the β grain size in the case of a higher heating rate 50 °Cs⁻¹ is much smaller than that in the case of 20 °Cs⁻¹ as there was not enough time for grain growth.

The disadvantage of high heating rates is that it also results in micro-chemical non-uniformity of the β-phase; leading to a heterogeneous microstructure and a large variation in mechanical properties [32]. Therefore, a heating rate of 20 °Cs⁻¹ is most commonly used for β-Ti (heating rates of 10 °Cs⁻¹ are less commonly employed as such rates can result in significant of β grain growth) [34].

1.3.3.6 Texture of β-Ti after annealing

Singh et al. [19] investigated the changes in the texture of a LCB-Ti Ti-10V-4.5Fe-1.5Al during annealing. The material was processed by β solution treatment at 825°C for 8 hrs, water quenching and cold rolling to 60% thickness reduction and annealing for 2 hrs at 820°C and 860°C in order to reach the fully recrystallised state. The resulting texture of the annealed specimen at 820°C shows the
{001}\langle110\rangle orientation components and a weak $\gamma$-fiber while that of the specimen annealed at 860°C displayed the complete {001} fiber (Fig. 1.29).

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{fig129.png}
\caption{$\phi_2 = 45^\circ$ ODF section of Ti-10V-4.5Fe-1.5Al after $\beta$ solution treatment, cold rolling to 60\% thickness reduction and annealing at (a) 820°C and (b) 860°C [19].}
\end{figure}

The authors supposed that the presence of a strong {111}\langle110\rangle component in $\gamma$-fiber is because of both, a high nucleation rate (due to the high Taylor factor) and the high growth rate (due to the favorable growth relationship with the {113}\langle110\rangle rolling component) of {111}\langle110\rangle component.

Suwas et al. [22] investigated the texture evolution in an LCB-Ti Ti-10V-4.5Fe-1.5Al alloy after heat treatment in the $\beta$-phase field, cold rolling to 80\% thickness reduction conventionally and via cross-rolling and annealing to 800°C for 1hr to obtain fully recrystallised microstructures. The resulting recrystallised texture did not change significantly from that of the original deformation texture (Fig. 1.30).
Fig. 1.30: $\phi_2 = 45^\circ$ ODF section of Ti-10V-4.5Fe-1.5Al after $\beta$ solution treatment, cold rolling to 80% thickness reduction and annealing at 800$^\circ$C for (a) conventional rolling, (b) cross-rolling with larger grain sizes (left) and smaller grain sizes (right) in order [22].

The recrystallised texture was characterised by the weakening of the $\gamma$-fiber in the conventionally rolled and cross-rolled samples; with less intensity $(f(g))$ recorded in the former compared to the later. The weakening of the $\gamma$-fiber was explained such that the $\gamma$-fiber has a higher tendency to recrystallise due to its high Taylor factor in terms of stored energy. The severe weakening in intensity of the $\gamma$-fiber in the conventionally rolled sample was due to its higher stored energy in comparison to the cross-rolled sample.

Gurao et al. [23] also undertook research on an LCB Ti-10V-4.5Fe-1.5Al alloy using thermo-mechanical processing similar to [22]. However, the resulting recrystallised texture was different from that in [22]. In this case, the recrystallised texture exhibited an increase in strength of the $\gamma$-fiber. However, the overall changes in texture were similar to the recrystallised texture of Ti-10V-4.5Fe-1.5Al obtained after 60% cold rolling and annealing at 860$^\circ$C and reported in [19]. The $\gamma$-fiber strengthening effect was attributed to the higher cold rolling thickness
reduction (from 60% to 80%) and a resultant decrease in the recrystallisation temperature.

Recently, there have been a number of publications that revealed the recrystallisation texture in shape memory alloys based on the β-Ti system by Kim et al. [25] (Ti–22Nb–6Ta alloy), Inamura [26] (Ti–Nb–Al), Hosoda et al. [27] (Ti–Nb–Al). In these studies, materials were homogenised at 1000°C for 7200 s, severely cold rolled between 90% and 99% thickness reduction and annealed at temperatures ranging from 600°C to 1000°C for varying times between 60s and 3600s (Fig. 1.31).

![Fig. 1.31](image)

**Fig. 1.31:** $\phi_2 = 45^\circ$ ODF section of Ti–22Nb–6Ta alloy after heat treating at 900°C with the heating rate of 1.8 °Cs$^{-1}$ with prior cold rolling to thickness reductions of (a) 90%, (b) 95%, (c) 99% [25].

The recrystallisation texture components obtained in these studies comprised the \{112\}(110) component located on the α-fiber (Fig. 1.31). This type of recrystallisation texture was very different from that obtained in studies on LCB-Ti [19], [22] and [23] in which the α-fiber was absent in the annealed material.

**1.3.4 Ageing treatment**

Ageing heat treatments are used to improve the mechanical strength of β-Ti alloys. The increase in strength after ageing is due to the precipitation of the α-phase which is distributed uniformly throughout the β matrix. In this case, the volume fraction and morphology of the α precipitates are the key factors that impact the
strength of these alloys. In general, a uniform microstructure with small β grain sizes after recrystallisation produces a fine and uniform α+β microstructure after ageing; thus resulting in the optimised balance between strength and ductility [35].

Ivasishin et al. [36] investigated the effect of grain size after recrystallisation, the effect of heating rate to peak ageing temperature and the effect of peak ageing temperature on microstructure of three β-Ti alloys namely, LCB-Ti, Ti-15-3 and VT22. In this study, these alloys were β solution treated and water quenched to obtain coarse β grain with an average grain size of 200 μm. Moreover, these alloys also were treated via thermo-mechanical processing comprising β solution treatment, deformation (cold drawing for LCB-Ti, cold rolling for Ti-15-3 and hot rolling for VT22) and continuous heating to receive fine β grains with average size of 10 μm (for LCB-Ti and Ti-15-3) or 50 μm (for VT22). Thereafter, the ageing treatment was applied on these alloys with various heating rates (between 0.01 and 20 °Cs⁻¹) to peak ageing temperatures ranging between 500°C and 600°C. The result of this study indicated that with the exception of LCB-Ti, both Ti-15-3 and VT22 showed more ageing response in the fine grained condition (Fig. 1.32).

Fig. 1.32: Precipitation hardening kinetics at 538°C for coarse β grains (closed symbols) and fine β grains (open symbols) conditions of VT22, LCB-Ti and Ti-15-3 alloys at the heating rate to peak ageing temperature of 0.25 °Cs⁻¹ [36].
Slow heating rates to peak ageing temperature result in a fine $\alpha$ precipitation which increases mechanical strength. In the case of VT22 and LCB-Ti, isothermal $\omega$ precipitation was formed at a slow heating rate of 0.25 °Cs$^{-1}$; which acted as nucleation sites for $\alpha$ precipitation (Figs. 1.33(a, c)). However, at a higher heating rate of 20 °Cs$^{-1}$, the $\omega$ precipitation was avoided; which lead to the precipitation of coarse plate-type $\alpha$ phase (Figs. 1.33(b, d)).

Ti-15-3 exhibited $\beta$-phase separation rather than the formation of isothermal $\omega$ precipitation during ageing. At the heating rate of 0.25 °Cs$^{-1}$, there was insufficient time for the separation of the $\beta$-phase to develop prior precipitation. In this case, the non-uniformity of precipitation occurred due to its much slower response to the formation of fine $\alpha$ precipitation compared to the two other alloys (Fig. 1.34).

![Microstructures of LCB-Ti after recrystallisation and ageing at 538°C for 2 h at the heating rate to ageing temperature of (a, c) 0.25 °Cs$^{-1}$, (b, d) 20 °Cs$^{-1}$. (a, b) TEM micrographs and (c, d) optical micrographs [36].](image-url)
Ivasishin et al. [35] revealed the effect of ageing temperature on the microstructure of LCB-Ti. At high ageing temperatures, coarse $\alpha$ precipitation was formed resulting in a decrease in strength. For example, when ageing temperature was increased from 520°C to 560°C, coarse $\alpha$ laths appeared in the LCB-Ti (Fig. 1.35).

**Fig. 1.34:** Microstructure of Ti-15-3 after recrystallisation and ageing at 538°C for 4h (SEM micrograph) [36].

**Fig. 1.35:** Microstructures of LCB-Ti after recrystallisation and ageing for 8 h at (a, b) 520°C, (c) 538°C, (d) 560°C. (TEM micrographs) [35].
1.4 Mechanical properties of β-Ti alloys

Ivasishin et al. [35] investigated the mechanical properties of four high-strength β-Ti alloys, namely Ti-LCB, Ti-15-3, β-21S and VT22. These alloys were β solution treated for 0.5h at 950°C (VT22), 850°C (Ti-LCB) and 900°C (Ti-15-3 and β-21S), then cold rolled (VT22 and Ti-15-3) or cold drawn (Ti-LCB and β-21S) to thickness reductions from 30 to 80% and annealed at heating rates of 10 or 20 °Cs⁻¹ to peak temperatures between 815°C and 950°C in order to obtain fully recrystallised microstructures (Table 1.5).

Table 1.5: Thermo-mechanical processing, grain size and mechanical properties of β-Ti alloys after recrystallisation [35].

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cold deformation (%)</th>
<th>Continuous heating</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Heating rate (°Cs⁻¹)</td>
<td>Recrystallisation temperature (°C)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>β grain size (µm)</td>
</tr>
<tr>
<td>VT22</td>
<td>30</td>
<td>20</td>
<td>950</td>
</tr>
<tr>
<td>Ti-15-3</td>
<td>70</td>
<td>10</td>
<td>860</td>
</tr>
<tr>
<td>β-21S</td>
<td>80</td>
<td>10</td>
<td>880</td>
</tr>
<tr>
<td>LCB-Ti</td>
<td>70</td>
<td>20</td>
<td>815</td>
</tr>
</tbody>
</table>

It can be seen that VT22 showed the lowest ductility and LCB-Ti presented with high strength and reasonable ductility. With specific regard to LCB-Ti a good combination of strength and ductility can be achieved when its average recrystallised grain sizes ranges between 10 and 50µm.

It is known that the mechanical properties of β-Ti alloys can be improved; resulting in an increase in mechanical strength by downstream ageing treatments. An increase in strength after ageing is due to the precipitation of the α-phase which is uniformly distributed throughout the β matrix. After ageing, the volume fraction and morphology of the α precipitates impact on the strength of β-Ti alloys. Ivasishin et al. [12] characterised the mechanical properties of coil LCB-Ti that was previously solution treated either in a conventional furnace at 820°C for 0.5 hr (in
order to obtain a β grain size of 90 µm) or by rapid heating (in order to achieve a β grain size of 20 µm), then water quenched, followed by cold drawing to the reductions of 50 and 70% and rapid heating at 20 °C s⁻¹ and finally ageing. Processing parameters and mechanical properties of the samples are summarised in Table 1.6. It is found that the smaller β grain size results in higher strength and ductility. It was also revealed that the higher cold drawing reduction resulted in a uniform microstructure which facilitated the increase in strength and ductility.

**Table 1.6:** Mechanical properties of coil LCB-Ti in different conditions of thermo-mechanical processing [12].

<table>
<thead>
<tr>
<th>Solid solution</th>
<th>Reduction</th>
<th>Recrystallisation</th>
<th>β grain size (µm)</th>
<th>Ageing</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>RA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furnace</td>
<td>50</td>
<td>Rapid heating</td>
<td>30</td>
<td>538°C, 8h</td>
<td>1395</td>
<td>1425</td>
<td>24</td>
</tr>
<tr>
<td>Rapid heating</td>
<td>50</td>
<td>Rapid heating</td>
<td>12</td>
<td>538°C, 8h</td>
<td>1420</td>
<td>1470</td>
<td>22.5</td>
</tr>
<tr>
<td>Rapid heating</td>
<td>70</td>
<td>Rapid heating</td>
<td>7</td>
<td>538°C, 8h</td>
<td>1475</td>
<td>1490</td>
<td>25.5</td>
</tr>
<tr>
<td>Rapid heating</td>
<td>70</td>
<td>Rapid heating</td>
<td>7</td>
<td>520°C, 8h</td>
<td>1600</td>
<td>1625</td>
<td>20.0</td>
</tr>
<tr>
<td>Rapid heating</td>
<td>70</td>
<td>Rapid heating</td>
<td>7</td>
<td>500°C, 8h</td>
<td>1615</td>
<td>1690</td>
<td>13.5</td>
</tr>
</tbody>
</table>

In summary, a uniform microstructure with small β grain sizes after recrystallisation produces a fine and uniform α+β microstructure after ageing; thus resulting in the balance of strength and ductility [35]. The mechanical properties of the four alloys in [35] for each condition namely recrystallisation, recovery and ageing are summarised in Fig. 1.36.
In this figure, the area surrounded by the dashed lines shows the points with UTS ≥ 1500 MPa and elongation ≥ 8% [35].

1.5 Hall-Petch relationship

In general, reducing the grain size improves the strength of a material due to the increase in the grain boundary area which hinders the movement of dislocations. In this regard, Hall-Petch relationship originally found in the 1950s can be used to estimate tensile yield strength based on grain size [37, 38]:

\[
\sigma_y = \sigma_0 + k_{HP}d^{-1/2}
\]  

(1.1)

where, \(\sigma_y\) tensile yield strength, \(\sigma_0\) is the friction stress described as the internal resistance to the motion of a dislocation through the crystal lattice and is defined approximately as yield strength of a coarse, non-textured polycrystalline material, \(k_{HP}\) is the constant related to the strength of grain boundaries.

Eq. (1.1) is applicable to fully recrystallised microstructures which comprise mostly high angle grain boundaries (HAGBs). However, as a result of deformed or
partially recrystallised microstructures containing large amounts of low angle grain boundaries, the Eq. (1.1) needs to be modified in order to account for the effect of subgrain boundary strengthening. Thus, the modified Hall-Petch equation in [39, 40], which has been applied to severely deformed materials, can be used to estimate the strength of partially recrystallised microstructures:

\[
\sigma_y = \sigma_0 + \sigma_{LAGBs} + \sigma_{HAGBs}
\]

\[
\sigma_y = \sigma_0 + M \alpha b \sqrt{1.5 S_v \theta_{LAGB} (1 - f_{HAGB})} + k \sqrt{f_{HAGB} \over d_{HAGB}}
\]  

(1.2)

where, \( \sigma_{LAGBs} \) and \( \sigma_{HAGBs} \) are the strengthening of contributions subgrain and grain boundaries, respectively, \( M \) is the average Taylor factor, \( \alpha \) is a constant, \( b \) is Burger vector, \( S_v \) is the boundary area per unit volume of LAGBs, \( k \) is the constant representing the strength of HAGBs, \( f_{HAGB} \) is the fraction of high angle grain boundaries, \( \theta_{LAGB} \) is the average misorientation of low angle grain boundaries and \( d_{HAGB} \) is the grain size.

1.6 Summary

From the literature review, the effects of cold deformation and heating rate on the microstructure and texture in \( \beta \)-Ti alloys have been discussed. The influence of grain size and ageing parameters on the microstructure and mechanical properties of LCB-Ti was revealed. However, recrystallisation behavior of \( \beta \)-Ti after heavily cold working has not been studied to date. Cold deformation and recrystallisation textures have been calculated but different studies have reported contradictory data. Consequently, the evolution of texture in LCB-Ti remains ambiguous. Therefore, the aim of this study is two-fold: (i) to investigate the evolution of microstructure and texture of LCB-Ti alloy after \( \beta \) solid solution treatment, cold deformation and rapid heating and, (ii) to evaluate the effects of cold deformation and heating rate on the microstructure and texture after annealing.
Chapter 2

Experimental procedure

2.1 Material and processing

LCB-Ti a nominal composition of Ti-1.52Al-6.78Mo-4.44Fe-0.150 (in wt.%) was melted and initially processed by the TIMET Corporation, USA and then hot rolled into ø54 mm diameter rods by the Perriman Company, USA.

In order to negate the influence of preliminary history, the rod was solid solution treated at 850 °C for 1800 s (above the β transus of 790 °C) followed by water quenching. Consequently, a single phase, metastable β microstructure with an average β-grain size of 190±10 µm was obtained.

The quenched rods were cold drawn at room temperature to 80% and 90% reductions in cross-section which resulted in final diameters of ø10.8 mm and ø5.4 mm, respectively. For the purposes of this study, the samples will hereafter be referred to CD80 and CD90, respectively.

The cold drawn rods were then cut into 60 mm long samples and rapidly heat treated via resistance heating to peak temperature as follows. The CD80 samples were rapid resistance heated to peak temperatures of 780, 815 and 840 °C while the CD90 samples were rapidly heated to peak temperatures of 710, 720, 730, 750 and 820 °C. In order to evaluate the effect of imparted strain on the recrystallisation kinetics, both CD80 and CD90 conditions were rapidly heated at a constant heating rate of 20 °Cs⁻¹ (Fig. 2.1). Once again, after reaching each chosen peak temperature, the samples were immediately water quenched.

The above TMP was carried out at the Institute for Metal Physics National Academy of Sciences, Ukraine.
After annealing process, central parts of those specimens were wire-cut along their axial length, rough ground up to 15µm silica paper, fine polished using 25ml OP-S colloidal silica + 2ml H₂O₂ + 3ml NH₃ solution and electropolished on a Lectropol-5 with a Struers A3 electrolyte (600ml methanol, 360ml butoxyethanol, 60ml perchloric acid and 2ml Vogel’s sparbeize) at 50V, ~1.2mA for 20 seconds. Finally, the electropolished surface was passivated using Kroll’s reagent (3ml HF + 6ml HNO₃ + 100ml distilled water). These samples were used for X-ray diffraction (XRD) and Electron Back-Scattering Diffraction (EBSD) studies.

**2.3 Electron back-scattering diffraction**

EBSD is a standard technique to examine the microstructure and micro-texture of crystalline materials. The technique provides a range of microstructural information such as misorientation, grain size, grain boundary statistics, recrystallised/deformed fraction and phase identification. The technique can also be employed to define the preferred orientation (texture) of polycrystalline materials.
EBSD is carried out on a Scanning Electron Microscope (SEM) equipped with a phosphor screen, a charge-coupled device (CCD) camera and a computer which used to control the scanning system. The measurement process is briefly described as follows. A primary electron beam is accelerated to raster across the finely polished surface of the specimen which is tilted at 70° relative to the normal of the primary electron beam. The diffracted electrons creating the pattern of Kikuchi lines are captured on the phosphor screen and digitized via image processing to index the EBSD pattern.

**Fig. 2.2:** A schematic of the EBSD measurement process [41].

The microstructures of the cold drawn and rapidly heated samples were characterised by EBSD. The EBSD mapping was conducted at the center of the cylindrical cross-section using a JEOL 7001F Field Emission Gun – Scanning Electron Microscope (FEG-SEM) fitted with a Nordlys-II™ EBSD detector operating at 15kV, 5nA, 15mm working distance, 186.5 mm detector distance. Depending on the sample condition, the step size was varied between 0.15 and 0.35 μm.

Initial post processing of the raw data consisted of removing misindexes and wild spikes and addressing pseudo-symmetry issues using the HKL – Channel 5™ software package. Low-angle boundaries (LAGBs) comprise misorientations between 2° and 15° while high-angle boundaries (HAGBs) extend from 15° onwards. The average internal misorientation (IM) cut-off angle was calculated via...
the cumulative distribution of the grain orientation spread (GOS) criterion [42]. Thereafter an in-house developed methodology [21] was applied to deconstruct the EBSD map into four end subsets: (i) deformed, (ii) recovered, (iii) newly nucleated, and (iv) growing grains fractions. In order to define these four subsets, the grain orientation spread (GOS) criterion was first applied to calculate the critical internal misorientation ($\theta_C$) of the entire EBSD map and thereafter, to divide the map into three initial units comprising: (i) structures with GOS angle over $\theta_C$, (ii) structures with GOS angle under $\theta_C$ but surrounded by features that have a GOS angle above $\theta_C$ and (iii) structures with GOS angle under $\theta_C$. Units (i) and (iii) clearly belong to the unrecrystallised and recrystallised fractions, respectively. On the other hand, unit (ii) still comprises possible recrystallised structures surrounded by a highly unrecrystallised matrix. To extract the recrystallised structures from unit (ii), a threshold was applied via an aspect ratio criterion with a maximum equal to the arithmetic mean plus the standard deviation of the grains of the recrystallised subset. Following this, the average internal misorientation criterion was re-applied to all the (sub)grains of this fraction with a maximum limit equal to $\theta_C$. The substructures that fulfilled both of the above criteria were then added to the recrystallised fraction, while the remainder was regarded as belonging to the unrecrystallised fraction. Thereafter, if the cut-off criterion is applied to the normalised cumulative distribution of the unrecrystallised (sub)grains, it can be subdivided into recovered (slope < 1) and deformed (slope = 1) fractions. The recovered grains are morphologically similar to deformed grains in terms of their sizes and aspect ratios but have lower internal misorientation angles. Newly nucleated grains (which are a subset of the recrystallised/softened fraction) are defined as the microscopic features with: (i) a lower limit of equivalent circle diameter (ECD) equal to three times the step size; (ii) an upper limit of ECD equal to the mean of the log-normal frequency distribution and; (iii) at least one HAGB. Consequently, growing grains comprise softened structures with sizes between the mean and maximum of the log-normal frequency distribution.
Fig. 2.3: Schematic diagram illustrating the approach used to deconstruct the EBSD maps of the partially recrystallised conditions.

2.4 Bulk texture calculation

The process to obtain ODFs is described in brief in Fig. 2.4. First, pole figure, background and defocusing scans were conducted to obtain raw intensity data, background intensity data and defocusing function, respectively. Afterward, the raw intensity data from pole figure scanning was corrected by taking into account the background and defocusing errors in order to obtain corrected intensity data. Following this, the corrected intensity data was calculated by using Resmat software to obtain ODFs of bulk texture. Finally, these ODFs were plotted with high quality images by using Matlab software.
**Fig. 2.4:** Schematic diagram showing procedure for data acquisition and calculation of bulk texture ODFs.

### 2.4.1 Pole figure measurement

In the present study, the pole figure scanning was conducted on central circle of steady tilting angle ($\alpha$). The values of $\alpha$ were in the range between $0^\circ$ and $85^\circ$ and those of $\beta$ were ranging from $0^\circ$ to $360^\circ$ with scanning steps of $5^\circ$ for both $\alpha$ and $\beta$. Thus, $I_{\text{measured}}(\alpha, \beta)$ was obtained by scanning ($\alpha \times \beta$) = $18 \times 73 = 1314$ points on an incomplete pole figure. The Fig. 2.5 demonstrates how the reflected intensities can be determined via either: continuous (Fig. 2.5(a)) or discrete (Fig. 2.5(b)) scanning methodologies.

Pole figure scanning was conducted on a PANalytical Xpert–PRO MRD goniometer with a Cu tube at 40 kV and 45 mA and the window size of the X-ray beam was $2 \times 2$ mm$^2$. The three incomplete pole figures ($\alpha=0^\circ$-$85^\circ$) $\{1 1 0\}$, $\{2 0 0\}$ and $\{2 1 1\}$ were obtained for selected samples of this study.
During the process of pole figure scanning, there are several errors that change the intensities of the reflected peaks such as background, defocusing, absorption and irradiated volume errors. In the present study, the correction of absorption errors is unnecessary as the studied samples are thicker than the penetration depth of the X-rays. On the other hand, background intensities can be produced by unsuitable wavelengths of monochromatic X-rays, the interference of materials that are in the path of the X-rays between the tube, sample and detector [43]. This problem can be eased by choosing proper wavelengths of monochromatic X-rays or eliminating the air and electronic noise in the working area [44]. However, the largest contributors to the background error are incoherent scattering and fluorescence. This drawback can be reduced by using a suitable tube with wavelength higher than the K-absorption edge of the specimen. Choosing smaller receiving slits is another way to reduce the background error. However, this increases the defocusing error that will be discussed later. In fact, even though the experimental procedure is setup appropriately with suitable parameters that reduce background intensities, this error cannot be removed completely. Therefore, it is necessary to carry out a correction that subtracts the background intensities from the measured ones. To correct the pole figure data, Eq. (2.1) is applied [43]:
$I_{corr} = I_{measured}(\alpha, \beta) - BG(\alpha)$

where, $I_{measured}(\alpha, \beta)$ is the intensities obtained from measuring a pole figure, $BG(\alpha)$ is the background intensities.

While $I_{measured}(\alpha, \beta)$ depends on both $\alpha$ and $\beta$ angle, $BG(\alpha)$ only depends on the $\alpha$ angle such that at a constant value of $\alpha$, the background intensities are stable. The background intensities were measured on both sides of the Bragg peaks and were then averaged to obtain the appropriate values [43].

Regarding the defocusing error which causes the decrease in measured intensities of the reflected peaks at the receiving slit, there are many causes that result in this error such as the roughness of sample surface, inaccurate location of a sample in the goniometer or incorrect alignment of the goniometer itself [45]. These causes can be reduced by undertaking good sample preparation, regular goniometer alignment and correct sample adjustment. However, the main cause that contributes the most to this error is the change of the tilt angle $\alpha$ during pole figure scanning [45]. When $\alpha$ increases from 0° to 85°, the area that the incident beam covers on the surface of a sample elongates gradually. Moreover, and as shown in Fig. 2.6, this area is also distorted into different shapes at various Bragg angles ($\theta$).

![Fig. 2.6: Visualisation of the change in shape of the irradiated area at different values of tilting angle (\(\alpha\)) and Bragg angle (\(\theta\)) [43].](image)

As a result, the intensities of the reflected peaks are enlarged but the entire reflected beam cannot be captured by detector through the receiving slits. Thus,
the obtained intensities are lower than the real intensities when $\alpha$ is above $0^\circ$ (Fig. 2.7) [43].

![Diagram](image)

**Fig. 2.7**: (a) The widening of the inflected peak as tilting angle $\alpha$ increases, (b) Visualisation showing the defocusing error [43].

The defocusing error due to the changes of in tilt angle ($\alpha$) and Bragg angle ($\theta$) cannot be eliminated during pole figure scanning. Also, this error due to the goniometer alignment and sample setting cannot be totally removed. However, this error can be corrected for after the scanning is completed. The theoretical correction method is impossible because it is too complicated to establish and apply a theoretical correction applied for any case. The conventional method for the correction of defocusing error is using the correction function ($U(\alpha)$) created from an un-textured (random) powder sample. This function shows the dependence of normalised intensity ($I/I_{\alpha=0^\circ}$) on the change in tilt angle ($\alpha$) (in the
range between $0^\circ$ and $85^\circ$) at each value of Bragg angle ($\theta$). Following this, the measured intensities are corrected using Eq. (2.2) [43]:

$$I_{\text{corr}} = \frac{I_{\text{meas}}(\alpha, \beta) - BG(\alpha)}{U(\alpha)}$$

(2.2)

However, preparing a powder sample free of texture is a difficult and time-consuming process. In addition, the defects and phases in the powder microstructures influence the shape of the inflected peaks. As a result, the preparation of a random sample is an even more intricate and complicated process because microstructure and alloy make-up of the random sample should be similar to that of the investigated sample. In addition to the defocusing error, irradiated volume errors could also occur when the tilting angle ($\alpha$) changes during pole figure scanning. Due to the effect of the irradiated volume, the reflected intensities increase as tilting angle $\alpha$ progresses towards higher values.

Therefore, it is crucial to consider the combined effect of these two errors (defocusing and irradiated) when $\alpha$ changes. Liu et al. [46] introduced the defocusing correction method using interpolation (or extrapolation) from obtained data of available Al and Cu powder random samples. In this study, the total effect of defocusing and irradiated volume was found to be similar to the effect of the defocusing error (Fig. 2.8).

**Fig. 2.8:** The changes in intensity due to defocusing and irradiated volume effect in NiTi film [46].

In [46], the relationship between the normalised intensity and the Bragg angle ($\theta$) was recorded at different values of tilting angle ($\alpha$) for angular step of $5^\circ$ in the
range from 0° to 80°. Thereafter, the linear relationship between normalised intensity and the Bragg angle (θ) was found at constant tilting angles α via polynomial fitting functions (Fig. 2.9).

![Fig. 2.9: The dependence of defocusing error on Bragg angle θ at (a) α = 60°, (b) α = 75° [46].](image)

By this method, the correction function \( U(\alpha) \) can be determined without the need for a random sample of the same studied material. However, in a major limitation of the above technique, the linear relationship for \( U(\alpha) \) only covers the value of θ in the range between 0° to approximately 45°. In the case of material with peak Bragg angle (θ) beyond 45°, this defocusing correction cannot be applied.

In the present work, a more robust defocusing correction method was developed in-house by increasing the range of θ values. Five existing random samples of α-Ti, Cu, Al, austenitic stainless steel and ferritic stainless steel were used for pole figure scanning at various values of Bragg angle (θ) ranging between 15° and 60°. Afterward, for each θ-angle, the normalised intensities \( (I/I_{\alpha=0}) \) or defocusing correction factor at different values of tilting angle α from 0° to 85° were calculated. Following this, the linear relationships between normalised intensity and angle θ at different values of α from 0° to 85° were determined by fitting the data. For example, the Fig. 2.10 shows the linear fit obtained between the normalised intensity and Bragg angle (θ) with respect to tilting angle α = 0°, 75°,
80° and 85°.

By using the linear fit, the defocusing correction factors of β-Ti alloy were calculated at the three Bragg angles of 19.4°, 28° and 35.1° which correspond to the \{110\}, \{200\} and \{211\} planes, respectively (Fig. 2.11).

**Fig. 2.10:** The dependence of defocusing correction factor on Bragg angle $\theta$ at $\alpha = 0°, 75°, 80°$ and $85°$.

**Fig. 2.11:** The dependence of defocusing correction factor on tilting angle $\alpha$ in β-Ti alloy.
After background and defocusing correction, the samples of the studied material LCB-Ti alloy were scanned to obtain their incomplete pole figures ($\alpha = 0^\circ$ to $85^\circ$). ODFs were determined from these incomplete pole figures. Following this, the complete pole figures were calculated from the ODF. In this study, ODFs of various LCB-Ti samples have been plotted using programs written by the Matlab software package (Fig. 2.4).

2.5 Microhardness test

The Vickers microhardness of CD90 and CD80 samples were measured using an automated Leco M-400-H1 tester with an approximately 2N (200g) load for 10 s. Ten random microhardness measurements were made on the polished surface of each sample and averaged for each sample condition.
Chapter 3

Results

3.1 Effects of cold deformation

3.1.1 Hardness and bulk stored energy

During annealing, the decrease in hardness is due to the combined softening effects of recovery and recrystallisation. With respect to this study, the relationship between the change in hardness and the rapid resistance heating peak temperature at the heating rate of 20 °C·s⁻¹ for the CD80 and CD90 samples is illustrated in Fig. 3.1(a). In general, the small decrease in hardness during the first stage is usually correlated with recovery while the significant drop in hardness during the second stage is ascribed to recrystallisation. The fraction softened \( S_f \) can be estimated from the hardness using the relation:

\[
S_f = \frac{H_R - H_T}{H_R - H_0}
\]

(3.1)

where, \( H_R \) is the hardness after cold drawing, \( H_T \) is the instantaneous hardness at temperature \( T \) and \( H_0 \) is the hardness of the recrystallised, coarse grained, water quenched material. Alternatively, if it is assumed that boundary misorientation is shared between the various subsets of the deconstructed EBSD maps, the total fraction softened can be estimated via the equation [47]:

\[
S_f = \sum A_f \left[ \frac{HAGB_f - HAGB_{def}}{HAGB_{gg} - HAGB_{def}} \right]
\]

(3.2)

where, \( A_f \) and \( HAGB_f \) denote the map area fractions and HAGB percentages for each of the four subsets and \( HAGB_{gg} \) and \( HAGB_{def} \) denote the HAGB percentages for the growing grain and deformed subsets, respectively. The values of the fraction softened using Eqs. (3.1 and 3.2) as well as the EBSD map area fraction of the recrystallised microstructure for the CD80 and CD90 samples are presented in
Figs. 3.1(b, c). While the softened fraction estimates from Eqs. (3.1 and 3.2) are close, a large disparity between these two trends and the EBSD -returned recrystallised area fraction is noted especially at 780 and 815 °C for CD80 (Fig. 3.1(b)) and 720 and 750 °C for CD90 (Fig. 3.1(c)), respectively. On the other hand, the values from all three estimates are comparable at 840 °C (CD80) and 820 °C (CD90). If Eq. (3.2) is reapplied without accounting for the contribution from the recovered subset, values close those from the EBSD map area fraction are obtained.

The above underlines the predominant influence of recovery during the early stages of rapid heating. To further emphasise this point, the map area fraction recrystallised is plotted against the fraction softened estimated from Eq. (3.1) in Fig. 3.1(d). The black line in the figure represents the theoretical limiting case where the map area fraction recrystallised is equal to the fraction softened. The trends from both the CD80 and CD90 samples show that for a majority of the rapid heating treatment, recrystallisation (or the replacement of the original deformation microstructure) severely lags softening (or processes that comprise recrystallisation and more predominantly, the clean-up of the internal defects induced during cold drawing). Moreover, this variation between the two competing processes of recrystallisation and softening becomes even more apparent with the higher imparted strain associated with greater cold drawing reduction (see the difference in the shape of the curves between CD80 and CD90 in Fig. 3.1(d)).

1 Alternatively, Eq. (3.2) can also be used along the abscissa if it is applied using all four subsets (see Eq. (3.2) and Figs. 3.1(b and c)).
Fig. 3.1: Dependence of (a) Vickers hardness (VHN) on the rapid heating peak temperature. The comparison between the fraction softened or the map area fraction recrystallised and the correlations obtained using Eqs. (3.1 and 3.2) for (b) CD80 and (c) CD90 LCB-Ti. (d) The rate of change between the EBSD map area fraction recrystallised versus the fraction softened estimated from hardness data (Eq. (3.1)). (e) The relation between stored energy calculated using Eq. (3.3) for CD80 and CD90 LCB-Ti.
Since the disparity in the map area fraction recrystallised estimate can be attributed to: (i) the methodology of deconstruction, and (ii) the fact that the EBSD maps do not account for the density of statistically stored dislocations, they cannot provide information related to the overall softening behaviour during rapid heating and are primarily restricted to making distinctions between recrystallised and unrecrystallised microstructures only. Consequently, sections of this work related to describing the evolution of various aspects of the partially recrystallised microstructures use the map area fraction recrystallised estimate along the abscissa. To ensure brevity, the partially recrystallised samples are referred to by the magnitude of cold drawing reduction followed by their estimated map area fraction recrystallised in brackets. For example, the CD80 sample with 6.6% of its map area fraction recrystallised is denoted as CD80(0.07).

The bulk stored energy \( (E_u) \) can be approximated from the total dislocation density \( (\rho) \) and the line energy of dislocations \( (E) \). In this case, the Taylor equation relation to the hardness and dislocation density was applied such that [48]:

\[
E_u = \rho E = \frac{1}{9} \left( \frac{H_x - H_o}{M \alpha G b} \right) \left( \frac{Gb^2}{2} \right) = \frac{1}{18G} \left( \frac{H_x - H_o}{M \alpha} \right)^2
\]

(3.3)

where, \( M = 3.05 \) is the Taylor factor, \( \alpha = 0.3 \) is a constant, \( G = 43.98 \) GPa is the average shear modulus and \( b = 2.87 \times 10^{-10} \) m is the Burgers vector. The values of bulk stored energy estimated from Eq. (3.3) are shown in Fig. 3.1(e). As seen from the graph and similar to the reduction in VHN, the bulk stored energy declines rapidly and almost linearly between the onset (780 and 710 °C for CD80 and CD90, respectively) and end (840 and 820 °C for CD80 and CD90, respectively) of recrystallisation.

### 3.1.2 Changes in microstructure and morphology

The representative microstructures of the CD80 and CD90 samples are illustrated in Fig. 3.2 in terms of the grain orientation spread (or GOS) criterion. While the red colour denotes deformed and recovered structures, the grey colour denotes newly nucleated and growing recrystallised grains. In Figs. 3.2(a, c, e), the CD80 samples
are rapid resistance heated at the heating rate of \(20^\circ\cdot s^{-1}\) to 780, 815 and 840 °C peak annealing temperature, respectively. In the case of Figs. 3.2(b, d, f), the CD90 samples are rapid resistance heated at the same heating rate as CD80 to 720, 750 and 820 °C peak annealing temperature, respectively. The four partially recrystallised samples namely CD80(0.07), CD80(0.48), CD90(0.04) and CD90(0.37) are deconstructed with band contrast into deformed, recovered, newly nucleated and growing grain subsets as shown in Figs. 3.3(a-d), Figs. 3.4(a-d), Figs. 3.5(a-d) and Figs. 3.6(a-d), respectively.

Irrespective of the magnitude of cold drawing, the characteristically heterogeneous microstructures after partial recrystallisation (Fig. 3.2 and Figs. 3.3-3.6 (a, b)) were found to contain coarse unrecrystallised grains with large aspect ratios (between 3.2-3.8 for CD80(0.07) and 3.2-4.6 for CD90(0.04), respectively) separated by packets of fine unrecrystallised grains with even larger aspect ratios (between 6.2-7.0) as well as approximately equiaxed recrystallised grains. In most cases, the coarse unrecrystallised grains contained in-grain shear bands which were found to be typically inclined between 20° and 45° to the drawing direction (map horizontal). A fewer number of shear bands were noted in the CD80 samples as compared to the CD90 samples as a direct result of the lower strain associated with lesser cold drawing reduction.

The effect of strain and grain growth is clearly illustrated when the equivalent circle diameter (ECD) and aspect ratio of subgrains (LAGBs + HAGBs) and grains (HAGBs only) are plotted for the CD80 (Figs. 3.7(a, c)) and CD90 (Fig. 3.7(b, d)) samples, respectively. It can be seen that the ECD values of the growing grain subsets in the case of the CD80 samples increase almost monotonically with the onset of recrystallisation (Fig. 3.7(a)). On the other hand, the ECD values of the growing grain subsets of the CD90 samples remains approximately constant until 36.8% of the map area is recrystallised (Fig. 3.7(b)) and increases only during the later stages of recrystallisation. As a result of grain growth, the average grain size of the CD80(0.92) sample was approximately 1.5 times higher than that of CD90(0.90) sample.

If the number density of newly nucleated grains are plotted (Figs. 3.7(e, f)), the lower cold drawing reduction (CD80) leads to approximately half the density of
newly nucleated grains after 48% of its map area is recrystallised (Fig. 3.7(e)) as compared to the CD90 sample after 36.8% of its map area is recrystallised (Fig. 3.7(f)). Similar trends and ratios are obtained when the number density of the newly nucleated subgrains is plotted instead.
Fig. 3.2: Selected maps based on GOS of (a, c, e) CD80 and (b, d, f) CD90 LCB-Ti after rapid heating to peak temperatures of (a) 780, (b) 720, (c) 815, (d) 750, (e) 840 and (f) 820 °C. The map area fractions recrystallised are (a) 6.6%, (b) 3.9%, (c) 48%, (d) 36.8%, (e) 92.4% and (f) 90.2%. Grey for small and red for large GOS values. DD is drawing direction; d1 and d2 are two indistinguishable and interchangeable directions of the cylindrical wire drawn specimens.
Fig. 3.3: Band contrast maps of CD80(0.07) LCB-Ti after rapid heating to a peak temperature of 780 °C deconstructed into (a) deformed, (b) recovered, (c) newly nucleated, (d) growing grain subsets and (e) recrystallised and unrecrystallised boundary pairs. Grey = LAGBs, black = HAGBs.

DD is drawing direction; d1 and d2 are two indistinguishable and interchangeable directions of the cylindrical wire drawn specimens.
Fig. 3.4: Band contrast maps of CD80(0.48) LCB-Ti after rapid heating to a peak temperature of 815 °C deconstructed into (a) deformed, (b) recovered, (c) newly nucleated, (d) growing grain subsets and (e) recrystallised and unrecrystallised boundary pairs. Grey = LAGBs, black = HAGBs.

DD is drawing direction; d1 and d2 are two indistinguishable and interchangeable directions of the cylindrical wire drawn specimens.
**Fig. 3.5:** Band contrast maps of CD90(0.04) LCB-Ti after rapid heating to a peak temperature of 720 °C deconstructed into (a) deformed, (b) recovered, (c) newly nucleated, (d) growing grain subsets and (e) recrystallised and unrecrystallised boundary pairs. Grey = LAGBs, black = HAGBs.

DD is drawing direction; d1 and d2 are two indistinguishable and interchangeable directions of the cylindrical wire drawn specimens.
Fig. 3.6: Band contrast maps of CD90(0.37) LCB-Ti after rapid heating to a peak temperature of 750 °C deconstructed into (a) deformed, (b) recovered, (c) newly nucleated, (d) growing grain subsets and (e) recrystallised and unrecrystallised boundary pairs. Grey = LAGBs, black = HAGBs. DD is drawing direction; d1 and d2 are two indistinguishable and interchangeable directions of the cylindrical wire drawn specimens.
Fig. 3.7: Evolution of (a and b) equivalent circle diameter, (c and d) aspect ratio of subgrains (LAGB+HAGB) and grains (HAGB) and (e and f) density of newly nucleated (sub)grains in partially recrystallised (a, c, e) CD80 and (b, d, f) CD90 LCB-Ti.

The above observations are similar to an earlier report that optically measured the variation in grain size after cold drawing an LCB-Ti alloy to 50% and 70% reduction and annealing to complete recrystallisation via rapid heating at $-120 \, ^\circ C \, s^{-1}$ [12]. In that study, the grain size of the 50% cold drawn sample was approximately 1.75 times larger than that of the 70% cold drawn sample. This was attributed to the lower dislocation density in the 50% cold drawn sample resulting in fewer defect sites for preferential nucleation. Of even greater interest is the fact that the CD80(0.92) sample presented with a greater non-uniformity in recrystallised grain size compared to the CD90(0.90) sample such that relatively coarse grains were distributed among even finer grains. The non-uniformity of the recrystallised microstructure obtained after small magnitudes of cold working was also noted in a VT-22 $\beta$-Ti alloy after $\beta$ solid solution treatment, cold rolling to 40% thickness reduction and continuous heating at the rate of $5 \, ^\circ C \, s^{-1}$ [11]. If the results of the present study are combined with the conclusions from [11], it suggests that the severity of prior cold working has an even greater influence on the distribution of
the final grain size in the recrystallised microstructure than the rate of rapid heating employed during annealing.

The histograms depicting the misorientation distribution of the various subsets for the partially recrystallised CD80 and CD90 samples are shown in Figs. 3.8(a-d). In the case of Figs. 3.8(e, f), the misorientation distribution of the full map is presented as the maps record 92.4% (CD80(0.92)) and 90.2% (CD90(0.90)) of their area fractions as recrystallised. As per the definitions used for map deconstruction, it is clear that the deformed subsets contain a greater fraction of LAGBs compared to the recovered fraction (compare the red and green histograms in Figs. 3.8 (a-d)).
Fig. 3.8: Misorientation distributions in the (a, c, e) CD80 and (b, d, f) CD90 LCB-Ti after rapid heating to peak temperatures of (a) 780, (b) 720, (c) 815, (d) 750, (e) 840 and (f) 820 °C. Random denotes the McKenzie distribution for non-textured materials.
The changes in the fraction of high-angle boundaries ($f_{HAGBs}$) and the average misorientation ($\theta_{AVG}$) as a function of the map area fraction recrystallised are shown in Figs. 3.9(a, b) for the CD80 and CD90 samples, respectively. Although the proportion of high-angle boundaries and average boundary misorientation angle in the newly nucleated and growing grain subsets increases concurrently with greater recrystallisation, the tendencies of boundary development and misorientation accumulation between these two subsets are markedly different in the case of the CD80 and CD90 samples. The newly nucleated grains in CD80(0.07) return slightly lower and higher $f_{HAGBs}$ and $\theta_{AVG}$ –values, respectively compared to their CD90(0.04) counterparts. On the other hand, the growing grains in CD80(0.07) have higher $f_{HAGBs}$ and $\theta_{AVG}$ –values than those of the CD90(0.04) condition. Irrespective of the $f_{HAGBs}$ and $\theta_{AVG}$ –values at the start of the rapid heating process, the newly nucleated and growing grain fraction of the CD90 samples show a relatively faster rate of increase in HAGB fraction and misorientation accumulation throughout recrystallisation. The result of these distinctions is seen in Figs. 3.8(e, f). While the misorientation histogram in the case of the CD80 sample still contains a relatively higher proportion of LAGBs (Fig. 3.8(e)), the histogram of the CD90 sample closely resembles the McKenzie random distribution (Fig. 3.8(f)).
Fig. 3.9: Dependence of (a and b) fraction HAGBs (left-hand axis) and average boundary misorientation (right-hand axis) on the progress of recrystallisation in (a) CD80 and (b) CD90 LCB-Ti.

It can be pointed out that the variation in the fraction of high-angle boundaries and the rate of accumulation of average misorientation in the recrystallised microstructure between the CD80 and CD90 samples also suggests differences in the growth rate of this fraction during rapid heating (see Section 3.1.3).

3.1.3 Specific interfacial boundaries, local stored energy estimates and apparent growth rates

The specific interfacial areas per unit volume ($S_v$) can be more accurately determined from EBSD map using double-dilation of the four subsets (The interface between the unrecrystallised and recrystallised fraction is illustrated in Figs. 3.3-3.6(e)). Here, $S_v$ is expressed by the equation [47]:

$$S_v = \frac{4}{\pi} \left( \frac{L_i}{A} \right) = \frac{2f_i}{\pi \cdot \text{step size}}$$

(3.4)

where, $L_i$ is the total interface length, $A$ is the total map area and $f_i$ is the fraction of pixels making up the interface. The standard error was determined for each map as the difference between the total of the $f_i$ values of the four subsets against the
pixel percentage of the interface between the recrystallised and unrecrystallised fractions. The values from Eq. (3.4) are displayed in Figs. 3.10(a, b) for CD80 and CD90, respectively. As expected in a severely cold drawn material undergoing discontinuous recrystallisation via rapid heating, it is observed that the rate of increase in the specific interfacial area of the CD90 samples is much higher than that of the CD80 samples. A qualitative check for the three-dimensional spheroidal growth of the recrystallising (sub)grains is utilised, linearity and non-linearity are shown in the CD80 and CD90 samples, respectively (Figs. 3.10(c,d)).

**Fig. 3.10:** Evolution of (a and b) specific interfacial area, (c and d) dependence of $S_v/(1-A_{f})$ on $-\ln(1-A_{f})$ in (a, c) CD80 and (b, d) CD90 LCB-Ti.
From the misorientation histogram of the specific interface, the local stored energy per unit volume associated with the dislocation boundaries \( (E_b) \) can be defined by modifying the Read-Shockley equation such that \([28, 49]\):

\[
E_b = S_v \cdot C_{\text{L}} \cdot C_p \cdot \bar{\gamma} = S_v \cdot C_{\text{L}} \cdot C_p \cdot \sum_{2}^{62.8}(\gamma(\theta) f(\theta))
\]

with \( \gamma(\theta) = \gamma_m (\theta / \theta_m)[1 - \ln(\theta / \theta_m)] \) for \( \theta \leq \theta_m \)

or \( \gamma(\theta) = \gamma_m \) for \( \theta > \theta_m \)

where, \( C_{\text{L}} \) is the ratio of the map areas of the two subsets making up the specific interface, \( C_p \) is the ratio of the number of pixels at a specific interface versus the unrecrystallised-recrystallised interface, \( \bar{\gamma} \) is the average energy per unit boundary area, \( f(\theta) \) is the boundary fraction for a given misorientation, \( \gamma_m = 0.55 \text{ J} \cdot \text{m}^{-2} \) \([50]\) is the energy per unit area of a HAGB, \( \theta \) is the boundary misorientation and \( \theta_m = 15^\circ \) is the critical misorientation denoting HAGBs above which the energy per unit area is independent of the misorientation angle.

The lower value of boundary stored energy in the CD80(0.07) sample (Fig. 3.11(a)) as compared to the CD90(0.04) sample (Fig. 3.11(b)) is attributed to the former having undergone a smaller cold drawing reduction. On the other hand, the rather large standard deviation in the particular case of the CD90(0.04) condition is due to the higher standard error carried over from double dilation (see above). In turn, the standard error can be attributed to: (i) the greater inhomogeneity in the morphology of the deformed and recovered (sub)grains due to the more pronounced orientation gradients in the CD90 condition, and (ii) the relative “coarseness” of the step size used for mapping.

Although Fig. 3.11 show that the boundary stored energy declines throughout the rapid heating process for both CD80 and CD90 conditions, the rate of decline between them are again different. While CD80 samples record a gradual decease in boundary stored energy, the CD90 samples return a drastic reduction in \( E_b \) values until the CD90(0.37) condition followed by a more steady decline up to the CD90(0.90) condition. Interestingly, it is noted that the values of boundary stored
energy between the various interfaces in the case of CD90 samples tends to remain higher than those obtained for the CD80 case for the most part of the rapid heating process. This could be ascribed to the greater strain as a result of higher cold drawing reduction and the higher rate of increase in the $f_{HAGBs}$ and $\theta_{AVG}$ values of the recrystallised fraction in CD90 samples.

Fig. 3.11: Evolution of specific interfacial boundary stored energy on the progress of recrystallisation in (a) CD80 and (b) CD90 LCB-Ti.

Since the present cold drawn LCB-Ti has been subjected to rapid heating under non-isothermal annealing conditions, a real growth rate cannot be defined. In [47], a modification to the Cahn-Hagel equation was suggested in order to derive an apparent growth rate of the various recrystallisation fronts such that [51, 52]:

$$G = \frac{1}{S_v} \left( \frac{dX}{dt} \right) = \frac{1}{S_v} \left( \frac{dX}{dT} \frac{dT}{dt} \right)$$

(3.6)

where, $dX/dt$ is the slope of the linear second stage (corresponding to recrystallisation) of the sigmoidal recrystallised fraction versus temperature plot and $dT/dt$ is the heating rate during annealing. In that study, the application of Eq. (3.6) is justified on the basis that the rate of increase of the recrystallised fraction closely matches the rate of softening calculated from hardness data when warm
and cold rolled low carbon Cr steel was subjected to non-isothermal annealing at a comparatively much slower heating rate of $4 \times 10^{-2} \, ^\circ C \cdot s^{-1}$ (refer to Table 1 in [47]).

However, as seen in Fig. 3.1(d) for the present LCB-Ti case of rapid heating at 20 $^\circ C \cdot s^{-1}$, the rate of change in the area fraction of the recrystallised grains (including newly nucleated and growing grain subsets) severely lags the rate of change in softening values (the latter including both, recovery and recrystallisation effects). Thus, the calculation of the $G$-values needs to account for the difference between the rates at which: (i) the deformed grains in the unrecrystallised fraction undergo recovery and, (ii) the increase in the area fraction of the recrystallised microstructure occurs. In order to account for the above effects and specific to the EBSD maps, a further modification to the original Cahn-Hagel equation is suggested in this study such that:

$$G = \frac{1}{S_y} \left( \frac{dA_x}{dt} \right) = \frac{1}{S_y} \left( \frac{dA_x}{dS_f} \cdot \frac{dS_f}{dT} \cdot \frac{dT}{dt} \right)$$

(3.7)

where, $A_x$ is the area fraction of recrystallised grains from the EBSD map. The values of the apparent growth rate in CD90 and the CD80 samples are illustrated in Fig. 3.12. While the highest rates of apparent growth were found to occur at the interface between the deformed and newly nucleated or growing grain subsets, the trends between the CD80 and CD90 conditions are different. While the apparent growth rate of the deformed–newly nucleated interface of the CD80 samples show an increase only after the CD80(0.48) condition, the apparent growth rate of the deformed–growing grain interface remains approximately constant throughout the rapid heating process (Fig. 3.12(a)). On the other hand, a linear increase in the apparent growth rates of the deformed–newly nucleated or growing grain interface up to the CD90(0.90) condition is seen in the case of the CD90 samples (Fig. 3.12(b)). While the values of apparent growth rate for these interfaces are very large, it should also be kept in mind that the rate at which deformed grains undergo recovery is also very high and that the process of recovery is concurrent to recrystallisation.
Thus it is more important to note that the apparent growth rate of the unrecrystallised-recrystallised and recovered-recrystallised interfaces is by comparison, rather small. While the former is more representative of the “average” apparent growth rate across all interfaces, the latter denotes the apparent growth rates of the interface from subsets that comprise the majority of the map area fraction. In this regard, the CD80 samples return slightly faster rates of apparent growth rates than the CD90 samples. This observation is in agreement with the lesser magnitude of imparted strain in the case of the CD80 sample providing fewer barriers to boundary migration during recrystallisation.

Thus, from Figs. 3.2 to 3.12 it is apparent that the rapid heating of both CD80 and CD90 samples is marked by concurrent and competing processes involving recovery, nucleation and grain growth. The relative rates of these processes tend to be governed by the magnitude of imparted strain during cold drawing.

![Fig. 3.12: Evolution of growth rate on the progress of recrystallisation in (a) CD80 and (b) CD90 LCB-Ti.](image)

### 3.1.4 Apparent activation energy

The mobility of the new grain boundaries formed during recrystallisation can be expressed as a function of boundary velocity and the driving pressure for recrystallisation as follows:
Here the estimated boundary velocity is analogous to the apparent growth rate from Eq. (3.7) (Figs. 3.12). Conventionally, the driving pressure is expressed in the terms of the subgrain size and misorientation by utilising the classical Read-Shockley equation [28]. In the present case, double dilation of the various subsets results in a specific boundary interfacial area estimate which comprises both, low and high-angle boundaries. Thus, Eq. (3.5) can be applied as an estimate of the driving pressure from the subgrain population towards primary recrystallisation.

When the values of mobility calculated from Eq. (3.8) are plotted as a function of the peak temperature \(1/T\) in Kelvin, an approximately linear best-fit relationship is obtained for the various specific interfaces (Fig. 3.13). In this figure, it should be noted that the recrystallising fronts common to CD80 and CD90 have been combined together. Since the mobility of these boundaries is temperature dependent, they usually obey an Arrhenius–type relationship of the form:

\[
M = M_0 \exp \left( -\frac{Q_a}{RT} \right)
\]

where, \(M_0\) is the pre-exponential factor, \(Q_a\) is the apparent activation energy for boundary migration, \(R\) is the gas constant (8.3144621 J·K\(^{-1}\)·mol\(^{-1}\)) and \(T\) is the rapid heating peak temperature in Kelvin. If the equation of the best-fit line defines an exponential relationship, the value of the constant within the exponent term (= \(-Q_a/R\)) can be used to solve for the apparent activation energy.
The apparent activation energies for boundary migration across the various specific interfaces are shown in Table 3.1. As expected, the value of apparent activation energy at the deformed-recrystallised (newly nucleated or growing grains) interface is the lowest and suggests the relative ease of nucleation and growth across such interfaces, respectively. On the other hand, the apparent activation energy across the recovered-recrystallised interface is even higher. This observation is in agreement with expectations that the driving pressure for recrystallisation reduces with the lowering of the total dislocation density during recovery. With respect to the recrystallised grains, the above trend suggests that their growth via boundary migration across the recovered microstructure will tend to be increasingly sluggish as recrystallisation progresses. The direct consequence of this phenomenon can be seen clearly in the case of the CD80(0.92) sample (Fig. 3.2(e)) which presents with remanent unrecrystallised (recovered) substructures.

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**Fig. 3.13:** Specific interfacial boundary mobility averaged over all misorientations as a function of rapid heating peak temperature in CD80 and CD90 Ti-LCB.
Table 3.1: Apparent activation energy ($Q_a$, kJ·mol$^{-1}$) for boundary migration across specific interfaces during the rapid heating of LCB-Ti.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Unrecrystallised/ Recrystallised</th>
<th>Deformed / Newly nucleated</th>
<th>Deformed / Growing grains</th>
<th>Recovered / Newly nucleated</th>
<th>Recovered / Growing grains</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>518.7 ± 45.5</td>
<td>292.2 ± 79.4</td>
<td>303.0 ± 79.2</td>
<td>463.5 ± 49.8</td>
<td>483.5 ± 50.1</td>
</tr>
</tbody>
</table>

3.1.5 Evolution of the bulk and micro-texture

Fig. 3.14(a) is a schematic of the $\phi_2 = 45^\circ$ ODF section for cold drawn bcc metals shown in Bunge's notation and characterised by the $\alpha$ fiber. This fiber comprises orientations with their $\langle 110 \rangle$ axes parallel to the drawing direction (DD) and are located from $(001)[\bar{1}10]$ to $(110)[\bar{1}01]$ orientation corresponding to $\phi = 0^\circ$-$90^\circ$ at $\phi_1 = 0^\circ$ on the $\phi_2 = 45^\circ$ ODF section. The $(001)[\bar{1}01]$ and $(111)[0\bar{1}1]$ orientations that are equivalent to $(001)[\bar{1}01]$ and $(111)[\bar{1}01]$ orientations, respectively also belong to the $\alpha$-fiber.

The bulk textures of the CD80 samples after rapid heating to 780, 815 and 840 °C are shown in Figs. 3.14(b-d) while the bulk textures of the CD90 samples after cold drawing and rapid heating to 720, 750 and 820 °C are shown in Figs. 3.14(e-h), respectively. In Fig. 3.14(b), the ODF of the CD80(0.07) sample is characterised by a complete $\alpha$-fiber with intensity maxima for the $\{001\}\langle 110 \rangle$ followed by $\{111\}\langle 110 \rangle$ equivalent orientations. On the other hand, the ODF of the 90% cold drawn sample is denoted by an incomplete $\alpha$-fiber with an intensity maxima located close to the $(223)[1\bar{1}0]$ orientation component (Fig. 3.14(e)). With the onset of rapid heating and with characteristics similar to both, cold drawn CD90 and CD80(0.07) samples, the ODF of the CD90(0.04) sample shows an incomplete $\alpha$-fiber and an intensity maxima for the $\{001\}\langle 110 \rangle$ equivalent orientations (Fig. 3.14(f)).
Fig. 3.14: $\phi_2 = 45^\circ$ ODF sections of the (a) ideal body-centred cubic drawing and recrystallisation fibers, (b-d) CD80 and (e-h) CD90 LCB-Ti bulk textures after rapid heating to peak temperatures of (b) 780, (c) 815, (d) 840, (f) 720, (g) 750 and (h) 820 °C. The ODF in (e) is the cold drawn condition for CD90 LCB-Ti. Contour levels = 1×.
With the progress of recrystallisation during rapid heating, the maxima in the texture intensity of the ODFs decline in both, CD80 and CD90 samples. The ODFs of the CD80(0.48) and CD90(0.37) samples (Figs. 3.14(c, g)) are mostly similar as they record the development of the major \( \{111\} \langle 110 \rangle \) and minor \( \{001\} \langle 010 \rangle \) and \( \{110\} \langle 001 \rangle \) orientation components. In terms of their differences, the CD80(0.48) sample return fairly weaker \( \{001\} \langle 110 \rangle \) equivalent orientations compared to the CD90(0.37) sample.

Although the ODFs of the CD80(0.92) and CD90(0.90) samples (Figs. 3.14(d, f)) depict the \( \{001\} \langle 110 \rangle \) equivalent orientations, a marked difference in the relative volume fraction of these orientations is apparent. This observation alludes to variations in the rate of texture evolution between CD80 and CD90 samples. For instance, while the ODF of the CD80(0.92) sample continues to show weak intensities for the \( \{111\} \langle 110 \rangle \) equivalent orientations, the ODF of the CD90(0.90) sample mainly comprises the \( \{001\} \langle 110 \rangle \) equivalent orientations along with an extremely weak \( \{110\} \langle 001 \rangle \) orientation component.

The microtextures of the various subsets obtained from deconstruction of the EBSD maps for the CD80(0.07), CD80(0.48), CD90(0.04) and CD90(0.37) conditions are shown in Figs. 3.15(a-d), 3.15(e-h), 3.16(a-d) and 3.16(e-h), respectively. It is apparent that the microtextures obtained from EBSD are unable to fully capture all the features in the bulk texture. Since the deformation in LCB-Ti tends to be heterogeneous, the areas mapped by EBSD are only representative of changes occurring in localised areas of the specimen. However, the main orientation components are common to both, bulk and micro textures. In particular, the ODFs of the CD80(0.07) sample (Figs. 3.15(a-d)) depict an approximately complete \( \alpha \)-fiber and is similar to the bulk texture in Fig. 3.14(b).
Fig. 3.15: $\phi_2 = 45^\circ$ ODF sections of the (a, e) deformed, (b, f) recovered, (c, g) newly nucelated and (d, h) growing grain fractions in CD80 LCB-Ti after rapid heating to peak temperatures of (a-d) 780 and (e-h) 815 °C. The map area fractions recrystallised for (c, d) and (g, h) are 6.6% and 48%, respectively. Contour levels = 1×.
Fig. 3.16: $\phi_2 = 45^\circ$ ODF sections of the (a, e) deformed, (b, f) recovered, (c, g) newly nucleated and (d, h) growing grain fractions in CD90 LCB-Ti after rapid heating to peak temperatures of (a-d) 720 and (e-h) 750 °C. The map area fractions recrystallised for (c, d) and (g, h) are 3.9% and 36.8%, respectively. Contour levels = 1×.
3.2 Effect of heating rate

3.2.1 Effect on microstructure

In order to examine the effect of the heating rate, the sample CD90 were heated at two different heating rates of 10 °C.s\(^{-1}\) and 20 °C.s\(^{-1}\). The microstructures after annealing of the sample with the heating rate of 10 °C.s\(^{-1}\) is shown in the Fig. 3.17.

![Image](image.png)

**Fig. 3.17:** Selected band contrast map of CD90 LCB-Ti after rapid heating to peak temperatures of 820 °C at the heating rate of 10 °C.s\(^{-1}\). Grey = LAGBs, black = HAGBs.

As seen from the Fig. 3.2(f) and Fig. 3.17, the microstructures at 820 °C after the heating rate of 20 °C.s\(^{-1}\) (90\% area fractions recrystallised) and after the heating rate of 10 °C.s\(^{-1}\) (95\% area fractions recrystallised) are relatively homogeneous with equiaxed grains having low aspect ratios (between 1.6 and 1.7). However, an increase in the heating rate from 10 to 20 °C.s\(^{-1}\) produced a 1.7 times finer grain sized microstructure.
3.2.2 Effect on texture

Texture of CD90 samples after annealing at different heating rate to 820 °C are displayed in Fig. 3.18.

Fig. 3.18: $\phi_2 = 45^\circ$ ODF sections of the bulk texture for CD90 LCB-Ti after rapid heating to peak temperatures of 820 °C at the heating rate of 10 °C.s$^{-1}$. Contour levels = 1x.

The bulk texture components of the two samples heated to 820 °C at the heating rate of 20 °C.s$^{-1}$ (Fig. 3.14(h)) and 10 °C.s$^{-1}$ (Fig. 3.18) are nearly the same as both ODFs are characterised by the \{001\}$<110>$ orientations. While the \{111\}$<110>$ component is non-existent in the sample heated at the rate of 10 °C.s$^{-1}$, it is extremely weak for the higher heating rate of 20 °C.s$^{-1}$.

3.3 Hall-Petch relationship

The dependence of yield strength on the grain size is usually expressed by the conventional Hall-Petch [37, 38] relationship such that [37, 38]:

$$\sigma_y = \sigma_0 + k_{HP}d^{-1/2}$$

(3.10)
where, \( \sigma_Y \approx H_V/M \) \((H_V\) is Vickers hardness and \( M\) is the average Taylor factor determined from the EBSD maps \), \( \sigma_o \) is the friction stress, \( k_{HP}\) is the constant related to the strength of grain boundaries.

In order to define \( \sigma_o \), the Eq. 3.10 was employed for the three fully recrystallised comprising a CD80 sample annealed to 840 °C at 20 °Cs\(^{-1}\) and two CD90 samples annealed to 820 °C at 10 °Cs\(^{-1}\) and 20 °Cs\(^{-1}\), respectively. The fitted linear relationship between \( d^{-1/2} \) and \( \sigma_Y \) is shown in Fig. 3.19. From this, the values of \( \sigma_o \) and \( k_{HP}\) were found as 880 MPa and 534 MPa.\(\mu\)m\(^{0.5}\) (~0.53 MPa.m\(^{0.5}\)), respectively.

**Fig. 3.19:** The relationship between yield strength and EBSD-returned grain diameter.

In the partly recrystallised metals, the microstructure comprises two types of boundaries: (i) lamellar boundaries (geometrically necessary boundaries, GNBs) and (ii) cell boundaries (incidental dislocation boundaries, IDBs) [53]. These two types of boundaries result in boundary strengthening (or HAGBs strengthening) and dislocation strengthening (or LAGBs strengthening), respectively in the contribution to flow stress. Therefore, the yield strength of the partially recrystallised metals can be defined by the modified Hall-Petch equation [39, 40] such that:
\[ \sigma_Y = \sigma_0 + \sigma_{\text{LAGBs}} + \sigma_{\text{HAGBs}} \]

\[ \sigma_Y = \sigma_0 + M \alpha b \sqrt{1.5 S_v \theta_{\text{LAGB}} (1 - f_{\text{HAGB}})} + k \frac{f_{\text{HAGB}}}{d_{\text{HAGB}}} \]  \hspace{1cm} (3.11)

where, \( \sigma_0 \) is determined by the classical Hall-Petch relation (Eq. 3.10) (\( \sigma_0 = 880 \text{ MPa} \)), \( M \) is the average Taylor factor determined from the EBSD maps, \( \alpha \) is a constant (= 0.3), \( G \) is the shear modulus (~ 44 MPa), \( b \) is Burger vector (= \( 2.87 \times 10^{-10} \text{ m} \)), \( S_v \) is the boundary area per unit volume of LAGBs (~ \( \pi/2d_{2\sim15^\circ} \)), \( k \) is the constant presenting the strength of HAGBs and is optimised by minimising squared difference between the experimental and predicted tensile strength values, \( f_{\text{HAGB}} \) is the fraction of high angle grain boundary (misorientation above 15\(^\circ\)), \( \theta_{\text{LAGB}} \) is the average misorientation of low angle grain boundary (misorientation in the range between 2\(^\circ\) and 15\(^\circ\)) and \( d_{\text{HAGB}} \) is the grain size of high angle grain boundary.

The changes in the tensile strength of CD80 and CD90 during recrystallisation are shown in the Fig. 3.20. As seen from the graph, the result shows agreement between the predicted and experimental values. The error between the predicted and experimental values is in the range of \( \pm 3\% \). The optimal values of \( k \) are 0.23 and 0.32 MPa.m\(^{0.5}\) for CD90 and CD80, respectively. These values are expectedly smaller than \( k_{\text{HP}} = 0.53 \text{ MPa.m}^{0.5} \) calculated by the classical Hall-Petch relation from Eq. 3.10 and can be attributed to the decrease of LAGBs during recrystallisation.

The contributions of friction stress, LAGBs and HAGBs strengthening to true stress are illustrated in the Fig. 3.21. While \( \sigma_0 \) contributes heavily to the strength of LCB-Ti via solid solutioning, the contribution of LAGBs is more prominent than that of HAGBs during the initial stages of recrystallisation. As recrystallisation progresses, the contribution of the LAGBs declines with the decrease of the total dislocation density. On the other hand, the contribution of the HAGBs increases slightly as recrystallisation proceeds due to the increase in the fraction of HAGBs.
**Fig. 3.20:** The dependence of predicted and experimental tensile yield strength ($\sigma_Y$) of the CD90 and CD80 LCB-Ti alloy.

**Fig. 3.21:** The contributions of the $\sigma_0$, LAGBs and HAGBs strengthening to true stress in the CD90 and CD80 LCB-Ti alloy.
4.1 Effect of cold deformation

4.1.1 Variations between the softened and map area fraction recrystallised estimates

As seen in Figs. 3.1b and c, the differences between the softened fraction estimates based on hardness (Eq. (3.1)) and the EBSD boundary data (Eq. (3.2)) and the recrystallised map area fraction estimates are significant. This can be explained as follows. Since hardness is a measure of the total dislocation content in the material, the reduction in its value during rapid heating can be directly correlated with the decline of the statistically stored and geometrically necessary dislocations as a result of concurrent recovery and recrystallisation.

While it is possible that the area mapped by EBSD is insufficient to provide reliable global statistics and that the EBSD measurements are limited in terms of spatial and angular resolution, close correlation with the map recrystallised area fraction estimate is achieved in the partially recrystallised samples of both, CD80 and CD90 LCB-Ti when Eq. (3.2) is reapplied without including the high-angle boundary fraction of the recovered subset. It should be noted that implicit in Eq. (3.2) are the following assumptions: (i) that boundaries are shared between the various subsets, and (ii) that the deformed grains do not contribute to softening.

Thus it is more likely that the above disparity is the direct result of the definitions used in the analysis process. For example, the deconstruction of the EBSD maps defines the recovered fraction as those structures that have the external boundary morphology resembling that of deformed grains but have undergone a significant

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clean-up of their internal dislocation content and present with lower internal misorientation. Thus, they are regarded as being part of the unrecrystallised fraction. On the other hand, the recrystallised area fraction estimate strictly accounts for those “new” structures that are the result of discontinuous recrystallisation. As a consequence, the estimated recrystallised area fractions do not include the softening component caused by concurrent recovery.

As shown in [47] for the case of low carbon steel, the disparity between the softened and recrystallised area fractions can be overlooked when their values are in close proximity. Under such circumstances, close correspondence with the ideal linear trend denoted by the black line of Fig. 3.1(d) is obtained. However, in the present case of LCB-Ti, recovery plays a more dominant role during the rapid heating process and results in a pronounced deviation from linearity up to CD80(0.48) and CD90(0.37). In this regard, if the curves between CD80 and CD90 are compared, it is clear that LCB-Ti undergoes far greater recovery and lesser recrystallisation with higher cold drawing strain. As expected, the role of recovery decreases towards the end of the rapid heating process as CD80(0.92) and CD(0.90) samples return approximately equivalent softened and recrystallised area fraction estimates. Thus, the key role played by recovery in reducing the overall bulk stored energy for a majority of the rapid heating process becomes apparent (Fig. 3.1(e)).

4.1.2 Specific interfacial area, boundary stored energy and apparent growth rates

The calculation of the specific $S_v$ (Figs. 3.10(a and b)) using the double-dilation approach (Fig. 3.3-3.6(e)) in conjunction with Eq. (3.4) provides a more accurate estimate of the interfacial area between any two dissimilar fractions (such as the unrecrystallised and recrystallised subsets) than the conventional stereological approach that utilises the approximate $3/d_{ECD}$ (where, $d_{ECD}$ is the (sub)grain diameter) relationship for a particular subset. In turn, the specific $S_v$ can be reapplied to deduce the spheroidal tendency of the recrystallising grains, the interfacial boundary stored
energy and the apparent growth rates applicable to the various recrystallisation fronts Figs. 3.10(c-d), Fig. 3.11 and Fig. 3.12, respectively.

The dependence of $S/(1-A_p)$ on $-\ln(1-A_p)$ shows linearity and non-linearity in the case of CD80 and CD90 samples, respectively (Figs. 3.10(c, d)) and corroborates the EBSD map data which shows slightly greater aspect ratios for the recrystallised grains in the CD80(0.92) sample than the CD(0.90) sample (Figs. 3.2(e, f)) [54-56]. While the specific boundary interfacial stored energy declines monotonically with the progress of recrystallisation in both, CD80 and CD90 conditions (Figs. 3.11), higher values of standard deviation were returned for the deformed and recrystallised (newly nucleated and growing grains) interfaces of the CD90(0.04) sample. Although the primary reason for this scatter in the data can be attributed to the small area fraction of recrystallised (sub)grains, it also serves to highlight the limitations of the large area EBSD mapping in general and the current deconstruction technique in particular.

The specific $S_y$ returned by double-dilation is a strictly localised estimate that only accounts for the pixels between two dissimilar fractions. Given that the specific $S_y$-value depends on the fraction of pixels making up the interface in question, it follows that further sub-division of maps into smaller deformed, recovered, newly nucleated and growing grain subsets decreases the fraction of pixels making up the interfaces. In the present case of severely cold drawn CD90(0.04), the combined effects of curvature in the vicinity of the boundaries, the relative “coarseness” of the map step size and the variation in the area occupied by individual (sub)grains (measured in pixels) serve to increase the inaccuracy in estimating the boundary interface during the double dilation of the subsets. When these inaccuracies are carried over and combined with the standard deviation in misorientation data (when estimating the interfacial boundary stored energies via the modified Read-Shockley relationship, Eq. (3.5)) or with the variations in the rate of change in the area fraction recrystallised (when deducing the apparent growth rates via the modified Cahn-Hagel relationship, Eq. (3.7)), the errors for these calculations are by extension, exaggerated even further.
With respect to the apparent growth rates shown in Figs. (3.12), the highest growth rates are found at the interface between the deformed-newly nucleated and deformed-growing grain subsets and are indicative of the boundaries encountering fewer barriers to their migration across these particular recrystallisation fronts [51, 52, 55, 57]. Here two critical aspects deserve mention. First, the growth rates across deformed and recrystallising interfaces is expectedly higher than those across recovered and recrystallising interfaces due to the higher driving pressure for recrystallisation across the former interfaces. However, it should also be remembered that the LCB-Ti of this study is undergoing concurrent recovery to a far greater extent for a majority of the rapid annealing process. Therefore it is more realistic to state that the discontinuously recrystallised grains are almost never in contact with deformed boundaries for too long at any particular instance of recrystallisation as these boundaries are steadily transformed into recovered boundaries at much higher rates than the progress of grain growth occurs. Thus, in all likelihood the growth of most of the recrystallised grains occurs at the rather limited rates defined by the driving pressure difference across the common interface with recovered substructures. Second, apart from differences in the average value of the log-normal tendencies of the (sub)grain population of the total recrystallised fraction, there is no underlying physical criterion to sub-divide it into newly nucleated and growing grain subsets. The deliberate creation of a subset comprising newly nucleated grains can be justified on the basis of tracking the number density of such sized (sub)grains (Figs. 3.7(e, f)), their accumulation in misorientation (Figs. 3.8(a-d) and Fig. (3.9)) and the accompanying changes in interfacial area (Figs. 3.10(a, b)) during rapid annealing. However, when the actual average grain size obtained after rapid annealing (Fig. 3.7(a, b)) are compared with the rather high values of apparent growth for the “newly nucleated grains” of the CD80(0.92) and CD(0.90) samples, it is probable that the latter are simply a statistical artefact introduced as a result of the definitions used during deconstruction. Thus adequate care needs to be undertaken when interpreting the veracity of the data obtained from the deconstruction of the EBSD maps.
4.1.3 Apparent activation energy

The efficacy of deconstructing the EBSD map into the four subsets and the use of the double dilation approach is illustrated in the calculation of the apparent activation energy for boundary migration across the various recrystallisation fronts (Eqs. (3.8 and 3.9), Table 3.1). In agreement with previous calculations based on the entire subgrain population [58], the value of \( Q_b = 518.7 \pm 45.5 \text{ kJ mol}^{-1} \) across all unrecrystallised-recrystallised interfaces is expectedly larger than those obtained from bulk grain-size measurement techniques.

However, it is more interesting to compare the values of apparent activation energy between the deformed – recrystallising and the recovered – recrystallising interfaces. Here the \( Q \) values for all four such interfaces are close to the activation energy for self-diffusion of \( \beta \)-Ti (\( Q_s = 145-315 \text{ kJ mol}^{-1} \) [59, 60]). This suggests that the processes of boundary migration just after nucleation and during later grain growth are predominantly diffusion controlled. Again, two crucial aspects deserve mention. First, the activation energy across deformed-recrystallising interfaces is smaller than across recovered-recrystallising interfaces. This observation is in agreement with expectations of a higher driving pressure for recrystallisation in the case of the deformed-recrystallising interfaces (compare these boundary stored energy values with those for other interfaces in Figs. (3.11). Second, and irrespective of the type of the unrecrystallised fraction (deformed or recovered subsets) on the opposite side of the interface, the average value of apparent activation energy for boundary migration is slightly smaller in the case of the newly nucleated grains than the growing grains. In this regard, a recent report on TG6 and Ti17 \( \beta \)-Ti alloys observed an increase in the activation energy for grain growth during extended isothermal soaking time [61] and attributed the phenomenon to the solute drag effect.

In the present case of LCB-Ti, a size based sub-division of the recrystallised microstructure into subsets comprising newly nucleated and growing grains is justified on the basis that it automatically separates out grains with a size (or correspondingly, time) advantage over their smaller counterparts for any particular
instance of partial recrystallisation. This detail should be remembered when noting
the sharp increases in the number density and specific interfacial area of newly
nucleated (sub)grains (Figs. 3.7(e, f) and 3.10(a, b)) and their relatively highest
apparent growth rates (Fig. (3.12)). Thus, it is plausible that during the early stages of
growth just after nucleation, the boundary migration velocity of the newly nucleated
grains is faster than the diffusion of solute atoms. As these boundaries are relatively
free of the solute drag effect, they consequently present with lower activation energy
values. Here emphasis is placed on the fact that such boundaries can only be
considered as “relatively free” of the solute drag effect. Although the rate of self-
diffusion tends to be fast in β-Ti alloys, the diffusivity of alloying elements like Fe is
greater by almost two orders of magnitude [62]. In turn, the enhanced diffusivity
caused by the higher concentration of Fe (4.5 wt.%) in the present LCB-Ti alloy can
also manifest as: (i) higher boundary migration rates in the case of the recrystallised
microstructure and, (ii) greater recovery effects in the unrecrystallised volume [63].
Both these parameters are also critical in determining the overall growth kinetics of
the recrystallising microstructure.

In the case of the growing grains, the lowering of their driving pressure for growth
(compare the differences in the boundary stored energy and apparent growth rates
between the deformed-newly nucleated and deformed-growing grain interfaces in
Figs. (3.11) and (3.12)) is ongoing with the recovery of the deformed grains with
whom they increasingly share a common interface. Thus greater rapid heating time
coupled with a lowering in the driving pressure for growth and the enrichment of
solute atoms at such boundaries results in a slightly more pronounced solute drag
effect. Consequently, further growth is achieved only by increasing the activation
energy of the boundaries of the growing grains.

The above evidence can also be correlated with the earlier remark citing other studies
that suggested limiting the rate rapid heating to $20^\circ\text{C.s}^{-1}$ in β-Ti alloys; as faster
heating rates lead to microchemical non-uniformities which caused microstructural
inhomogeneities and resultantly large variations in mechanical properties [64]. Thus,
if the rapid heating rates were increased, the greater variation between the relatively faster boundary migration velocity of the newly nucleated grains and the rate at which solute diffusion to the boundary occurs could serve to exacerbate microchemical non-uniformities in the bulk recrystallising microstructure.

4.1.4 Evolution of the bulk and micro texture

While the bulk cold drawn texture of CD80(0.07) sample presents with a relatively more uniform and complete \( \alpha \) partial fiber, the CD90(0) and CD90(0.04) samples return an incomplete \( \alpha \) partial fiber with orientation clusters along the \( \phi \)-axis. However, all three samples exhibit local intensity maxima at \( \sim (223)[1\overline{1}0], \sim (115)[\overline{1}10] \) and \( \{001\langle 110 \rangle \) as well as comparatively weaker intensities for the equivalent \( \{111\langle 011 \rangle \) orientations. The above is in general agreement with literature–based descriptions of the conventional texture after the cold drawing of bcc materials [65]. Alternatively, the fact that the bulk recrystallisation textures of CD80(0.48), CD80(0.92), CD90(0.37) and CD90(0.90) samples after rapid heating continue to show the characteristic \( \langle 110 \rangle \) partial fiber is also in agreement with other texture studies that detail the recrystallisation texture of low carbon steel wire [66-68].

It should be kept in mind that the micro-textures shown in Figs. 3.15 for CD80(0.07) (a-d) and CD80(0.48) (e-h) and Figs. 3.16 for CD90(0.04) (a-d) and CD90(0.37) (e-h) are unable to reproduce all of the orientation features of the bulk textures shown in Figs. 3.14 (b, c) and 3.14(f, g) due to the limitations of beam time availability as well as the rather localised nature of the EBSD map with respect to the microstructural heterogeneity of the bulk sample. Nonetheless, the micro-texture –based ODFs are able to capture the basic tendency of texture evolution during rapid annealing.

The ODFs of the deformed and recovered subsets of the CD80(0.07) and CD90(0.04) conditions are presented in Figs. 3.15 and 3.16(a, b), respectively. The difference in the intensity of the \( \alpha \) partial fiber is of particular interest here as the intensities of the
recovered subsets are lower than those of the deformed subsets. During these initial stages of recrystallisation, the deformed grain orientations belong mostly to the α fibre (Figs. 3.15 and 3.16(a)). While the deformed subset of the CD80(0.07) sample exhibits significantly higher intensities for the \{001\}\langle110\rangle orientations and weaker intensities for the \{111\}\langle110\rangle orientations, the deformed grains of the CD90(0.04) sample returns higher intensities for the \{111\}\langle110\rangle followed by the \{001\}\langle110\rangle orientations. More significantly, this particular ODF also shows the extremely weak presence of the \langle110\rangle[001] orientation. The intensities are greater in the case of the CD90 samples due to the higher magnitude of cold drawing.

The recovered grain orientations belong predominantly to the \langle110\rangle[0\bar{1}1] and \langle111\rangle[0\bar{1}1] orientations as the stored energies associated with these orientations are higher than other orientations along the \langle110\rangle||DD fiber (Figs. 3.15 and 3.16(b)). However, the presence of recovered \{001\}\langle110\rangle and Goss [(110)[001]] orientations with their nominally low stored energies is also noted in the case of both, CD80(0.07) and CD90(0.04) samples.

As recrystallisation progresses, the unrecrystallised ODFs of the CD80(0.48) and CD90(0.37) samples both show the disappearance of the α partial fiber. However, the deformed and recovered grains of the CD80(0.48) sample (Fig. 3.15(e, f)) comprise the \{001\}\langle110\rangle orientations while the unrecrystallised grains of the CD90(0.37) sample (Fig. 3.16(e, f)) exhibit both, \{111\}<110> (with much stronger intensities for the \langle111\rangle[0\bar{1}1] compared to the \langle111\rangle[1\bar{1}0] orientation) and \{001\}\langle110\rangle orientations. This leads to two important observations: (i) that on a local scale, rapid annealing results in regions where a marked depletion of \{111\}\langle110\rangle orientations occurs and, (ii) that approximately mid-way through the rapid annealing process, the evolutionary rates for the disappearance of the unrecrystallised matrix in the CD80 and CD90 samples varies rather greatly. The overall effect of the above distinctions in tendency is clearly seen when the bulk ODF of the CD80(0.92) sample (Fig. 3.14(d)) is compared with that of the CD90(0.90) sample (Fig. 3.14(h)).
In the ODFs of the newly nucleated and growing grain subsets of the CD80(0.07) and CD90(0.04) conditions (Figs. 3.15 and 3.16(c, d)), general trends similar to those seen for the unrecrystallised grains are noted. At this initial stage of recrystallisation, the newly nucleated grains of the CD80(0.07) and CD90(0.04) samples (Fig. 3.15 and 3.16(c)) present with the strongest intensity for the \([110][\bar{1}10]\) component followed by the \{111\}(110) orientations (with comparatively higher intensities for the \((111)[0\bar{1}1]\) compared to the \((111)[1\bar{1}0]\) orientation). However, weak intensities for the \{001\}(110) and Goss (\((110)[001]\)) orientations are also noted in the newly nucleated grains of both cold drawn samples. The same orientations are also visible in the ODFs of the growing grains (Figs. 3.15 and 3.16(d)) such that the strongest intensities are seen for the \((110)[\bar{1}10]\) and \((111)[\bar{1}10]\) orientation components. Similar to the unrecrystallised and newly nucleated grains, the growing grains also present with \{001\}(110) and Goss (\((110)[001]\)) orientations.

The ODFs of the newly nucleated and growing grains of the CD80(0.48) and CD90(0.37) samples (Figs. 3.15 and 3.16(g, h)) are again similar to their unrecrystallised counterparts. In agreement with the orientations seen in the ODFs of the deformed and recovered subsets of the CD80(0.48) sample, the recrystallised subsets also depict the nucleation and growth of the \{001\}(110) orientations. Similarly, the orientations of the newly nucleated and growing grains of the CD90(0.37) samples also show the predominant development of the \((111)[0\bar{1}1]\) orientation, weaker intensities for the \{001\}(110) orientations and the weakest intensities for the \((110)[001]\) component.

When comparing the bulk partially recrystallised textures (Figs. 3.14(b-d, f-h)) with the micro-textures shown in Figs. 3.15 and 3.16 or when comparing between the unrecrystallised and recrystallised micro-textures of the various subsets of the CD80 and CD90 samples, it is immediately apparent that with the progression of rapid annealing, an overall weakening in texture intensity occurs and that the orientations tend to be distributed over a larger angular range. In general, the above features are in
broad agreement such that the first orientations to be affected by rapid annealing are of the high stored energy \{1\over 0\} \langle 1\over 0\rangle and \{1\over 1\} \langle u\over v\over w\rangle types.

As seen in the EBSD maps of the CD80(0.07), CD80(0.48), CD90(0.04) and CD90(0.37) samples (Figs. 3.2(a, c, b and d)) as well as the deconstructed subsets shown in Fig. 3.3 (for CD80(0.07)) and Fig. 3.5 (for CD90(0.04)), nucleation occurs mainly at grain boundaries, with relatively few and limited nucleation events located at regions of localised higher strain such as shear bands. The evolution of the recrystallisation texture described above is more or less consistent with the conventionally accepted stored energy and Taylor factor (M) considerations; where the \{0\over 0\} < \{1\over 1\over 2\} < \{1\over 1\} < \{1\over 1\over 0\} orientations are ranked from lowest to highest stored energy and M value [69-73]. However, variations from the above generalisations are also noted.

During the initial stages of annealing, the orientations of the newly nucleated fractions in both the CD80 and CD90 samples are largely centered around the high stored energy \{1\over 1\over 0\}\langle 1\over 1\rangle and \{1\over 1\\} \langle 1\over 1\rangle orientation components. However, right from the very beginning to approximately mid-way through the rapid annealing process, the weak presence of low \{0\over 0\}\langle 1\over 1\rangle and even lower \{1\over 1\\}\langle 0\over 0\rangle stored energy orientation components is detected. To this end, the CD90 samples heated at 20 and 10 °Cs⁻¹ to approximately full recrystallisation (Fig. 3.14(h) and 3.18(a)) also show the weak \{0\over 0\}\langle 1\over 1\rangle and even weaker \{1\over 1\\}\langle 0\over 0\rangle orientation components.

In the literature, the operation of high-energy nucleation involving the subgrain growth and coalescence mechanisms that are favoured near grain boundaries, shear bands, particles and other high dislocation density sites leads to the formation of the \{1\over 1\} or \{1\over 1\} components during recrystallisation [74]. On the other hand, low-energy nucleation occurs via the bulging of a facet of a pre-existing grain boundary and is associated with strain-induced boundary migration. It favours the formation of \{0\over 0\} and \alpha-fiber orientation components [75]. Thus, irrespective of the cold working, the CD80 and CD90 samples present with recrystallisation textures that is reminiscent of both, high-energy and low-energy nucleation mechanisms.
At the center of the sample cross-section where the EBSD maps were conducted, localised areas can undergo axisymmetric deformation during cold drawing. The prevalence of such deformation conditions can promote the formation of \(\{001\}(110)\) orientations during recrystallisation via rapid annealing [76-80]. Similar to current observations, earlier studies have also found that the nucleation and subsequent growth of small, equiaxed grains with this orientation occurs in a necklace-type morphology at the grain boundaries and around pan-caked and elongated deformed grains that have undergone significant recovery [81, 82]. In the case of rapid annealing, the occurrence of significant recovery has been shown to severely hinder the initiation and progress of recrystallisation during the initial stages of heating by reducing the dislocation density in the vicinity of grain boundaries and the driving pressure available for recrystallisation. In this regard, since the \(\{001\}(110)\) deformed grains do not have significant deformation-induced intragranular orientation gradients, the rate of nucleation of recrystallised grains with \(\{001\}(110)\) orientation will also be smaller as the formation of more mobile, high-angle boundaries is restricted [83].

On the other hand, the recrystallisation of the Goss component ((110)[001]) carried over from the unrecrystallised matrix is characteristic of the heterogeneous nature of induced deformation in severely cold drawn \(\beta\)-Ti. Typical of bcc materials, the presence of deformed grains with the stable Goss orientation occurs in small volume fractions and is due to the prevalence of strong shear -type deformation conditions during cold drawing and the build-up of local regions of high equivalent strain [84]. As shown in previous studies on commercial steel grades [85] and other non-oriented electrical steels [86], the nucleation of this component in the recrystallised fraction is exacerbated under conditions of rapid annealing. Thus in local regions, the limited recovery experienced by some deformed grains results in their unreleased stored elastic energy providing the driving force for nucleation of low stored energy orientation components. As seen in the this work, islands of newly nucleated grains with the Goss orientation grow into the heavily deformed matrix [87]. However, with
the progress of recrystallisation, the intensity of the Goss component also reduces in the ODFs of the recrystallised grains. This is ascribed to the fact that with rapid annealing to higher peak temperatures, the deformed grains in the material undergo even further recovery such that the nucleation and growth of such components at favorable locations with higher elastic stored energy reduces drastically.

4.2 Effect of heating rate

Unlike isothermal annealing in which β-grain grow non-uniformly and there is the likelihood of precipitation of the α and ω phases, continuous heating of LCB-Ti with the heating rate above 5 °C.s⁻¹ provides an overall finer recrystallised β-grain microstructure and eliminates the tendency of α and ω-phase precipitation [11]. In the present study, heating rates of 10 and 20 °C.s⁻¹ resulted in a single phase microstructure with fine β-grain sizes. However, rapid heating to the same peak annealing temperature of 820 °C at the lower heating rate of 10 °C.s⁻¹ produced 1.7 times larger recrystallised β-grain size. Other words, the high heating rate proved more beneficial in ensuring smaller β-grain sizes after recrystallisation. This phenomenon was also observed in LCB-Ti [12] after two heating rates of 20 °C.s⁻¹ and 50 °C.s⁻¹ such that the grain size of the samples after heating at 20 °C.s⁻¹ were approximately two times larger than those produced after heating at 50 °C.s⁻¹ to the same peak annealing temperature in the range between 950 and 1000 °C. This may be attributed to the greater time available for grain growth at lower heating rates.
Chapter 5
General conclusions and future work

5.1 General conclusions

In the present study, the microstructure and texture of the LCB-Ti alloys after TMP including β solution treatment at 850 °C for 1800 s, cold drawing to 80 and 90% reduction in diameter and rapid resistance heating to select peak temperatures at rates of 10 and 20 °C.s⁻¹ were examined by a combination of EBSD and XRD. For the first time, the EBSD maps of an LCB β-Ti alloy have been separated into deformed, recovered, newly nucleated and growing grain subsets by applying a novel in-house developed deconstruction methodology. Furthermore, XRD was also used to obtain the bulk texture of the LCB β-Ti alloy using another in-house developed experimental methodology that calculates the experimental ODFs after accounting for background, irradiated volume and defocusing errors. The technique to correct for defocusing errors was developed such that the correction function \( U(\alpha) \) can be determined without the need for a random sample of the same studied material. The microhardness of LCB-Ti alloys after each annealing conditions was measured by the Vickers hardness test. The correlation between microstructure and mechanical properties was estimated by the modified Hall-Petch equation. To the best of the author's knowledge, the latter has also been employed for the first time in β-Ti alloys. The research also includes the effect of cold working and heating rate on the microstructure and texture of LCB-Ti. The relationship and the interplay between microstructure, morphology, grain boundary characteristics and the estimated mechanical properties have also been characterised. The results provide new insights into the recrystallisation and grain growth behaviour of LCB-Ti during rapid annealing as follow:
(1) Static discontinuous recrystallisation occurred during the rapid annealing of CD80 and CD90 LCB-Ti.

(2) During the rapid annealing of heavily cold drawn LCB-Ti, recovery plays an important role during the initial stages of recrystallisation. As annealing progresses, role of recovery reduces very gradually.

(3) The heterogeneous microstructures after partial recrystallisation were found to contain coarse unrecrystallised grains with large aspect ratios separated by packets of fine unrecrystallised grains with even larger aspect ratios as well as approximately equiaxed recrystallised grains. In most cases, the coarse unrecrystallised grains contained in-grain shear bands which were found to be typically inclined between 20° and 45° to the drawing direction. A fewer number of shear bands were noted in the CD80 samples as compared to the CD90 samples as a direct result of the lower strain associated with lesser cold drawing reduction.

(4) The newly nucleated (sub)grains in CD80 and CD90 samples are formed mostly at the grain boundaries of deformed grains; with few nucleation events occurring at the in-grain shear bands. The density of newly nucleated (sub)grain in the CD90 samples is approximately double the density of newly nucleated (sub)grains in the CD80 samples due to the higher defect density in the former as a result of greater cold drawing reduction. In the nearly full recrystallised condition, the average equivalent grain size of the CD80(0.92) sample is nearly 1.5 times larger than that of the CD90(0.90) sample.

(5) The apparent growth rates and activation energy across deformed-recrystallising interfaces is higher than those across recovered-recrystallising interfaces due to the higher mobility and driving pressure for recrystallisation across the former interfaces. The value of apparent activation energy for boundary migration in the case of the
newly nucleated grains is slightly smaller than the growing grains due to the observation of solute drag effects in the latter case.

(6) The bulk and micro-textures of cold drawn and recrystallised conditions in CD80 and CD90 LCB-Ti after rapid heating showed the characteristic ⟨110⟩ fiber of cold drawn bcc materials.

(7) During the initial stages of annealing, the orientations of the newly nucleated fractions in both the CD80 and CD90 samples are largely centered around the high (⟨110⟩[110] and ⟨111⟩[110]) and low (⟨001⟩⟨110⟩ and ⟨110⟩[001]) stored energy orientation components. However, right from the very beginning to approximately mid-way through the rapid annealing process, the weak presence of low stored energy ⟨001⟩⟨110⟩ and ⟨110⟩[001] orientation components was detected. To this end, the CD90 samples heated at 20 and 10 °Cs⁻¹ and the CD80 sample heated to 20 °Cs⁻¹ to approximately full recrystallisation also showed the persistence of ⟨001⟩⟨110⟩ and ⟨110⟩[001] orientation components.

(8) An increase of heating rate from 10 to 20 °C.s⁻¹ to 820 °C resulted in a 1.7 times smaller final recrystallised grain size and no difference in final recrystallised texture.

(9) Microstructural statistics from EBSD were used in the modified Hall-Petch equation were applied to fit and estimate strength of Ti-LCB alloy. The fit calculated from the Hall-Petch relationship is in general agreement with the values determined experimentally from Vickers hardness. The contribution from LAGBs to the true stress was found to decrease with the progress of recrystallisation.

5.2 Future work

In order to facilitate greater understanding of the interplay between: (i) grain refinement via TMP, (ii) recrystallisation behaviour and the adopted experimental strategies and, (iii) the overall evolution of mechanical properties, the following recommendations can be suggested for future work:
- Beta solid solutioning could be conducted by rapid heating at heating rates of 10 °C.s\(^{-1}\) to 850 °C in order to eliminate the primary α-phase in the initial microstructure and achieve an even finer β grain size after this step.

- Because of the feasibility of room or elevated temperature severe plastic deformation (SPD) techniques such as equal-channel angular pressing (ECAP), large strains could be imparted to the LCB-Ti in order to obtain even higher stored energies for subsequent recrystallisation. Also, since SPD techniques have different strain paths, the effect of the initial deformation microstructure produced by the different strain paths on the subsequent recrystallisation behaviour could be studied in detail.

- As microstructural evolution corresponds very closely with the changes in the temperature at which the various TMP steps are undertaken, in-situ heating experiments in the field-emission gun scanning electron microscope (FEG-SEM) or X-ray diffractometer could be used to observe the evolution of the microstructure and crystallography of LCB-Ti samples during plane strain, uniaxial deformation and/or annealing.
References


