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Microstructure, texture and vacancy-type defects in severe plastic deformed aluminium alloys

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University of Wollongong

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Microstructure, Texture and Vacancy-type Defects in Severe Plastic Deformed Aluminium Alloys

A thesis submitted for the award of the degree of

Doctor of Philosophy

from
UNIVERSITY OF WOLLONGONG

by

Lihong Su
BEng, MEng

School of Mechanical, Materials and Mechatronic Engineering
Faculty of Engineering

2012
Declaration

I, Lihong Su, declare that this thesis, submitted in fulfilment of the requirements for the award of Doctor of Philosophy, in the School of Mechanical, Materials and Mechatronic Engineering, University of Wollongong, Australia, is wholly my own work unless otherwise referenced or acknowledged, and has not been submitted for qualifications at any other university or academic institution.

Lihong Su
August 2012
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Abstract

Severe plastic deformation (SPD) as a technique to process ultrafine grained (UFG) materials has attracted significant interest over recent years. Equal channel angular pressing (ECAP) and accumulative roll bonding (ARB) are two of the most important SPD techniques. Despite that many studies have been conducted on both techniques, some research areas have still not been explored deeply, such as the influence of low pressing temperature on ECAP, the deformation behaviour of the same material in monotonic material sheets or laminated composite sheets during ARB and the evolution of point defects during SPD. The present work investigates the microstructure, mechanical properties, texture, and vacancy-type defects evolution in detail in UFG aluminium alloys processed by ECAP and ARB.

The UFG AA1050 billets were processed by ECAP at both room temperature (RT-ECAP) and cryogenic temperature (CT-ECAP) to study the effect of cryogenic temperature on the properties of the materials. Substantial grain refinement was achieved after both RT-ECAP and CT-ECAP deformation. The average grain size after CT-ECAP is comparable but slightly smaller than after RT-ECAP and the CT-ECAP processed samples have a higher hardness and tensile strength than the RT-ECAP processed samples deformed to the same strain. A high fraction of vacancy-type defects were detected in both RT-ECAP and CT-ECAP deformed samples by positron annihilation lifetime spectroscopy (PALS). The increased hardness of the CT-ECAP processed samples can be attributed to the existence of bulk mono- and di-vacancies in the samples. The textures developed after CT-ECAP are completely different from those after RT-ECAP. Three fibres $f_1C$, $f_2C$ and $\gamma_C$ can be used to characterise the texture components developed for CT-ECAP processed samples.
The UFG AA1050 sheets were processed by ARB. The properties of AA1050 after ECAP and ARB deformation to the same level of strain were compared. The grain size of the AA1050 after ARB is smaller than those after RT- and CT-ECAP to the same strain, while the hardness and tensile strength are higher, indicating that the ARB process is more efficient in grain refinement and strengthening compared with ECAP. The positron lifetime of the ARB processed AA1050 is smaller than those for the RT- and CT-ECAP processed AA1050 due to the elevated deformation temperature during ARB.

The UFG AA1050 sheets, AA6061 sheets and AA1050/AA6061 composite sheets were processed by ARB. The properties of AA1050 and AA6061 deformed in the monotonic material sheets and the composite sheets were compared and it has been found that the grain size and the hardness values of the AA1050 and AA6061 layers in the composite sheets are similar to those in the corresponding monotonic sheets. The tensile strengths of the composite sheets agree well with the rule of mixture. The positron lifetimes of the ARB processed AA1050/AA6061 composites are also in between those of the ARB processed AA1050 and AA6061 sheets.
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<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>ANSTO</td>
<td>Australian Nuclear Science and Technology Organisation</td>
</tr>
<tr>
<td>ARB</td>
<td>Accumulative roll bonding</td>
</tr>
<tr>
<td>BCC</td>
<td>Body-centred cubic</td>
</tr>
<tr>
<td>CCW</td>
<td>Counter-clock-wise</td>
</tr>
<tr>
<td>CDRX</td>
<td>Continuous dynamic recrystallisation</td>
</tr>
<tr>
<td>CMWP</td>
<td>Conventional multiple whole profile</td>
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<tr>
<td>CT-ECAP</td>
<td>Cryogenic temperature ECAP</td>
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<tr>
<td>DPD</td>
<td>Dynamic plastic deformation</td>
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<tr>
<td>DR</td>
<td>Distorted region</td>
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<tr>
<td>DSC</td>
<td>Differential scanning calorimetry</td>
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<tr>
<td>DTM</td>
<td>Diffusion trapping model</td>
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<tr>
<td>EBSD</td>
<td>Electron backscatter diffraction</td>
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<tr>
<td>ECAP</td>
<td>Equal channel angular pressing</td>
</tr>
<tr>
<td>ED</td>
<td>Extrusion direction</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy dispersive X-ray spectroscopy</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy dispersive X-ray</td>
</tr>
<tr>
<td>FCC</td>
<td>Face-centred cubic</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full width at half maximum</td>
</tr>
<tr>
<td>GB</td>
<td>Grain boundary</td>
</tr>
<tr>
<td>GNB</td>
<td>Geometrically necessary boundaries</td>
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<tr>
<td>HAGB</td>
<td>High angle grain boundary</td>
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<tr>
<td>HPT</td>
<td>High pressure torsion</td>
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<tr>
<td>HRTEM</td>
<td>High resolution TEM</td>
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<tr>
<td>IDB</td>
<td>Incidental dislocation boundaries</td>
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<tr>
<td>IPF</td>
<td>Inverse pole figure</td>
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<tr>
<td>LAGB</td>
<td>Low angle grain boundary</td>
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<tr>
<td>MMC</td>
<td>Metal matrix composites</td>
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<tr>
<td>MWP</td>
<td>Multiple whole profile</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
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<td>----------------------------------</td>
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<tr>
<td>ND</td>
<td>Normal direction</td>
</tr>
<tr>
<td>NR</td>
<td>Non-distorted region</td>
</tr>
<tr>
<td>ODF</td>
<td>Orientation distribution function</td>
</tr>
<tr>
<td>OM</td>
<td>Optical microscopy</td>
</tr>
<tr>
<td>PALS</td>
<td>Positron annihilation lifetime spectroscopy</td>
</tr>
<tr>
<td>PDW</td>
<td>Polygonised dislocation wall</td>
</tr>
<tr>
<td>PF</td>
<td>Pole figure</td>
</tr>
<tr>
<td>PSC</td>
<td>Plane strain compression</td>
</tr>
<tr>
<td>PTB</td>
<td>Partially transformed boundary</td>
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<tr>
<td>RD</td>
<td>Rolling direction</td>
</tr>
<tr>
<td>RER</td>
<td>Residual electrical resistivity</td>
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<tr>
<td>RT-ECAP</td>
<td>Room temperature ECAP</td>
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<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
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<tr>
<td>SFE</td>
<td>Stacking fault energy</td>
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<tr>
<td>SAD</td>
<td>Selected area diffraction</td>
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<tr>
<td>SPD</td>
<td>Severe plastic deformation</td>
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<tr>
<td>SRS</td>
<td>Strain rate sensitivity</td>
</tr>
<tr>
<td>STM</td>
<td>Standard trapping model</td>
</tr>
<tr>
<td>TD</td>
<td>Transverse direction</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>UFG</td>
<td>Ultrafine grained</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate tensile strength</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray diffraction</td>
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Chapter 1 Introduction

The average grain size of materials generally plays a significant role on the mechanical properties of all crystalline materials. According to the well-known Hall-Petch equation, the strength increases with a reduction in the grain size [1]. Thus fabricating materials with a grain size in the nano-range (less than 100 nm) or ultrafine range (between 100 nm and 1 μm) is an attractive approach to increase the strength of materials [2].

Severe plastic deformation (SPD) is used to process ultrafine grained (UFG) materials from coarse grained metals and alloys [3]. The process is capable of generating large plastic strains that significantly refines the microstructure without altering the external dimensions of the specimens. Many different SPD processing techniques have been developed, including equal channel angular pressing (ECAP) [4-16], high pressure torsion (HPT) [17-23], multi-directional forging [24-28], accumulative roll bonding (ARB) [29-39], and many others. All of these procedures are capable of introducing large plastic strains and obtaining significant microstructural refinement in bulk crystalline materials. Of these various procedures, ECAP and ARB are the most commonly used techniques.

Aluminium and its alloys have great potential in automotive and aerospace industry due to their light-weight nature, good formability, good corrosion resistance and low cost [40-42]. However, the application is limited mostly due to their low strength compared with other metallic materials. Therefore, using SPD techniques to improve the strength of aluminium and its alloys should expand their usage in these industries.
Chapter 1 Introduction

Over recent years, ECAP has successfully been applied to aluminium and its alloys to achieve high strength in these materials. The influence of various ECAP parameters on the properties has been well studied, including the number of pressings, ECAP die geometry, processing route, pressing speed, pressing temperature and the application of back pressure. However, studies on the influence of pressing temperature were mostly reported at room temperature and higher, and investigations of pressing at lower than room temperatures have been seldom reported. In this work, AA1050 was processed by ECAP at both room temperature and cryogenic temperature in order to study the effect of cryogenic temperature on the properties of the materials. The evolution of vacancy-type defects during ECAP deformation was investigated as it was reported that vacancy-type defects have a great influence on the strengthening of the materials but has rarely been studied in detail.

ARB offers an opportunity to produce multilayered composites with two different starting materials. To date a number of laminated composites processed by ARB have been reported, including the same metal based materials as well as different metals. However, differences in the deformation behaviour of the two starting materials in the composite with respect to their monotonic material laminates have not been thoroughly compared. In this work, AA1050 and AA6061 were processed by ARB to produce three kinds of laminates: AA1050 sheets, AA6061 sheets and AA1050/AA6061 composites. The properties for the three kinds of laminates were investigated and the deformation behaviour of AA1050 and AA6061 in the monotonic material laminates and the composites was compared.

This thesis consists of 9 chapters as follows:
Chapter 2 presents a brief overview of two of the SPD techniques: ECAP and ARB, and the evolution of the microstructure, mechanical properties, texture, and vacancy-type defects during deformation.

Chapter 3 describes the starting materials, the experimental procedures and the types of testing equipment used for characterisation.

Chapter 4 presents the results of AA1050 subjected to room temperature ECAP (RT-ECAP). The evolution of the microstructural and mechanical properties, texture, and vacancy-type defects with the number of RT-ECAP passes are presented. Vacancy-type defects evolution and their influence are discussed in detail.

Chapter 5 focuses on the results of AA1050 subjected to cryogenic temperature ECAP (CT-ECAP). The evolution of the microstructural and mechanical properties, texture, and vacancy-type defects with the number of CT-ECAP passes are analysed and are compared with those after RT-ECAP.

Chapter 6 shows the investigation of AA1050 sheets after ARB processing. The results of microstructural and mechanical properties, texture, and vacancy-type defects are presented. The properties of the AA1050 sheets are compared with the AA1050 deformed by ECAP to the same strain.

Chapter 7 shows the results of ARB processed AA6061 sheets. The results of microstructural and mechanical properties, texture, and vacancy-type defects are compared with those of the AA1050 sheets. Through-thickness heterogeneities of microstructure, hardness and texture due to high roll surface roughness are well discussed.
Chapter 8 studies the properties of ARB processed AA1050/AA6061 laminated composites. The properties of AA1050 and AA6061 layers are compared with their monotonic material laminates presented in Chapters 6 and 7. The microstructures of the AA1050/AA6061 interfacial areas and the bonding properties are presented in detail. Comparison of the mechanical properties and vacancy-type defects with the monotonic material laminates are discussed in detail.

Chapter 9 summarises the research findings obtained in this thesis, and some suggestions for future work are also offered.
Chapter 2 Literature review

SPD has been applied to metals and alloys to obtain UFG and nanocrystalline materials for many years. Ultra high plastic strains are introduced into bulk metallic materials during SPD and substantial grain refinement can be achieved, which significantly strengthens the materials. ECAP and ARB are two of the most widely used SPD techniques.

Aluminium and its alloys have attracted attention for many years as promising structural materials for automotive and aerospace applications due to their lightweight nature, good formability, good corrosion resistance and low costs. The present work has been conducted on aluminium alloys AA1050 and AA6061. Therefore, the following review is focused on UFG aluminium and its alloys processed by ECAP and ARB.

2.1 Equal channel angular pressing

2.1.1 Equal channel angular pressing deformation

ECAP was first developed by Segal et al. to homogenise the microstructures of cast billets [4]. The principle of ECAP is illustrated schematically in Fig. 2.1. During ECAP, a sample is pressed through a die with two equal cross section channels intersected with each other. The inner angle at the intersection is defined as $\Phi$, the outer arc of curvature is $\Psi$, and both of the angles are adjustable. The sample, in the form of a rod or bar, is machined to fit within the channel and is pressed through the die using a plunger. The pressing is repeatable because there is no change in the cross-sectional dimension, unlike other conventional deforming processes such as rolling,
Chapter 2 Literature review

extrusion and drawing. Large strains can be accumulated in bulk material by repetitive ECAP deformation.

![Fig. 2.1 Schematic illustration of the ECAP process.](image)

One parameter used to measure the degree of deformation is the equivalent strain imposed on materials after ECAP. The equivalent strain after \( N \) passes can be expressed by the relationship [43]:

\[
\varepsilon_N = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi \csc \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) \right]
\]  

(2.1)

where \( \varepsilon_N \) is the equivalent strain, \( N \) is the number of passes, \( \Phi \) is the inner angle of the intersection, and \( \Psi \) is the outer curvature of the die.

It can be seen from Eq. (2.1) that the equivalent strain depends on the number of ECAP passes, and the inner and outer angle of the die. The channel angle \( \Phi \) was studied from 60° up to 157.5° and it was found that deformation was more intense at a lower angle, but it was easier to deform when the channel angle was higher [44-46]. The presence of the outer angle of curvature \( \Psi \) will increase the microstructural inhomogeneities but its effect will be compensated by the “dead
zone” observed in many experiments [47-49]. Φ=90° and Ψ=20° is the most frequently used die geometry. Each pressing using this type of die corresponds to an equivalent strain of approximately 1. Repeating ECAP pressing several times will result in a large accumulated strain and refine the grains substantially. There are other factors influencing the ultimate microstructure and mechanical properties of the materials besides accumulated strain, such as the processing route [50-57], pressing speed [58, 59], pressing temperature [8, 60-63] and the use of back-pressure [64-67].

2.1.2 Processing route of equal channel angular pressing

There are four processing routes in ECAP: route A where the sample is pressed repetitively without any rotation, route B_A where the sample is rotated 90° in alternate directions between consecutive passes, route B_C where the sample is rotated 90° in the same way between each pass and route C where the sample is rotated by 180° between passes, as depicted in Fig. 2.2. [68]. The effect of the processing route on the material properties has been well studied [50-57]. Models of grain refinement and shear characteristics for different routes were developed and compared with the experimental results [55-57]. It has been found that when using route B_C, the material is shear deformed in sequence on four planes of the material, and this is repeated after every 4 passes. Therefore, route B_C leads most rapidly to an array of reasonably equiaxed ultrafine grains with high angle grain boundaries (HAGBs) and has been widely used thereafter [50, 69-73].
2.1.3 Microstructure evolution during equal channel angular pressing

The microstructural evolution of pure aluminium deformed by ECAP using route B\textsubscript{C} has been systematically studied. It has been found that elongated subgrains with low angle grain boundaries (LAGBs) were formed at low strains and equiaxed grains with HAGBs of about 1 µm were formed at high strains (from the fourth pass on), as can be seen in Fig. 2.3.
Fig. 2.3 TEM microstructure and corresponding SAD patterns in pure Al after route B\textsubscript{C} ECAP deformation for (a) 1, (b) 2, (c) 3 and (d) 4 passes [50].

Chang et al. [7] studied the boundary morphologies of the generated boundaries during ECAP deformation. As can be seen in Fig. 2.4, the grains developed from an elongated shape into an equiaxed shape with increasing strain. At the same time, the grain size and the dislocation density in the grain interiors decreased. As indicated in Fig. 2.4(d), three types of boundaries were observed in the 8-pass room temperature ECAP processed samples, namely the polygonised dislocation wall (PDW), the partially transformed boundary (PTB) and the grain boundary (GB).
The polygonised dislocation walls transformed into partially transformed boundaries and then into grain boundaries with increasing strain. It was concluded that most of the generated boundaries had transformed into grain boundaries at an equivalent strain of 8, and 95% of the grain boundaries had a misorientation angle larger than $10^\circ$ [7].

![TEM micrographs showing the structure evolution at an equivalent strain of (a) 2, (b) 4, (c) and (d) 8 [7].](image)

Although in theory the ECAP process can repeat many times, in practice, 8 pressings are sufficient for most materials to obtain a saturated state. Metals and alloys cannot be refined indefinitely due to dynamic recovery during processing. A grain size of $\sim1 \mu m$ was reported for pure aluminium after ECAP processing of 4 and more passes [46, 74, 75]. Therefore, other experimental conditions need to be attached to the ECAP processing steps or other techniques need to be combined with ECAP for further grain refinement.
2.1.4 Influence of deformation temperature on equal channel angular pressing

The effect of the pressing temperature on the microstructure and mechanical properties of ECAP processed specimens were studied extensively [8, 60-63]. Generally, as the temperature increases, pressing becomes easier but the equilibrium grain size would be larger [8, 60-63], as can be seen in Fig. 2.5 and Fig. 2.6. However, different conclusions were made on the distribution of HAGBs with temperature. Although an increase in the fraction of HAGBs was reported in Ref. [62], most of the experiments showed a decrease in the fraction of HAGBs [8, 61, 63]. Generally, the lowest possible pressing temperature should be used to achieve substantial grain refinement, even though specimens are easier to press at high temperature [60]. Most of the reported ECAP deformation of commercial purity aluminium was conducted at room temperature [51, 76-80].

![Fig. 2.5 Microstructures and associated SAD patterns for pure Al after ECAP deformation through a 90° die using route Bc up to 6 passes, at (a) room temperature, (b) 100 °C, (c) 200 °C and (d) 300 °C [61].](image-url)
Fig. 2.6 TEM micrographs of commercial purity aluminium after ECAP deformation through a 120° die with route A up to 8 passes at (a) 25 °C, (b) 100 °C, (c) 200 °C, (d) sketch of HAGB distribution in (c), (e) 250 °C, and (f) sketch of HAGB distribution in (e) [63].

The steady state grain size of pure aluminium subjected to ECAP at room temperature can be achieved after approximately 4 passes. Further pressings can only change the grain shape to equiaxed and increase the fraction of high angle grain boundaries but cannot refine the grain size [46, 75]. For aluminium, high
dynamic recovery occurs at room temperature, which limits the grain size. To further refine the microstructure, additional plastic deformation by cold rolling at the temperature of liquid nitrogen was always combined with ECAP at room temperature or a slightly elevated temperature [81-91]. The use of liquid nitrogen can restrict the dynamic recovery that opposes the accumulation of dislocations and grain boundaries. Therefore, rolling at the temperature of liquid nitrogen enables refine the grain size to levels beyond what can be achieved by ECAP at room temperature. Fig. 2.7 shows the microstructure of Al-0.13%Mg alloy deformed by ECAP and followed by plane strain compression (PSC) deformation at room temperature and lower. It shows that PSC further refines the ECAP processed microstructure and the grain size decreases with decreasing PSC temperature [87, 88].

**Fig. 2.7** EBSD map showing the UFG structure of Al-0.13Mg alloy after ECAP deformation through a 120° die with route A up to 15 passes (a) as-ECAP processed, and followed by PSC to a strain of 2.1 at (b) 298 K, (c) 213 K and (d) 77 K [87].
During deformation at cryogenic temperature, dislocation gliding is suppressed and other deformation techniques are effective. Li et al. reported that when pure copper was deformed by dynamic plastic deformation (DPD) at cryogenic temperature, plastic deformation was achieved by twinning and shear banding at medium to high strains [92]. It was also reported by Subramanya Sarma et al. that metals with intermediate stacking fault energies (SFEs) were deformed by twinning during cryogenic temperature rolling but by dislocation slipping during room temperature rolling [93]. The textures of materials with high SFEs after cryogenic deformation were also transferred to typical textures of materials with low SFEs [88].

ECAP deformation at cryogenic temperature was also reported by Mabuchi et al. [94], Chen et al. [95] and Chen et al. [96]. It was reported that high dislocation density was observed in the grain interiors after ECAP at cryogenic temperature due to the restraining of dynamic recovery. Cryogenic temperature ECAP processed samples also have a higher hardness than those after room temperature ECAP processing [95]. Twins were observed in Al-1 wt.% Mg alloy for the first time [96].

2.2 Accumulative roll bonding

2.2.1 Accumulative roll bonding deformation

ARB as a kind of SPD technique has been drawing a lot of attention since it was first introduced by Saito et al. [97]. Using the same equipment as conventional rolling, ARB is considered to be one of the most promising methods for manufacturing UFG sheet materials [98-100]. The ARB process is shown schematically in Fig. 2.8. During ARB, rolling is conducted on two sheets with the same dimensions and which have been stacked together beforehand. The rolling process not only provides large
plastic deformation but also bonds the two sheets together. The bonded specimen of each cycle is prepared by cutting, surface degreasing, brushing, and stacking together for the next cycle [97, 98]. The reduction in thickness after each pass is approximately 50% and the increase in width is negligible, thus the thickness of the bonded materials should remain the same during the whole rolling process. Since in theory the number of repetitions is endless, it is possible to achieve ultrahigh strain with the ARB process.

![Diagram of ARB process](image)

**Fig. 2.8** Schematic illustrations of the ARB process.

Assume the sample is deformed in a plain strain condition, the equivalent strain $\varepsilon_{eq}$ can be calculated as follows [98]:

$$
\bar{\varepsilon}_{eq} = \frac{2}{\sqrt{3}} n \ln \frac{h_0}{h} = \frac{2}{\sqrt{3}} n \ln \frac{1}{1-r}
$$

(2.2)

where $h_0$ is the initial thickness of the stacked sheets, $h$ is the thickness after roll-bonding, $r$ is the reduction in thickness per cycle and $n$ is the number of ARB cycles. The reduction per ARB cycle is usually $r = 50\%$, which results in an equivalent strain of 0.8 per cycle.

**2.2.2 Microstructure evolution during accumulative roll bonding**

ARB processed materials generally have grains elongated to rolling direction, and with grains less than 1 µm in size. Two types of boundaries were identified: the lamellar boundaries were approximately parallel to the rolling direction and the
short transverse boundaries interconnected the lamellar boundaries. The spacing of both boundaries decreased with an increasing number of ARB cycles [101]. Large misorientation exists between the lamellar grain boundaries. However, the interconnecting grain boundaries consist of a mixture of low- and high-angle boundaries [101]. Fig. 2.9 shows the typical microstructures of ARB processed AA6061 with different cycles.

![TEM microstructures of ARB processed AA6061 by (a) 2, (b) 4, (c) 6 and (d) 8 cycles](image)

Fig. 2.9 TEM microstructures of ARB processed AA6061 by (a) 2, (b) 4, (c) 6 and (d) 8 cycles [42].

### 2.2.3 Shear strain distribution of accumulative roll bonding processed sheets

Since the ARB deformation is usually conducted without any lubrication to improve bonding, a redundant shear strain is introduced close to the surfaces of the sheet. The final shear strain distribution is complex because half the surfaces of the sheets move to the centre in the following cycle and then move progressively outwards.
with further deformation. Shear strain distribution through the sheet thickness of the ARB processed materials were investigated by embedded pin tests (shown in Fig. 2.10) and finite element simulation [42, 102-104]. The typical through-thickness shear strain distribution is shown in Fig. 2.11. It can be seen that the shear strain is high close to the surface areas and the layer interfaces. The shear strain distributes inhomogeneously through the thickness, which will lead to inhomogeneous distribution of microstructure, hardness and texture throughout the thickness of the ARB processed sheets [104-107].

![Embedded pin](image)

**Fig. 2.10** Optical microstructures of the flections of the embedded pin in the AA1100 sheet after 1-cycle ARB at room temperature without lubricant [102].
Chapter 2 Literature review

2.2.4 Processing laminated composites by accumulative roll bonding

ARB was developed to introduce large strains into sheet materials. However, as the process also bonds two pieces of materials together, the starting materials can be the same or dissimilar metals or alloys. Thus this process can also be used to bond two different kinds of materials so that different properties of the two dissimilar materials would combine. Many studies have been done in this area [108-119].

Dissimilar metal based materials were used as starting materials in ARB to produce multilayered composites. Al/Mg [108], Al/Cu [109], Al/Ni [110], Al/Zn [111], Al/Ti [120], Cu/Ag [112], Cu/Zr [112], Fe/Cu [113] and other laminated composites have

Fig. 2.11 Shear strain distribution through the thickness of AA1100 after (a) 1, (b) 2, (c) 4 and (d) 8-cycle ARB deformation [102].
been produced by ARB. Different grades of Al based alloys were also used to process laminated composites [114, 118, 119, 121-123]. Metal matrix composites (MMCs) such as Al/Al₂O₃ [124-126], Al/B₄C [127-129] and Al/SiC [130-132] were also fabricated by ARB by spreading the particles between the interfaces and repeating the ARB process many times to ensure that the particles were uniformly distributed in the metal matrix.

One factor of the layered composites that is different from monotonic laminates is that the initial properties and the deformation behaviour of the two dissimilar materials are different, which induces different microstructure and mechanical properties of the two different layers. Fig. 2.12 shows an example of the ARB processed AA1050/AA5754 composite [118]. AA5754 was used as the core material and AA1050 was used as lateral materials. As can be seen in Fig. 2.12(b) and (c), the microstructures and hardness of the two alloys after roll bonding are quite different. This difference can also result in necking and fracturing of the harder layers, as has been observed in many studies [108, 110, 114, 122, 133]. Some researchers used this phenomenon to produce composites with fragments of the harder layers distributed in the softer layer matrix [111, 115-117, 120], as can be seen in Fig. 2.13.
Fig. 2.12 (a) Schematic drawings for the production of AA1050/AA5754 sandwich, (b) SEM micrograph of sandwich-like laminates at the interface and (c) the hardness profile across the interface of AA1050/AA5754 laminates [118].

Fig. 2.13 SEM backscattered images of Ti/Al multilayered composites after different numbers of ARB cycles [120].
2.3 Grain refinement during severe plastic deformation

When a metal is plastically deformed, significant changes can be observed in the microstructure, the size and shape of the grains, the relative fraction and distribution of HAGBs and LAGBs, and the density and distribution of dislocations. The grain shape is determined by the applied strain and strain path during deformation, thus the route B\_C ECAP and HPT deformation will result in near equiaxed grains [61, 134]. However, the ARB deformed samples always have a pancake shaped structure [103]. Generally, as the SPD process proceeds, the grain size decreases, while the volume fraction of grain boundaries increases, and the fraction of HAGBs also increases. The dislocation density increases at the beginning stage of SPD and decreases when the deformation proceeds to high strains [135]. The grain interiors are generally clean of dislocations at high strains [7].

When plastic deformation is subjected to a high stacking fault energy (SFE) polycrystalline metal, dislocation slip is the major deformation mechanism. A few slip systems operate and generate a large amount of dislocations. These dislocations exist within the grain interiors and form thick dislocation walls at low plastic strains. The dislocation walls further agglomerate into subgrain boundaries with subsequent plastic deformation. For SPD techniques like ECAP and ARB, subgrains surrounded by thick dislocation walls formed can be observed after only a single pass deformation, since the strain introduced in a single pass is huge. The subgrains and dislocation walls act as barriers to dislocation propagation and the misorientation angles between the subgrains gradually increase with subsequent plastic deformation. The subgrain boundaries gradually evolve into grain boundaries with further plastic strain, and most of the dislocations inside the subgrain interiors annihilate into the boundaries, which in turn leaves the grain...
boundaries clearly defined with high a misorientation angle and the grain interiors with a low dislocation density. The microstructure evolution with strain for the SPD process is shown schematically in Fig. 2.14.

![Diagram of microstructural evolution](image)

**Fig. 2.14** Schematic illustration of microstructural evolution during SPD, (a) homogeneous distribution of dislocations, (b) elongated cell formation, (c) dislocations blocked by subgrain boundaries, (d) break up of elongated subgrains and (e) reorientation of subgrain boundaries and formation of ultrafine grain size [136].

Another microstructure evolution mechanism during SPD is continuous dynamic recrystallisation (CDRX). This was used to explain grain refinement of metals severely deformed at a certain elevated temperature [137-139]. The theory consists of the formation of LAGBs and transformation of LAGBs into HAGBs. It is shown schematically in Fig. 2.15. As with the mechanism above, LAGBs are formed
by the rearrangement of accumulating dislocations and it is favourable in the shear direction, which results in elongated subgrains along the shear direction [140-142]. Such subgrain boundaries are the result of coarse slip associated with shear banding and have been termed as geometrically necessary boundaries (GNB). Transverse LAGBs are generated by dislocation rearrangement within the elongated subgrains to form fairly equiaxed shaped subgrains and they have been termed as incidental dislocation boundaries (IDB). The LAGBs aligned in the shear direction have higher misorientation angles than the transverse LAGBs at low strains [9]. With increasing strain, mobile dislocations move across the subgrains and are trapped by the subgrain boundaries which results in an increase in the misorientation. Extensive rotation of subgrains converts them into fine grains with HAGBs. Another type of subgrains which contains parts of the original HAGBs are considered to be a nucleus (shown in Fig. 2.15(c)) and are transformed into fine grains by converting all the LAGBs into HAGBs. Extensive grain rotation occurs in this type of structure which accelerates the evolution of LAGBs into HAGBs (shown in Fig. 2.15(d)).

The ARB process is similar to the conventional rolling process and the grain refinement mechanism is shown schematically in Fig. 2.16. This process is generally similar to that shown in Fig. 2.14 and Fig. 2.15, including subgrain formation, grain subdivision and transformation of LAGBs into HAGBs. The difference is that the grain boundaries formed in the rolling process tend to align in the rolling plane and eventually form a banded microstructure which is elongated along the rolling direction and has a large aspect ratio.
Fig. 2.15 Schematic representation of grain refinement in AA1421 in ECAP deformation, (a) formation of arrays of equiaxed subgrains alternating with elongated subgrains, (b) interaction of LAGB with lattice dislocations resulting in progressive increase in their misorientation, (c) a nucleus in 3D subgrain structure and (d) rotation of (sub)grains facilitates the transformation of LAGBs into HAGBs [143].

Fig. 2.16 Schematic illustration of the development of microstructure during cold rolling or plane strain compression, (a) initial grain structure, (b) subgrains and grain subdivision, (c) alignment of HAGBs, (d) ribbon grain structure and (e) break-up of ribbon grains by large second phase particles [144].
2.4 Mechanical behaviour of severe plastic deformed materials

It is well known that SPD can produce materials with an extremely high strength compared to their coarse grained counterparts. However, the ductility of the SPD processed materials is extremely low, which restricts their practical applications [46, 97, 145]. This so-called “paradox of ductility and strength” (as shown in Fig. 2.17) is applicable for nearly all UFG materials produced by SPD [81].

![Fig. 2.17 Normalised yield strength versus percentage elongation for UFG metals [81].](image)

The increased strength of UFG materials can be attributed to strain strengthening and grain refinement strengthening [146-148]. The strength $\sigma$ can be expressed by [148]:

$$\sigma = \sigma_0 + \sigma_\rho + \sigma_b$$

(2.3)

where $\sigma_0$ is the friction stress, $\sigma_\rho$ is the contribution from dislocations and $\sigma_b$ is the contribution from boundaries with medium to high boundary angles. Strain strengthening is based on the assumption of dislocation strengthening, where the strengthening is proportional to dislocation density ($\sigma_\rho \propto \rho^{1/2}$). The relationship of
strength and grain size can be specified by the conventional Hall-Petch relationship which is given by:

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

(2.4)

where \( \sigma_y \) is the yield stress, \( \sigma_0 \) is the lattice friction stress and \( k_y \) is the constant of yielding. It shows that the strength increases with a decreasing grain size \( (\sigma_b \propto d^{-1/2}) \). It is reported that for UFG materials deformed to low strains, strain strengthening is the major strengthening mechanism. At high strains, the increment of strength is mostly due to grain refinement and strain strengthening plays a less effect [146].

The ductility is controlled by two parameters: work hardening and strain rate sensitivity (SRS). Work hardening is caused by the accumulation of crystalline defects such as dislocations, which makes further deformation harder [81]. However, for UFG and nanostructured materials, the accumulation of dislocations is difficult because the generated dislocations will quickly annihilate into the grain boundaries due to the small grain size. Therefore, SPD processed materials exhibit little work hardening stage in the stress-strain curve and neck immediately after yielding. An example is shown in Fig. 2.18. It can be seen that for both ECAP and ARB processed samples, the strength increases significantly and continues to increase with the number of cycles, but the ductility drops drastically only after a single pass processing and then stays about the same. The sudden drop of strength at uniform elongation can be explained by plastic instability [149], which occurs when the Cosidère criterion is fulfilled [150]:

\[ \sigma \geq \frac{d\sigma}{de} \]  

(2.5)
where $\sigma$ and $\varepsilon$ are the true stress and true strain, respectively. For UFG materials, the strength ($\sigma$) is high and the strain hardening rate ($\frac{d\sigma}{d\varepsilon}$) is low. As a result, plastic instability happens at an early stage of tensile deformation, which limits the elongation.

![Engineering stress-strain curves](image)

**Fig. 2.18** Engineering stress-strain curves of (a) ECAP processed IF steel [151] and (b) ARB processed pure aluminium [146].

The interaction between dislocations and grain boundaries is identified as the main mechanism for the SRS and an enhanced interaction between dislocations and grain boundaries reinforces the SRS. Generally, materials that possess a high percentage of HAGBs exhibit a high SRS, and materials with a high SRS are expected to be able to resist localised deformation and have good ductility. Experiments showed that an increase in SRS was observed with increasing strain for face-
centred cubic (FCC) materials subjected to SPD [152, 153]. SRS is an important parameter for the superplasticity observed in some UFG materials [154-156]. Troger et al. conducted experiments on 6xxx aluminium alloys and found that the SRS reached a maximum of 0.5 at 540 °C, which resulted in an elongation of 375% [41].

Wang et al. prepared nanocrystalline copper through rolling at temperature of liquid nitrogen and further annealing, which improved the uniform elongation markedly without sacrificing much of the strength [86]. This result is one of the few points outside the “paradox of ductility and strength” shown in Fig. 2.17. After that, rolling at cryogenic temperature and subsequent annealing have been carried out on many materials, including commercial purity aluminium [157], AA2219 [158], AA5083 [159], AA6063 [160], Cu-Zn and Cu-Al alloy [161], pure V [90], pure Ni [162], etc. However, these experiments only exhibited a slight increase in ductility with a sacrifice of tensile strength.

Plastic deformation in coarse grained materials proceeds by dislocation slip. However, for UFG and nanocrystalline materials, the traditional deformation mechanism by dislocation slipping still reigns during the subsequent tensile deformation. Other deformation mechanisms such as grain boundary sliding and enhanced grain rotation are also proposed to take effect [163]. Grain boundary sliding and enhanced grain rotation were assumed to facilitate with dislocation sliding and twinning during the SPD of copper through 16 passes ECAP and titanium through 5 revolutions HPT [164]. The formation of deformation twins and their extensive intersections facilitated the accumulation of dislocations and resulted in the simultaneously enhanced strength and ductility of UFG Cu-Al alloys [165].
However, it is difficult to explain some of the phenomena observed by the above-mentioned mechanisms. Huang et al. [166] observed hardening by annealing and softening by subsequent deformation of nanostructured aluminium, which is in contrast to coarse grained metals where annealing leads to a decrease in strength and deformation leads to hardening. The same phenomenon was also reported for nanocrystalline nickel [167]. Horita et al. reported hardening by annealing in ultrafine grained AA1100 and AA3004 [168], and they also observed grain coarsening and a decrease of dislocation density in the mean time, which would normally lead to softening instead of hardening. This indicates that other hardening mechanisms besides dislocation hardening and grain boundary hardening must be operating. It is necessary to investigate the deformation mechanism of UFG materials.

2.5 Texture of severe plastic deformed materials

When crystalline materials are subjected to plastic deformation, the grains always tend to align in certain crystallographic orientations with respect to the overall geometry of the sample. The tendency of grains distributed at particular orientations is known as the texture of materials. The texture controls the formability of crystalline materials which means that understanding of the texture evolution is extremely important.

2.5.1 Texture representation

The texture of materials can be represented in a number of ways, among which the pole figure (PF), the inverse pole figure (IPF) and the orientation distribution function (ODF) are very common. PFs are developed by stereographic projection of crystallographic orientations onto a great circle plane of the reference sphere.
sample coordinate system is chosen to be the specific poles for the figure. For instance, the rolling direction (RD) is usually chosen to be the north pole and the transverse direction (TD) is the west pole for rolling symmetry. By contrast, IPF is the projection of the sample coordinate onto a plane which is specified with the crystal coordinate system. For cubic materials, IPF is normally presented in a unit triangle of <0 0 1>, <0 1 1> and <1 1 1> crystal directions. A detailed analysis of the texture evolution is always presented by ODFs, which separate the components that partially overlap in the PFs, allowing for a clearer comparison of components and fibres. Three Euler angles $\varphi_1$, $\Phi$ and $\varphi_2$ are defined using Bunge’s convention [169] and are plotted to represent texture orientations.

For cubic crystal structured materials, the orientation of the crystal with respect to the specimen is represented by \(\{h \ k \ l\}<u \ v \ w>\), where \(\{h \ k \ l\}\) stands for the Miller indices of the crystallographic plane parallel to the macroscopic deformation plane and \(<u \ v \ w>\) is the crystallographic direction parallel to the macroscopic deformation direction. Texture components in ECAP are defined as \(\{h \ k \ l\}\) plane parallel to extrusion direction (ED)-transverse direction (TD) plane and \(<u \ v \ w>\) direction parallel to ED. In the ARB process, however, the definition is \(\{h \ k \ l\}\) plane parallel to the rolling plane (RD-TD) and \(<u \ v \ w>\) direction parallel to the rolling direction (RD).

### 2.5.2 Texture measurement

Texture analysis is generally divided into macrotexture and microtexture analysis depending on the scale of the measured area. Macrotexture is measured from a large number of grains and refers to the bulk texture of a particular sample. Normally, macrotexture is measured by X-ray diffraction (XRD) or neutron
diffraction methods. Microtexture takes texture of individual grains and is studied by Kikuchi patterns with the electron diffraction method under an electron microscope. Electron backscatter diffraction (EBSD) under scanning electron microscopy (SEM) is more commonly used than study with transmission electron microscopy (TEM).

Both the XRD and neutron diffraction methods are based on Bragg’s law:

\[ n\lambda = 2d \sin \theta \]  

(2.6)

where \( n \) is the order of reflection, \( \lambda \) is the wavelength of the beam, \( d \) is the spacing of the diffracted planes and \( \theta \) is the corresponding Bragg angle.

The pole figures are scanned on equidistant concentric circles with a constant polar angle (0–90°) and azimuth angle (0–360°), at a step of 5° typically. For the XRD reflection mode, a polar angle 90° cannot be reached in practice, so the XRD measurement is usually measured up to a polar angle of 85°, while neutron diffraction can take measurements of the whole range. Besides this difference, neutron diffraction is a better way of representing bulk texture as the penetration depth for neutrons are much deeper than X-rays.
Fig. 2.19 (a) Sketch of polar angle $\alpha$ and azimuth angle $\beta$ in scanning setup and (b) equal-angle scanning grids for pole figure measurements (shown in steps of 20°, with conventional rolling coordinate system) [170].

EBSD is a technique that allows crystallographic information from samples to be obtained under SEM. As can be seen in Fig. 2.20, in EBSD, an electron beam hits a tilted crystalline specimen (the tilt angle is ~60–70°), and the diffracted electrons form a specific Kikuchi pattern on a fluorescent screen. The Kikuchi pattern is characteristic of the crystal structure and orientation. After further processing, some information of the sample can be obtained, including the crystal orientation and grain boundary misorientation. A quantitative representation of the sample microstructure can be obtained when the scanning is conducted in a grid across the surface of a polycrystalline sample.
2.5.3 ECAP texture

ECAP deformation is often considered to be near simple shear deformation along the intersection plane of the two channels. The ideal ECAP texture is the same as the texture for simple shear but counter-clock-wise (CCW) rotated by $\theta = \Phi/2$ around TD. The ideal texture components and fibres for simple shear and $\Phi=90^\circ$ ECAP are shown in $\{1 1 1\}$ pole figures in Fig. 2.21 and in $\phi_2=0^\circ$ and $\phi_2=45^\circ$ ODF sections in Fig. 2.22.

![Schematic diagram of a typical EBSD installation in an SEM](image)

**Fig. 2.20** Schematic diagram of a typical EBSD installation in an SEM [140].

![Pole figures showing the ideal orientations and fibres of (a) simple shear and (b) ECAP deformation](image)

**Fig. 2.21** $\{1 1 1\}$ pole figures showing the ideal orientations and fibres of (a) simple shear and (b) ECAP deformation [171].
Fig. 2.22 Relationship between the ideal positions of (a) simple shear and (b) ECAP deformation in FCC materials [172].

As a monoclinic symmetry of the texture components is expected for ECAP processed samples, an ODF section with $\varphi_2=0\text{--}180^\circ$ interval is shown for 1-pass ECAP processed aluminium (Fig. 2.23 (a)). For multi-pass ECAP processed samples, the symmetrical properties are related to the deformation route. Suwas et al. reported that the monoclinic symmetry was maintained for the samples processed by route A and route C, whereas the symmetry was lost for samples processed by route B [173]. Fig. 2.23(b) shows the ODFs of 4-pass ECAP processed aluminium and it can be seen that the monoclinic symmetry was not observed.

Three fibres denote f1, f2 and f3 are used to represent the fibres in the ECAP processed samples. As can be seen in Fig. 2.23, the f1 fibre starts at $A_{1\theta}$, passes through $A_{\theta}$ or $\bar{A}_{\theta}$ and ends at $A_{2\theta}^*$. The f2 fibre starts at $C_{\theta}$ orientation, passes through $B_{\theta}$ or $\bar{B}_{\theta}$, then $A_{\theta}$ or $\bar{A}_{\theta}$ and ends at the $A_{1\theta}^*$ orientation. The f3 fibre is symmetrical about the f2 fibre, starts at $A_{1\theta}^*$, then passes through $A_{\theta}$ or $\bar{A}_{\theta}$ and through $B_{\theta}$ or $\bar{B}_{\theta}$ and then ends at the $C_{\theta}$ orientation.
2.5.4 ARB texture

The texture of aluminium (FCC materials with high SFE) after conventional rolling is dominated by Copper, Brass and S components, and α and β fibres [175], as can be seen in Fig. 2.24. The α fibre connects the Goss and Brass components, both with \(<1 1 0>\) parallel to sheet normal, while the β fibre, which is called the skeleton line...
connecting the Brass, Copper and S components, is in space with \(<1\ 1\ 0>\) tilted 60° towards the rolling direction [175], as shown in Fig. 2.24(b).

![Fig. 2.24](a) \(\{1\ 1\ 1\}\) pole figures showing the ideal positions of S, Brass and Copper components [140], and (b) Sketch of the position of \(\alpha\) and \(\beta\) fibres in Euler space [170].

The texture of ARB processed materials has the same merits as those after conventional rolling, such as a strong \(\beta\) fibre, which was always observed [103, 104, 106, 125, 176-180]. However, the texture developed during ARB deformation is also different from the conventional rolling texture as the ARB texture show shear-type components near the surface [106, 125, 177-180], a strong texture gradient through the thickness [106] and generally weaker intensities [37, 97]. Typical ARB texture for AA1050 and AA6061 after different cycles of ARB processing is shown in Fig. 2.25. It can be seen that the strong initial Cube texture has been transformed into a combination of Copper, Rotated Cube and Brass components.
ARB texture is generally characterised by a strong Copper (or Dillamore) component dominated β fibre close to the centre of the sheets and Rotated Cube dominated shear-type components near the surface [31, 103, 104, 176-186]. The surface shear texture disappears as the surface area moves to the centre in the subsequent ARB cycle [106, 177-179]. Prangnell et al. [178, 179] and Li et al. [106] predicted that shear-type components Rotated Cube, Rotated Copper, E and F would rotate to rolling type components D, Copper and Brass when deformed under plane strain compression. The development of shear texture and the penetration depth of the shear strain are mainly affected by the strain rate, the friction between the rolls and the sheet, the rolling temperature, roll gap geometry and the material properties [177].

2.6 Characterisation of the defects in ultrafine grained materials

The defects in aluminium generated by SPD include grain boundaries, dislocations, vacancies, etc. The fraction of the grain boundaries in the UFG materials is large compared to their coarse grained counterparts. Detailed knowledge of these
defects, including their morphologies, densities and distribution can lead to a better understanding of the properties of SPD processed materials. TEM, EBSD, XRD, differential scanning calorimetry (DSC), residual electrical resistivity (RER), positron annihilation lifetime spectroscopy (PALS) and many other techniques are used to analyse the defects in SPD processed materials.

2.6.1 TEM technique

TEM is a conventional and widely used technique for material characterisation. The grain boundaries can easily be seen, the average grain sizes can be directly determined, and dislocations can easily be observed by TEM. Misorientation between grains can easily be determined by measuring the tilting angle between two grains. The dislocation density can also be determined quantitatively using a line intercept method, or by measuring the total line length of the dislocations and averaging it over the measured volume [187]. However, measuring the dislocation density in grains by TEM is not simple and there is a limit. TEM is usually able to determine the dislocation density up to $5 \times 10^{14} \text{ m}^{-2}$ with the weak beam technique [188], but when the dislocation density is higher than $10^{15} \text{ m}^{-2}$, it is difficult to measure it with TEM. However, many severely deformed metals and alloys have dislocation density higher than $10^{15} \text{ m}^{-2}$ [189-191]. As a result, the quantitative measurement of dislocation densities of SPD processed materials by TEM is not applicable for SPD processed materials most of the time.

Moreover, vacancy-type defects are not easy to be detected by TEM, unless they form big vacancy clusters such as vacancy Frank loops and stacking fault tetrahedras. It is reported that SPD can introduce aggregates of vacancy-type defects because of the intense dislocation interactions. However, the size and
density of vacancy-type defects in ordinary SPD processed materials [192] are not comparable to those observed in thin foils [193-195], which makes it harder to be observed under TEM. Additionally, irradiation by high-energy electrons in TEM can damage the sample. TEM observation of HPT deformed Al-3% Mg alloy revealed that defects changed with electron irradiation [196]. It can be concluded that TEM is not an effective technique for vacancy-type defects characterisation.

2.6.2 X-ray profile analysis

X-ray and neutron diffraction techniques are powerful to determine the microstructural details of plastic deformed materials. The scan can be conducted over a fairly large part of the sample, especially with neutron diffraction, thus the average values obtained from the diffraction methods can be used to represent the entire sample.

XRD $\theta$–$2\theta$ scan is always performed on a polished surface with optimum parameters to provide a good quality XRD pattern. The peak parameters, including peak intensity, peak position, full width at half maximum and integral breath, contain information of the material structure. Further analysis of the pattern will reveal details of the microstructure such as the lattice parameter, crystalline size, microstrain, dislocation density and dislocation type [197-200]. X-ray diffraction profile analysis has been used to determine the dislocation densities of the severely deformed metals such as commercial purity Al [135, 201, 202], Al alloy [203], Cu and alloys [189, 190, 204, 205] and Ti [190].

Peak broadening is considered to be caused by grain size refinement and lattice microstrain in addition to instrumental broadening. Using that the crystallite size
and microstrain can be calculated. The grain size can simply be determined from Scherrer formula [206]:

$$w_f = \frac{0.9\lambda}{D \cos \theta_0}$$  \hspace{1cm} (2.7)

and the lattice microstrain can be calculated by the Strokes and Wilson formula [206]:

$$w_f = 2\xi \tan \theta_0$$  \hspace{1cm} (2.8)

where $w_f$ is the width at half maximum intensity excluding instrumental broadening, $\lambda$ is the wavelength, $D$ is the average crystallite size, $\theta_0$ is the Bragg angle and $2\xi$ is the effective lattice microstrain.

There are many disadvantages of the two equations in practice, since both the lattice microstrains and fine grains contribute to the peak broadening in most of the plastically deformed materials. Therefore, many researchers have been taking effort to modify the formulas in order to make them applicable to different materials. The whole profile fitting methods such as Rietveld refinement, have been widely used in structure refinement of diffraction patterns by X-ray, neutron and synchrotron diffraction to determine the phase composition, lattice parameters, etc [207]. The multiple whole profile (MWP) was developed by Ungár et al. [199] by modifying the theory of Williamson-Hall method [197] and Warren-Averbach method [198], which was later updated to conventional multiple whole profile (CMWP) [200] and has been applied to many materials to determine crystallite size, microstrain, dislocation density, etc.

The X-ray measurements provide the coherently scattering domain size (crystallite size), which is often smaller than the grain or subgrain size obtained by TEM [188,
It has been shown that for bulk UFG materials produced by SPD, the grain size determined by TEM is about four to six times larger than the crystallite size obtained by X-ray line profile analysis [203, 208]. This is because XRD measures the coherent diffraction domains which include not only grains and subgrains but also the dislocation cells which are separated from each other by small differences in orientation, typically $1^\circ$–$2^\circ$ [188, 208]. However, TEM observation normally gives the sizes of grains with relatively high angle grain boundaries. Apart from which, obtaining the dislocation density from XRD profiles is much easier than performing quantitative dislocation density analysis under TEM. As a result, XRD profile analysis is usually combined with TEM techniques to investigate the structural properties of plastically deformed materials. TEM provides direct images of grain size and dense dislocations but other parameters like microstrain is always measured by XRD.

### 2.6.3 Positron annihilation lifetime spectroscopy

Positron annihilation lifetime spectroscopy (PALS) is a non-destructive technique to study defects in solids, especially open-volume defects such as vacancies, vacancy clusters and dislocations [209]. Upon thermalising in a solid, a positron will diffuse through the lattice and eventually annihilate with an electron to produce two 511 keV $\gamma$-quanta. However, if the solid contains open-volume defects (such as vacancies and dislocations), then the positron can localise in the attractive potential (attractive because of the absence of repulsive nuclei) provided by these sites. Open-volume defects have an electron density that is lower than average. As the positron lifetime is a function of electron density, when a positron becomes localised at these sites its lifetime increases with respect to the defect-free material. For example, in well-annealed aluminium, the positron lifetime is approximately
165 ps [210]. When the material is deformed to create atomic vacancies, the lifetime increases to 240 ps [210]. This lifetime indicates the size of the open volume, with larger vacancy clusters and voids exhibiting even longer lifetimes [211].

Typical positron lifetime spectra obtained in as-grown and plastically deformed Silicon are shown in Fig. 2.26. It can be seen that the deformed sample is located significantly higher, which indicates the existence of long-lived lifetime components. Like XRD, obtaining structure information from PALS requires analysing the raw experimental data. Many types of software have been developed to perform spectra fitting, such as Lifspecfit, LT, MELT and PALSfit [212]. A quantitative interpretation of PALS data can extract information of defect type, defect size, as well as defect density in the material.

![Experimental positron lifetime spectra obtained in as-grown and plastically deformed Silicon](image)

**Fig. 2.26** Experimental positron lifetime spectra obtained in as-grown and plastically deformed Silicon [209].
Because the positron lifetime is sensitive to the electron density at the trapping site, different lifetimes that are characteristic for different types of defects can be recorded. PALS is well-suited to study the defect structure and evolution during deformation, and has been used to study the defects in solids for over 30 years. Recently, PALS was also used to investigate the defects in UFG metals processed by SPD, and it is always combined with TEM, XRD and other techniques to investigate the defect evolution during SPD [213-219]. The PALS examination also proved the inhomogeneous dislocation distribution in SPD processed metals and the presence of vacancy-type defects. PALS was used to analyse HPT deformed copper which contains a high density of dislocations and small vacancy clusters corresponding to 5 and 10 mono-vacancies [216]. It’s reasonably easy to understand that microvoids exist in UFG copper and iron, since both metals have low SFE [219]. Vacancy-type defects associated with dislocations and alloy atoms were found in an Al-Cu-Mg-Mn alloy, but no microvoids were proven to have existed [220].

2.7 Summary and scope of the thesis

ECAP and ARB are two of the most frequently used SPD techniques and have received considerable attention in recent years. The techniques have the advantages of being able to introduce large amounts of plastic strain without changing the overall dimensions of the samples. Substantial grain refinement can be achieved by these techniques and the strength of the materials can be enhanced significantly.

Microstructure, mechanical properties and texture evolution of the ECAP and ARB processed samples have been investigated extensively over the years. The mechanical behaviour of samples processed by ECAP and ARB are similar, having
high strength and low ductility. The microstructure and texture developed during ECAP and ARB are different in that ECAP can process fairly large samples and obtain an equiaxed UFG microstructure which is difficult to obtain with regular plastic deformation, whereas ARB has the advantage of manufacturing large scaled sheet samples. The microstructures of the sheets after ARB are pancake shaped and are elongated along the rolling direction, which is similar to the microstructure after normal rolling but much finer. The texture developed during ECAP is the same as 45° CCW rotation of ideal simple shear texture. The ARB texture, on the other hand, is similar to the rolling texture with more through-thickness heterogeneity due to the interfaces between the layers.

To date, ECAP deformation has been conducted mostly at room temperature or higher where the microstructure refinement is saturated after about four passes. Many studies have been carried out by combining rolling at room temperature or lower with ECAP to further refine the microstructure, but the influence of a low ECAP temperature on the properties of the materials was not investigated. In the present study, ECAP has been conducted at both room temperature and cryogenic temperature to investigate the influence of ECAP temperature on the microstructure, mechanical properties and texture evolution during deformation.

ECAP deformation is close to simple shear deformation and ARB deformation is similar to traditional rolling deformation, which is plane strain compression in the sheet centre and simple shear close to the surface. In this study, AA1050 is deformed by both ECAP and ARB, and the properties of the materials after deformation by ECAP and ARB to the same strain have been compared. As the ARB process is usually conducted without any lubricant, the simple shear deformation close to the surface is more severe than traditional rolling. In addition, because the
materials at the surface areas are consecutively cycled to the centre and quarter-thickness areas with further processing, the ARB technique offers an opportunity to develop sheet materials with a higher fraction of shear type textures. In the present study, the rolls are deliberately treated so they have high roughness to enhance the shear deformation close to the surface areas, and the texture distribution throughout the thickness of the sheets has been investigated in detail.

ARB is able to roll-bond two pieces of different materials together to form laminated composites. Many studies have been done in this area but the difference in the deformation behaviour of the materials in the composites and in the monotonic material sheets was not reported. In this study, AA1050 and AA6061 were processed by ARB to obtain monotonic material sheets and laminated AA1050/AA6061 composite. The deformation behaviours of the three kinds of sheets have been compared in detail.

Some unusual phenomena were observed for UFG materials and many studies have suggested that vacancy-type defects also played an important role in the mechanical behaviour of the UFG materials. Vacancy-type defects in SPD processed samples were investigated in a few previous works, but not in detail. In the present study, PALS was used to investigate the evolution of the vacancy-type defects during the ECAP and ARB process.
Chapter 3 Experimental and analytical instruments

This chapter includes a detailed description of starting materials, experimental instruments and the analytical methodology used in the current study.

3.1 Sample processing

3.1.1 Equal-channel angular pressing

The material used in ECAP was commercially pure aluminium AA1050. Billets were cast and heat treated at 456 °C for 1 h and then air cooled to room temperature. The chemical composition of the alloy is shown in Table 3.1. The microstructure of the annealed material is shown in Fig. 3.1. The average grain size of the material before ECAP is 491 μm.

Table 3.1 Chemical composition of AA1050 and AA6061 used in this thesis.

<table>
<thead>
<tr>
<th></th>
<th>wt %</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Cr</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA1050</td>
<td></td>
<td>0.25</td>
<td>0–0.4</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td>0.03</td>
<td></td>
<td>Balance</td>
</tr>
<tr>
<td>AA6061</td>
<td>0.4– 0.8</td>
<td>0.7</td>
<td>0.15–0.4</td>
<td>0.15</td>
<td>0.8–1.2</td>
<td>0.25</td>
<td>0.15</td>
<td>0.04–0.35</td>
<td>Balance</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 3.1 Optical microstructure of AA1050 before ECAP.
The ECAP instrument and specimens processed by ECAP are shown in Fig. 3.2. The ECAP die has a cross-section of $20 \times 20$ mm$^2$, an intersection angle of 90° and an outer arc of curvature of 20°, which provides an equivalent strain of approximately 1 per ECAP pass. The billets were pressed up to 8 passes via route B$_C$ at a speed of 7 mm/s, with one group pressed at room temperature and the other group cooled with liquid nitrogen between each pass.

![Fig. 3.2 Photos of (a) ECAP instrument and (b) ECAP processed specimens.](image)

### 3.1.2 Accumulative roll bonding

The materials used in ARB are commercial aluminium alloy AA1050 and AA6061 (Yuanhang Metallic Materials Factory, Shenzhen, China). The as-received AA1050 and AA6061 both have thickness of 1.5 mm, and were in hot rolled state and T6 state, respectively. The chemical composition of both alloys is shown in Table 3.1. The AA1050 was heat treated at 450 °C for 1 h and the AA6061 was heat treated at 500 °C for 2 h before ARB. The microstructure of the annealed materials is shown in Fig. 3.3. The grain sizes of the annealed AA1050 and AA6061 are 95.8 μm and 36.3 μm, respectively.
Fig. 3.3 Optical microstructure of (a) AA1050 and (b) AA6061 before ARB.

Annealed AA1050 and AA6061 were used as starting materials for ARB. Before ARB, sheet materials with a dimension of $1.5 \times 50 \times 400 \text{ mm}^3$ (thickness $\times$ width $\times$ length) were cut from the original sheets, with the longitudinal direction parallel to the original rolling direction. Three groups of sheets were processed, one group using AA1050 as primary materials, one using AA6061 and another group using a combination of AA1050 and AA6061 as staring materials, as shown in Fig. 3.4. Three types of sheets: AA1050, AA6061 and AA1050/AA6061 were produced.

The roll diameter was 125 mm and the rolling speed was 196 mm/s. Prior to each rolling cycle, the rolls were cleaned by acetone and the roll gap and speed were set to the required setting. One side of the sample was cleaned with acetone and wire-brushed. Two pieces of the samples were then stacked and welded together at one end, pre-heated in a furnace at 200 °C for 3 min, and then rolled with a nominal reduction of $\sim50\%$ under dry condition, which gives an accumulated Von Mises equivalent strain of about 0.8. This pre-heating temperature was chosen to achieve good hardening and bonding simultaneously [221]. The rolled samples were cut into two halves and stacked together again to perform the next cycle. The above procedure was carried out for five cycles to achieve an accumulative strain of about 4. Because the rolled sheets had small edge cracks after roll-bonding at two cycles.
and higher, the edges were trimmed to conduct the following cycle. As a result, the samples after higher cycles of ARB are narrower. The photographs of the rolling mill and specimens processed by ARB are shown in Fig. 3.5.

![Fig. 3.4 Schematic illustration of ARB deformation in this work.](image)

![Fig. 3.5 Photos of (a) rolling mill for ARB deformation and (b) ARB processed specimens.](image)

### 3.2 Mechanical property tests

#### 3.2.1 Hardness tests

Vickers micro-hardness was measured with a Leco hardness testing machine to show the mechanical behaviour before and after deformation. The samples were ground and polished to a Struers OPS finish to ensure a flat surface before conducting the hardness test. Average hardness values were obtained by averaging 10 hardness measurements.
3.2.2 Tensile tests

Tensile tests were conducted with an Instron 5566 testing machine at room temperature. Tensile specimens with a dog bone shape were used.

3.3 Microstructure characterisation

3.3.1 Optical microscopy

The optical microstructures were observed with a Leica DMRM microscope. The samples were round and polished with a Struers TegraPol-21 polishing machine to an OPS finish and then etched with Barker’s reagent. Micrographs were taken under both normal light and polarised light. The Video Pro 32 Visual Analysis software was used for further analysis. The grain size was measured using the linear intercept method.

3.3.2 Scanning electron microscopy

The fracture surfaces of the tensile failed samples were examined using a JEOL 6490 SEM. Precipitate compositions were analysed with the energy dispersive X-ray (EDX) detector equipped in the SEM. The operation voltage was chosen as 20 kV and the working distance was set at 10 mm.

3.3.3 Transmission electron microscopy

TEM micrographs were obtained with a JEOL 2011F microscope equipped with LaB₆ filament operating at 200 kV. Thin foils for TEM examination were prepared by twin-jet electron polishing using an electrolyte of 25% nitric acid in methanol at –20 °C. Grain size measurements were performed on several images. The linear intercept method was used to measure the grain size and the measurements were
performed on several images. Precipitate compositions were estimated from the Energy dispersive X-ray spectroscopy (EDS) results detected using an ultra-thin window EDX detector.

### 3.4 Texture measurement

#### 3.4.1 X-ray diffraction

The texture of the deformed samples was measured by X-ray diffraction using X’Pert MRD goniometer (shown in Fig. 3.6) with Cu Kα radiation at 45 kV and ~40 mA. The samples were ground and polished with a Struers OPS finish to ensure a flat surface. Incomplete pole figures from 0 to 85° of \{1 1 1\}, \{2 0 0\} and \{2 2 0\} were measured in each case. Complete pole figures and ODFs were calculated using X’Pert texture software after defocusing and background correction.

![Fig. 3.6](image)

**Fig. 3.6** Photo of X’Pert MRD goniometer with texture measurement setup.

#### 3.4.2 Neutron diffraction

Texture of the cryogenic temperature ECAP processed samples were measured using the Kowari standard Eulerian goniometer (shown in Fig. 3.7) at Australian Nuclear Science and Technology Organisation (ANSTO). Complete pole figures of \{1
1\), \{2\ 0\ 0\}, \{2\ 2\ 0\}, \{3\ 1\ 1\} and \{2\ 2\ 2\} peaks were measured. At a wavelength of 1.67 Å the diffraction peaks were positioned at 42°, 49°, 71°, 86° and 91°, respectively. ODFs were calculated from the measured pole figures by an open MATLAB toolbox MTEX.

![Figure 3.7](image)

**Fig. 3.7** Photo of KOWARI instrument with texture measurement setup in ANSTO.

### 3.5 Defect characterisation by positron annihilation lifetime spectroscopy

In order to examine the defect evolution during SPD, PALS measurements were carried out on the SPD processed samples using a fast-fast spectrometer (shown in Fig. 3.8), with detectors consisting of a BC418 plastic scintillator coupled to a Burle 8850 photomultiplier tube. The positron source consisted of ~30 µCi of $^{22}\text{NaCl}$ sealed in 7 µm thick Kapton foil (grade HN from Goodfellow), and was sandwiched between two identical samples. The time resolution of the system is about 220 ps, as determined from analysing of a spectrum of high-purity annealed nickel. The spectra were comprised of at least 2 million counts. A pure aluminium sample annealed at 600 °C for one hour was measured to determine the source lifetime.
and intensity. Shukla et al. [222] developed a program called MELT 4.0 to analyse the positron lifetime. This program uses the maximum entropy method to extract positron lifetime distribution without an assumption of the number of lifetime components. It has been proven that MELT 4.0 is especially appropriate for evaluating short positron lifetimes [223]. In this study, the MELT 4.0 program has been used to analyse the measured spectra.

Fig. 3.8 Photo of positron lifetime spectrometer in the University of Western Australia.
Chapter 4 Equal channel angular pressing of AA1050 at room temperature

ECAP has been extensively studied over various types of metals and alloys, such as Al and alloys [154, 224-227], Cu and alloys [67, 196, 228, 229], Ti and alloys [230-232], Mg and alloys [233-235], steels [151, 236, 237], Ni [238-240] and Nb [241]. Aluminium and alloys are the most frequently studied materials and different properties of ECAP deformed aluminium and alloys have been reported. The microstructure [242-244], mechanical properties [243-245] and texture [173, 246, 247] evolution of aluminium and alloys during ECAP deformation were frequently reported. It was also reported that the microstructure was extensively refined after ECAP deformation, and excellent properties like high strength, improved fatigue behaviour, and superplasticity were obtained.

Defects such as grain boundaries [7, 248-250] and dislocation densities [80, 135, 251] were also reported, vacancy-type defects in ECAP processed Al, however, were rarely studied [192, 220]. In this chapter, ECAP was conducted on AA1050 at room temperature for up to 8 passes. The microstructures of ECAP processed samples were observed by TEM. The hardness and tensile tests were used to evaluate the evolution of mechanical properties. Average textures were measured by XRD and the vacancy-type defects evolution was investigated by PALS.

4.1 Experimental procedure

Annealed AA1050 was cut into 20 × 20 × 100 mm³ billets and then pressed through an ECAP die up to 8 passes via route B_C at room temperature. The pressing speed
was 7 mm/s. The samples after 1, 2, 4, 6 and 8 RT-ECAP passes were used for the testings were named RT-1, RT-2, RT-4, RT-6 and RT-8, respectively.

Vickers micro-hardness was measured using a load of 100 g for 12 s. The hardness values were taken on the cross sections of the RT-ECAP processed samples. The average hardness was calculated by averaging 10 values.

Tensile specimens with a gauge dimension of $8 \times 3 \times 1.5$ mm$^3$ (ED × TD × ND) were cut from the RT-ECAP processed materials with the tensile axis parallel to the pressing direction. An initial strain rate of $5 \times 10^{-4}$ s$^{-1}$ was used.

TEM micrographs were taken on the ED-ND plane of RT-ECAP processed samples. Thin foils were sliced from the plane perpendicular to the pressing direction, mechanically ground to less than 200 μm thick, and then twin-jet electron polished with Struers Tenupol polishing machine in a solution of 20% nitric acid and 80% methanol at −20 °C. 1, 2, 4, 6 and 8-pass RT-ECAP processed samples were investigated by TEM. SAD patterns were taken using a 20 μm diameter aperture. Grain size measurements were performed on several pictures measuring over 100 grains.

The average texture of RT-ECAP processed samples was measured by XRD on the cross-sections of the samples. Specimens with a dimension of 20 mm (ND) by 20 mm (TD) were cut from the RT-ECAP processed samples and then ground and polished with a Struers OPS finish. Incomplete pole figures from 0 to 85° of {1 1 1}, {2 0 0} and {2 2 0} were measured in each case. Complete pole figures and ODFs were plotted using the MTEX program.

DSC analysis was performed on a 7-pass RT-ECAP processed sample in METTLER TOELDO TGA/DSC-1 thermogravimetric analyser protected with flowing Argon. Disk
samples of were heated isochronally at 3, 5 and 8 °C/min to 400 °C and air cooled to room temperature. The recovery stage was analysed.

PALS measurements were carried out on the 1, 4 and 8-pass RT-ECAP processed samples. Two specimens with the same dimension of 20 × 20 × 2 mm³ (ND × TD × ED) were cut from the RT-ECAP processed samples. The positron source ~30 µCi of ²²NaCl was sealed in a 7 µm thick Kapton foil and sandwiched between the two identical samples. The measured lifetime spectra were analysed using the MELT 4.0 program. Lifetimes of vacancy-type defects were investigated.

**4.2 Microstructure evolution of AA1050 during RT-ECAP deformation**

The microstructures of the RT-ECAP deformed samples are shown in Fig. 4.1, and Table 4.1 lists the grain sizes. Fig. 4.1 (a) shows that the microstructure of RT-1 includes regions containing networks of dislocation cells with high dislocation densities. The microstructure of RT-2 shows an array of grains and subgrains aligned in a certain direction. The SAD patterns were obtained from an area of approximately 5 µm² and the patterns of RT-1 and RT-2 are similar, in that both patterns indicate one preferential zone axis and the individual spots from the zone axis pattern are slightly diffused. This is consistent with the selected areas for electron diffraction comprising grains separated by predominately LAGBs. The mean linear size of the dislocation cells in these samples is approximately 600 nm.

It can be seen in Fig. 4.1(c) that a nearly equiaxed grain structure has developed for the sample RT-4. The dislocation density is quite low in most grain interiors. The SAD pattern indicates several different grain orientations and grain structures with a higher proportion of HAGBs. The sample RT-6 shows a more refined microstructure than RT-4 and the grains are clearly aligned in a certain direction.
The grains in the sample RT-8 are reasonably equiaxed, as shown in Fig. 4.1(e), and most of the grains appear to be free of dislocations in the interiors and are surrounded by HAGBs. The microstructure is more uniform than that developed for RT-6.

Fig. 4.1 TEM micrographs of (a) RT-1, (b) RT-2, (c) RT-4, (d) RT-6 and (e) RT-8.
Table 4.1 Grain size of RT-ECAP processed samples measured from TEM micrographs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>RT-1</th>
<th>RT-2</th>
<th>RT-4</th>
<th>RT-6</th>
<th>RT-8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (nm)</td>
<td>683</td>
<td>582</td>
<td>608</td>
<td>570</td>
<td>551</td>
</tr>
</tbody>
</table>

The grain size of RT-8 is slightly smaller but close to the size of the dislocation cells and subgrains of RT-1. Iwahashi et al. reported that the ultimate grain size of the materials after several passes of RT-ECAP was decided by the banded structure obtained from the first RT-ECAP pass [50]. A single pass RT-ECAP gave a band structure that comprised of many subgrains within each grain. For route Bc, the band structure of the subgrains was still noticeable after the second pass, but not evident after 3 and more passes. The equiaxed grains with grain size similar to the band width of RT-1 can be obtained after 4-pass RT-ECAP and further deformation will reduce the dislocation density within the grain interiors and increase the angle of grain boundaries.

4.3 Mechanical properties of RT-ECAP processed AA1050

The results of hardness measurements on the RT-ECAP processed samples are shown in Fig. 4.2. There is a significant increase in hardness after 1 and 2-pass RT-ECAP deformation, which increases with the number of passes although at a much slower rate.
Fig. 4.2 Hardness evolution of AA1050 with the number of RT-ECAP passes.

Fig. 4.3 shows the tensile strength and uniform elongation of the RT-ECAP processed samples. The tensile strength shows the same trend as that of the hardness, with a significant increment in the first two RT-ECAP passes and then levels off. There is a slight drop from the sixth to the eighth pass. The initial material has a high uniform elongation, which drops drastically after 1-pass RT-ECAP deformation. The uniform elongation slightly increases as the RT-ECAP deformation proceeds, but the values remain relatively low compared to the initial material.

Fig. 4.3 The relationship of (a) tensile strength and (b) uniform elongation with the number of RT-ECAP passes.
4.4 Texture evolution of AA1050 during RT-ECAP deformation

The \{111\} pole figure and \(\phi_2=0^\circ\) ODF section of the initial sample is shown in Fig. 4.4. It can be seen that the initial material has a strong Cube texture. The measured \{111\} PFs for 1, 4 and 8-pass RT-ECAP processed samples are shown in Fig. 4.5. Remarkable changes in texture can be observed after only 1-pass RT-ECAP deformation. ECAP deformation is generally considered as near simple shear deformation in the intersection plane of the two channels. The ideal ECAP texture components are the same as those of simple shear rotating by 45°. Fig. 4.5(d) adopts the ideal ECAP texture components and fibres from Ref. [171].

![Initial texture of AA1050 before ECAP](image)

**Fig. 4.4** Initial texture of AA1050 before ECAP (a) \{111\} PF and (b) \(\phi_2=0^\circ\) ODF section.

It can be seen that the intensities of the texture increases from the first to the fourth pass, and then decreases. The overall intensity of RT-8 is stronger than RT-1. It can be concluded from the PFs that the textures of RT-1 and RT-8 are close to ideal, while the deviation from ideal texture is obvious for RT-4. This deviation can
be attributed to shear plane changing that occurred during sample rotating for route B_c. The ECAP deformation is considered to be similar to the ideal simple shear deformation, which indicates a monoclinic symmetry of the texture components. It is reported that for the 1-pass ECAP processed samples, monoclinic symmetry is observed. However, for the multi-pass ECAP processed samples, some reported monoclinic symmetry while others reported a loss of such symmetry [173, 174, 252, 253]. In the current experiment, it can be seen from the PFs that the monoclinic symmetry has been maintained for RT-1 and RT-8, but not for RT-4.

Fig. 4.5 {1 1 1} pole figures of (a) RT-1, (b) RT-4, (c) RT-8 and (d) (from Ref. [171]) ideal orientations and partial fibres for FCC materials after a single pass ECAP deformation.

According to Li et al. [171], ECAP texture can be derived from negative simple shear texture by a CCW rotation of $\theta=\Phi/2=45^\circ$ (for ECAP of $\Phi=90^\circ$ die) around TD, which
is equivalent to increasing the Euler angle $\varphi_1$ of simple shear orientations by $\theta=45^\circ$ while the other two Euler angles ($\Phi$ and $\varphi_2$) remain the same. The ODFs of the 1, 4 and 8-pass ECAP processed samples are shown in Fig. 4.6 and the ideal orientations and fibres are listed in Table 4.2.

Fig. 4.6(a) RT-1
Fig. 4.6(b) RT-4
Chapter 4 Equal channel angular pressing of AA1050 at room temperature

The f1 fibre, which is \{1 1 1\}_θ partial fibre, starts at \(A_{1θ}^*\), passes through \(A_θ\) or \(\overline{A}_θ\) and ends at \(A_{2θ}^*\). The f2 fibre consists of \(<1 1 0>_θ\) partial fibre and \{1 1 1\}_θ partial fibre, the former starts at the \(C_θ\) orientation, passes through \(B_θ\) or \(\overline{B}_θ\) and then meets the latter at \(A_θ\) or \(\overline{A}_θ\) and ends at the \(A_{1θ}^*\) orientation. The f3 fibre is
symmetrical about the f2 fibre, starts at $A_{1\theta}^*$, passes through $A_\theta$ or $\overline{A}_\theta$ and through $B_\theta$ or $\overline{B}_\theta$ and then ends at the $C_\theta$ orientation. The $C_\theta$ orientation has the maximum intensity and is followed by $A_\theta / \overline{A}_\theta$ and $B_\theta / \overline{B}_\theta$. The $A_{1\theta}^*$ and $A_{2\theta}^*$ orientations have the minimum intensity. Similar orientation patterns and intensity distributions were observed for 1-pass ECAP processed pure aluminium [174], AA6016 [252] and Cu [229].

After a single RT-ECAP pass, the texture is in correspondence with the ideal texture. All the components can be observed and are almost at their exact ideal positions. $C_\theta$ orientation has the maximum intensity and the other components are more or less equally distributed along the fibres. It can be seen in Fig. 4.6(b) that the texture of RT-4 has shifted considerably from the ideal texture. The texture components are still distributed along the three fibres but are apparently less uniform and more incomplete. Instead of showing strong intensity at the $C_\theta$ orientation, the strongest component of RT-4 is close to $A_{2\theta}^*$ but has shifted along the fibres from its ideal position. Components $A_{1\theta}^*$ and $C_\theta$ are much weaker and $A_\theta$, $\overline{A}_\theta$, $B_\theta$, and $\overline{B}_\theta$ are not observed at all. A similar deviation of texture for the 4-pass ECAP processed sample was also observed elsewhere [174, 252, 253]. However, the textures observed in these references are not consistent and are slightly different from the texture measured in this work. It can be concluded that unlike a single pass RT-ECAP deformation, the texture developed after 4-pass RT-ECAP is sensitive to the exact deformation history since the shear deformation cannot be guaranteed to be exactly the same for different groups of experiments. The texture of RT-8 is more similar to the ideal texture than RT-4. $A_{1\theta}^*$ and $A_{2\theta}^*$ which shifted a lot for RT-4 have been shifted back to the ideal position. Components $B_\theta$ and $\overline{B}_\theta$ are still absent but $A_\theta$ and $\overline{A}_\theta$ do appear. The intensity distributions along the fibres are more uniform.
than RT-4 but less than RT-1. The $C_\theta$ orientation has the highest intensity and the intensities of $A_{2\theta}^*$, $A_\theta$ and $\overline{A}_\theta$ are similar but weaker than $C_\theta$. This result suggests that the sample after 8-pass RT-ECAP has reached a steady state.

**Table 4.2** Ideal orientations and fibres for FCC materials after a single pass ECAP deformation [174].

<table>
<thead>
<tr>
<th>Notation</th>
<th>Euler angles (°)</th>
<th>Belong to fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{1\theta}^*$</td>
<td>80.26/260.26 45 0</td>
<td>${1 1 1}_\theta$</td>
</tr>
<tr>
<td>$A_{2\theta}^*$</td>
<td>97.74/189.74 45 0</td>
<td>${1 1 1}_\theta$</td>
</tr>
<tr>
<td>$A_\theta$</td>
<td>45 35.26 45</td>
<td>${1 1 1}_{&lt;1 1 0&gt;\theta}$</td>
</tr>
<tr>
<td>$\overline{A}_\theta$</td>
<td>225 35.26 45</td>
<td>${1 1 1}_{&lt;1 1 0&gt;\theta}$</td>
</tr>
<tr>
<td>$B_\theta$</td>
<td>45/165/285 54.74 45</td>
<td>$&lt;1 1 0&gt;\theta$</td>
</tr>
<tr>
<td>$\overline{B}_\theta$</td>
<td>105/225/345 54.74 45</td>
<td>$&lt;1 1 0&gt;\theta$</td>
</tr>
<tr>
<td>$C_\theta$</td>
<td>45/315 45 90</td>
<td>$&lt;1 1 0&gt;\theta$</td>
</tr>
</tbody>
</table>

**Table 4.3** Definition of fibres f1, f2 and f3 [172].

<table>
<thead>
<tr>
<th>Notation of fibre</th>
<th>Orientations in fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>f1</td>
<td>$A_{1\theta}^<em>-A_\theta/\overline{A}<em>\theta-A</em>{2\theta}^</em>$ ${1 1 1}_\theta$ partial fibre</td>
</tr>
<tr>
<td>f2</td>
<td>$C_\theta-B_\theta/A_\theta/\overline{A}<em>\theta-&lt;1 1 0&gt;\theta$ partial fibre; $\overline{A}</em>\theta/A_\theta-A_{1\theta}^*$ ${1 1 1}_\theta$ partial fibre</td>
</tr>
<tr>
<td>f3</td>
<td>$C_\theta-B_\theta/\overline{B}<em>\theta-&lt;1 1 0&gt;\theta$ partial fibre; $A</em>\theta/\overline{A}<em>\theta-A</em>{2\theta}^*$ ${1 1 1}_\theta$ partial fibre</td>
</tr>
</tbody>
</table>

The global texture of RT-1 was also measured by neutron diffraction. The ODF figure shown in Fig. 4.7 reveals that the texture measured by neutron diffraction is quite similar to that measured by XRD, except that the relative intensities of orientations are different. Fig. 4.6(a) shows that in the XRD measured texture, $C_\theta$ component has the strongest intensity, but the results of neutron diffracted, as can be seen in Fig. 4.7, shows that the $B_\theta/\overline{B}_\theta$ component has the maximum
intensity and the intensity of the $C_\theta$ component is weaker. The $A_{1\theta}^*$ and $A_{2\theta}^*$ components have similar intensities when measured by XRD, but $A_{1\theta}^*$ appears to be stronger than $A_{2\theta}^*$ in the neutron diffracted result. It has been reported that the $A_{1\theta}^*$ component is stronger than the $A_{2\theta}^*$ component due to the negative simple shear [171, 254], so it can be concluded that the texture measured by neutron diffraction represents the texture of the RT-ECAP processed samples more precisely.

Fig. 4.7 ODF of RT-1 measured by neutron diffraction.
4.5 Vacancy-type defects evolution of AA1050 during RT-ECAP deformation

4.5.1 Investigation of vacancy-type defects by differential scanning calorimetry

Fig. 4.8 shows the DSC curves of the 7-pass RT-ECAP processed sample at heating rates of 3, 5 and 8 °C/min, respectively. Two exothermic peaks appear in Fig. 4.8(a) and they significantly overlap in Fig. 4.8(b) and (c). Gaussian fittings with two peaks, named by P1 and P2, were performed, and are shown by dashed lines. The peaks P1 and P2 are centred at about 100–122 °C and 142–188 °C, and have been identified as the annealing of vacancies and dislocations, respectively [255].

![DSC curves](image)

**Fig. 4.8** DSC curves of 7-pass RT-ECAP processed sample at different heating rates: (a) 3 °C/min, (b) 5 °C/min and (c) 8 °C/min.
Fig. 4.9 shows the activation energy $Q$ as a function of the energy dependent transformed fraction $Y$ for the peaks P1 and P2. It can be seen that at the early stage of the peak P1, $Q$ increases from 0.47 eV at $Y=10\%$ to 0.51 eV at $Y=20\%$, and then decreases with $Y$. This trend is similar to Ref. [256], in which the migration of vacancies has been identified as being responsible for the reaction. The activation energy for the early stage peak P1 is close to, but smaller than the migrating energy of mono-vacancy (0.57–0.62 eV [256]. This implies that vacancy migration dominates at the early stage of the peak P1 but it is not a unique reaction. There should be another exothermal reaction responsible for the decrease of activation energy.

![Graph showing activation energy Q as a function of energy dependent transformed fraction Y.](image)

**Fig. 4.9** Activation energy $Q$ as functions of the energy dependent transformed fraction $Y$.

After the vacancies are activated, randomly moving vacancies can meet both dislocations and other vacancies. If the vacancies diffuse to the core of a dislocation, the dislocation climbs and will finally annihilate into the sinks, leading to a reduced dislocation density. If a mono-vacancy meets another, the smallest vacancy cluster, di-vacancy, will be created. If a vacancy meets an existing vacancy cluster, the cluster will grow to become a stacking fault tetrahedral, vacancy Frank loop, or even a void. Stacking fault tetrahedral and vacancy Frank loop are more
stable [257]. They may not be mobile at the temperature range of the peak P1. The formation energy of a stationary vacancy cluster includes two parts: migration energy of the mono-vacancies and binding energy between the mono-vacancies and the existing vacancy clusters. As the vacancy cluster grows up, the formation energy will decrease due to the continuous binding. Therefore, the activation energy decreases at the middle and final stages of the peak P1. This conclusion is consistent with Ref. [258], which states that at small concentrations of vacancies the annealing process is controlled by the migration energy of single vacancies, whereas at large densities of vacancies, the annealing process is controlled by the migration energy of vacancies and the binding energy to form vacancy clusters. The large stationary vacancy clusters will act as pinning centres and hinder the slip motion of the dislocations, results in enhanced strength. The activation energy for the peak P2 is much smaller than the migration energy of the mono-vacancy at the early stage. This is consistent with Matsukawa and Zinkle’s observation that the effective migration energy for the glissile vacancy cluster is much less than that for mono-vacancy [259].

4.5.2 Investigation of vacancy-type defects by positron annihilation lifetime spectroscopy

The DSC analysis shown in Section 4.5.1 indicated that vacancy-type defects exist in RT-ECAP processed samples. As PALS is a more effective way to investigate the vacancy-type defects, PALS was conducted to 1, 4 and 8-pass RT-ECAP processed samples.

A fully annealed aluminium sample was used as a reference and the positron lifetime analysis of this sample reveals three lifetime components: 162 ps, 357 ps
and 1770 ps. The first component (162 ps) is located within the range reported for
the perfect lattice of aluminium [211, 260]. The second and third components can
be attributed to positron source annihilations. Staab et al. [261] stated that there
are three different source contributions to the positron lifetimes. The first one is
due to the fraction of the positrons annihilating in the support foil. McGuire and
Keeble studied the positron lifetime in Kapton support foil [262] and found that the
positron lifetime is ~369 ps for Kapton. The second source contribution is due to
the radioactive salt and the interface between the foil and the sample. This
contribution provides a lifetime of ~350 ps from the analysis of the experimentally
measured spectra [261]. The third source contribution, which generates a lifetime
of about 1500−2500 ps, is due to the annihilation of positrons at the surfaces [261].
The value of the third source lifetime depends on the size of the samples in
comparison to the source size and the surface roughness of the samples. The
second measured lifetime component (357 ps) in the annealed sample locates
between the first (369 ps) and second (350 ps) source lifetimes. Since the two
source lifetimes are too close to be separated in practice, it is reasonable to
conclude that the second measured lifetime component is induced by the mixture
of the first and second types of source contributions. It is also clear that the
measured longest lifetime of 1770 ps is the third type of source lifetime. The
intensities for the second and third measured lifetimes are 15.0% and 0.2%,
respectively. They are close to those obtained from the experimental results and
Monte-Carlo simulations in Ref. [261]. The measured positron spectra of all ECAP
processed samples are corrected by subtracting the source components (second
and third lifetimes of the annealed sample).
Two positron lifetime components were obtained for the RT-ECAP processed samples: a short lifetime $\tau_1$ and a long lifetime $\tau_2$. The results are shown in Fig. 4.10. The error bars in the figures indicate the values of the full width at half maximum (FWHM) calculated by MELT. All of the $\tau_1$ values are smaller than the bulk Al lifetime (~162 ps), while the $\tau_2$ values vary between 220 ps and 240 ps. It is known that vacancies can form by collisions of moving dislocations and they can easily diffuse into the dislocation cores. The positron lifetime of vacancies in the dislocation cores, which are also called vacancies associated with dislocations, is different from that of mono-vacancies in the bulk [263-265]. Petersen et al. [263] reported a lifetime of ~220 ps in Al single crystal subjected to an uniaxial tensile strain of up to 28 %, where dislocations were practically the sole type of defect produced. Therefore, 220 ps is regarded as the positron lifetime in vacancies associated with dislocations. A similar result has been obtained by Hidalgo et al. [260], where two Al single crystal samples with different crystallographic orientations were strained 27% under tension. The positron lifetimes were in the range of 215–220 ps. In addition, Rajainmäkit and Linderoth [264] measured a lifetime spectrum for Al single crystal at 327 °C. At this temperature it is known that thermal mono-vacancies are present, and the lifetime of bulk mono-vacancies was found to be 246 ps. Fluss et al. reported a similar lifetime of 244 ps for bulk mono-vacancies in high-purity polycrystalline measured at temperatures higher than 350 °C [265]. Furthermore, Häkkinen et al. [266] studied the positron annihilation characteristics of vacancies in Al using molecular dynamics and found that vacancy-type defects associated with $\frac{1}{2}\langle 112 \rangle$ type full dislocations have positron lifetimes of 224–225 ps, while the positron lifetime in a bulk mono-vacancy is 252 ps. Therefore, it has been generally accepted that vacancies associated with
dislocations have a lifetime of ~220 ps, while bulk mono-vacancies have a lifetime of ~245 ps in Al.

![Graph](image)

**Fig. 4.10** Measured positron lifetimes of RT-ECAP processed samples (a) long lifetime and (b) short lifetime.

Fig. 4.10(a) shows that all RT-ECAP processed samples possess a $\tau_2$ value between 220 and 240 ps. It should be noted that it is difficult, if not impossible, for the lifetime analysis program to distinguish these two lifetimes with such a small difference. Therefore, it is reasonable to conclude that the measured long lifetime values for all RT-ECAP processed samples are the average lifetimes mainly due to positron annihilation in vacancies associated with dislocations and bulk mono-
vacancies. Thus the fractions of these two vacancy-type defects can be estimated by \( f_1 \times 220 + f_2 \times 245 = \tau_2 \) and \( f_1 + f_2 = 1 \), where \( f_1 \) is the fraction of vacancies associated with dislocations and \( f_2 \) is the fraction of bulk mono-vacancies. The calculated fractions are listed in Table 4.4. Sample RT-1 has a \( \tau_2 \) value of 221 ps, which is close to the lifetime for vacancies associated with dislocations (220 ps). The calculated \( f_1 \) value for this sample is 96%, indicating that vacancies associated with dislocations are dominant. Samples RT-4 and RT-8 have \( \tau_2 \) values around 240 ps. The \( f_2 \) values for RT-4 and RT-8 are 80% and 71%, respectively. This indicates that bulk mono-vacancies are the major vacancy-type defects in these samples and a certain amount of vacancies associated with dislocations still exist. Fig. 4.10(b) shows the short positron lifetime (\( \tau_1 \)) for the RT-ECAP processed samples. The short positron lifetime can give a rough indication of the concentration of vacancy-type defects. A small \( \tau_1 \) value indicates a high defect concentration [217]. It can be seen that \( \tau_1 \) decreases monotonically with increasing number of ECAP passes for the RT-ECAP processed samples.

**Table 4.4** Fractions of vacancy-type defects for RT-ECAP processed samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction (%)</th>
<th>Vacancies associated with dislocations (( f_1 ))</th>
<th>Mono-vacancies in the bulk (( f_2 ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT-1</td>
<td>95.6</td>
<td>4.4</td>
<td></td>
</tr>
<tr>
<td>RT-4</td>
<td>20.0</td>
<td>80.0</td>
<td></td>
</tr>
<tr>
<td>RT-8</td>
<td>29.2</td>
<td>70.8</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 4.11 depicts the intensities (\( I_2 \)) of the long positron lifetime. \( I_2 \) significantly increases from sample RT-1 to RT-4 and then slightly increases for RT-8. \( I_2 \) is related to the concentrations and distributions of the vacancy-type defects. A detailed analysis is presented in Section 4.6.
4.6 Discussion

Two mechanisms governing point defect formation during plastic deformation have been proposed [257, 267]. The first is, if a positive dislocation and a negative dislocation come together and terminate one plane apart, a string of vacancies will be generated. If moving positive and negative dislocations terminate at the same plane, a string of self-interstitials will be generated. The second is, if two moving dislocations cross, jogs on dislocations can be produced. Since the jog is subjected to the frictional drag under deformation, the jog velocity is lower than the velocity of a non-jogged dislocation segment. Under these conditions, a non-conservative jog motion should result in the attendant production of point defects (vacancies or self-interstitials). The formation energy for a self-interstitial (2.59 eV) is approximately four times that for a vacancy (0.68 eV) [268], which suggests that it is hard to form self-interstitials in aluminium. Even if self-interstitials can be generated, they will easily annihilate in the sinks because the activation energy for the migration of a self-interstitial (0.1 eV [257]) is much smaller than that for a vacancy (0.61–0.64 eV [268, 269]). Therefore, it is expected that the concentration of self-interstitials is negligible and vacancy-type defects are the common point defects.
defects in aluminium subjected to plastic deformation. During ECAP deformation
the dislocations move and meet other dislocations and they act as both vacancy
sources and vacancy sinks. When a dislocation encounters a bulk mono-vacancy,
the mono-vacancy becomes a vacancy associated with the dislocation and moves
along the dislocation core by pipe diffusion. The vacancy associated with the
dislocation then annihilates when it reaches a vacancy sink, such as a jog or grain
boundary.

The standard trapping model (STM) has been widely used to determine the defect
concentrations from positron lifetime data for metals and semiconductors [270].
This model assumes that defects are distributed homogeneously in the material.
However, TEM observations in Fig. 4.1 show that dislocations are distributed
inhomogeneously in RT-ECAP processed samples. A high dislocation density can be
seen in the vicinity of the boundaries of dislocation cells and grains, while their
interiors exhibit a relatively low dislocation density. This indicates that the STM
cannot be directly applied to these samples. Dupasquier et al. [271] have
developed a diffusion trapping model (DTM), which considers the positron
diffusion from grain interiors to grain boundaries. This model assumes that the
material is formed by identical spherical domains of radius $R$ and that open-volume
defects only concentrate in a distorted region (DR) of constant thickness ($\delta$) at the
surfaces of the domains. This model was used for the analysis of the positron
lifetime spectra in a fine-grained Al-Ca-Zn alloy [271]. However, the assumption of
defect-free grain interiors (non-distorted region (NR)) is inconsistent with
observations in UFG materials [192, 217]. Microvoids and vacancy clusters have
been observed by TEM inside the grain interiors of UFG Cu produced by HPT [217]
and the UFG Al deformed by room temperature ECAP followed by liquid nitrogen
rolling [192]. Čížek et al. [217] have improved the DTM by considering the spatial distribution of vacancy-type defects in UFG Cu. They assumed that after thermalisation the positrons enter the material uniformly. Due to the high dislocation density and nearness to the grain boundary, all the positrons inside the DR were assumed to annihilate into the sinks. Since the grain diameter is comparable to the positron diffusion length, only a fraction of the positrons annihilate in the NR in a free or trapped state, while the rest of the positrons inside the NR will diffuse from the NR to the DR.

In this study we followed the framework of the DTMs developed by Dupasquier et al. [271] and Čížek et al. [217]. The UFG material is assumed to be formed by identical spherical domains of radius $R$, which are divided into two regions: the boundary region with a spherical shell shape (DR) and the interior region with a spherical core shape (NR). The thickness of the DR is defined as $\delta$, which gives the radius of the NR as $R-\delta$. In addition, we make two assumptions that differ from the previous DTMs:

(1) Čížek et al. observed microvoids and assumed that microvoids were situated in the NR. Our positron lifetime analysis did not indicate large vacancy-type clusters. Vacancies associated with dislocations and bulk mono-vacancies are the major vacancy-type defects in our samples and bulk di-vacancies also appear in some samples.

(2) Dupasquier et al. [192] and Čížek et al. [217] assumed that vacancies associated with dislocations are located only in the DR due to high dislocation density near the grain boundaries in the fine and ultrafine grained materials. However, we note that the concentration of vacancy-
type defects depends not only on the dislocation density but also on the history of the dislocation-dislocation interactions. The macro plastic deformation requires dislocations passing through the whole grain, which leaves a non-uniform distribution of vacancy-type defects over the grain. The current study assumes that vacancy-type defects exist in both the DR and NR with identical fractions \( f_1, f_2 \) and \( f_3 \). But the defects concentrations are different in the DR and NR. The concentration of vacancy-type defects is defined as \( C_D \) and \( C_N \) in the DR and NR, respectively.

There are three rate equations governing the diffusion and annihilation of positrons in the UFG samples:

First, the density of free positrons \( (n) \) in the NR is given by:

\[
\frac{\partial n}{\partial t} = D_+ \left( \frac{\partial^2 n}{\partial r^2} + \frac{2}{r} \frac{\partial n}{\partial r} \right) - (\lambda_B + K_N)n \tag{4.1}
\]

where \( t \) is time, \( r \) is the radial distance from the centre of the domain, \( D_+ \) is the positron diffusion coefficient, \( \lambda_B \) is the bulk annihilation rate for a free positron, \( K_N \) is the positron trapping rate of vacancy-type defects in the NR.

(1) The density of positrons \( (n_N) \) trapped in the defects situated in the NR is expressed by

\[
\frac{\partial n_N}{\partial t} = -\lambda_{trap} n_N + K_N n_a \tag{4.2}
\]

where \( \lambda_{trap} \) is the annihilation rate for trapped positrons and \( n_a \) is the average free positron density in the NR, which can be determined by

\[
n_a(t) = \frac{3 \int_0^{R-\delta} n(r,t) r^2 dr}{(R-\delta)^3} \tag{4.3}
\]
(2) The density of positrons \(n_D\) trapped in the defects situated in the DR is given by

\[
\frac{\partial n_D}{\partial t} = -\lambda_{\text{trap}} n_D + K_D n|_{r=R-\delta} \tag{4.4}
\]

where \(K_D\) is the positron trapping rate of the vacancy-type defects in the DR.

The initial condition for Eq. (4.1) is

\[
n(r, 0) = \frac{3(1-\eta)}{4\pi(R-\delta)^3} \tag{4.5}
\]

where \(\eta\) is the volume fraction of the DR, which can be calculated from

\[
\eta = \frac{R^3-(R-\delta)^3}{R^3} \tag{4.6}
\]

It has been assumed that all positrons diffusing through the interface between the NR and the DR will annihilate in the defects in the DR. This yields a boundary condition at \(r = R - \delta\) [217]:

\[
K_D 4\pi (R-\delta)^2 \delta n|_{r=R} = -4\pi (R-\delta)^2 D_+ \left( \frac{\partial n}{\partial r} \right)_{r=(R-\delta)} \tag{4.7}
\]

where

\[
\left( \frac{\partial n}{\partial r} \right)_{r=(R-\delta)} = -\frac{\alpha}{(R-\delta)} n_{r=(R-\delta)} \tag{4.8}
\]

and

\[
\alpha = \frac{K_D \delta (R-\delta)}{D_+} \tag{4.9}
\]

Solving Eqs. (4.1), (4.2) and (4.4) with the initial condition (Eq. (4.5)) and boundary condition (Eq. (4.8)) yields the following expression for the total number of positrons which annihilate in both the NR and the DR [217].
Chapter 4 Equal channel angular pressing of AA1050 at room temperature

\[ N(t) = 3(1 - \eta) \alpha \frac{D_+}{(R - \delta)^2} \sum_{k=1}^{\infty} a_k \left( \frac{\lambda_k - \lambda_{\text{trap}}}{(\lambda_k - \lambda_B - K_N)(\lambda_k - \lambda_{\text{trap}})} \right) e^{-\lambda_k t} 
+ \left( \eta + 3(1 - \eta) \alpha \frac{D_+}{(R - \delta)^2} \sum_{k=1}^{\infty} a_k \left( \frac{\lambda_k - \lambda_B}{(\lambda_k - \lambda_B - K_N)(\lambda_k - \lambda_{\text{trap}})} \right) \right) e^{-\lambda_{\text{trap}} t} \]  \hspace{1cm} (4.10)

where \( \beta_k \) is the \( k^{th} \) solution of

\[ \beta_k \cot \beta_k + \alpha - 1 = 0 \]  \hspace{1cm} (4.11)

\[ a_k = \frac{2\alpha}{\beta_k^2 + \alpha (\alpha - 1)} \]  \hspace{1cm} (4.12)

\[ \lambda_k = \lambda_B + K_N + \frac{\beta_k^2 D_+}{(R - \delta)^2} \]  \hspace{1cm} (4.13)

It can be seen that the first term of \( N(t) \) consists of infinite exponential components, which cannot be resolved in spectra in any practical experimental situation [217, 271]. If a weighted average is used to represent the first component, Eq. (4.10) can be approximated by [217]:

\[ N(t) = (1 - I_2) e^{-\lambda_1 t} + I_2 e^{-\lambda_2 t} \]  \hspace{1cm} (4.14)

with

\[ \lambda_1 = \frac{1}{\tau_1} = \frac{\sum_{k=1}^{\infty} i_k}{\sum_{k=1}^{\infty} i_k t_k} \]  \hspace{1cm} (4.15)

\[ \lambda_2 = \frac{1}{\tau_2} = \lambda_{\text{trap}} \]  \hspace{1cm} (4.16)

\[ I_2 = \eta + 3(1 - \eta) \alpha \frac{D_+}{(R - \delta)^2} \sum_{k=1}^{\infty} a_k \left( \frac{\lambda_k - \lambda_B}{(\lambda_k - \lambda_B - K_N)(\lambda_k - \lambda_{\text{trap}})} \right) \]  \hspace{1cm} (4.17)

where

\[ t_k = \left( \lambda_B + K_N + \frac{\beta_k^2 D_+}{(R - \delta)^2} \right)^{-1} \]  \hspace{1cm} (4.18)
\[ i_k = 3(1-\eta)\alpha \frac{D_+}{(R-\delta)^2} \sigma_k \frac{\lambda_B-\lambda_{trap}}{(\lambda_k-\lambda_B-K_N)(\lambda_k-\lambda_{trap})} \]  

(4.19)

Dupasquier et al. [271] suggested that in practice the truncation at \( k>\alpha \) in Eqs. (4.15) and (4.17) gives an acceptable result. We use a truncation of \( k>2\alpha \) to achieve a higher accuracy. The domain used in the model is not necessary to be a grain. Since the domain is related to the dislocation density [217], it should be the dislocation cell if the dislocation cells exist in the sample. TEM observations show that the dislocation cells are the major microstructural characteristics in sample RT-1 and they have evolved to grains after 4 RT-ECAP passes. The mean linear size of the dislocation cells in these samples is similar to that of the grains in other samples in that both are in the order of \( d = 600 \) nm. Therefore, a constant \( R \) is set to 338 nm for all the samples by \( R = d/\sqrt{\pi} \). The positron diffusion coefficient \( D_+ \) is a property of the bulk material. It has been set to 123 nm\(^2\)/ps [211]. Eqs. (4.17), (4.18) and (4.19) predict the short positron lifetime \( (\tau_1) \), long positron lifetime \( (\tau_2) \) and intensity for the long positron lifetime \( (I_2) \), respectively. There are three unknown parameters in these equations: \( K_N, K_D \) and \( \delta \), which are determined by matching the predicted \( \tau_1, \tau_2 \) and \( I_2 \) values with the experimental results. The following relative uncertainty has been used to determine the calculation accuracy:

\[
\text{uncertainty} = \left| \frac{\tau_1^p - \tau_1^m}{\tau_1^m} \right| + \left| \frac{\tau_2^p - \tau_2^m}{\tau_2^m} \right| + \left| \frac{I_2^p - I_2^m}{I_2^m} \right| 
\]  

(4.20)

where the superscript \( p \) represents the predicted values and the superscript \( m \) represents the values obtained from the positron lifetime analysis, as listed in Table 4.4.

The calculated \( K_N, K_D, \delta \) values and calculation uncertainties have been tabulated in Table 4.5. Sample RT-1 has the largest uncertainty of 2.83%, while the
calculation errors of other samples are less than 0.25%. This indicates that the calculation accuracies for all the samples are acceptable. The thickness of the DR (δ) varies from sample to sample. Sample RT-1 has a small value of δ (10.2 mm). As the deformation increases, δ increases to 106.8 mm in the sample RT-4 and then decreases to 52.9 mm in the sample RT-8.

Table 4.5 Calculated $K_N$, $K_D$, δ and calculation uncertainty for RT-ECAP processed samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$K_N$ ($\times 10^{10}$ s$^{-1}$)</th>
<th>$K_D$ ($\times 10^{10}$ s$^{-1}$)</th>
<th>δ (nm)</th>
<th>Uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT-1</td>
<td>1.101</td>
<td>0.580</td>
<td>10.2</td>
<td>2.83</td>
</tr>
<tr>
<td>RT-4</td>
<td>0.419</td>
<td>0.988</td>
<td>106.8</td>
<td>0.138</td>
</tr>
<tr>
<td>RT-8</td>
<td>4.337</td>
<td>5.389</td>
<td>52.9</td>
<td>0.035</td>
</tr>
</tbody>
</table>

The positron trapping rate $K$ is proportional to the defect concentration $C$:

$$K = \mu C \quad (4.21)$$

where $\mu$ is the specific trapping rate, which is expressed by

$$\mu \cong \sigma v \quad (4.22)$$

where

$$v = \sqrt{\frac{8kT}{\pi m}} \quad (4.23)$$

is the average diffusion speed of the positron, $\sigma$ is the trapping cross section and $m$ is the mass of the positron [272].

At $T = 300$ K $v$ is 1.076 m/s. It has been assumed that a mono-vacancy possesses the same volume as a single atom in the perfect material, respectively. The trapping cross section for a mono-vacancy can be calculated by the same method used in Ref. [272] using the aluminium lattice constant of 4.05 Å. This value is
determined to be 0.0787 nm² for a mono-vacancy. Vacancies associated with dislocations are assumed to have the same trapping cross section as bulk mono-vacancies. Therefore, the specific trapping rate is \( \mu_1 = 8.342 \times 10^{-9} \text{ cm}^3/\text{s} \) or \( 5.097 \times 10^{14} \text{ s}^{-1} \text{ atom}^{-1} \) for bulk mono-vacancies and vacancies associated with dislocations. The specific trapping rate obtained for mono-vacancies is comparable to the value \((4 \pm 1 \times 10^{14} \text{ s}^{-1} \text{ atom}^{-1})\) determined from the formation entropy [265]. By considering the multiple components of the vacancy-type defects in the NR and DR, Eq. (4.21) can be rewritten as

\[
K_N = (f_1\mu_1 + f_2\mu_1 + f_3\mu_1)C_N 
\]

\[
K_D = (f_1\mu_1 + f_2\mu_1 + f_3\mu_1)C_D 
\]

where \( C_N \) and \( C_D \) are the concentrations of vacancy-type defects in the NR and DR, respectively.

The calculated \( C_N \)s and \( C_D \)s for the RT-ECAP processed samples are shown in Fig. 4.12. For the sample RT-1, \( C_D \) is lower than \( C_N \). Vacancies associated with dislocations are the dominant vacancy-type defects in this sample and they can easily annihilate in the jogs on the dislocations. The inter-jog distance can be estimated by \( \left(\frac{v_j}{v}\right)^{1/2} \) [273], where \( \rho \) is the dislocation density, \( v_j \) is the lateral jog velocity and \( v \) is the mean dislocation velocity. The high dislocation density near the grain boundaries results in a short inter-jog distance, leading to a smaller \( C_D \) in the DR. In addition, vacancies near the grain boundaries annihilate easily to the latter, which is also responsible for the lower \( C_D \) in the DR and the small width of the DR (\( \delta \)) as indicated in Table 4.5. In the sample RT-4, \( C_D \) is larger than \( C_N \), indicating that bulk vacancies tend to migrate to the grain boundaries due to the low dislocation density in the NR and barriers may exist to prevent vacancies from
annihilating into the grain boundaries. The possible barriers sealing grain boundaries may be high-energy and non-equilibrium configurations [274] along the grain boundaries and/or nano-sized precipitates in the grain boundaries [269]. The continuous migration of vacancies and the grain boundary sealing effect result in an enlarged $\delta$ in the sample RT-4. After 8 RT-ECAP passes, the defect concentrations in both the NR and DR have been increased significantly. The vacancy concentration in the DR exceeds $1 \times 10^{-4}$, which is comparable to the vacancy concentration at melting temperatures for most metals [258]. This can be contributed to the significantly strengthened grain boundary sealing effect caused by the heavy deformation. Moreover, the reduced dislocation density in the grain interiors results in less vacancy migration. This means that vacancies tend to concentrate in the vicinity of the grain boundaries, leading to a reduced $\delta$ in the sample RT-8 compared to the sample RT-4. The high vacancy concentration in the sample RT-8 confirms the conclusion of Ref. [275], which also identified a high vacancy concentration in aluminium deformed by more than 6 RT-ECAP passes. In this case DSC and XRD were employed.

![Graph](image)

**Fig. 4.12** Calculated $C_N$ and $C_D$ for RT-ECAP processed samples.
4.7 Summary

AA1050 was processed by ECAP deformation at room temperature and the evolution of the microstructure, mechanical properties, texture and vacancy-type defects were investigated. The following summaries can be made.

(1) Substantial grain refinement was achieved for AA1050 by RT-ECAP deformation. The average grain size achieved after 8-pass RT-ECAP is about 550 nm and the grains have equiaxed shapes with high fraction of HAGBs.

(2) A significant increase in hardness and tensile strength occurred after the first pass RT-ECAP deformation and followed by a steady increase with subsequent passes. In the meantime, the uniform elongation dropped dramatically after 1-pass RT-ECAP and stayed at similar levels with further deformation.

(3) The texture developed after 1-pass RT-ECAP shows similar features as theoretical negative shear textures. After 4-pass RT-ECAP deformation, the textures deviated from the ideal texture quite markedly, and the components are much less uniform and more incomplete. The texture of the 8-pass RT-ECAP processed sample is more similar to the theoretical texture than the 4-pass sample.

(4) A high fraction of vacancy-type defects were detected in RT-ECAP deformed samples by PALS. A trapping model has been proposed and the positron trapping rate and vacancy-type defects concentration were calculated. Vacancies associated with dislocations and bulk mono-vacancies are identified in the RT-ECAP deformed samples. Vacancies associated with dislocations dominate in the sample RT-1, while bulk mono-vacancies become the dominant vacancy-type defects in the samples RT-4 and RT-8.
Chapter 5 Equal channel angular pressing of AA1050 at cryogenic temperature

Over the last two decades, many researchers have studied the effect of pressing temperature on the microstructure and mechanical properties of ECAP processed specimens. Basically, as the temperature increases, the pressing becomes easier but the equilibrium grain size would be larger, and the fraction of HAGBs also decreases [60, 61]. As for aluminium and its alloys, dynamic recovery during plastic deformation is one important factor that restricts the microstructure refinement since aluminium has a high SFE.

Plastic deformation at cryogenic temperature is believed to be capable of depressing the dynamic recovery and refine the grain size to smaller levels. Wang et al. prepared nanocrystalline Cu by rolling at temperature of liquid nitrogen and further annealing [84, 86]. This procedure resulted in a bimodal structured material with excellent properties. A marked improvement in uniform elongation was achieved without sacrificing much of the strength. Cryogenic temperature rolling has been carried out to many materials since, including commercial purity aluminium [157], AA2219 [158], AA5083 [159], AA6063 [160, 276], Cu-Zn and Cu-Al alloy [158, 161], pure V [90] and pure Ni [162]. However, these experiments only exhibited a slight increase in ductility with a sacrifice of tensile strength. The same technique does not seem to work well with the other metals and no significant improvement has been reported since then.

Plastic deformation at cryogenic temperature such as cryo-rolling, was also combined with ECAP by some researchers to obtain high strength and ductility simultaneously [81-83, 86]. A lower pressing temperature is supposed to be able to
further refine the microstructure although it is easier to press specimens at high temperature. However, ECAP deformation at low temperature has not attracted much attention.

In this chapter, ECAP deformation up to 8 passes was conducted to AA1050 at cryogenic temperature. The microstructure, mechanical properties, texture and defects evolution of cryogenic temperature ECAP processed samples were analysed in detail and were compared with room temperature ECAP processed results. The microstructures of the CT-ECAP processed samples were observed by TEM and the mechanical properties were tested by tensile and hardness measurements. The texture evolution was investigated by measuring the global texture using neutron diffraction, and the vacancy-type defects evolution was analysed by PALS.

5.1 Experimental procedure

The material used for CT-ECAP processing was the same annealed AA1050 as used for RT-ECAP and had the same dimension. The billets were pressed up to 8 passes via route B_C with liquid nitrogen cooling between each pass. The billets were immersed in liquid nitrogen immediately after one ECAP pass and were kept there for more than one hour before the next pass. The time between cooling and pressing was kept as short as possible to minimise the temperature rise. The billets were left at ambient temperature after all the pressings were finished and allowed the temperature to rise up. CT-1 to 8 was named for the CT-ECAP processed samples in the following contents.

The mechanical properties were also examined by Vickers micro-hardness and tensile tests. Sample preparation and the testing methods were the same as that listed in Section 4.1. TEM observations were conducted to the 1, 2, 4, 6 and 8-pass
CT-ECAP processed samples on thin foil specimens prepared in the same methods as the RT-ECAP deformed samples.

CT-ECAP textures were measured using Kowari standard Eulerian goniometer in ANSTO. Complete pole figures of \{1 1 1\}, \{2 0 0\}, \{2 2 0\}, \{2 1 1\} and \{2 2 2\} peaks were measured. In order to examine the texture evolution during CT-ECAP deformation, the texture measurements were conducted on the 1, 2, 3, 4, 6 and 8-pass CT-ECAP processed samples. The samples were cut into 20 × 20 × 20 mm$^3$ in size and the texture was measured on the RD-ND plane. The ODFs were calculated from the measured pole figures with the MTEX program.

In order to examine the defect evolution during the CT-ECAP deformation, PALS measurements were carried out on the 1, 4 and 8-pass CT-ECAP processed samples. The same method as for the RT-ECAP deformed samples was used.

### 5.2 Microstructure evolution of AA1050 during CT-ECAP deformation

TEM images of the CT-ECAP processed samples are shown in Fig. 5.1. It can be seen from the figures that the microstructure of CT-1 is dominated by networks of dislocation cells and subgrains. The mean linear size of the dislocation cells is approximately 600 nm, as listed in Table 5.1. Similarly, CT-2 contains subgrains with LAGBs and high dislocation densities. The grains are still not well defined. Fig. 5.1(c) shows that the microstructures of CT-4 are almost equiaxed and have a relatively high dislocation density in some grain interiors. The microstructure of CT-6 is similar to CT-4 with HAGBs and low dislocation density in the grain interiors. CT-8 shows a nearly equiaxed microstructure and the grain interiors have less dislocations than that of CT-4 and CT-6. The grain size decreases with increasing number of the CT-ECAP passes but it almost levels off after the fourth cycle.
deformation, which suggests that the refinement limits were reached at the fourth CT-ECAP cycle and further deformation is unable to further refine the structure.

![TEM micrographs of (a) 1, (b) 2, (c) 4, (d) 6 and (e) 8-pass CT-ECAP processed AA1050.](image)

**Fig. 5.1** TEM micrographs of (a) 1, (b) 2, (c) 4, (d) 6 and (e) 8-pass CT-ECAP processed AA1050.

**Table 5.1** Grain size of CT-ECAP processed samples measured from TEM micrographs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>CT-1</th>
<th>CT-2</th>
<th>CT-4</th>
<th>CT-6</th>
<th>CT-8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (nm)</td>
<td>590</td>
<td>554</td>
<td>445</td>
<td>422</td>
<td>409</td>
</tr>
</tbody>
</table>
5.3 Mechanical properties of CT-ECAP processed AA1050

The results of hardness measurements on the CT-ECAP processed samples are shown in Fig. 5.2. There is a significant increase in hardness after 1-pass of CT-ECAP deformation. After that the hardness increases with the number of passes in a much slower rate up to the sample after 4-cycle CT-ECAP and then slightly decreases when the deformation proceeds to the eighth pass.

![Hardness evolution of AA1050 with the number of CT-ECAP passes.](image)

The tensile strength and uniform elongation of the CT-ECAP processed samples are shown in Fig. 5.3. The evolution of the tensile strength follows the same trend, but slightly different from that of the hardness. Instead of the hardness being saturated after 4-pass CT-ECAP, the tensile strength continues to increase with the number of CT-ECAP passes. The uniform elongation drops markedly after only 1-cycle CT-ECAP and increases slightly thereafter but stays in a low value level.
Fig. 5.3 The relationship of (a) tensile strength and (b) uniform elongation with the number of CT-ECAP passes.

5.4 Texture evolution of AA1050 during CT-ECAP deformation

The average textures of the CT-ECAP processed samples were measured by neutron diffraction and the \{1 1 1\} pole figures are shown in Fig. 5.4. It can be seen that the textures of the CT-ECAP deformed samples are completely different from that of the RT-ECAP processed samples, when compared to the pole figures shown in Fig. 4.5. As observed in Fig. 4.5, the 1-pass and 8-pass RT-ECAP processed samples show monoclinic symmetry as ECAP deformation is close to simple shear deformation. However, there is no monoclinic symmetry in the CT-ECAP processed samples. It can be inferred that the deformation at cryogenic temperature caused this difference.

It can be seen from the pole figures that the initial Cube orientation rotates mostly around TD in a CCW direction with 1-pass CT-ECAP deformation, and the components are scattered along the rotation paths. The ideal texture of RT-ECAP through a 90° die is considered to be the ideal simple shear texture with 45° CCW rotation. The texture of CT-1 can be considered as Cube texture with a CCW rotation of about 35° around TD and some rotation along ED, which can be seen more clearly in the \{2 0 0\} pole figure shown in Fig. 5.5. During the second pass CT-
ECAP deformation, the major components seem to further rotate along TD but the rotation angle is much smaller. The major features of the 3 and more passes CT-ECAP processed samples are similar to CT-2, with only small variations of the relative intensities. The overall intensities of CT-1, CT-2 and CT-3 are close to each other, but the intensities are much smaller for the higher pass CT-ECAP processed samples.

Fig. 5.4 {1 1 1} pole figures of CT-ECAP processed samples.
Chapter 5 Equal channel angular pressing of AA1050 at cryogenic temperature

Fig. 5.5 \{2 0 0\} pole figure of CT-1.

The ODF figures of the CT-ECAP processed samples are shown in Fig. 5.6. It can be seen from the figures that the initial Cube texture has been completely transformed into other components after CT-ECAP deformation, and the original orthorhombic symmetry of the initial Cube texture has also been lost. The main features of the textures of CT-1, CT-2, CT-3 and CT-4 are different, which shows the evolution of the textures with the number of CT-ECAP passes. The textures of CT-4, CT-6 and CT-8 are quite similar, which indicates that the texture is stabilised at the fourth cycle and does not change much with further pressings.

The texture components of CT-1 and CT-4 are analysed in detail. The main components are marked in the ODF figures in Fig. 5.6(a) and (d) and in the \{1 1 1\} pole figures shown in Fig. 5.7. The orientations observed in $\varphi_2=0^\circ$ and $45^\circ$ are listed in Table 5.2 with the Euler angles and Miller indexes and are plotted in the two ODF sections in Fig. 5.8. The ECAP texture is usually measured on the ED-ND plane but is denoted by \{h k l\} $<u v w>$ with \{h k l\} parallel to the ED-TD plane and $<u v w>$ parallel to ED to compare with the simple shear texture. The same method is used to identify the CT-ECAP processed textures. The texture was measured and plotted...
on the ED-ND plane but is denoted with \(<h k l>\) parallel to ND and \(<u v w>\) parallel to ED.
Fig. 5.6(a) shows that the texture of CT-1 can be considered as initial Cube components that rotate along $\varphi_1$ for around 35° and along $\Phi$ for around 20°. The rotation can also be considered as rotation along $[1\ 3\ 6]$ for about 40°. The components are more or less uniform, with the highest intensity at orientations close to $(5\ 6\ \bar{2})[0\ 1\ 3]$ and $(1\ 0\ 3\ 9)\ [\bar{3}\ 1\ \bar{3}]$. Two fibres denoted as $f_{1C}$ and $f_{2C}$ are marked in Fig. 5.6(a). The $f_{1C}$ fibre starts from $(5\ 6\ \bar{2})[0\ 1\ 3]$, goes through...
(8\quad12\quad3)\quad[3\quad\overline{2}\quad0],\quad(0\quad1\quad\overline{2})\quad[5\quad\overline{4}\quad\overline{2}],\quad\text{and finishes at}\quad(14\quad25\quad\overline{2})\quad[7\quad\overline{4}\quad\overline{1}].\quad f_{2c}\quad\text{starts from}\quad(10\quad\overline{3}\quad9)\quad[\overline{3}\quad1\quad\overline{3}],\quad\text{passes through}\quad(\overline{20}\quad\overline{14}\quad19)\quad[\overline{4}\quad3\quad\overline{2}],\quad(\overline{10}\quad\overline{13}\quad12)\quad[\overline{9}\quad6\quad\overline{1}],\quad\text{and ends at}\quad(\overline{1}\quad\overline{3}\quad2)\quad[\overline{4}\quad2\quad1].\quad\text{The} f_{1c}\quad\text{and} f_{2c}\quad\text{fibres are marked with light orange and blue lines in the\{1\quad1\quad1\}\ pole figures shown in Fig. 5.7(a). It can be seen that the texture formed at the first CT-ECAP pass can be characterised by orientations along the two fibres.}
After the second pass CT-ECAP, the intensities of the orientations (3 0 1)[0 1 0] and (2 5 1 4 2)[4 7 1] in \( \varphi_2 = 0^\circ \), and orientation (2 3 3 1 2 0)[3 1 5] in \( \varphi_2 = 45^\circ \) decrease considerably. The major orientations and the \( f_{1C} \) and \( f_{2C} \) fibres shift slightly from the positions of CT-1. The intensities of the orientations along the \( f_{1C} \) and \( f_{2C} \) fibres are less uniform than CT-1 and the \( f_{2C} \) fibre is more complete. Another fibre with \( \varphi_1 = 330^\circ \) to 60°, \( \Phi = 75^\circ \) and \( \varphi_2 = 45^\circ \) also develops. The intensity of this fibre is
low but it distributes quite uniformly. The overall intensity increases slightly compared to CT-1.

![Diagram](image)

Fig. 5.6(e) CT-6 Max=7.7
The texture of CT-3 differs a lot from CT-1 and CT-2. The intensities of the major orientations (5 6 2) [0 1 3] and (10 3 9) [3 1 3] in CT-1 and CT-2 decrease significantly and split into two components close to the original position. The $f_{1C}$ and $f_{2C}$ fibres still can be seen but the intensities of the orientations along $f_{1C}$ are weak and appear to be uniform. However, the intensities of the orientations along $f_{1C}$ are non-uniform and have the highest intensity at (23 34 33) [3 3 1] ($\phi_2=160^\circ$, $\phi_1=90^\circ$).
Φ=45°, ϕ₂=60°). The Φ=75° fibre still exists and appears to be non-uniform, with the highest intensities at an orientation close to (40 23 34)[5 12 14] (ϕ₁=45°, Φ=75°, ϕ₂=45°).

The texture of CT-4 is closer to CT-2 but the overall intensity is markedly weaker. The f₁c fibre has high intensities at orientations close to (5 6 2)[0 1 3] and low intensities close to (7 7 2)[1 1 0] and (21 25 3)[7 6 1]. The f₂c fibre is more complete, which starts from orientation (10 3 9)[3 1 3] (ϕ₁=220°, Φ=70°, ϕ₂=0°), passes through (20 14 19)[4 3 2] (ϕ₁=210°, Φ=45°, ϕ₂=15°), (10 13 12)[9 6 1] (ϕ₁=190°, Φ=40°, ϕ₂=30°), (1 3 2)[4 2 1] (ϕ₁=160°, Φ=40°, ϕ₂=45°), (23 34 33) [3 3 1] (ϕ₁=160°, Φ=45°, ϕ₂=60°), (20 31 37)[5 8 4] (ϕ₁=150°, Φ=52°, ϕ₂=75°), and ends at (3 25 21)[1 6 7] (ϕ₁=130°, Φ=80°, ϕ₂=90°). High intensities are close to the (10 3 9)[3 1 3] and (23 34 33)[3 3 1] orientations. The Φ=75° fibre is named γc and has extended to ϕ₁=290°–60° interval. The fibre has shifted 20° from the γ fibre often observed in FCC materials subjected to rolling deformation. The γc fibre is characterised by [5 5 2] parallel to TD. The position of the fibre is plotted with light green lines in the {1 1 1} pole figure shown in Fig. 5.7(b). It can be seen that the texture components can be described quite well by the f₁c, f₂c, and γc fibres.

The texture of CT-6 is similar to CT-4, in both the position and intensity of the orientations. The overall intensity of CT-8 decreases from CT-4 and CT-6. The intensities of the components along the f₁c and f₂c fibres are more uniform than the other samples. The γc fibre has extended to ϕ₁=270°–90° interval.

It can be concluded that the texture developed during CT-ECAP is completely different from that developed during RT-ECAP. The near ideal shear deformation
occurred during RT-ECAP does not happen for CT-ECAP. The reason is attributed to the low deformation temperature. A detailed discussion is given in Section 5.6.2.

![Fig. 5.7](image)

**Fig. 5.7** \{1 1 1\} pole figures of (a) CT-1 and (b) CT-4 with the positions of identified component.

**Table 5.2** Texture components observed in $\phi_2$=0° and 45° ODF sections in CT-ECAP processed samples.

<table>
<thead>
<tr>
<th>Notation</th>
<th>Euler angles (°)</th>
<th>Miller indices</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\phi_1$</td>
<td>$\phi$</td>
</tr>
<tr>
<td>△</td>
<td>35</td>
<td>20</td>
</tr>
<tr>
<td>▽</td>
<td>110</td>
<td>85</td>
</tr>
<tr>
<td>▲</td>
<td>220</td>
<td>70</td>
</tr>
<tr>
<td>▼</td>
<td>297</td>
<td>8</td>
</tr>
<tr>
<td>★</td>
<td>130</td>
<td>80</td>
</tr>
<tr>
<td>□</td>
<td>310</td>
<td>8</td>
</tr>
<tr>
<td>☉</td>
<td>160</td>
<td>40</td>
</tr>
<tr>
<td>★</td>
<td>298</td>
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<td>★</td>
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<td>43</td>
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<tr>
<td>⊙</td>
<td>355</td>
<td>12</td>
</tr>
<tr>
<td>⊙</td>
<td>292</td>
<td>50</td>
</tr>
<tr>
<td>⊙</td>
<td>340</td>
<td>75</td>
</tr>
</tbody>
</table>
5.5 Vacancy-type defects evolution of AA1050 during CT-ECAP deformation

Fig. 5.9 shows the short lifetime \( \tau_1 \) and long lifetime \( \tau_2 \) of the CT-ECAP processed samples. The \( \tau_2 \) values are in the range of 240 to 250 ps, which are significantly higher than the values for RT-ECAP processed samples. The fractions of these two vacancy-type defects can be estimated by \( f_1 \times 220 + f_2 \times 245 = \tau_2 \) and \( f_1 + f_2 = 1 \), and the calculated fractions are listed in Table 5.3. CT-1 and CT-8 have \( \tau_2 \) values around 241 ps, which is between 220 and 245 ps. The \( f_2 \) values for both CT-1 and CT-8 are larger than 80%, indicating that bulk mono-vacancies are the major vacancy-type defects in these samples and a certain number of vacancies associated with dislocations still exist. Sample CT-4 exhibits a larger \( \tau_2 \) value of 251 ps, which is greater than that for bulk mono-vacancy. It is known that the positron lifetime increases as the size of vacancy-type defects increases. Thus, \( \tau_2 = 251 \) ps indicates that vacancy clusters exist in the sample. The positron lifetime of a di-
vacancy is 273 ps for Al [277]. Therefore, it is reasonable to assume that both bulk mono-vacancies and bulk di-vacancies exist in the sample CT-4 and the fraction of vacancies associated with dislocations is relatively small. By assuming that $f_1$ is negligible the fractions of bulk mono-vacancies and di-vacancies are 81.1% and 19.9%, respectively, determined by solving the equations $f_2 \times 245 + f_3 \times 273 = \tau_2$ and $f_2 + f_3 = 1$ with $f_3$ being the fraction of bulk di-vacancies. Fig. 5.9(b) shows that the short positron lifetime ($\tau_1$) increases from the sample CT-1 to CT-4 and then decreases from CT-4 to CT-8.

![Diagram](image)

**Fig. 5.9** Measured positron lifetimes of CT-ECAP processed samples (a) long lifetime and (b) short lifetime.
Table 5.3 Fractions of vacancy-type defects for CT-ECAP processed samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction (%)</th>
<th>Vacancies associated with dislocations ($f_1$)</th>
<th>Mono-vacancies in the bulk ($f_2$)</th>
<th>Di-vacancies in the bulk ($f_3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CT-1</td>
<td>14.0</td>
<td>86.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>CT-4</td>
<td>-</td>
<td>81.1</td>
<td>18.9</td>
<td></td>
</tr>
<tr>
<td>CT-8</td>
<td>15.2</td>
<td>-</td>
<td>84.8</td>
<td>18.9</td>
</tr>
<tr>
<td></td>
<td>(22.86)*</td>
<td>(70.30)</td>
<td>(6.84)</td>
<td></td>
</tr>
</tbody>
</table>

* The values in the brackets are the results when di-vacancies are assumed to exist in the sample CT-8.

Fig. 5.10 shows the intensities ($I_2$) of the long positron lifetime. It can be seen from the figure that CT-1 has the largest $I_2$ value, followed by a drastic decrease in CT-4 and then a slight decrease in CT-8.

![Intensity of long positron lifetime for CT-ECAP processed samples.](image)

5.6 Discussion

5.6.1 Comparison of microstructure and mechanical properties of RT- and CT-ECAP processed AA1050

ECAP deformation at cryogenic temperature is believed to be capable of depressing the dynamic recovery which will result in further grain refinement and strengthening of the materials. A comparison of the hardness (Fig. 4.2 and Fig. 5.2)
and tensile strength curves (Fig. 4.3 and Fig. 5.3) clearly depicts that the samples subjected to cryogenic processing have a higher hardness and tensile strength than those deformed at room temperature. The uniform elongation, however, shows basically no difference in either the trend or the values.

It can be seen in Fig. 4.1 and Fig. 5.1 that the microstructural features in the sample RT-1 and CT-1 are similar, thus no immediate differences can be distinguished by TEM at this stage. Both samples consist of a network of dislocation cells with a high dislocation density surrounded by low-angle boundaries. It can be seen that the dislocation density is quite low in most grain interiors of the sample RT-4. The grains in RT-4 appear to be fairly equiaxed and are surrounded by relatively high angle grain boundaries. However, the microstructure of the sample CT-4 is less equiaxed and shows a relatively high dislocation density in some grain interiors, which indicates that CT-4 is less evolved than RT-4. The grain size of CT-4 is slightly smaller than RT-4. This suggests that the dislocation cells have developed into grains or subgrains. The grains in RT-8 are reasonably equiaxed and most of the grains are free of dislocations in the interiors and surrounded by high angle grain boundaries. The microstructure of CT-8, however, shows a less equiaxed grain shape. The average grain size values of samples RT-8 and CT-8 are similar, which reveals that no obvious difference can be drawn from TEM observation at this magnitude. Although deformation at cryogenic temperature is expected to suppress dynamic recovery and further refine the microstructure, this has not achieved for ECAP deformation of commercial purity aluminium.

The microstructure evolution of the RT-ECAP process includes several stages: The first pressing substantially reduces the grain size and results in parallel bands of subgrains with dense dislocations inside. The microstructure after the second
pressing contains mainly large grains with a high dislocation density containing subgrains divided by LAGBs. After the fourth pass, many of the subgrain boundaries evolve into HAGBs and the grains change into reasonably equiaxed ultrafine grains, where the dislocation density reaches the highest value. For the 8-pass RT-ECAP processed sample, the microstructure evolves into equiaxed grains with HAGBs and most of the grain interiors are free of dislocations. The average dislocation density decreases slightly compared to the 4-pass RT-ECAP processed sample but it is still higher than that of the first few pass processed samples [278, 279].

It is well known that plastic deformation conducted at cryogenic temperature can suppress dislocation movement. This can induce two kinds of effects. First, as the dislocation movement is suppressed, it also suppresses the microstructure development and the grain refinement. This appears as the microstructures of the CT-ECAP processed samples are less evolved. The second effect is that the annihilation of the dislocations at sinks such as grain boundaries is also suppressed. This appears as the higher dislocation density in the grain interiors of the CT-ECAP deformed samples, whereas for the RT-ECAP deformed samples, the dislocations are easy to annihilate into the grain boundaries. This is especially so for those after four or more passes of RT-ECAP of which the grain sizes are small. The microstructure of RT-8 comprises equiaxed grains with lower density of dislocations than CT-8, as can be seen in Fig. 5.11(a) and (b). CT-8 also contains some grains with dislocation cells in the grain interiors and subgrains (Fig. 5.11(c)), which are the characteristics of the 2 or 3-pass RT-ECAP processed samples. It is reasonable to expect that the dislocation density in CT-8 is higher than RT-8.
5.6.2 Comparison of textures of RT- and CT-ECAP processed AA1050

The textures developed from RT-ECAP and CT-ECAP are completely different from each other, as can be seen from the pole figures shown in Fig. 4.5 and Fig. 5.4, and the ODFs shown in Fig. 4.6 and Fig. 5.6. The RT-ECAP textures can be well described by the f1, f2 and f3 fibres and the CT-ECAP textures can be expressed by the f1C, f2C and γC fibres. Only the intensities of the components along these fibres vary with the number of ECAP passes.

The textures of the multi-pass CT-ECAP processed samples are similar to the 1-pass deformed texture, with only variations of intensities. This phenomenon is just like the texture evolution of RT-ECAP processed samples. The difference is for RT-ECAP processed samples, for the first pass, the texture is closer to the ideal texture and
more complete than the textures of the multi-pass samples. However, the texture of the first pass deformed sample is less complete for the CT-ECAP processed samples, which is believed to be caused by the suppressed microstructure and texture evolution due to the low deformation temperature.

The first pass RT-ECAP texture usually has monoclinic symmetry, which is determined by the near simple shear deformation nature [228, 229, 280, 281]. The symmetry of multi-pass RT-ECAP textures depends on the processing route. Monoclinic symmetry was reported for route A [173, 282, 283] and most of route C [173, 229, 280]. For route B_A and B_C, however, the textures developed are highly dependent on the number of passes. In this work, monoclinic symmetry is lost for 4-pass RT-ECAP texture but is maintained for 8-pass texture. Monoclinic symmetry after 4-pass RT-ECAP was lost for many materials reported in the literature [173, 284].

The effect of the pressing temperature on ECAP texture evolution has not been reported a lot. The effect of temperature on texture evolution in a novel ECAP process was investigated by Qin et al. up to 513 K [285]. They found that the texture did not change much when the pressings were conducted at 353 and 433 K, but it changed remarkably when deformed at 513 K. ECAP deformation was rarely conducted at lower than room temperatures. The textures for lower than room temperature ECAP deformation were not reported in the literature, but the textures for rolling and simple shear deformation at low temperatures have been reported before. Generally speaking, low temperature makes the texture move to an alloy type texture [286].
For both rolling and simple shear deformation, ‘pure metal’ and ‘alloy’ type textures are used to describe the textures as the opposite extremes. High SFE metals and deformation at high temperatures usually result in pure metal type textures, whereas low SFE metals or alloys and deformation at low temperatures result in alloy type textures. The transition from pure metal to alloy type texture can be achieved by decreasing the deformation temperature [286]. Copper texture and Brass texture are regarded as the pure metal and alloy type texture in rolling, respectively. In simple shear, the difference between alloy type texture and pure metal texture is the replacement of A fibre by D fibre.

Ideal RT-ECAP texture is the same as the ideal texture of simple shear by a 45° CCW rotation. CT-ECAP texture can also be regarded as the texture of simple shear at cryogenic temperature by a 45° CCW rotation. Therefore, the texture of simple shear at cryogenic temperature can be obtained by rotating the CT-ECAP texture by 45° in a clockwise manner. However, the texture obtained in this way does not match with the D fibre, which is supposed to be the texture for simple shear at low temperatures. This suggests that the grain rotations that the materials have undergone during CT-ECAP are not the same as during RT-ECAP. More detailed tests are suggested for the CT-ECAP samples in order to find out the development process for CT-ECAP textures.

5.6.3 Comparison of vacancy-type defects of RT- and CT-ECAP processed AA1050

The fractions of vacancies associated with dislocations and bulk mono-vacancies are related to the dislocation density, the concentration of bulk mono-vacancies and the diffusion coefficient \(D\) of bulk mono-vacancies. A higher dislocation density or a higher concentration of bulk mono-vacancies means that the bulk
mono-vacancies need to traverse a shorter distance before encountering a dislocation, resulting in a higher $f_1$ and a lower $f_2$. A higher $D$ indicates that a larger fraction of bulk mono-vacancies can readily migrate to the vicinity of the dislocation core and incorporate into the dislocation structure. This also results in a higher $f_1$ value. $D$ can be described by the following equation, $D = D_0 e^{-E_M/kT}$ [257], where $D_0$ is a constant, $E_M$ is the activation energy for the migration of a bulk mono-vacancy, $k$ is the Boltzmann constant and $T$ is the absolute temperature. It is clear that the vacancy diffusion coefficient increases with temperature. Thus, when the samples are deformed at room temperature $D$ is high. The TEM image in Fig. 4.1(a) shows that the sample RT-1 is characterised by a high dislocation density. Both a high vacancy diffusion coefficient and a high dislocation density result in a larger fraction of vacancies associated with dislocations in RT-1, as shown in Table 4.4. However, when the deformation is carried out at cryogenic temperature (sample CT-1), the vacancy diffusion coefficient is low. Even though the sample CT-1 is also characterised by a high dislocation density, the low vacancy diffusion coefficient results in a lower fraction of vacancies becoming associated with dislocations. After 1-pass at cryogenic temperatures most mono-vacancies retain their identity as localised vacancy-type defects in the bulk rather than vacancies associated with dislocations. A fraction of bulk mono-vacancies may be annealed out during the subsequent temperature rise to room temperature. However, the temperature range for annealing of vacancy-type defects after low temperature deformation is appreciably higher than that after other vacancy-producing processes, such as irradiation [267]. The consequence is that the supersaturated bulk mono-vacancies remain in the sample CT-1 as reported in Table 5.3. Birnbaum
[267] has also found that the vacancies in the sample deformed at 77 K are not incorporated into the dislocation structure.

After 4 ECAP passes at room temperature the bulk mono-vacancies, rather than the vacancies associated with dislocations, become the dominant vacancy-type defects, as shown in Table 4.4. The reason can be attributed to the low dislocation density in the sample. The TEM observation in Fig. 4.1(c) shows that the sample RT-4 is characterised by relatively high angle grain boundaries and a low dislocation density in the grain interiors. This suggests that compared to the sample RT-1, the bulk mono-vacancies in RT-4 have less opportunity to meet dislocations, resulting in a higher fraction of bulk vacancies. For cryogenic ECAP processing, the dislocation density in the grain interiors decreases from the first pass to the fourth pass. However, since a high fraction of bulk mono-vacancies were generated in the first cryogenic ECAP pass, the concentration of bulk mono-vacancies increases continuously due to an accumulation of the dislocation-dislocation interactions. When the concentration of bulk mono-vacancies is high enough the mono-vacancies should combine to form di-vacancies since the binding energy of two vacancies is positive [267]. Therefore, di-vacancies are likely to be the second major type of vacancy-type defects in the sample CT-4. Di-vacancies in aluminium are more mobile than mono-vacancies because the activation energy for migration is lower (0.42–0.5 eV for di-vacancies [269] compared to 0.61–0.64 eV [135, 269] for mono-vacancies). This indicates that some di-vacancies may annihilate in the grain boundaries.

Both the sample RT-8 and CT-8 are characterised by high angle grain boundaries and a low dislocation density in the grain interiors. It has been found that the dislocation density in the 8-pass ECAP processed samples is lower than that in the
4-pass ECAP processed samples [135]. However, \( f_1 \) increases and \( f_2 \) decreases in RT-8 compared to RT-4. This indicates that the sample RT-8 has a high concentration of bulk mono-vacancies. The low \( \tau_1 \) value for RT-8 shown in Fig. 4.10(b) has confirmed this conclusion. Details of the analysis will be given in the following text. Since the long positron lifetime of the sample CT-8 is located between the bulk mono-vacancies and vacancies associated with dislocations, \( f_1 \) and \( f_2 \) for CT-8 have been determined as 15.2\% and 84.8\%, respectively. This result came from the measured long positron lifetime while assuming that the fraction of bulk di-vacancies is negligible. A medium amount of di-vacancies were identified in the sample CT-4. It is difficult to conclude that di-vacancies disappear completely in the sample CT-8 even though they are more mobile than bulk mono-vacancies. However, the fraction of di-vacancies (\( f_3 \)) could not be determined from the single long positron lifetime value.

\( K_N, K_D, \delta \) values and calculation uncertainties were calculated by the same method used in Section 4.6 and the results are listed in Table 5.4. The values for CT-ECAP processed samples can be compared with those for RT-ECAP processed samples listed in Table 4.5. CT-1 has a large \( \delta \) value, while the \( \delta \) values of the sample CT-4 and CT-8 are close to those of their room temperature counterparts.

### Table 5.4 Calculated \( K_N, K_D, \delta \) and calculation uncertainty for CT-ECAP processed samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( K_N \times 10^{10} \text{s}^{-1} )</th>
<th>( K_D \times 10^{10} \text{s}^{-1} )</th>
<th>( \delta ) (nm)</th>
<th>Uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CT-1</td>
<td>1.157</td>
<td>0.790</td>
<td>110.98</td>
<td>0.0526</td>
</tr>
<tr>
<td>CT-4</td>
<td>0.367</td>
<td>0.246</td>
<td>111.03</td>
<td>0.242</td>
</tr>
<tr>
<td>CT-8</td>
<td>1.889</td>
<td>3.284</td>
<td>54.26</td>
<td>0.216</td>
</tr>
</tbody>
</table>

It has been assumed that a mono-vacancy possesses the same volume as a single atom in the perfect material. The trapping cross section for a mono-vacancy is
determined to be 0.0787 nm\(^2\) in Section 4.6. By assuming that a di-vacancy has the same volume as double atoms and using the same method as in Ref. [272], the trapping cross section for a di-vacancy can be calculated and the value is 0.1249 nm\(^2\). Therefore, the specific trapping rate is \(\mu_{2v} = 1.388 \times 10^{-8} \text{ cm}^3/\text{s} \) or \(8.481 \times 10^{14} \text{ s}^{-1}\text{atom}^{-1}\) for bulk di-vacancies.

By applying Eq. (4.24) and (4.25), the \(C_N\) and \(C_D\) can be calculated for the CT-ECAP processed samples and the results are shown in Fig. 5.12. These results can be compared with the RT-ECAP processed samples shown in Fig. 4.12. It can be concluded that the sample CT-1 has slightly higher vacancy concentrations than RT-1. It should be kept in mind that the dominant defects in the sample CT-1 are bulk mono-vacancies rather than vacancies associated with dislocations. The higher concentration of bulk mono-vacancies in the grain interiors of the sample CT-1 introduce a higher driving force for vacancy annihilation to the grain boundaries, leading to a larger \(\delta\) in the sample CT-1 than the sample RT-1. After 4 ECAP passes at cryogenic temperatures, the vacancy concentrations in both the DR and NR have decreased due to the formation of di-vacancies. Since two mono-vacancies form one di-vacancy, the generation of di-vacancies reduces the total number of defects. In addition, bulk di-vacancies are more mobile than bulk mono-vacancies and some bulk di-vacancies will annihilate at the grain boundaries. After 8 cryogenic passes, barriers that seal the grain boundaries have been generated, for the same reasons as the sample RT-8, resulting in supersaturated vacancy-type defects. The sample CT-8 possesses lower vacancy concentrations than the sample RT-8 because di-vacancies may exist in the sample CT-8. The change of \(\delta\) values in the sample CT-4 and CT-8 should be the same as the sample CT-4 and CT-8, respectively, since the two groups of samples have similar \(\delta\) values after 4 ECAP passes.
5.6.4 Influence of vacancy-type defects on hardness

It can be concluded that the CT-ECAP processed samples are harder than the RT-ECAP processed samples by comparing Fig. 4.2 and Fig. 5.2. However, an examination by TEM revealed no significant difference in the microstructure between the two groups of samples. It is well known that dislocation pinning by vacancy-type defects can cause material hardening. Birnbaum stated that the yield stress is related to vacancy-type defects which retained their identity on arrival at the dislocation but not the defects which have become incorporated into the dislocation structure [267]. This indicates that bulk mono- and di-vacancies, rather than vacancies associated with dislocations, are the effective dislocation pinning centres. Due to the interaction between the effective pinning vacancies and dislocations, the material strength or hardness ($H$) is proportional to the concentration of the effective pinning vacancy-type defects and $D^{2/3}$ [267], where $D$ is the diffusion coefficient of the effective vacancy-type defect. We defined a parameter $\xi$ to consider the combined influence of the defect concentration and diffusion coefficients of the effective pinning vacancies on the hardness.
\[ \xi = f_2 \tilde{C}D_{1v}^{2/3} + f_3 \tilde{C}D_{2v}^{2/3} \]  

(5.1)

\[ \tilde{C} = \eta C_D + (1 - \eta)C_N \]  

(5.2)

where \( \tilde{C} \) is the overall concentration of vacancy-type defects. The subscripts \( 1v \) and \( 2v \) represent bulk mono- and di-vacancies, respectively. \( D \) can be expressed as [274]

\[ D = ga^2v_o \exp \left( \frac{S_M}{k} \right) \exp \left( -\frac{E_M}{kT} \right) \]  

(5.3)

\[ v_o = \frac{1}{a} \left( \frac{E_M}{m_a} \right)^{1/2} \]  

(5.4)

where \( g \) is a geometrical factor, \( a \) is the lattice constant, \( m_a \) is the mass of the atom, \( v_o \) is the attempt frequency associated with the jump of an adjacent atom into the vacancy, \( S_M \) is the entropy of migration of vacancy-type defects and \( E_M \) is the activation energy of migration of vacancy-type defect. In aluminium, \( g \) is 1 for a mono-vacancy and 1/6 for a di-vacancy, and \( S_M \) is set to 0.8\( k \) and 1.2\( k \) for mono- and di-vacancies, respectively [274], where \( k \) is the Boltzmann constant. \( E_M \) for a mono-vacancy is in the range of 0.61–0.64 eV [268, 269], while \( E_M \) for a di-vacancy is in the range of 0.42–0.5 eV [269]. In this work we have used \( E_M = 0.62 \) eV for a mono-vacancy and \( E_M = 0.46 \) eV for a di-vacancy.

Based on the definition of the parameter \( \xi \), an equation has been developed to describe the influence of vacancy-type defects on hardness:

\[ H = H_0(1 + A\xi) \]  

(5.5)

where \( A \) is a constant representing the dislocation pinning effect by vacancy-type defects and \( H_0 \) denotes the hardness of a vacancy-free sample. It is expected that \( A \) is a constant for all the samples, while \( H_0 \) depends on the dislocation density and
grain size. Since TEM observations did not show any significant differences in the microstructures of the RT- and CT-ECAP processed samples subjected to the same number of ECAP passes, we assume that the hardness difference between these samples is due to the dislocation pinning caused by bulk mono- and di-vacancies. Therefore, Eq. (5.5) can be expressed as

$$\frac{H_C}{H_R} = \frac{1 + A\xi_C}{1 + A\xi_R}$$  \hspace{1cm} (5.6)$$

where the superscripts $C$ and $R$ denote the CT-ECAP processed sample and the RT-ECAP processed sample subjected to the same deformation, respectively. $H_C$s and $H_R$s are shown in Fig. 4.2 and Fig. 5.2 and $\xi_C$s and $\xi_R$s can be determined by Eq. (5.1) using the values listed in Tables 4.4, 4.5, 5.3 and 5.4. As mentioned above, bulk di-vacancies may exist in the sample CT-8 but the fraction of di-vacancies could not be determined from the long positron lifetime. Therefore, only the constant $A$ for 1 and 4-pass samples has been calculated. The value $A = 1.53 \times 10^8$ was obtained for the sample RT-1 and CT-1 and $A = 1.52 \times 10^8$ was obtained for RT-4 and CT-4. These two results are in close agreement confirming that bulk mono- and di-vacancies play a significant role in the hardening of UFG materials. If we use the average of the above $A$ values and the long positron lifetime, the fractions of three types of vacancy-type defects for the sample CT-8 can be determined using

$$f_1 \times 220 + f_2 \times 245 + f_3 \times 273 = \tau_2$$

and Eq. (5.6). This gives the values $f_1 = 22.86\%$, $f_2 = 70.3\%$ and $f_3 = 6.84\%$. This result confirms our assumption that bulk di-vacancies exist in the sample CT-8 and contribute to the materials hardening.

### 5.7 Summary

AA1050 was subjected to ECAP deformation at cryogenic temperature. The microstructure, mechanical properties, texture, and vacancy-type defects evolution
of the CT-ECAP processed samples were investigated and compared with the same materials after RT-ECAP deformation. The summaries are listed as follows.

(1) Substantial grain refinement was achieved for the AA1050 after CT-ECAP deformation and the grain size is comparable but slightly smaller than the RT-ECAP processed samples. The RT-ECAP processed samples have an equiaxed microstructure after 4 and 8 pass ECAP while the CT-ECAP processed samples have slightly elongated grains and the dislocations in the grain interiors of CT-ECAP processed samples are higher.

(2) The CT-ECAP processed samples have higher hardness and tensile strength values than the RT-ECAP processed samples subjected to the same deformation, but the ductility of the two kinds of samples are similar to each other.

(3) The textures developed after CT-ECAP are completely different from that after RT-ECAP. The monoclinic symmetry observed for the 1-pass RT-ECAP processed sample is not observed for CT-ECAP processed samples. Three fibres \( f_{1C} \), \( f_{2C} \) and \( \gamma_C \) are used to characterise the texture components developed for the CT-ECAP processed samples.

(4) Three types of vacancy-type defects have been identified in the CT-ECAP processed samples: vacancies associated with dislocations, bulk mono-vacancies and bulk di-vacancies. The bulk mono-vacancies are the major vacancy-type defects in the CT-ECAP processed samples and bulk di-vacancies appear in the highly strained samples (CT-4 and CT-8).

(5) The increased hardness of the CT-ECAP processed samples can be attributed to the existence of bulk mono- and di-vacancies existing in these samples. These two types of defects are the major vacancy-type defects that can work as dislocations.
pinning centres and induce hardening. A model has been proposed to relate the hardness of the materials with the concentrations of vacancy-type defects and diffusion coefficients of bulk mono- and di-vacancies.
Chapter 6 Accumulative roll bonding of AA1050 sheets

ARB has been used to process various kinds of metallic materials successfully. Many aluminium based alloys have been processed by ARB in the literature, such as commercial purity aluminium [33, 34, 97, 101, 102, 105, 184, 287-290], AA2xxx [114], AA3xxx [181, 185, 288], AA5xxx [98, 291], AA6xxx [34, 42, 105, 292-294], AA7xxx [295] and AA8xxx [33, 37, 177, 296, 297]. All of the ARB processed aluminium alloys showed a submicron sized banded microstructure with high strength and low ductility compared with their coarse grained counterparts. The deformation inhomogeneity through the thickness direction was reported several times [42, 146, 298] but it has not been studied in detail. Comparison of different SPD techniques has been reported but not often and the comparison was not thorough [293, 299].

In this Chapter, AA1050 sheets were processed by ARB using rolls with high surface roughness (Ra~2 μm). The evolution of microstructure, mechanical properties, texture and vacancy-type defects were examined. The properties of the materials are compared with those processed by ECAP to the same levels of strain.

6.1 Experimental procedure

AA1050 sheets were produced by ARB using annealed AA1050 sheets as the starting materials. Two pieces of the sheets were stacked and welded together at one end, pre-heated in a furnace at 200 °C for 3 min and then rolled with a nominal reduction of 50% under dry condition, which gives a Von Mises equivalent strain of about 0.8. Note that the actual rolling temperature was lower than 200 °C. The
rolled samples were cut into two halves and stacked together again to perform the next cycle. The above procedure proceeded for five cycles to achieve an accumulative strain of about 4.

The mechanical properties were tested by tensile and hardness tests. The hardness measurement was taken using a load of 25 g and a dwell time of 12 s. The measurement was taken on the RD-ND plane along the thickness direction with 50 μm distance of two adjacent indents. Through-thickness hardness distributions and average hardness values were obtained.

Tensile specimens with a 25 mm gauge length and a 6 mm gauge width were processed with the longitudinal direction parallel to the rolling direction of the ARB processed samples. Tensile tests were conducted at ambient temperature and with an initial strain rate of $10^{-3}$/s.

The samples for optical microstructures were prepared by grinding and polishing to an OPS finish and then etching with Barker’s reagent. The microstructures were taken on the longitudinal cross-section (RD-ND plane) for the annealed and ARB processed samples.

TEM micrographs were taken on the RD-ND plane for the ARB processed samples. The 1, 3 and 5-cycle ARB processed samples were conducted to TEM observation. Grain size measurements were performed on several pictures measuring over 300 grains. Only the grain size normal to the rolling direction was measured.

The texture of the annealed AA1050 was measured by XRD on the surface of the RD-TD plane. For ARB processed samples, the through-thickness texture gradient was investigated by measuring the textures at the surface, quarter-thickness and the centre of the samples on the RD-TD plane. Specimens with a dimension of 10
mm (RD) by 10 mm (TD) were cut from the ARB processed sheets and then ground and polished to a Struers OPS finish to the surface, quarter-thickness and centre positions. The average texture was measured on the RD-ND plane by stacking 7 pieces of sheets together along the normal direction to form a surface of around 10 mm (RD) by 10 mm (ND). The incomplete pole figures from 0 to 85° of \{1 1 1\}, \{2 0 0\} and \{2 2 0\} were measured in each case. The 1, 3 and 5-cycle ARB processed samples were chosen to do texture measurement to investigate the texture evolution during ARB deformation. The complete pole figures and ODFs were calculated using X’Pert texture software after defocusing and background correction. The orientation intensities \(f(g)\) of the texture components along the major rolling and shear fibres were calculated.

In order to examine the defect evolution during ARB, PALS measurements were carried out on the 1, 3 and 5-cycle ARB processed samples using a fast-fast spectromter. The positron source consisted of ~30 µCi of \(^{22}\)NaCl sealed in 7 µm thick Kapton foil (grade HN from Goodfellow), and was sandwiched between two identical ARB processed samples. The samples were cut from the centre of the ARB processed sheets and ground with a 1200 grit SiC paper. The sample size was 20 mm (RD) by 20 mm (TD) by the thickness (~1.5 mm). The time resolution of the system is about 220 ps. The spectra comprised at least 2 million counts. An Al sample annealed at 600 °C for one hour was measured to determine the source lifetime and intensity. The MELT 4.0 program has been used to analyse the measured spectra.
6.2 Microstructure evolution of AA1050 sheets during ARB deformation

The AA1050 sheets before ARB had a fully annealed homogeneous microstructure, as shown in Fig. 3.3(a). The optical and TEM microstructures of the 1, 3 and 5-cycle ARB processed AA1050 sheets are shown in Fig. 6.1. The optical micrographs were taken on the RD-ND plane close to the thickness centre. It can be seen in Fig. 3.3(a) that the grains after 1-cycle ARB are strongly elongated along the rolling direction and the grain thicknesses decrease drastically compared to the initial microstructure. The grain thickness reduction after 3 and 5-cycle ARB is less substantial and the grains are strongly elongated along the rolling direction.

The microstructure of AA1050 after 1-cycle ARB comprises slightly elongated subgrains and dense dislocation cells. The grains after 3 and 5-cycle ARB are still slightly elongated and are aligned along a certain direction. The grain size reduces further compared to that after 1-cycle ARB, but the difference is not as substantial as in the optical micrographs. Most of the grains are clearly defined with high angle boundaries after 5-cycle ARB, while some grains involved a high density of dislocations at the grain interiors. This is the typical microstructure of ARB processed commercial purity aluminium [33, 101]. The angle of the grain boundaries increases with the number of ARB cycles, as can be indicated by the trend to more complex and diffused diffraction patterns.

The grain sizes of the 1, 3 and 5-cycle ARB processed AA1050 sheets measured from TEM micrographs are approximately 465 nm, 406 nm and 300 nm, respectively. These grain size values are slightly higher than those reported for commercial purity Al [33, 101, 300], and the microstructure of the AA1050 appears
to be more equiaxed compared to similar alloys reported in the literature [33, 101, 300]. This is believed to be due to pre-heating before rolling, which accelerates the recovery process and prevents grain refinement.

**Fig. 6.1** (a)–(c) Optical micrographs and (d)–(f) TEM micrographs of 1, 3 and 5-cycle ARB processed AA1050 sheets.

### 6.3 Mechanical properties of ARB processed AA1050 sheets

#### 6.3.1 Tensile properties

Tensile test results of AA1050 sheets before and after 1 to 5-cycle ARB deformation are shown in Fig. 6.2. The engineering stress-strain curves (Fig. 6.2(a)) show that
the stress increases and the elongation decreases after ARB deformation. The average values of ultimate tensile strength (UTS) and uniform elongation of the ARB processed AA1050 sheets are shown in Fig. 6.2(b). These values were calculated by averaging over three samples. The figure shows that after 1-cycle of ARB, the UTS of the sheet is about 172 MPa, which is about twice the strength of AA1050 before ARB deformation (84.8 MPa). The tensile strength increases with the number of ARB cycles from the second to the fifth cycle ARB, although at a slower rate. The value increases up to 243 MPa after 5-cycle ARB, which is about three times the initial value. This indicates that the materials are efficiently strengthened by ARB deformation. The uniform elongation after 1-cycle ARB is about 1.5%, which drops substantially compared to the original material and then stays about the same with further ARB cycles.

Unlike coarse grained metals, the UFG materials have tensile curves that peak immediately after yielding, which results in low ductility. The high strength and low ductility in tensile tests is the typical behaviour of UFG materials. Most of the reported UFG materials are typically several times stronger than their coarse grained counterparts, but at the same time, the elongation to failure is no more than a few percent [164, 301]. The strain hardening rate of the ARB processed sheets is higher than the original annealed material at low true strain but it decreases rapidly at the very early stage. For the initial annealed materials, on the other hand, the strain hardening rate remains at a relative high value even at a strain as high as 30%. The rapid decrement in the strain hardening rate in the ARB processed sheets is responsible for the early deformation instability and early fracture.
Fig. 6.2 (a) Engineering stress-strain curves, (b) Tensile strength and uniform elongation and (c) strain hardening rate of 1 to 5-cycle ARB processed AA1050 sheets.
6.3.2 Hardness

The average hardness evolution of AA1050 sheets with the number of ARB cycles is shown in Fig. 6.3. The trend of average hardness with the number of ARB cycles is similar to that of tensile strength. The hardness increases considerably after 1-cycle ARB and increases further with more ARB cycles, but at a lower rate. This increment continues up to 5-cycle ARB deformation without saturation, as reported for many materials, which is due to pre-heating before ARB [300]. The hardness doubles after about 3-cycle ARB.

Fig. 6.3 Average hardness values of 1 to 5-cycle ARB processed AA1050 sheets.

Fig. 6.4 shows the through-thickness optical micrographs of 1, 3 and 5-cycle ARB processed AA1050 sheets and the hardness distribution throughout the thickness direction. As can be seen from the through-thickness microstructure, good bonding with no delamination between the sheets was attained at each cycle of ARB. Fig. 6.4(a) shows that after 1-cycle ARB, the grain size around the centre of the sheet is much larger than that close to the surface. The microstructures of the 3 and 5-cycle ARB processed sheets are more uniform and the grain sizes are much smaller.
Fig. 6.4 Through-thickness hardness distribution of (a) 1, (b) 3 and (c) 5-cycle ARB processed AA1050 sheets.
The hardness along the thickness direction was measured and it shows that after 1-cycle ARB the hardness has the lowest values around the centre of the sheet and gradually increases towards the surface. This behaviour was observed previously and was explained by the redundant shear strain near the surface [42]. The hardness distribution is in correspondence with the grain size distribution. The hardness distribution after 3-cycle ARB is more uniform than that after 1-cycle ARB, with high values close to the surfaces and interfaces. The microstructure appears to be uniform at the magnification of the optical microscope. The higher hardness close to the surfaces are due to the redundant shear strain caused by friction between the sheet and the rolls, and the higher hardness close to the layer interfaces are due to wire brushing before each cycle ARB and the redundant strain from the previous cycle. The hardness distribution after 5-cycle ARB is more uniform since the microstructure is also distributed more uniformly and the interfaces are close to each other.

6.4 Texture evolution of AA1050 sheets during ARB deformation

The $\varphi_2$=0°, 45° and 65° ODF sections of the AA1050 sheets before ARB are shown in Fig. 6.5. The initial texture of the annealed AA1050 is dominated by Cube orientation, which is the typical annealed texture of aluminium alloy and is in correspondence with the fully annealed microstructure shown in Fig. 3.3(a).

The Euler angles of the texture components and fibres observed in this work are shown in Table 6.1 and Table 6.2, respectively. The Cube texture is the major texture in the annealed samples before ARB deformation, while $\alpha$ fibre composed of Goss and Brass orientation, and $\beta$ fibre composed of Brass, Copper/Dillamore and S/R orientations are the major rolling type texture. Since the surfaces of the
rolling sheets generally undergo shear deformation, shear type orientations such as E, F, Rotated Cube and Rotated Copper are often observed in ARB processed materials. The positions of the texture components and important fibres observed in this work are shown in the \( \varphi_2=0^\circ, 45^\circ \) and \( 65^\circ \) ODF sections in Fig. 6.6.

**Fig. 6.5** \( \varphi_2=0^\circ, 45^\circ \) and \( 65^\circ \) ODF sections of annealed AA1050 before ARB. (Contour levels 0.5x)

<table>
<thead>
<tr>
<th>Notation</th>
<th>Miller indices {h k l}&lt;u v w&gt;</th>
<th>Euler angles (°)</th>
<th>Belong to fibre</th>
<th>Texture type</th>
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<tr>
<td>Cube</td>
<td>{0 0 1}&lt;1 0 0&gt;</td>
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<td>( \phi )</td>
<td>( \varphi_2 )</td>
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<tr>
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<td>{0 1 1}&lt;1 0 0&gt;</td>
<td>0</td>
<td>45</td>
<td>0</td>
</tr>
<tr>
<td>Brass</td>
<td>{0 1 1}&lt;2 1 1&gt;</td>
<td>35</td>
<td>45</td>
<td>0</td>
</tr>
<tr>
<td>Copper</td>
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<td>90</td>
<td>35</td>
<td>45</td>
</tr>
<tr>
<td>D(Dillamore)</td>
<td>{4 4 11}&lt;11 11 8&gt;</td>
<td>90</td>
<td>27</td>
<td>45</td>
</tr>
<tr>
<td>R</td>
<td>{1 2 4}&lt;2 1 1&gt;</td>
<td>57</td>
<td>29</td>
<td>63</td>
</tr>
<tr>
<td>S</td>
<td>{1 2 3}&lt;6 3 4&gt;</td>
<td>59</td>
<td>37</td>
<td>63</td>
</tr>
<tr>
<td>E (A)</td>
<td>{1 1 1}&lt;1 1 0&gt;</td>
<td>0/60</td>
<td>55</td>
<td>45</td>
</tr>
<tr>
<td>F (A1*/A2*)</td>
<td>{1 1 1}&lt;1 1 2&gt;</td>
<td>90/30</td>
<td>55</td>
<td>45</td>
</tr>
<tr>
<td>Rotated Cube (C)</td>
<td>{0 0 1}&lt;1 1 0&gt;</td>
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<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Rotated Copper (B)</td>
<td>{1 1 2}&lt;1 1 0&gt;</td>
<td>0</td>
<td>35</td>
<td>45</td>
</tr>
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Table 6.2 Characteristic rolling and shear type fibres in FCC materials.

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Fibre axis</th>
<th>Euler angles (°)</th>
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<tr>
<td></td>
<td></td>
<td>$\phi_1$</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>$&lt;1 1 0&gt;//ND$</td>
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</tr>
<tr>
<td>$\beta$</td>
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<tr>
<td>$\tau$</td>
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<td>90</td>
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<tr>
<td>${1 1 1}/\gamma$</td>
<td>$&lt;1 1 1&gt;//ND$</td>
<td>0–90</td>
</tr>
<tr>
<td>$&lt;1 1 0&gt;$</td>
<td>$&lt;1 1 0&gt;//TD$</td>
<td>0</td>
</tr>
</tbody>
</table>

Fig. 6.6 Schematic representation of the important texture components and fibres in FCC materials.

6.4.1 Texture evolution of AA1050 sheets with the number of ARB cycles

Fig. 6.7 shows the $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the average texture measured on the stack of RD-ND planes of the AA1050 sheets after 1 to 5-cycle ARB deformation. It can be seen from the figures that the Rotated Cube texture developed after 1-cycle ARB and remains strong in intensity up to 5-cycle ARB. The $\beta$ fibre starts to develop from the first cycle ARB and the intensity increases with the number of ARB cycles, along with weak Brass and strong Copper and S orientations. Weak shear type orientation Rotated Copper is observed throughout the ARB deformation. Weak E is observed in the 1-cycle ARB processed sample and evolves to F from the second cycle on. It can be concluded that the Copper, S and
Rotated Cube orientations are the most stable orientations in the ARB processed sheets.

Fig. 6.7 $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the average texture of the 1 to 5-cycle ARB processed AA1050 sheets. (Contour levels 0.5x)
The intensities of the textures after ARB deformation are much weaker than the annealed sample. The maximum intensity is located at the Rotated Cube orientation and the intensity increases from 1 to 4-cycle ARB processed sample and drops slightly at the fifth cycle.

6.4.2 Through-thickness texture distribution of ARB processed AA1050 sheets

In order to investigate deformation asymmetry during the ARB process, the texture distribution throughout the thickness direction was measured. Fig. 6.8 shows the $\varphi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of texture measured at the centre, quarter-thickness and the surface of the AA1050 sheets after 1, 3 and 5-cycle ARB. It can be seen in Fig. 6.8 that texture gradient exists in all the samples measured.
Fig. 6.8 $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of texture measured at the centre, quarter-thickness and surface of the (a)1, (b)3 and (c)5-cycle ARB processed AA1050 sheets. (Contour levels 0.5×)

The texture in the centre of the 1-cycle ARB processed sample is characterised by the initial Cube texture, $\alpha$ fibre and $\beta$ fibre developed during the rolling process. The intensities of the Cube orientation and different orientations along the $\alpha$ and $\beta$
fibres are quite uniform. At the quarter-thickness position, Rotated Cube orientation is dominant, together with weak initial Cube texture and other shear type texture such as Rotated Copper and \{1 1 1\} fibre. The texture at the surface of the 1-cycle ARB processed sample is near identical with the quarter-thickness, with a strong Rotated Cube orientation and weak \{1 1 1\} and <1 1 0> fibres.

For the 3-cycle ARB processed sheet, the initial Cube texture has almost disappeared. The central position is characterised by the β fibre with Copper as the strongest orientation and Brass as the weakest, while Copper has shifted towards the D orientation. The texture of the quarter-thickness position is composed of strong shear component Rotated Cube, and strong rolling components Copper and S. The texture at the surface position is typical shear type texture, with a strong Rotated Cube orientation and weak \{1 1 1\} and <1 1 0> fibres, which is almost the same as the texture of the 1-cycle ARB processed sample at the surface position.

The textures at different positions for the 5-cycle ARB processed sample are similar to that of the 3-cycle sample. It is characterised by rolling type texture (β fibre) at the centre, shear type texture at the surface (strong Rotated Cube orientation and weak \{1 1 1\} and <1 1 0> fibres) and a combination of rolling and shear type textures at the quarter-thickness position (strong Rotated Cube, Copper and S). This type of texture is the stabilised texture of the ARB processed sheets.

In order to interpret the texture evolution and distribution of the ARB processed AA1050 sheets in detail, the strength variations of the main texture components along main fibres are summarised in Fig. 6.9 and Fig. 6.10. The α, β and τ fibres which are always analysed in rolling deformation are calculated for all the positions. The \{1 1 1\} and <1 1 0> fibres, which are typical shear fibres, are shown for the
surface and average textures. Strong texture gradient throughout the thickness can be observed in the figures.

Fig. 6.9 α, β and τ fibres of texture measured at the centre, quarter-thickness, surface and the average texture of the 1, 3 and 5-cycle ARB processed AA1050 sheets. (Black represents 1-cycle ARB processed sample, red 3-cycle and blue 5-cycle, same for Fig. 6.10)

The texture at the centre of all the 1, 3 and 5-cycle ARB processed samples is mainly composed of rolling texture. The initial Cube orientation evolves into rolling texture with uniform intensities along the α and β fibres and then evolves into a Copper dominated β fibre. The intensities of the Brass and Copper orientations
increase with the number of ARB cycles, while the intensities of S increase from 1 to 3-cycle and then decrease from 3 to 5-cycle ARB. The intensities of Copper orientation are much stronger than S and Brass and the highest intensity is located between Copper and D for the 1-cycle ARB processed sample and moves towards the D and Rotated Cube orientation with further deformation.

The textures of 1 and 3-cycle ARB processed samples at the quarter-thickness are similar to each other. The Rotated Cube orientation is dominant and the intensity increases from 1 to 3-cycle ARB. The texture of the 5-cycle ARB processed sample is composed of Copper dominated β fibre and Rotated Cube. The intensity of Copper is higher than Rotated Cube and the maximum intensity of the Copper orientation has shifted towards D position.

![Texture plots](image)

**Fig. 6.10** {1 1 1} and <1 1 0> fibres of texture measured at surface and the average texture of the 1, 3 and 5-cycle ARB processed AA1050 sheets.
The textures at the surface of the 1, 3 and 5-cycle ARB processed sheets are quite similar. The β fibre has disappeared completely and Rotated Cube dominated shear type textures are observed. As can be observed from the τ fibre in Fig. 6.9 that the intensity of Rotated Cube reduces after 3 and 5-cycle ARB and the intensity of the F component increases with increasing ARB cycles, as can be seen in the {1 1 1} fibre in Fig. 6.10. The intensity of the F component is comparable to that of Rotated Cube.

The average textures are a combination of rolling texture mainly β fibre and shear texture mainly Rotated Cube. The intensities of the orientations along the β fibre and that of the Rotated Cube orientation increase with the number of ARB cycles. The β fibre is Copper dominated and the intensities of the Copper orientation are always slightly weaker than those of Rotated Cube. Other shear components such as E, F and Rotated Copper are extremely weak in the average texture.

6.5 Vacancy-type defects evolution of AA1050 sheets during ARB deformation

The PALS results of 1, 3 and 5-cycle ARB processed AA1050 sheets are shown in Fig. 6.11. The τ₁ values are smaller than the bulk lifetime, which indicates annihilation into the bulk. The τ₂ values lie between the values of vacancies associated with dislocations and mono-vacancies in the bulk, but are closer to the former. It can be seen that the τ₂ value increases from 1 to 3-cycle ARB processed AA1050 and then slightly decreases from 3 to 5-cycle sample. The long lifetime τ₂ also has the maximum intensity for the 3-cycle sample while that for the 1 and 5-cycle ARB processed samples are lower. However, the differences between the τ₂ values and the intensities are not substantial. The τ₂ values lie between 221 ps and 228 ps and
the intensities are between 92% and 94%, which indicates that all three samples have similar types of defects type and concentration. The fractions of $\tau_1$ and $\tau_2$ can be estimated by $f_1 \times 220 + f_2 \times 245 = \tau_2$ and $f_1 + f_2 = 1$. The results are listed in Table 6.3.

![Positron lifetime results of ARB processed AA1050 sheets](image)

**Fig. 6.11** Positron lifetime results of ARB processed AA1050 sheets (a) short lifetime, (b) long lifetime and (c) intensity of long lifetime.
Table 6.3 Fractions of vacancy-type defects for ARB processed AA1050 sheets.

<table>
<thead>
<tr>
<th>Number of ARB cycles</th>
<th>Vacancies associated with dislocations ($f_1$)</th>
<th>Mono-vacancies in the bulk ($f_2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>92.4</td>
<td>7.6</td>
</tr>
<tr>
<td>3</td>
<td>69.6</td>
<td>30.4</td>
</tr>
<tr>
<td>5</td>
<td>85.6</td>
<td>14.4</td>
</tr>
</tbody>
</table>

It can be seen that the fraction of vacancies associated with dislocations $f_1$ for 1 and 5-cycle ARB processed AA1050 are dominant while $f_1$ for the 3-cycle ARB processed sample is around 70%, indicating that vacancies associated with dislocations are the major vacancy-type defects whereas there are a fairly large amount of mono-vacancies in the bulk.

For ARB processed sheets, the bonding interfacial areas are different from the bulk because there might be oxide and some contaminations, or even voids if the bonding quality is poor. However, long lifetimes corresponding to vacancy clusters or voids have not been detected in the ARB processed AA1050 sheets, which indicates that large vacancy clusters or voids do not exist along the interfaces. It can be inferred that the sheets are well bonded.

6.6 Discussion

AA1050 has been deformed by RT-ECAP, CT-ECAP and ARB in this work. The properties of AA1050 after the three kinds of deformation up to the same strain level of ~4 (4-pass RT- and CT-ECAP and 5-cycle ARB) are compared and discussed in the following.

Although the starting materials of the three types of deformation are fully annealed AA1050 and the accumulative strains of the samples discussed in this
section are all around 4, the strain paths of the three types of deformation are different, which leads to a different combination of properties. The average grain size of the 5-cycle ARB processed AA1050 is about 300 nm, which is significantly smaller than the 4-pass RT- and CT-ECAP processed AA1050, as listed in Table 6.4. Note that the grain sizes are averaged from the widths of the slightly elongated grains. The TEM micrographs of RT-4, CT-4 and 5-cycle ARB processed AA1050 are shown in Fig. 4.1(c), Fig. 5.1(c) and Fig. 6.1(f), respectively. It can be seen that the grains of all three samples are slightly elongated, among which the microstructure of RT-4 is more equiaxed than the others. The average hardness and tensile strength of the three samples are also listed in Table 6.4, which shows that the hardness and tensile strength of the 5-cycle ARB processed AA1050 is considerably larger than the ECAP processed samples. The average hardness of 4-pass ECAP processed samples almost doubles the initial value whereas the value increases to about 2.5 times the initial value for the 5-cycle ARB processed sample. It can be concluded that ARB deformation is more efficient in microstructure refinement and strengthening. Note that the ARB deformation in this work was conducted at an elevated temperature, which may accelerate the dynamic recovery during deformation and restrict the microstructure refinement and strengthening. The efficiency of ARB deformation would be more obvious if the ARB process was conducted at room temperature or lower. Cherukuri et al. conducted ARB and ECAP deformation on AA6061 up to the same accumulative strain at the same temperature and found that ARB processed samples had higher hardness and strength [293], which is consistent with the results of the present work.
Table 6.4 Comparison of AA1050 deformed by ECAP and ARB up to a strain of ~4.

<table>
<thead>
<tr>
<th></th>
<th>4-pass RT-ECAP</th>
<th>4-pass CT-ECAP</th>
<th>5-cycle ARB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (nm)</td>
<td>608</td>
<td>445</td>
<td>300</td>
</tr>
<tr>
<td>Hardness (Hv)</td>
<td>48.8</td>
<td>53.5</td>
<td>63.7</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>145.2</td>
<td>155.4</td>
<td>243</td>
</tr>
<tr>
<td>Short lifetime $\tau_1$ (ps)</td>
<td>122.3</td>
<td>143.8</td>
<td>21.5</td>
</tr>
<tr>
<td>Long lifetime $\tau_2$ (ps)</td>
<td>240</td>
<td>250.3</td>
<td>223.6</td>
</tr>
<tr>
<td>Intensity of long lifetime $I_2$</td>
<td>86.6</td>
<td>81.7</td>
<td>92.0</td>
</tr>
</tbody>
</table>

The textures of the three types of samples are completely different from each other due to different strain paths and deformation temperatures. The ideal deformation of ECAP is simple shear and that of ARB is plane strain compression at the centre and simple shear close to the surface. Since the rolls used in this work were deliberately treated to have high surface roughness, the simple shear deformation close to the surface area is more severe (The influence of high roll roughness is discussed in Section 7.6). The texture components that resulted from the simple shear deformation in ARB are actually the same as the ideal components from simple shear. They appear in different positions of the ODFs due to the different representation used in rolling and simple shear deformation. The difference is in ARB, rotated Cube component is always the strongest, while in ECAP, the $C_0$ component (CCW rotated from Rotated Cube component of simple shear) is the strongest for the 1-pass deformed sample but not for the multi-pass samples. In general, the textures of ECAP and ARB are not comparable since the positions of shear plane and shear direction are different with respect to the sample geometry.

The long positron lifetime $\tau_2$ of the 5-cycle ARB processed AA1050 is 223.6 ps, which is much smaller than those for RT-4 and CT-4, as listed in Table 6.4. The lifetimes of 4-pass RT-ECAP processed and 5-cycle ARB processed AA1050 are
between the values of vacancies associated with dislocations and mono-vacancies in the bulk, with the former closer to mono-vacancies and the latter closer to vacancies associated with dislocations. The lifetime of 4-pass CT-ECAP processed AA1050 is larger than the value of mono-vacancies in the bulk. The intensities of the long lifetime $I_2$ for all three samples are higher than 80%. It can be found from the lifetime values that vacancies associated with dislocations are the dominant vacancy-type defects in the 5-cycle ARB processed sample, whereas bulk mono-vacancies are dominant in the 4-pass RT-and CT-ECAP processed samples. The fraction of the positrons annihilated in the bulk of the 5-cycle ARB processed sample is smaller than the ECAP processed samples, indicating a large fraction of defects in the ARB processed sample. The defect fraction is difficult to be detected by other methods. As can be seen from the TEM micrographs, the dislocation densities of all three samples are high but it is not possible to determine which sample has the higher fraction.

The ARB process is more efficient in grain refinement and strengthening, as stated above. However, the accumulation of vacancy-type defects in ARB deformation seems less efficient. The major reason might be because of the elevated deformation temperature for ARB. The lifetime values obtained in this work are also smaller than the lifetimes of pure aluminium processed by ARB at room temperature [302]. Although the stress state in the SPD processed sample is able to restrict the mobility of vacancies, the intermittent heating temperature used in this work is high enough for thermal activation of dislocations, the movement of which would result in vacancy annihilation to the dislocations or other defects like the grain boundaries. Since the concentration of mono-vacancies is smaller for the 5-cycle ARB processed AA1050, the number of vacancy pinning centres is therefore
less than the 4-pass RT- and CT-ECAP processed samples. The higher hardness and strength of the 5-cycle ARB processed AA1050 is most probably resulted from grain refinement hardening due to the smaller grain size compared with the ECAP processed samples.

6.7 Summary

The results from this detailed study of the microstructure, mechanical properties, texture, and vacancy-type defects evolution of AA1050 sheets processed by ARB are summarised as follows.

(1) AA1050 sheets with good bonding were obtained after ARB deformation and substantial grain refinement and significant strengthening were achieved.

(2) After 1-cycle ARB, the hardness has the lowest values around the centre of the sheet and gradually increases when moves to the surface. The hardness distribution after 3 and 5-cycle ARB is more uniform than that after 1-cycle ARB, with the high values close to the surfaces and interfaces. The hardness distribution is in correspondence with the grain size distribution.

(3) During the ARB process, the intensities of the texture increase with the number of ARB cycles. Copper dominated β fibre and Rotated Cube component are the most favourable texture in the ARB process. For the average texture, the Rotated Cube component is the most significant component for all the ARB processed samples.

(4) Vacancies associated with dislocations are dominant for 1 and 5-cycle ARB processed AA1050, while for the 3-cycle ARB processed AA1050, vacancies associated with dislocations are the major vacancy-type defects but there exists a
fairly large number of bulk mono-vacancies. Long lifetimes corresponding to vacancy clusters or voids are not detected, which indicates that the interfaces are well bonded.

(5) The grain size of the AA1050 after ARB is smaller than those after RT- and CT-ECAP to the same strain and the hardness and tensile strength are higher, which indicates that the ARB process is more efficient in grain refinement and strengthening compared with ECAP. The long positron lifetime of the ARB processed AA1050 is much smaller than those for RT- and CT-ECAP processed AA1050 which is probably due to the elevated deformation temperature during ARB.
Chapter 7 Accumulative roll bonding of AA6061 sheets

Aluminium alloys have a wide usage in the industry for its light-weight nature. Aluminium alloys with UFG structure with extraordinary mechanical properties have a high potential in the engineering application. However, some of the applications are restricted due to the poor drawability of the aluminium alloy sheets. The deep drawability is determined by Lankford value \( r \) [303, 304]. The average \( r \) value is defined as \( \bar{r} = \left( r_0 + 2r_{45} + r_{90} \right)/4 \), and the variation of the \( r \) value in the plane of the sheet is characterised by \( \Delta r \) value. Materials with a higher \( \bar{r} \) value and a lower \( \Delta r \) value generally have good formability. The Lankford value of aluminium alloy sheets is generally low due to the texture developed by conventional rolling, which is mainly composed of \( \beta \) fibre. This results in rolled sheets with poor deep drawability. After annealing, \( \beta \) fibre always transforms to Cube orientation. These components have a low \( \bar{r} \) value and high \( \Delta r \) value, which are not favourable for drawing [304]. However, materials like low carbon steel usually have \( \gamma \) fibre after rolling and annealing. \( \gamma \) fibre is also known as \( \{1 1 1\} // ND \) fibre, which is composed of components E and F. Both the components have high \( \bar{r} \) value and low \( \Delta r \) value and are more favourable for deep drawing [304]. A lot of effort has been applied to modify the texture developed by rolling to improve shear texture fraction, such as asymmetrical rolling [305, 306], rolling at high frictions between the sheet and rolls [104] and using rolls with small or large draughts [307-309].

ARB technique offers an opportunity to control the textures and the plastic anisotropy of sheet materials. The texture evolution throughout the rolled sheets...
has been studied by many researchers, and textures developed in ARB are generally characterised by rolling-type components at the centre and shear-type components near the surface [34, 105, 106, 122, 177, 184, 185, 310]. As stated above, the shear texture is more favourable for deep drawing [304]. However, the texture developed in ARB process is not uniform. In the ARB process, the rolled samples were cut into two halves and stacked together again to perform the next cycle. The surface of the previous cycle is going to be deformed at the centre in the next cycle. Several studies showed that the shear type texture would rotate to rolling texture when deformed at the centre. As the materials at the surface continuously being cycled into the centre, and the materials in the centre are moving progressively outwards during the following cycles, which are combined with the effect of shear deformation at the surface and plane strain compression at the centre, texture anisotropy through the thickness would develop, which make it difficult to develop uniform shear texture [179].

In this Chapter, AA6061 was processed by ARB using rolls with high surface roughness ($R_a \approx 2 \mu m$). The evolution of microstructure, mechanical properties, texture and vacancy-type defects were examined.

### 7.1 Experimental procedure

AA6061 sheets were produced by ARB by using annealed AA6061 sheets as the starting materials. The processing procedure was the same as the ARB deformation of AA1050 sheets described in Section 6.1. A total of 5 cycles of ARB was conducted to achieve an accumulative strain of about 4.

The mechanical properties were tested by tensile and hardness tests. The same testing parameters as the AA1050 sheets were used. The microstructure of the ARB
processed AA6061 sheets was investigated by OM and TEM. The texture was measured by XRD using the same methods as for the AA1050 sheets. PALS measurements were also carried out in order to examine the defect evolution during ARB. The 1, 3 and 5-cycle ARB processed AA6061 sheets were used for all the testings. The samples were prepared by the methods used for the AA1050 sheets listed in Section 6.1 for each of the testings. The same techniques for data analysis were also used in this Chapter.

7.2 Microstructure evolution of AA6061 sheets during ARB deformation

The initial microstructures of AA6061 sheets before ARB comprises of pancake shaped grains, which are slightly elongated along the original rolling direction, as shown in Fig. 3.3(b). Dense precipitates can be seen in AA6061, which are not observed in AA1050. Optical and TEM micrographs of the 1, 3 and 5-cycle ARB processed AA6061 are shown in Fig. 7.1. The optical micrographs, taken close to the centre interfaces on the RD-ND surface, show elongated grains for the 1-cycle ARB processed AA6061 which are aligned along the rolling direction. As the number of ARB cycles increases, the grains become finer. A few shear bands can be observed in the 3-cycle ARB processed sample, which can be frequently identified in the 5-cycle ARB processed sample.
Chapter 7 Accumulative roll bonding of AA6061 sheets

Fig. 7.1 (a)–(c) Optical micrographs and (d)–(f) TEM micrographs of 1, 3 and 5-cycle ARB processed AA6061 sheets.

After 1-cycle ARB, AA6061 comprises subgrains and dense dislocation cells in the microstructure. The subgrain bands lie along a certain direction. After 3-cycle ARB, well-defined banded grain structures with clear boundaries have developed. The average band width is about 154 nm. Dense dislocations exist in most of the grain interiors. The 5-cycle ARB processed AA6061 shows elongated grains with HAGBs and most of the grain interiors are free of dislocations. The average band width has been refined to approximately 139 nm, which is slightly smaller than the 3-cycle ARB processed sample. The microstructure and grain sizes are comparable to those
obtained by other investigators [42, 300]. The SAD patterns have a trend of from single net patterns with a few diffused spots to discontinuous ring patterns. This indicates that LAGBs are dominated for the low cycle ARB deformed samples which evolves into HAGBs as the ARB process proceeds.

### 7.3 Mechanical properties of ARB processed AA6061 sheets

#### 7.3.1 Tensile properties

Fig. 7.2 shows the tensile test results of AA6061 before and after 1 to 5-cycle ARB. The stress increases considerably and the elongation decreases sharply after ARB deformation, as can be seen from the engineering stress-strain curves in Fig. 7.2. Average values of ultimate tensile strength and uniform elongation of the ARB processed AA6061 sheets are shown in Fig. 7.2(b). The increment of tensile strength between each ARB cycle is fairly uniform, which is unlike the strengthening behaviour of the AA1050 sheets. The tensile strength increases up to 383 MPa after 5-cycle ARB, which is about three times the initial value. The uniform elongation after 1-cycle ARB is about 1.7% and it then remains the same with further ARB cycles. The tensile strength of the AA6061 sheet after 5-cycle ARB is much higher than the AA1050 sheet, while the uniform elongations of both sheets are at a similar level.
Fig. 7.2 (a) Engineering stress-strain curves and (b) tensile strength and uniform elongation and (c) strain hardening rate of 1 to 5-cycle ARB processed AA16061 sheets.
7.3.2 Hardness

The average hardness evolution of AA6061 sheets with the number of ARB cycles is shown in Fig. 7.3. The hardness increment with further ARB cycles is more significant than the tensile strength but at a lower rate. This increment continues up to 5-cycle ARB deformation and the hardness is approximately three times the initial value.

![Graph showing hardness vs. number of ARB cycles](image)

**Fig. 7.3** Average hardness values of 1 to 5-cycle ARB processed AA6061 sheets.

The optical microscopy graphs and through-thickness hardness of the 1, 3 and 5-cycle ARB processed AA6061 sheets are shown in Fig. 7.4. It can be seen in Fig. 7.4(a) that the band width of the grains around the centre of the 1-cycle ARB processed sheet is much larger than that close to the surface. The hardness along the thickness was measured and it indicates that the lowest values are around the centre of the sheet and they increases gradually when moves to the surface. This non-uniform deformation is caused by the redundant shear strain close to the surface induced by the friction between the sheet and rolls. Some simulations and experiments revealed that shear strain is zero at the centre and increases as it
moves towards the surface [102, 104, 298]. The increment from the quarter-thickness to the surface is more significant than from the centre to the quarter-thickness. The shear strain distribution is consistent with the hardness distribution, which implies that the non-uniform distribution of the microstructure and hardness is caused by the redundant shear strain. Fig. 7.4(b) and (c) show that both the hardness and the microstructure are more uniformly distributed for the 3 and 5-cycle processed sheets. The hardness values are similar to the shear strain distribution, with higher values at the surface and at the interfaces between two layers [102, 104, 298]. The grain size of AA6061 sheets is much smaller than the AA1050, as can be seen in the optical micrographs, especially for the 1-cycle ARB processed sheets.
Fig. 7.4 Through-thickness hardness distribution of (a) 1, (b) 3 and (c) 5-cycle ARB processed AA6061 sheets.
7.4 Texture evolution of AA6061 sheets during ARB deformation

The $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the AA6061 sheets before ARB are shown in Fig. 7.5. It can be seen that the initial textures of the annealed AA6061 are dominated by Cube orientation, which is similar to the annealed AA1050 and in good correspondence with the fully annealed microstructure shown in Fig. 3.3(b).

![Fig. 7.5 $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of annealed AA6061 before ARB. (Contour levels 0.5×)]

7.4.1 Texture evolution of AA6061 sheets with the number of ARB cycles

The $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the average texture measured on the stack of RD-ND planes for the AA6061 sheets after 1 to 5-cycle ARB deformation are shown in Fig. 7.6. The texture evolution of the AA6061 sheets with the number of ARB cycles is close to the AA1050 sheets.

Rotated Cube texture developed after 1-cycle ARB further increases in intensity up to 5-cycle. The $\beta$ fibre also becomes stronger with increasing number of ARB cycles. Strong Rotated Cube and Copper dominated $\beta$ fibre are obtained for the 5-cycle ARB processed sheet. Weak shear type orientations Rotated Copper, E and F are also observed throughout ARB deformation. Copper, S and Rotated Cube orientations are the most stable orientations in the ARB processed AA6061 sheets, which is the same as the AA1050 sheets. The intensity of the texture after 1-cycle
ARB is much weaker than the annealed sheet, which gradually increases with the number of ARB cycles.

**Fig. 7.6** $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the average texture of the 1 to 5-cycle ARB processed AA6061 sheets. (Contour levels 0.5×)
7.4.2 Through-thickness texture distribution of ARB processed AA6061 sheets

Fig. 7.7 shows the $\varphi_2=0^\circ$, 45° and 65° ODF sections of the centre, quarter-thickness and surface texture of AA6061 sheets processed by 1, 3 and 5-cycle ARB. The textures measured at the sheet centre of the 1, 3 and 5-cycle ARB processed sheets are dominant with a typical rolling texture with $\beta$ fibre. The centre plane of the 1-cycle ARB processed sample consists mainly of typical rolling texture components and the initial Cube component. Strong initial Cube texture, together with a weak $\alpha$ fibre composed of Goss and Brass, and a strong $\beta$ fibre composed of Brass, Copper and S were observed. The S and Cube components are the strongest while Goss is extremely weak. The component between Copper and D positioned at $(\varphi_1, \Phi, \varphi_2) = (90^\circ, 29^\circ, 45^\circ)$ is closer to D. The Brass component has shifted 10° along the ND direction.

After 3-cycle ARB, the texture of the centre plane changes to $\beta$ fibre together with a weak Rotated Cube texture. The strongest component changes from S after 1-cycle ARB to the D component, while both the S and Brass components shift about 10° from their ideal position. The initial Cube texture is fully transformed to other components and a weak Rotated Cube component has formed. After 5-cycle ARB, the $\beta$ fibre with strong D and S components and Rotated Cube component are the major texture. Rotated Cube component is much stronger than that of the 3-cycle ARB processed sample. From 1 to 5-cycle, the D component shifts along the TD direction, while the S component shifts further away from the ideal position with the number of ARB cycles.

It is found that a high texture gradient exists for the 1-cycle ARB processed sample. The texture at the quarter-thickness is composed of shear texture. A strong
Rotated Cube component and a weak E component, which are typical shear components, develop at an expense of the initial Cube texture. The texture is strongly scattered but without any sign of rolling texture. The textures at the quarter-thickness of the 3 and 5-cycle ARB processed samples are similar, with D and S dominated β fibre and strong Rotated Cube texture. The texture intensity of the 5-cycle processed sample is higher than the sample deformed after 3-cycle ARB.
Fig. 7.7 $\phi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of texture measured at the centre, quarter-thickness, surface and the average texture of the (a)1, (b)3 and (c)5-cycle ARB processed AA6061 sheets. (Contour levels 0.5x)

The surface of the 1-cycle ARB processed AA6061 is composed of a typical shear texture, with strong Rotated Cube and weak E, F and Rotated Copper components. The rolling texture components Brass, Copper and S are not developed at this
position. Rotated Cube, Rotated Copper and E form \(<1\ 1\ 0>\) fibre and E and F form \(\{1\ 1\ 1\}\) fibre, both of which are typical fibres developed by shear deformation due to the high friction between the sheet and the rolls. The textures at the surface of the 3 and 5-cycle ARB processed samples are similar. They are composed of a strong Rotated Cube component together with \(<1\ 1\ 0>\) fibre and \(\{1\ 1\ 1\}\) fibre, while the rolling components disappear completely, which proves that shear is the only deformation that occurs at the sample surface.

The texture intensity of the annealed AA6061 is weaker than the annealed AA1050, which is presumably caused by particle-simulated nucleation during annealing. The texture of the ARB processed AA6061 is slightly lower than the AA1050, because there are a large number of micron-sized precipitates in the AA6061, which induce an inhomogeneous deformation around them that weakened the texture [177].

The intensities of the texture components along the major rolling and shear fibres were calculated. The results are shown in Fig. 7.8 and Fig. 7.9. It can be seen in Fig. 7.8 that for the centre of the sheet, \(\beta\) fibre with S stronger than Copper and Brass was developed after 1-cycle ARB. The intensity of the S component decreases with increasing number of ARB cycles while the Copper component increases. In the meantime, the Copper component, which locates between Dillamore and Copper, shifts to the D component after 3-cycle ARB and then shifts further towards the Rotated Cube component. The intensity of the Brass component increases after 3-cycle ARB and then decreases from 3 to 5-cycle. The Rotated Cube component begins to develop at the centre plane after 3-cycle ARB and the intensity increases further after 5-cycle. The final intensity of the Rotated Cube component is similar to that of the Brass component.
The 1, 3 and 5-cycle ARB processed samples have similar fibres at the surface position. It is composed of Rotated Cube dominated \( \tau \) fibre and \( <1\,1\,0> \) fibre, and a weak \( \{1\,1\,1\} \) fibre. The intensities of the fibres are increasing with the number of ARB cycles, and the distribution of intensities along the \( \{1\,1\,1\} \) fibre varies with the number of ARB cycles. The intensity of the E component is higher than F after the first cycle while the F component is higher than E for the 3 and 5-cycle ARB processed samples. The intensity of these two components is weak compared with the Rotated Cube component.

It can be seen from the \( \alpha \), \( \beta \) and \( \tau \) fibres that the texture at the quarter-thickness of the 1-cycle ARB processed sheet is closer to that at the surface, while the textures of the 3 and 5-cycle ARB processed sheets are closer to that at the centre. The 3-cycle ARB processed sample is characterised with a relatively uniform \( \beta \) fibre, with Copper stronger than S and Brass. The intensity of Rotated Cube is slightly lower than Copper. The Copper component becomes stronger at the expense of some Brass for the 5-cycle sample compared with the 3-cycle sample. The intensity of the Rotated Cube also increases but still slightly lower than that of Copper. The textures of these two sheets at the quarter-thickness are composed of Copper dominated \( \beta \) fibre and Rotated Cube, which is closer to the corresponding textures at the centre. The texture at the quarter-thickness of the 1-cycle ARB processed sheet is significantly different from the 3 and 5-cycle ARB processed sheets. The texture at this position is composed of a strong Rotated Cube component, the intensity of which is comparable to that of the 5-cycle ARB processed sample, and weak \( \tau \) fibre.
Fig. 7.8 α, β and τ fibres of texture measured at the centre, quarter-thickness, surface and the average texture of the 1, 3 and 5-cycle ARB processed AA6061 sheets (Black represents 1-cycle ARB processed sample, red 3-cycle and blue 5-cycle, same for Fig. 7.9).
Fig. 7.9 {1 1 1} and <1 1 0> fibres of texture measured at the surface and the average texture of the 1, 3 and 5-cycle ARB processed AA6061 sheets.

The Rotated Cube component is the strongest for the average texture of all three samples, followed by Copper, which dominates the β fibre of the 3 and 5-cycle ARB processed samples. The intensities of most fibres of the 1-cycle sample are lower than the corresponding fibres of the 3 and 5-cycle ARB processed samples, among which the most obvious is β fibre, which is considerably weaker and more evenly distributed. The intensity of the {1 1 1} fibre for the 1-cycle ARB processed sample is higher than the 3 and 5-cycle ARB processed samples, which indicates that the {1 1 1} fibre developed at the first ARB cycle rotates into the Rotated Cube component or other rolling components with further deformation.

Although the {1 1 1} and <1 1 0> fibres exist at the surface position of all the ARB processed sheets, they appear in the average texture as partial <1 1 0> fibre and some individual components around E and F with very weak intensity. The E and F
components have a high $\bar{r}$ value and a low $\Delta r$ value and provide good drawability. Since Rotated Cube is the major shear component, which is more favourable to deep drawing than $\beta$ fibre but not as good as the components E and F, a certain improvement in deep drawability can be expected but not as dramatic.

### 7.5 Vacancy-type defects evolution of AA6061 sheets during ARB deformation

Fig. 7.10 shows the PALS results of 1, 3 and 5-cycle ARB processed AA6061 sheets. The $\tau_1$ values are at the similar level as the AA1050 sheets and are much smaller than the bulk lifetime, which indicates annihilation in the bulk. The $\tau_2$ values are between 217 ps and 222 ps, which are smaller than the AA1050 sheets. The values of the 1 and 3-cycle ARB processed AA6061 are similar and lie between the bulk lifetime and the lifetime of vacancies associated with dislocations but close to the latter. However, the $\tau_2$ value of the 5-cycle ARB processed AA6061 is slightly higher than the lifetime of vacancies associated with dislocations. The intensities of long lifetime $\tau_2$ are all over 95% and are higher than the ARB processed AA1050 sheets, especially the 5-cycle ARB processed AA6061, the $\tau_2$ intensity of which is higher than 99%. This indicates that most of the positrons are annihilated at defects, only a few are annihilated into the bulk. The fractions of vacancies associated dislocations for 1, 3 and 5-cycle ARB processed AA6061 are calculated to be 94.9%, 95.8% and 90.4%, respectively. This suggests that most of the vacancy-type defects in the ARB processed AA6061 are vacancies associated with dislocations. A detailed discussion is given in Section 8.6.4.
Fig. 7.10 Positron lifetime results of ARB processed AA6061 sheets (a) short lifetime, (b) long lifetime and (c) intensity of long lifetime.
### 7.6 Discussion

In this Chapter, AA6061 was processed by ARB using rolls with high surface roughness ($Ra\sim2$ μm) to increase the fraction of shear texture and improve deep drawability. The following discussion is focused on the texture distribution throughout the thickness of the ARB processed sheets.

As pointed out by many researchers, the textures developed by the ARB process are characterised by rolling texture at the centre and shear texture at the surface. It is well known that rolling texture has orthorhombic symmetry and shear texture has monoclinic symmetry. Therefore, it is expected that the centre and surface area would exhibit orthorhombic and monoclinic symmetry, respectively. However, most of the reported ARB texture at the surface showed orthorhombic symmetry [104, 177-179, 181, 185, 311]. Li et al. [106] pointed out that a monoclinic symmetry is expected for surface texture as the materials are deformed by simple shear. The texture at the quarter-thickness is a combination of rolling and shear texture. It is assumed that monoclinic symmetry would be displayed at this position. The $\varphi_2=45^\circ$ ODF figures of the surface and quarter-thickness positions up to $\varphi_1=180^\circ$ for 5-cycle ARB processed AA6061 sheets are shown in Fig. 7.11. Although monoclinic symmetry is expected, the orthorhombic symmetry is observed, as can be seen from Fig. 7.11. The same symmetry is also observed for the ARB processed AA1050 sheets and AA1050/AA6061 composites. Therefore, ODF figures up to $\varphi_1=90^\circ$ are shown in this work but without applying any symmetry.
In order to achieve good bonding, the ARB process is always conducted without any lubricant. As a result, a large amount of redundant shear strain is introduced into the area close to the surface due to the high friction between the sheet surface and the rolls. As the ARB process is repeated, the redundant shear strain distribution through the thickness becomes quite complicated, and the microstructure, hardness, and texture heterogeneity throughout the thickness are induced, as can be seen in Figs. 6.4, 6.9, 7.4 and 7.8.

ARB texture is characterised by a typical rolling texture of β fibre with the Cu component dominating. Moreover, Rotated Cube texture also forms due to shear deformation in the surface layer of the sheets during rolling. The development of shear texture during rolling is affected by the rolling temperature [221], roll gap geometry [307-309] and friction condition [104].

The pre heating temperature in the current work was 200 °C for 3 min to ensure that the sheets have good bonding and retain high strength in the meantime. Topic et al. [221] did experiments on the temperature effect on the bonding and tensile strength and showed that for AA6016, the optimum temperature for obtaining roll bonded sheets with a combination of good bonding and high strength is 230 °C. They stated that materials processed at 200 °C or lower resulted in poor bonding. In this experiment, good bonding has been achieved at 200 °C (discussed in Section
8.6.2. In Ref. [34, 105, 310], AA6016 was pre-warmed at 230 °C for 4 min before ARB (close to our experiments), and the average textures were measured by neutron diffraction. The results showed that the Rotated Cube component started to form after 4-cycle ARB, however, its intensity was considerably lower than that of Copper component even after the eighth cycle. By comparing their ODFs with the $\phi_2=45^\circ$ ODFs in Fig. 7.6, it can be found that the intensity of the Rotated Cube component in the current work is much higher. In fact, the average texture after 4 and 8-cycle ARB in Ref. [34, 105, 310] is more like the texture at the centre position of the 3-cycle ARB processed sample in our work. This proves that excessive shear texture has been retained in our ARB processed sheets, the processing temperature, however, is unlikely to be the key influence.

Texture inhomogeneity throughout the thickness of the rolled sheets is largely dependent upon the roll gap geometry. The roll gap geometry is represented by the $l/h$ ratio, where $l$ is the contact length of the rolls and the sheet, and $h$ is the mean thickness of the specimen before and after rolling [307-309]. Rolling with a large $l/h$ value is called large draughts rolling while that with a small $l/h$ value is small draughts rolling. It is considered that homogeneous textures develop when the $l/h$ value is between 0.5 and 5 and texture inhomogeneity happens for both large ($l/h>5$) and small draughts ($l/h<0.5$). The $l/h$ value in this experiment is 4.3, which implies that homogeneous texture should develop. However, as can be seen from previous figures, the texture developed in this experiment is strongly inhomogeneous throughout the cross section, which indicates that some other factor must influence the strain distribution.

Rolls of high surface roughness were deliberately chosen in this experiment to obtain high friction between the rolls and the sheet. The texture distribution of the
AA1050 sheets is similar to the AA6061 sheets, as can be seen from the figures in Sections 6.4 and 7.4. Therefore, only the texture inhomogeneity of the AA6061 sheets will be discussed in detail. The surface roughness of the 5-cycle ARB processed AA6061 sheet was measured in an area of 500 μm by 500 μm, as shown in Fig. 7.12. The average surface roughness is $R_a=1.9941 \, \mu m$, which is significantly larger than the surface roughness of the sheets from normal rolling [312].

![Surface roughness of 5-cycle ARB processed AA6061 sheet.](image)

Since the roll surface was deliberately chosen to have a high roughness, the friction between the rolls and the material is higher than usual. Friction might be the major factor that results in redundant shear strain. By conducting ARB in ultra-low carbon IF steel with and without lubrication, Kamikawa et al. [104] investigated the friction condition effect on the microstructure and texture evolution. The results showed that the shear strain without lubrication is significantly higher than with lubrication, which implies that high surface roughness could result in higher friction between the rolls and the material, which in turn leads to higher shear strain throughout the sheet. It is suggested that shear strain accelerates grain refinement and results in an inhomogeneous microstructure throughout the sheet thickness [102, 104, 298].

The texture developed in this work is comparable to the texture reported for aluminium alloy subjected to ARB deformation. The textures are generally characterised by rolling-type components at the centre and shear-type
components near the surface. Copper dominated $\beta$ fibre and strong Rotated Cube texture develops after a certain cycle ARB deformation. The intensity of the Copper component increases continuously with the number of ARB cycles, and shifts towards the Rotated Cube component. These characteristics are consistent with other work [34, 105, 106, 122, 177, 184, 185, 310]. The most significant difference of this experiment from the literature is the presence of a rather strong Rotated Cube component at all thickness positions as well as for the average texture.

Most of the reported texture for aluminium alloys at the ARB sheet centre is typical $\beta$ fibre [106, 181]. Only a few references reported a weak Rotated Cube component at the centre of the sheet [177, 185]. It was stated that the minor shear texture at the centre was from the shear texture at the surface of the last cycle. As in the ARB process, half the surfaces come to the centre by the cutting and stacking procedure, so the centre of the sample in the present state had been the surface in the previous cycles, which means the materials in the centre had gone through severe shear deformation. As predicted by Prangnell et al. [178, 179], the rolling texture at the centre of the sample would rotate towards shear texture when the material is deformed at the surface. The Rotated Cube component, which is formed at the surface, would rotate towards the Copper component when the material is deformed at the centre and subjected to plane strain compression. The latter is the real case that happens in the ARB process. Half the surfaces in the previous cycle stay at the surface while the other half move to the centre in the next cycle. The materials with shear texture deformed at the surface would result in enhanced shear texture while the materials with shear texture deformed at the centre would rotate towards Copper texture. The rotation from shear texture to Copper is not complete, as can be proved by our texture results, which results in some remnant
shear texture at the centre. In this experimental condition, the shear texture formed at the surface of the sample is so strong that it remains quite strong even after being deformed at the centre.

In Ref. [106], the simulation with a FC Taylor model shows that rolling texture components Copper, Brass and S evolve into shear texture components Rotated Cube, E, F and Rotated Copper are under shear deformation. Meanwhile, the shear components evolve into rolling components under plane strain compression. This simulation confirms that the rolling texture and shear texture can transform into each other during the ARB process, which in turn gives texture gradient. The simulation in Ref. [106] gave a more comprehensive prediction of the texture transformation under different strain history. However, as can be seen from many experimental results, the formation of Rotated Cube at the surface and Copper at the centre is more common than the other components. This is also the case with the results of this work where the Rotated Cube and Copper components are the most important shear and rolling components at all positions. This proves that the Rotated Cube and Copper components are the most favourable components during the ARB process.

Shear texture with strong Rotated Cube and weak <1 1 0> and {1 1 1} fibres was reported for the texture at the surface [106, 177], which is consistent with the results from this experiment. However, the intensities of the Rotated Cube component at the surface in this experiment are higher than the rolling texture components at the centre, which is different from both Ref. [177] and [106], in which the intensities of Rotated Cube at the surface are much lower than the rolling texture at the centre. This is due to the high friction between the rolls and material due to large surface roughness.
The quarter-thickness region shows both shear and rolling textures. Ref. [184] showed the texture of ARB processed AA1100 measured at the quarter-thickness. The rolling texture was only detected at up to 3-cycle ARB, while the Rotated Cube component started to develop after the fourth cycle and stayed weak compared to the Copper component, even after the sixth cycle. The texture developed in this work is quite different, as a strong Rotated Cube component exists at the quarter-thickness after the first cycle and remains strong with the number of ARB cycles. The difference is due to different processing parameters between the two experiments. Ref. [106] showed the pole figures at the quarter-thickness of the ARB processed AA1070, where it appeared that the texture at the quarter-thickness is a mixture of rolling and shear texture, with more shear type texture at lower cycles and more rolling texture at higher cycles, which is closer to the results in this experiment.

As was stated earlier that half the surfaces in the previous cycle moved to the centre in the next cycle and then to the quarter-thickness region in the following cycle. This proves from the texture at the quarter-thickness of the 1-cycle ARB processed sample that strong shear strain penetrates into the quarter-thickness so that the initial Cube component rotates into Rotated Cube. For the 3 and 5-cycle ARB processed samples, the textures at the quarter-thickness display both strong Copper and Rotated Cube components instead of only Rotated Cube. The reason can be attributed to two aspects. The first is that the penetration depth of the shear strain due to the friction between the rolls and the material has changed because the hardness of the materials has been improved. The second is that the starting texture at the quarter-thickness position has changed. The initial texture before the first cycle is Cube texture, which is unstable and easy to transform into
other components, whereas the starting texture at the quarter-thickness for the second cycle is typical rolling texture which has developed at the centre of the first cycle ARB. The rolling texture, β fibre in this case, is stable compared to the Cube texture during plastic deformation. Therefore, the rolling texture stayed at the quarter-thickness position for the higher cycle ARB deformed samples. Ref. [177] reported weak Rotated Cube at the centre and the surface but not at the quarter-thickness, which means that in their experimental condition, the shear strain does not penetrate deep enough to the quarter-thickness, thus the Rotated Cube component formed at the surface would retain a weak intensity at the centre when the material moves from the surface to the centre, but completely disappears when the material moves again from the centre to the quarter-thickness position.

For the average texture, most of the reported texture showed strong β fibre and weak Rotated Cube developed from the fourth cycle on [34, 105, 106, 179, 310]. In this experiment, however, the Rotated Cube has higher intensities than the Copper component and it begins to develop at the first cycle ARB. This again proves that the high surface roughness of the rolls does improve the penetration depth of the shear strain.

As shown in Fig. 7.8 and Fig. 7.9, the texture evolution in the quarter-thickness position is different from that in the centre or at the surface. The latter two shows small changes such as intensity increment and small angle rotation. The texture at the quarter-thickness showed a different evolution as the texture after the first cycle ARB is pure shear texture, while the texture after 3 and 5-cycle is a combination of rolling and shear. This is believed to be caused by the movement of the materials induced by the repeated cutting and stacking of the ARB process, as explained earlier.
To further explain this matter, the fractions of orientations near specific components were calculated from the ODF data, while allowing a 15° deviation from the ideal orientations. The fractions of Brass, S and Copper components were summed to be the rolling texture fraction. The shear texture fraction was calculated by summing the fractions of Rotated Cube, Rotated Copper, E and F components. The volume fractions of the rolling and shear texture in the different positions of the 1, 3 and 5-cycle ARB processed samples are shown in Fig. 7.13.

As can be seen from Fig. 7.13, both the fraction of the rolling and shear texture of the 3 and 5-cycle processed samples are extremely close at almost all the positions. The shear texture fraction of the 5-cycle ARB processed sample is slightly higher than the 3-cycle sample, especially at the centre position. The fraction of the rolling and shear texture for the 1-cycle processed sample, however, differs strongly from the higher cycle deformed samples.

The fraction of the rolling texture does not change much with the number of ARB cycles at the centre position. The fraction of shear texture, however, increases gradually with the number of ARB cycles. As the shear texture at the centre is the redundant of the shear texture from the previous cycle, the increment of the shear texture at the centre corresponds with the increment of shear texture at the surface.
For the quarter-thickness position, the fraction of rolling texture of the 1-cycle processed sample is much lower than the samples after 3 and 5-cycle ARB, where the fraction of shear texture is much higher. The reason has been given earlier. The consequence of this phenomenon is that the total fraction of the rolling texture, calculated from the average texture, of the 1-cycle processed sample is lower than the 3 and 5-cycle processed samples. The total fraction of shear texture, on the other hand, is about the same for all three samples. The distribution of the
microstructure with rolling and shear texture is completely different for the three samples. For the 1-cycle deformed sample, the grains with rolling texture are located close to the centre of the sheet, and the microstructure with shear texture is distributed from the quarter-thickness to the surface areas. The 3 and 5-cycle deformed samples have a more or less uniformly distributed shear texture throughout the thickness, but the rolling texture is only distributed from the centre to the quarter-thickness of the sheets. The shear texture distribution is also consistent with the shear strain and hardness distribution throughout the sheet.

### 7.7 Summary

The results from the detailed study of the through-thickness microstructure, mechanical properties and texture evolution of AA6061 sheets processed by ARB with rolls of high surface roughness and 200 °C pre-heating are summarised as follows.

1. AA6061 sheets with good bonding and substantial grain refinement and significant strengthening were achieved. The grain size of the AA6061 sheets is finer and more elongated than the AA1050 sheets.

2. The tensile strength, uniform elongation and hardness evolution of the AA6061 sheets are similar to those of the AA1050 sheets. The hardness and grain size distribution throughout the thickness direction of the AA6061 sheets also have similar trends to the AA1050 sheets. The tensile strength and hardness values of the AA6061 sheets are much higher than the AA1050 sheets, whereas the uniform elongation values are similar.

3. The texture intensities of the AA6061 sheets are generally weaker than the AA1050 sheets but the major texture components are similar. Strong Rotated Cube
texture develops at the sheet surface. Typical rolling texture is obtained at the sheet centre for the 1-cycle ARB processed sheet, and a combination of rolling texture and Rotated Cube texture is observed for the 3 and 5-cycle processed samples. The texture at the quarter-thickness is pure shear after 1-cycle ARB and a combination of rolling and shear texture after 3 and 5-cycle ARB because the initial texture before the first cycle ARB is Cube texture, which is easy to transform into the Rotated Cube component due to the shear strain penetrating from the sheet. The starting texture at the quarter-thickness for the higher ARB cycle is typical rolling texture, which is stable during ARB deformation. Therefore, the rolling texture remained at the quarter-thickness position for the higher cycle ARB deformed samples.

(4) The distribution of shear texture is closely related to the distribution of redundant shear strain throughout the thickness of the sheet. The grain refinement and hardness evolution along the thickness are also related to the shear strain distribution.

(5) The major vacancy-type defects in the ARB processed AA6061 are vacancies associated with dislocations. The \( \tau_2 \) values of the 1 and 3-cycle ARB processed AA6061 are similar and slightly smaller than the vacancies associated with dislocations, but the \( \tau_2 \) value of the 5-cycle ARB processed AA6061 is slightly higher than the lifetime of vacancies associated with dislocations.
Chapter 8 Accumulative roll bonding of AA1050/AA6061 composites

The materials obtained from ARB are quite different from those manufactured by other SPD methods such as ECAP or HPT, as the ARB processed materials are more like layered composites [38, 42]. The ARB process allows bonding of two different kinds of materials. Al/Mg [108], Al/Cu [109], Al/Ni [110], Al/Zn [111], Al/Ti [120], Cu/Ag [112], Cu/Zr [112], Fe/Cu [113] and other laminated composites have been produced by ARB.

It has been found that during the ARB process of two different materials, both materials deform in a similar manner at the first few ARB cycles. Afterwards, plastic instabilities occur earlier in the harder layers than the softer layers, and the harder material experiences necking and fracture as the number of ARB cycles increases. This deformation behaviour results in a homogeneously distributed fragmentation of the hard material in the soft material matrix [108, 110, 111, 114-116, 120]. The mechanical properties of the composite were influenced by the necking and rupture of the harder layers. Some work reported that the strength of the composite still increased despite the fracture of the harder layers [111, 115, 116, 120], while others reported that the strength of the composite dropped because of the rupture [108].

It is generally believed that the strength of the composite is determined by the mechanical properties of both materials by the rule of mixture. For the composite produced by ARB of dissimilar starting materials, the two materials certainly obtain different properties after ARB deformation. It has been reported that the two layers in the composite have different microstructures and different mechanical
properties measured mostly by hardness tests in separate layers [108, 111, 116, 118, 313]. Although few studies have been done, the strength of the composite can be predicted by the rule of mixture [120]. Yang et al. reported that the yield strength of the Ti/Al composite fitted well with the value obtained from the rule of mixture [120].

Two grades of the same base metals were also used as starting materials, such as Al/Al(Sc) [119], AA1050/AA5754 [118], AA6014/AA5754 [118] and AA2219/AA5086 [114]. As stated by Hausöl et al. [118], different aluminium alloys have different properties. For example, 6××× alloys have high strength, 5××× alloys have good formability and 1××× alloys have good surface quality. A combination of different types of aluminium alloys by accumulative roll bonding could result in materials with a combination of the preferential properties of the base materials. AA1050/AA5754 and AA6014/AA5754 sheets were produced by using AA5754 as the core material and AA1050 and AA6014 as the lateral materials, respectively. This result showed that the composites exhibited combinative positive properties of the single component materials [118]. Quadir et al. processed Al/Al(Sc) layered sheets by using commercial purity aluminium and aluminium with 0.3 wt.% Sc as starting materials. This showed that after annealing at 350 °C, Al and Al(Sc) layers showed coarse and fine microstructures, respectively [119]. It has been concluded that layered composites can exhibit positive properties of both starting materials and can produce composites with a layered microstructure and mechanical properties distribution.

In this chapter, commercial aluminium alloys AA1050 and AA6061 were used as starting materials in the ARB process to produce laminated composites with AA1050 and AA6061 in alternate layers. The microstructure and overall mechanical
properties of the composite and those of two different layers in the composite were investigated. The mechanical properties of the AA1050/AA6061 composites were also compared with those of AA1050 and AA6061 sheets processed by the same ARB process.

8.1 Experimental procedure

AA1050/AA6061 was produced by ARB using annealed AA1050 and AA6061 sheets as the starting materials. One piece of AA1050 and one piece of AA6061 were then stacked, welded together at one end, pre-heated in a furnace at 200 °C for 3 min and then rolled with a nominal reduction of 50% under dry condition. The rolled samples were cut into two halves and stacked together with AA1050 and AA6061 in the adjacent layer of each other to perform the next cycle. The above procedure was repeated for five cycles to achieve an accumulative strain of about 4.

The mechanical properties were tested by tensile and hardness tests. The measurement was taken on the RD-ND plane along the thickness direction with 50 μm distance between two adjacent indents for the 1 and 3-cycle ARB processed composites. The hardness of the 5-cycle ARB processed composite was taken deliberately within the AA1050 and AA6061 layers but not with a constant distance between indents since the microstructure was not uniform. The average hardness and through-thickness hardness distributions were both used for further analysis. The average hardness values of the AA1050 and AA6061 layers in the AA1050/AA6061 composites were obtained from averaging the indentations within the corresponding alloy layers, regardless of their positions.

Tensile tests were conducted with specimens of the same geometry and at the same strain rate as that for the AA1050 and AA6061 sheets. The yield strength,
ultimate tensile strength and uniform elongation were identified from the engineering stress-strain curves. True stress-strain curves and strain hardening rate were also calculated and were compared with the ARB processed AA1050 and AA6061 sheets.

The microstructure of the ARB processed AA1050/AA6061 composites was investigated by OM and TEM. The same sample preparation and similar analysing techniques as for the ARB processed AA1050 and AA6061 sheets were used.

The average texture of the ARB processed AA1050/AA6061 was measured by X-ray diffraction. The average texture was measured on the RD-ND plane by stacking 7 pieces of sheets together along the normal direction to form a surface of around 10 mm (RD) by 10 mm (ND). Local textures of the AA1050 and AA6061 layers in the centre of the 5-cycle ARB processed AA1050/AA6061 composites were studied by EBSD on the RD-ND surface. Samples were prepared by grinding and polishing to a Struers OPS finish. EBSD was undertaken with step sizes of 90 nm and micro-textures were depicted by HKL Channel-5 software over 15,000 grains.

PALS measurements were carried out on the 1, 3 and 5-cycle ARB processed AA1050/AA6061 composites and the results were compared with the ARB processed monotonic AA1050 and AA6061 sheets.

8.2 Microstructure evolution of AA1050/AA6061 composites during ARB deformation

8.2.1 Microstructure evolution with ARB process

Fig. 8.1 shows the optical micrographs of 1, 3 and 5-cycle ARB processed AA1050/AA6061 composite under polarised light. It can be seen that the grains
after ARB have a lamellar structure which are elongated along the rolling direction and the grain size has decreased substantially as compared to the annealed materials before ARB, as shown in Fig. 3.3. The grain size becomes finer with the increasing ARB cycles.

![Optical micrographs of AA1050/AA6061 composite after (a) 1, (b) 3, (c) 5-cycle ARB deformation.](image)

**Fig. 8.1** Optical micrographs of AA1050/AA6061 composite after (a) 1, (b) 3, (c) 5-cycle ARB deformation.

It can be seen that the interfaces of AA1050 and AA6061 after 1 and 3-cycle ARB are straight and the thicknesses of both the AA1050 layers and the AA6061 layers are uniform through the whole cross section. The interfaces become wavy after the fourth cycle (not shown here) and the thickness of the AA6061 layers becomes non-uniform along the rolling direction due to different flow properties of AA1050 and AA6061, which induces plastic instabilities around the interfaces. After 5-cycle
ARB, necking of the AA6061 layers continues and fracture happens at various places.

Shear zones are observed in the composite processed after 5-cycle ARB, as indicated by different colours in Fig. 8.1(c), and the angles of the shear zones with respect to the rolling direction have the highest value close to the thickness centre and decrease when moving towards the surfaces. The angle is ~30° at the thickness centre and it reduces to ~17° at the quarter-thickness and then reduces further as moving to the outside. The AA6061 layers close to the surfaces of the composite are not fractured, thus the shear bands are hardly seen near the two surfaces. A similar microstructure was also observed by Roy et al. [114]. During rolling deformation, the material near the sample surface is subject to large shear deformation while the material near the thickness centre is subject to large elongation deformation. The shear deformation does not introduce essential tensile stress in the material. However, in the elongation deformation, the softer materials elongate more than the harder materials, which induces tensile stress along the rolling direction in the latter and then breaks the hard materials when the tensile stress exceeds a certain level. This is why the fracture of the hard materials was observed near the thickness centre.

The average thicknesses of the AA1050 layers and AA6061 layers were obtained by the linear intercept method and are listed in Table 8.1. It is clear that from 1 to 4-cycle, the thickness reduction for both the AA1050 layer and the AA6061 layer is around 50%, whereas the thickness reduction of the AA6061 layer after 5-cycle ARB is less than 50%. This implies that the accumulated strain in the AA6061 layers is slightly lower than the AA1050 layers. It has been reported that once the fracture of the hard layers occurs, their thickness will level off when subjected to further
ARB deformation [110, 111, 115, 116], which suggests that in the present study, the thickness of AA6061 starts to level off at the fifth ARB cycle. The volume fraction of the AA6061 layers was calculated by dividing its thickness with the total thickness of the composite. It can be seen that the volume fraction of the AA6061 layers stayed around 50% for all the cycles. Note that the volume fraction of the 5-cycle ARB processed composite is an estimated value as the thickness distribution of the AA6061 layers was not uniform so it cannot be used to represent volume fraction.

Table 8.1 Average thickness, reduction and volume fraction of AA1050 and AA6061 layers in the ARB processed AA1050/AA6061 composites.

<table>
<thead>
<tr>
<th>Number of ARB cycles</th>
<th>AA1050 layers</th>
<th>AA6061 layers</th>
<th>Volume fraction of AA6061 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Thickness (µm)</td>
<td>Reduction (%)</td>
<td>Thickness (µm)</td>
</tr>
<tr>
<td>1</td>
<td>802.0±3.7</td>
<td>46.5</td>
<td>803.5±7.8</td>
</tr>
<tr>
<td>2</td>
<td>402.3±10.2</td>
<td>49.8</td>
<td>399.4±25.0</td>
</tr>
<tr>
<td>3</td>
<td>193.6±12.3</td>
<td>51.9</td>
<td>194.0±8.8</td>
</tr>
<tr>
<td>4</td>
<td>92.1±9.1</td>
<td>52.4</td>
<td>93.9±11.1</td>
</tr>
<tr>
<td>5</td>
<td>32.9±17.8</td>
<td>64.3</td>
<td>58.3±19.4</td>
</tr>
</tbody>
</table>

Fig. 8.2 shows TEM microstructures of the AA6061 and AA1050 layers in the 1–5 cycle ARB processed AA1050/AA6061 composites. The microstructures of the AA6061 and AA1050 layers are obviously different after the same cycle ARB. The grains of the AA6061 layers are more elongated with a smaller band width and a larger aspect ratio as compared to the AA1050 layers.

Both layers comprise subgrains and dislocation cells in the microstructure after 1-cycle ARB with the subgrain bands lying parallel to a certain direction which is most probably the rolling direction. After 3-cycle ARB, well-defined banded grain structures with clear boundaries develop in both layers, and the band widths
reduce further compared to that after 1-cycle ARB. The 5-cycle ARB processed samples show further grain refinement in both layers and HAGBs have developed as indicated by the SAD patterns.

The band widths were considered as the grain size and the values of the ARB processed AA6061 and AA1050 in both the monotonic sheets and composites are listed in Table 8.2. It can be seen from the table that the grain sizes of AA1050 in both the monotonic sheets and composites are at the same level, so is the AA6061,
which indicates that both alloys have undergone similar deformation and accumulated a similar amount of strain whether they are deformed in the monotonic sheets or in the composites.

Table 8.2 Average grain size of 1, 3 and 5-cycle ARB processed sheets.

<table>
<thead>
<tr>
<th>Grain size (nm)</th>
<th>1-cycle ARB</th>
<th>3-cycle ARB</th>
<th>5-cycle ARB</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA1050</td>
<td>465.5</td>
<td>406.3</td>
<td>300.2</td>
</tr>
<tr>
<td>AA6061</td>
<td>304.1</td>
<td>154.1</td>
<td>139.4</td>
</tr>
<tr>
<td>AA1050 layer in AA1050/AA6061</td>
<td>453.5</td>
<td>449.3</td>
<td>337.0</td>
</tr>
<tr>
<td>AA6061 layer in AA1050/AA6061</td>
<td>295.3</td>
<td>175.5</td>
<td>124.4</td>
</tr>
</tbody>
</table>

8.2.2 Precipitates in the AA6061 layers

Micron-sized precipitates in the AA6061 layers make it possible to distinguish the AA6061 layers from the AA1050 layers. Fig. 8.3 shows the optical micrographs of 3-cycle ARB processed AA1050/AA6061 composite after polishing to an OPS finish. Fig. 8.3(a) shows the cross-sectional image from which the two layers can be identified by the dense precipitates in the AA6061 layers. Fig. 8.3(b) shows an enlarged graph around the centre interface with both the AA6061 and AA1050 layers. Two types of precipitates can be identified from the figure. One type is gray in colour and is reported as Mg$_2$Si, and the other is Fe enriched precipitate and is black in colour [314]. Both precipitates are in the micron size range. The type, morphology and distribution of the precipitates after different number of ARB cycles were compared and they appear to be similar, therefore, it is not discussed in this thesis.
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Fig. 8.3 Optical micrographs of 3-cycle ARB processed AA1050/AA6061 composite at (a) low and (b) high magnification.

SEM X-ray mapping was carried out on the precipitates in the AA6061 layer of the 5-cycle ARB processed AA1050/AA6061 composite and the results are shown in Fig. 8.4. The white coloured precipitates contain Fe, Si, Cr, Cu, Co and O, while the black coloured precipitates contain predominately Mg, Si and O. By applying the spot analysis, the ratio of the atoms in the precipitates can be estimated. The atomic ratio of Mg and Si of the black coloured precipitates is found to be roughly 2:1, which is consistent with the phase Mg₂Si. The chemical composition of the white coloured precipitates can be inferred as (Fe, Cr)₃SiAl₁₂ from the estimated atomic ratio.

TEM micrographs and their corresponding EDS profiles of two typical micron-sized precipitates are shown in Fig. 8.5. As can be seen in Fig. 8.5(a) and (c), the long rod shaped precipitate is Si and O enriched, with smaller amounts of Al, Mg, Fe and Co. The faceted precipitate shown in Fig. 8.5(b) and (d) is rich in Fe, Co and Si, with smaller amounts of Al, Cr and Cu. It appears that both precipitates comprise (Fe, Co), Si and Al, with varying ratios of these elements.
Fig. 8.4 SEM X-ray mapping of the precipitates in the AA6061 layer of the 5-cycle ARB processed AA1050/AA6061 composite.
Besides the micron-sized precipitates, nano-sized precipitates were also observed in the AA6061 layers. Fig. 8.6(a) shows the microstructure of the AA6061 layers of the 5-cycle ARB processed AA1050/AA6061 composite. The nano-sized precipitates indicated by arrows. Fig. 8.6(b) shows the precipitates at a large magnification. The fine precipitates have a regular faceted shape and are about 50–150 nm in size. The coarse and fine precipitates in the AA6061 layer might behave as obstacles to dislocation movements and this may be one of the reasons for the higher hardness in this layer.
Fig. 8.6 Nano-sized precipitates in the AA6061 layers of the 5-cycle ARB processed AA1050/AA6061 composite.

The structure of a nano-sized precipitate was analysed by selected area electron diffraction and high-resolution TEM imaging and the results are shown in Fig. 8.7. It can be seen in Fig. 8.7(a) that the precipitate is about 50 nm in size and has a regular shape. Preliminary results obtained from several different SAD zone axis patterns indicate that the precipitate has a body-centred cubic (BCC) crystal structure and a lattice parameter of 1.284 nm. Fig. 8.7(b) shows a precipitate SAD pattern which can be indexed as [0 0 1] zone axis. Coherent relationship of the precipitate with the Al matrix can be described as \(<0 0 1>_{\text{precipitate}}//<0 1 1>_{\text{Al}}\) and \(\{2 0 0\}_{\text{precipitate}}//\{2 0 0\}_{\text{Al}}\). Note that the \(\{2 0 0\}\) spacing of the precipitate is three times that of the Al matrix, which indicates that the lattice constant of the precipitate is about three times that of aluminium. High-resolution TEM images of the precipitate are shown in Fig. 8.7(c) and (d) and their corresponding SAD patterns are shown in Fig. 8.7(e) and (f). Fig. 8.7(c) is observed along the [0 0 1] zone axis of the BCC structured precipitate and Fig. 8.7(d) along the [1 1 1] zone axis. The spacing of the \(\{1 1 0\}\) planes is measured and marked in both graphs.
Fig. 8.7 TEM characterisation of a nano-sized precipitate in the AA6061 layer of the 5-cycle ARB processed AA1050/AA6061 composite, (a) overall image (b) SAD pattern showing the coherency relationship with the aluminium matrix, (c) HRTEM image viewed along the [0 0 1] zone axis, (d) HRTEM image viewed along the [1 1 1] zone axis, (e) and (f) the corresponding SAD patterns of (c) and (d), respectively.

8.3 Mechanical properties of ARB processed AA1050/AA6061 composites

8.3.1 Tensile properties of ARB processed AA1050/AA6061 composites

Tensile test results of AA1050/AA6061 composites after 1 to 5-cycle ARB deformation are shown in Fig. 8.8 together with the annealed initial materials. As can be seen from the engineering stress-strain curves, that the stress increases and
the elongation decreases dramatically after ARB deformation, which is similar to the monotonic material sheets, as shown in Fig. 6.2(a) and Fig. 7.2(a). The tensile strength, uniform elongation and strain hardening rate are compared with the monotonic material sheets and are discussed in detail in Section 8.6.3.

![Graph showing engineering stress-strain curves of 1 to 5-cycle ARB processed AA1050/AA6061 composites.](image)

**Fig. 8.8** Engineering stress-strain curves of 1 to 5-cycle ARB processed AA1050/AA6061 composites.

### 8.3.2 Through-thickness hardness distribution of ARB processed AA1050/AA6061 composites

The through-thickness Vickers hardness values of the 1, 3 and 5-cycle ARB processed specimens are shown in Fig. 8.9 together with the corresponding optical micrographs. Note that the difference in thickness between the samples is due to the slight variation in reduction at each rolling cycle and the loss of materials through wire brushing during the surface treatment. As can be seen in Fig. 8.9(a), the hardness of the AA6061 layer is higher than the AA1050 layer and the hardness within the AA1050 and AA6061 layers is not homogeneous, having higher values near the surfaces. This phenomenon is similar to that observed in the monotonic material sheets and is believed to be due to the redundant shear deformation close
to the surfaces. The hardness distribution in the 3-cycle ARB processed AA1050/AA6061 composite appears to be similar to the 3-cycle ARB processed monotonic sheets, as shown in Fig. 6.4(b) and Fig. 7.4(b), the difference of which is the former is due to the different hardness of the AA1050 and AA6061 layers while the latter is due to the effect of the interfaces. The hardness of the 5-cycle ARB processed AA1050/AA6061 composite was deliberately measured on each layer of the laminates since the AA6061 layers are necked and fractured in some areas so that it is not uniformly distributed. Fig. 8.9(c) shows that the hardness of the AA6061 layers is about Hv 35 higher than the AA1050 layers. A clear jump at the interfaces is observed for both the 1 and 3-cycle ARB processed composite, which indicates that the two materials are directly bonded and there is no transition region [118]. Moreover, it can be seen that the hardness in the AA6061 layer is scattered more than the AA1050 layer, which is probably due to the dense precipitates in the AA6061 layers that may cause inhomogeneous microstructure and hardness variation.
Fig. 8.9 Through-thickness hardness distribution of (a) 1, (b) 3 and (c) 5-cycle ARB processed AA1050/AA6061 composites.
8.4 Texture evolution of AA1050/AA6061 composites during ARB deformation

The average texture was measured on a stack of RD-ND planes of the AA1050/AA6061 sheets after 1 to 5-cycle ARB deformation, and the $\varphi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the results are shown in Fig. 8.10. The average texture of the ARB processed AA1050/AA6061 composites is similar to the monotonic material sheets, as shown in Fig. 6.7 and Fig. 7.6. It can be seen from the figures that the Rotated Cube texture develops after 1-cycle ARB and remains strong in intensity up to 5-cycle ARB. The $\beta$ fibre begins to develop from the first cycle ARB and the intensity becomes stronger with increasing number of ARB cycles especially on the Copper and S orientations. Copper, S and Rotated Cube orientations are the most stable orientations in the ARB processed composites, which is also the same as the monotonic material sheets.

The strength variations of the main texture components along the main fibres are shown in Fig. 8.11. It can be seen from the figures that the texture components mixed with rolling and shear type develop after only 1-cycle ARB. Afterwards, the intensities of the components increase after 3-cycle ARB and remain almost the same after 5-cycle ARB. The major components are shear type component Rotated Cube and rolling type component Copper followed by the S component. The other components are much weaker than the major components.
The ODFs of the ARB processed AA1050 sheets, AA6061 sheets and AA1050/AA6061 composites (shown in Figs. 6.7, 7.6 and 8.10, respectively) show that the average textures of the three kinds of sheets after ARB deformation are
similar to one another. The through-thickness texture distribution of the AA1050/AA6061 composites is expected to be similar to the AA1050 and AA6061 sheets. The textures of the AA1050 and AA6061 layers in the AA1050/AA6061 composites are measured by EBSD at the corresponding layers in the centre of the 5-cycle ARB processed AA1050/AA6061 composite and the ODFs are shown in Fig. 8.12.

![Graphs showing texture distribution](image)

**Fig. 8.11** (a) α, (b) β, (c) τ, (d) γ (111) and (e) <110> fibres of the average texture of 1, 3 and 5-cycle ARB processed AA1050/AA6061 composites.
Fig. 8.12 $\varphi_2=0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the local texture of (a) AA1050 layer and (b) AA6061 layer in the centre area of 5-cycle ARB processed AA1050/AA6061 composite. (Contour levels 1×)

Fig. 8.12 shows that the textures developed in the AA1050 layer and the AA6061 layer are similar, and both are characterised with typical rolling texture and Rotated Cube component. In both layers, the Copper component is the strongest, followed by S and Rotated Cube, and the Brass component is relatively weak. As stated in Sections 6.4.2 and 7.4.2, the textures developed at the centre of the 5-cycle ARB processed sheets are a combination of Copper dominated $\beta$ fibre and Rotated Cube. The types of components and relative intensities in both layers of the AA1050/AA6061 composite are similar to the monotonic sheets at the same position. The $\tau$ fibre of AA1050 and AA6061 layers in the 5-cycle ARB processed AA1050/AA6061 composite is shown in Fig. 8.13. It can be seen that the intensity distribution along the $\tau$ fibre is similar for the AA1050 and AA6061 layers. The strongest intensity has shifted from the ideal position of Copper towards Rotated...
Cube. The intensity of Rotated Cube orientation is weaker than Copper/D. The intensity distribution of τ fibre of the AA1050 and AA6061 layers at the centre of the 5-cycle ARB processed AA1050/AA6061 composite, is similar to the 5-cycle ARB processed AA1050 and AA6061 sheets at the centre position, as shown in Fig. 6.9 and Fig. 7.8. The strongest intensities of Copper have also shifted towards Rotated Cube for the 5-cycle ARB processed AA1050 and AA6061 sheets. It can be concluded that the texture developed in the ARB processed AA1050/AA6061 composite is similar to that developed in the ARB processed monotonic material sheets. Although the microstructure and mechanical properties of the AA1050 and AA6061 layers in the AA1050/AA6061 composites are different from each other, the textures developed in the two layers are similar.

![Graph showing intensity distribution of Copper/D and Rotated Cube orientations with AA6061 and AA1050 layers]

**Fig. 8.13** τ fibre of AA1050 and AA6061 layers in the centre of the 5-cycle ARB processed AA1050/AA6061 composite.
8.5 Vacancy-type defects evolution of AA1050/AA6061 composites during ARB deformation

Fig. 8.14 shows the PALS results of the 1, 3 and 5-cycle ARB processed AA1050/AA6061 composites. The $\tau_1$ values are much smaller than the bulk lifetime, indicating annihilation into the bulk. The $\tau_2$ values are between the lifetime of vacancies associated with dislocations and bulk mono-vacancies but closer to the former. The values of the 1, 3 and 5-cycle ARB processed composites are similar. The intensities of long lifetime $\tau_2$ are all over 93%, indicating that most of the positrons are annihilated at defects.

The fractions of $\tau_1$ and $\tau_2$ can be estimated by $f_1 \times 220 + f_2 \times 245 = \tau_2$ and $f_1 + f_2 = 1$. The results are listed in Table 8.3. It can be seen that the fractions of vacancies associated with dislocations $f_1$ for the 1, 3 and 5-cycle ARB processed AA1050/AA6061 composites are over 80% and the values are quite close, which suggests that vacancies associated with dislocations are the major vacancy-type defects and the fractions of this type of defects are stable during ARB deformation.

Comparison of the vacancy-type defects in the ARB processed AA1050 sheets, AA6061 sheets and AA1050/AA6061 composites is discussed in detail in Section 8.6.4.
Fig. 8.14 Positron lifetime results of ARB processed AA1050 sheets (a) short lifetime, (b) long lifetime and (c) intensity of long lifetime.
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Table 8.3 Fractions of vacancy-type defects for ARB processed AA1050/AA6061 composites.

<table>
<thead>
<tr>
<th>Number of ARB cycles</th>
<th>Vacancies associated with dislocations ((f_1))</th>
<th>Mono-vacancies in the bulk ((f_2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>82.8</td>
<td>17.2</td>
</tr>
<tr>
<td>3</td>
<td>85.2</td>
<td>14.8</td>
</tr>
<tr>
<td>5</td>
<td>82.4</td>
<td>17.6</td>
</tr>
</tbody>
</table>

8.6 Discussion

8.6.1 Interfaces between the AA1050/AA6061 layers

Fig. 8.15 shows the micrographs of the 5-cycle ARB processed AA1050/AA6061 composite at higher magnification. The AA1050 and AA6061 layers can be identified by the dense micron-sized precipitates in the AA6061 layers. It is obvious that the AA6061 layers are irregular and wavy in shape, and fractured at various locations. The thicknesses of both layers vary from point to point. The fracture of AA6061 indicates that the elongation of the material during deformation exceeds the limit. The adjacent AA1050 materials then protrude and fill in the necked and fractured regions. As the hardening rate of AA6061 is significantly higher than that of AA1050, the strength of the AA6061 layers in the composite is higher than the AA1050 layers, the difference of which is reflected on the relative thinning of the two layers. The AA1050 layers thin down easier than AA6061 layers. At the first three cycles, the AA6061 layers are able to deform in the same way as the AA1050 layers. However, the deformation becomes inhomogeneous and the thickness of AA6061 layers starts to vary along the rolling direction at further ARB cycles. The AA6061 layers start to neck with further deformation and the AA1050 layers then protrude into the necked areas. Finally the AA6061 layers fracture at various locations and form the structure shown in Fig. 8.15.
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The shear zones have been shown in Fig. 8.1(c) and a closer view is shown in Fig. 8.15(a). The shear zones are the results of necking and fracturing in the AA6061 layers and flow in the AA1050 layers. It is reported that a layered microstructure can result in shear banding due to different flow properties in the different layers [122, 315].

![Fig. 8.15 Optical micrographs of 5-cycle ARB processed AA1050/AA6061 composite.](image)

TEM observations were conducted close to the bonding interfaces of the 5-cycle ARB processed composite. The micrographs are shown in Fig. 8.16. It can be seen that there are several morphologies of the interfaces. The AA1050 and the AA6061 layers are in close contact with each other in Figs. 8.16(a)-(c), whereas the two layers are separated by oxides and contamination induced small grains in Fig. 8.16(d). Bonded interfaces consisting of finer grains and oxides and precipitations were also reported in other studies [42, 287].
Fig. 8.16 (a)–(d) TEM micrographs close to the interfaces of the 5-cycle ARB processed AA1050/AA6061 composite, (e) an enlarged graph of the interfacial area in (d), and the SAD pattern of (f) AA1050 layer, (g) interfacial area and (h) AA6061 layer in (d).
In addition, there are dense micron-sized precipitates in the AA6061 layer (indicated by arrows in Fig. 8.16), which influence the metal flow during the rolling deformation and form a curved interface (Fig. 8.16(b)) or induce the rotation of the elongated grains to a certain angle with respect of the interface (Fig. 8.16(c)). It is believed that the morphologies of the interfaces would influence the bond quality. The finer grains induced by oxides and contamination cause high hardness values close to the interfaces [118, 287], which may improve bond toughness. Nano-sized secondary particles were intentionally added between the interfaces to enhance bond strength [316, 317]. However, the precipitates and particles close to the interfaces may act as origins of little cracks which later induce de-lamination and reduce bond strength. In the present work, the surfaces of the sheets were well brushed and cleaned to ensure that thick oxidation layers and large contaminations were removed. The more refined grains observed close to the interfaces due to thin oxides and contaminations lead to enhanced hardness and hence improve bond quality.

During ARB, bonding occurs by fracture of the oxide layers to allow the fresh metals to contact each other, which leads to two kinds of areas along the interface. Type I is direct contact of fresh metals (AA1050/AA6061 contact in this case), as shown in Fig. 8.16(a) and (c), while type II is original metal surfaces with thin oxide film layers in between. The oxide films, which are brittle, are easy to break and allow further refinement of the materials around the interface. Therefore, the grain size in this kind of interface is finer than the other areas. As can be seen in Fig. 8.16(d), the AA1050/AA6061 interfacial area is characterised by fine grains which can easily be distinguished from AA1050 but not AA6061, because the grain size at the interface is closer to the AA6061 layer. The interfacial areas with further refined grains are
localised and can only be observed in small parts of the interfaces. The grains in this type of interfacial areas are also more equiaxed, as can be seen from the enlarged micrograph in Fig. 8.16(e). It is believed that the grain refinement at the interface is caused by the oxide film and other contamination. The fraction of high angle grain boundaries around the interfacial area is also higher than the AA1050 and AA6061 layers, as indicated by the SAD patterns in Figs. 8.16(f)-3(h).

The development of the two kinds of interfacial areas is shown schematically in Fig. 8.17. Assuming that there are thin layers of oxide film at the surface of the starting materials and it distributes evenly along the surface. Some contamination would also exist at the surface even though it has been cleaned and wire brushed before ARB deformation. Both the oxide film and contamination would influence the deformation and the morphologies of the interfacial areas. As the oxide films have much lower ductility than aluminium, it is easy to fracture even at low cycles of ARB deformation, as shown in Fig. 8.17(b). The fresh metals where the oxide films fractured appear and closely bond with each other due to the pressure of the rolling deformation and further refine with the increasing ARB cycles. The areas where the oxide films are located between the metal layers will allow further refinement because the oxide films are harder than the metals. Therefore, the grain sizes of the materials around the type II interfacial areas are smaller than those around the type I interfacial areas and the metal matrix, as shown in Fig. 8.17(c). Besides the microstructures of the interfacial areas shown in Fig. 8.16 (type I in Figs. 8.16(a)-(c) and type II in Fig. 8.16(d)), the two types of interfacial areas can be observed with optical microscope, as shown in Fig. 8.18. Note that the thick interfacial areas are deliberately chosen to show the effect but the thicknesses of the type II interfacial areas are generally much smaller than that shown in Fig. 8.18.
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Fig. 8.17 Schematic diagram showing the development of two types of interface morphologies (a) Initial state: surface oxide and contamination influenced layers are shown as straight, (b) Deformation at low cycles: brittle surface layers break up and induce areas with direct contact of fresh metals (type I) and areas of original metals with brittle surface layers in between (type II), and (c) Deformation at high cycles: brittle surface layers allow further refinement of the materials around the interface.

Fig. 8.18 Type I and II interfacial areas observed in optical micrograph of the 5-cycle ARB processed AA1050/AA6061 composite.

8.6.2 Bonding properties of ARB processed sheets

The bonding properties between the two layers of the sheet have a crucial influence on the properties and applications of the ARB processed materials. Sound bonding is considered to be a crucial factor for successful ARB processed sheets. Surface treatment prior to roll bonding, such as degreasing and wire-brushing, removes the oxide layers, reveals fresh metals and allows good bonding between the layers. This is the most commonly used method to improve the bond quality. Besides, bonding is affected by the amount of deformation per rolling cycle, the types of metals, the rolling temperature, etc.
Influence of the state of the initial materials on bonding quality was investigated in this work. The initial AA1050 sheets in a hot rolled state and the AA6061 sheets in a T6 treated state were used as starting materials and were ARB processed after pre-heating at 250 °C for 10 min. The bonding quality differed a great deal from that of the samples with fully annealed starting materials.

Fig. 8.19 shows the cross sections of the 1 and 3-cycle ARB processed samples with the two types of starting materials observed by optical microscopy and the fracture surfaces of the 5-cycle ARB processed samples. The optical micrographs reveal that the bonding interfaces can be observed in both materials by the different contrast between the two types of alloys due to dense precipitates in the AA6061 layers. The interfaces are in closer contact and there is no delamination at the interface of the composites with fully annealed starting materials. However, for the composites with starting materials in the as-received state, delamination can be observed in some areas between the layers, shown as black lines along the interfaces in the graphs. This indicates that the bonding properties of the sheets with the starting materials in the fully annealed state are better than the other one. Delamination along interfaces on polished surfaces can always be observed in ARB processed sheets, but it is mostly characterised by black lines, and is not severe [38, 294, 297]. It is more obvious to be observed on the tensile failed surfaces [38, 109, 111, 114, 147]. Fig. 8.19(c) and (f) shows the tensile failed surfaces of the 5-cycle ARB processed composites from the starting materials of the two different states. It can be seen from the graphs that the fracture surface of the sample with the annealed starting materials is more like single layered sheets, where the two layers deform in a coherent way and the delamination is not as severe as the sample with the as-received starting materials. This again proves that the fully annealed starting
materials result in better bonding. The pre-heating temperature of the samples with the as-received starting materials is higher and the total reduction is also higher, as can be seen from the total thickness of the materials, both of which should result in better bonding. The difference between the bonding properties of the materials is believed to be due to the annealing process before ARB.

Vickers hardness of the as-received AA1050 and AA6061 sheets were 44.1 and 102.7, respectively. These values are considerably higher than the corresponding fully annealed materials (28.9 and 39.0, respectively). As a result, the as-received sheets are difficult to deform and form good bonding. Moreover, there is a huge difference in the hardness of the as-received AA1050 and AA6061, while the difference of the annealed AA1050 and AA6061 is less pronounced. Therefore, the relative reductions of the two materials per cycle are different for the two groups of ARB processed sheets, as can be seen from Figs. 8.19(a)–(d) that the volume fraction of AA1050 in the as-received starting materials group is smaller than that of AA6061, while it is approximately the same for the group with fully annealed starting materials.
Bond strength refers to the interfacial shear strength between two layers of the ARB processed sheet. There are several methods to determine the bond strength, such as the peeling test, tensile shear test and T-peel test. In this work, the tensile shear test adopted from Ref. [318] was used to test the bond strength. In this method, a rectangular sheet was first machined from the ARB processed materials.
Two narrow slots were milled to the centre interface along the thickness direction from opposite surfaces, as shown in Fig. 8.20.

![Schematic diagram of the sample for tensile shear test to determine bond strength.](image)

Fig. 8.20 Schematic diagram of the sample for tensile shear test to determine bond strength.

As the rolling cycle increases, the number of the interfaces increases. Previous research in our group showed that the centre interface, which is the newly formed interface, is the weakest of all the interfaces [316]. Therefore, we used the bond strength of the centre interface to evaluate the bonding properties of the whole workpiece. Tensile tests were conducted on the specimen and the bond strength was calculated according to

\[
\tau_B = \frac{F_B}{(L_B \times W_B)}
\]

(8.1)

where \(F_B\) is the measured load, \(L_B\) is the length of the areas for bond strength testing and \(W_B\) is the sample width.

The ratio of bond strength to tensile strength for the 1, 3 and 5-cycle ARB processed composites with the as-received starting materials is determined to be 0.24, 0.27 and 0.25, respectively. The values are greater than that reported for low carbon steels [318], and comparable to the previous research results from our group by adding nano-sized particles between the AA6061 layers [316]. The reason
may be due to the high roll roughness in this work and the use of AA1050 between the AA6061 layers which is easy to deform and improve the bond strength. As for the composites with fully annealed starting materials, the bond strength cannot be obtained from the specimens with the same geometry as the group with as-received starting materials, where the length of the interface for bonding test was 5 mm. The fracture occurred at the slots in the form of normal tensile fracture instead of tensile shear fracture along the interface, which indicates that the composites with the fully annealed starting materials have higher bond strength than those with the as-received starting materials. Samples with shorter interface for bonding test can be used for bond strength testing in future work.

It can be concluded from this section that the ARB processed AA1050/AA6061 composites have good bonding quality. Besides, the bonding quality of the composites should be in between the corresponding ARB processed AA6061 sheets and AA1050 sheets. However, it is reasonable to conclude that good bonding was achieved for both the ARB processed sheets since delamination is hardly observed on the polished surfaces. High roll roughness and elevated rolling temperature have played important roles in improving the bonding properties.

8.6.3 Comparison of mechanical properties of ARB processed AA1050/AA6061 composites with the monotonic material sheets

The tensile properties of the three kinds of ARB processed sheets are shown in Fig. 8.21 and Fig. 8.22. The error bars in Fig. 8.21 are smaller than the symbols. The tensile strength increases with the number of ARB cycles for all the sheets and the values after 5-cycle ARB are more than two times greater than the initial materials. Uniform elongation, on the other hand, decreases substantially after 1-cycle ARB
and remains almost unchanged with further deformation. The uniform elongation of all three types of materials is less than 3%. It is common for the ARB processed materials that necking starts at an early stage of deformation and results in early fracture and low ductility [42, 97, 111, 120].

Fig. 8.21 shows that the tensile strength of the AA6061 sheets is always the highest and that of the AA1050 sheets are the lowest, while the tensile strength of the AA1050/AA6061 composites is located in between the two monotonic material sheets. Curve (d) of Fig. 8.21 is calculated from the values of curves (a) and (b) by the rule of mixture \( \sigma = V_{1050} \sigma_{1050} + V_{6061} \sigma_{6061} \), where \( V_{1050} \) and \( V_{6061} \) are the volume fractions of AA1050 and AA6061, as shown in Table 8.1, \( \sigma_{1050} \) and \( \sigma_{6061} \) are the tensile strength of the AA1050 and AA6061 sheets, respectively. It is obvious that the directly measured tensile strength of the composite fits reasonably well with the value obtained by the rule of mixture. Note that in the present work the initial values for the rule of mixture are the tensile strength of the sheets processed by ARB from monotonic starting materials. However in Ref. [120], the values used for the rule of mixture were calculated from the average hardness measured on the two different layers of the composite. The fact that the measured tensile strength fits with the calculated ones indicates that both the materials AA1050 and AA6061 undergo a similar strain path regardless of whether they are deformed in the monotonic material sheets or in the composites. Fig. 8.21 shows that the difference between the measured tensile strength of the AA1050/AA6061 composite and the value from the rule of mixture for the 5-cycle ARB processed materials is greater than the other cycles, which can be attributed to the fact that the hard layers in the composite have less deformation after 5-cycle ARB, as indicated in Table 8.1. It is
expected that the tensile strength of the composite subjected to more than 5-cycle ARB would move away from the value of the mixture rule.

Fig. 8.21 Tensile strength values of ARB processed (a) AA6061 sheets, (b) AA1050 sheets, (c) AA1050/AA6061 composites and (d) AA1050/AA6061 composites calculated by rule of mixture. (0 ARB cycle corresponding to hardness of annealed materials, the same for the following figures)

Fig. 8.22 Uniform elongation of ARB processed (a) AA6061 sheets, (b) AA1050 sheets and (c) AA1050/AA6061 composites.
Fig. 8.23 shows the true stress-strain curves of the 5-cycle ARB processed sheets with monotonic and different starting materials, and the initial annealed AA1050 and AA6061 along with their strain hardening rate. In the initial annealed materials, the flow stress is low and strain hardening rate stays at a relative high value even at high strain. In the 5-cycle ARB processed sheets, the strain hardening rate rapidly decreases at a very early stage of true strain and meets the true stress-strain curve at the peak stress, which is considered to be the necking point according to Considére criterion shown in Eq.(2.5). The rapid drop in the strain hardening rate is considered to be the reason for early plastic instability and hence limited uniform elongation of the ARB processed materials [33].

![True stress-strain curves](image)

**Fig. 8.23** True stress-strain curves along with the strain hardening rate of (a) annealed AA1050, (b) annealed AA6061, (c) 5-cycle ARB processed AA6061 sheet, (d) 5-cycle ARB processed AA1050/AA6061 composite and (e) 5-cycle ARB processed AA1050 sheet.

Fig. 8.24 shows the effect of ARB deformation on the strain hardening rate, which is plotted against the true strain. The effect of ARB deformation can be seen by comparing the strain hardening rate of ARB processed samples for different cycles. As can be seen in Fig. 8.24(a), the strain hardening rate increases with the number
of ARB cycles. A similar phenomenon has been observed in Ref. [290]. This can be explained by decreasing grain sizes with the increasing number of ARB cycles due to the increasing accumulative strain. The high strain hardening rate in the samples with high accumulated strain is due to the smaller grain sizes in these samples, which makes it easier for dislocations to encounter grain boundaries, and then resist further deformation.

Fig. 8.24(b) shows the strain hardening rate of 5-cycle ARB processed samples with different starting materials along with the initial annealed materials. It can be seen that the initial hardening rate of the ARB processed samples shows a larger value than the corresponding annealed conventional sized samples, which can be explained by the grain size effect above. However, the annealed samples display positive strain hardening values to significant high strains (~34% for annealed AA1050 and ~24% for annealed AA6061), whereas the strain hardening rate of the ARB processed samples drops rapidly below zero after only a small plastic strain was subjected to the tensile specimens (less than 3% for all the cases). This explains the limited uniform elongation of the ARB processed samples. It also can be seen in Fig. 8.24(b) that 5-cycle ARB processed AA6061 has a higher strain hardening rate than that of the 5-cycle ARB processed AA1050 and the initial strain hardening rate of the 5-cycle ARB processed AA1050/AA6061 is located between the values of the two monotonic sheets, which is in good correspondence with the tensile strength values.
Fig. 8.24 Strain hardening rate of (a) 1, 3 and 5-cycle ARB processed AA1050/AA6061 composites and (b) annealed starting materials and 5-cycle ARB processed sheets with different starting materials.

The average microhardness of the ARB processed samples is shown in Fig. 8.25. The evolution of hardness with the number of ARB cycles is similar to the tensile strength. The hardness increases rapidly after the first cycle ARB and then the increment between each cycle becomes less significant. After 5-cycle ARB, the hardness becomes around two times greater than the initial values for both AA6061 and AA1050. The hardness of AA6061 is always higher than AA1050.
The tensile test takes the AA1050/AA6061 composite as a complete material, which makes it hard to evaluate the behaviour of the two different layers. However, in the hardness test, the values of the AA1050 and AA6061 layers of the composite can be measured separately. Curves (b) and (c) in Fig. 8.25 denote the average hardness values of the AA6061 and AA1050 layers in the composites, respectively. The figure shows that the average hardness of AA6061 single material sheets is close to the AA6061 layers in the AA1050/AA6061 composites, and so are the AA1050 materials. This confirms the conclusion obtained from the tensile strength values in that the materials AA1050 and AA6061 undergo similar deformation no matter they are deformed in the single material sheets or in the composites for up to 5 ARB cycles.

![Graph showing hardness values](image)

**Fig. 8.25** Average hardness values of ARB processed (a) AA6061 sheets, (b) AA6061 layer in AA1050/AA6061 composites, (c) AA1050 layer in AA1050/AA6061 composites and (d) AA1050 sheets.

The increment of strength/hardness with respect to the ARB cycles can also be considered as an increment with the decreasing grain size, as the grain size is decreasing with increasing number of ARB cycles in the current deformation. The
relationship of strength and grain size can be specified by the conventional Hall-Petch relationship which is given by Eq. (2.4).

The values of $\sigma_y$ are plotted against $d^{1/2}$ for the ARB processed sheets, as shown in Fig. 8.26. The conventional grain sized relationship was reported in Ref.[319] for pure aluminium and $k_y$ was 70 MPa $\mu$m$^{1/2}$. However, the $k_y$ values of the ARB processed sheets are different from the conventional sized aluminium. The deviation of conventional grain sized and UFG materials in the Hall-Petch relationship is commonly observed [23, 149, 290]. For the curves (a)–(d), the $k_y$ values are 124.57, 83.46, 169.70 and 139.68 MPa $\mu$m$^{1/2}$, respectively. These values are comparable to those reported for UFG aluminium [149, 290].

![Hall-Petch relationships of ARB processed](image)

**Fig. 8.26** Hall-Petch relationships of ARB processed (a) AA6061 sheets, (b) AA6061 layer in AA1050/AA6061 composites, (c) AA1050 layer in AA1050/AA6061 composites and (d) AA1050 sheets.
8.6.4 Comparison of vacancy-type defects between the ARB processed AA1050, AA6061 and AA1050/AA6061

To date, positron lifetime examination on ARB processed aluminium has rarely been reported. Pure aluminium [302] and AA8006 [320] are the two references on the ARB processed aluminium investigated by PALS and both work did not provide a detailed discussion. The types of defects in the ARB processed samples are supposed to be more than that in the ECAP processed samples because the interfaces between different layers in ARB processed sheets are also defects in the samples. However, this type of defects is not always able to be detected by PALS. Normally, voids are believed to exist along the interfaces. But the lack of any long lifetimes corresponding to voids in the PALS results suggests that there are no voids in the samples. The reason might be because of the good bonding quality of the sheets so that voids do not appear until after tensile deformation, as discussed in Section 8.6.2.

As shown in the previous chapters, vacancies associated with dislocations are the major vacancy-type defects but some bulk mono-vacancies for ARB processed AA1050 sheets still exist. However, most of the vacancy-type defects in the ARB processed AA6061 sheets are vacancies associated with dislocations. A very small fraction of bulk mono-vacancies can be extracted but it is so small that it can be neglected. The major microstructure difference of the ARB processed AA6061 and AA1050 sheets is the smaller grain size and the dense precipitates in the AA6061. The small grain size offers large fractions of grain boundaries which are supposed to be able to increase the possibility of positrons annihilating at vacancies associated with dislocations. However, the smaller $\tau_2$ values contradict this
assumption. Therefore, the precipitates in the AA6061 sheets must play an important role in the positron annihilation.

Precipitates in aluminium alloys can be described by three types: coherent, semi-coherent and incoherent [321]. All three types of precipitates can be detected with positron annihilation by combining lifetime measurements with Doppler broadening [321]. Principally, coherent precipitates can act as shallow positron traps, but for incoherent precipitates, trapping occurs at misfit interfaces between the matrix and the precipitates, which is comparable to grain boundaries and gives a similar lifetime value [211, 321]. The positron lifetime related to the coherent precipitates in aluminium alloys is reported to be around 210 ps in Ref.[211, 322], and 220 ps in Ref. [323]. In Ref. [324], positron annihilation was conducted to solution treated and aged and HPT deformed AA2017. Long lifetime $\tau_2$ is determined to be $\sim 225$ ps for the solution treated and aged sample and $\sim 240$ ps for the HPT deformed sample. It was stated that the positrons are trapped in Cu-vacancy complexes and $\theta'$-Al interfaces for the solution treated and aged sample. It can be concluded that the positron lifetime for coherent precipitates is slightly smaller than the lifetime for incoherent precipitates and is similar to the vacancies associated with dislocations.

The nano-sized precipitates in AA6061 are coherent with the matrix (shown in Section 8.2.2) and can be considered as shallow positron traps. The micron-sized precipitates, on the other hand, provide dense dislocations along the precipitates-matrix interfaces which give lifetimes that are characteristic of vacancies associated with dislocations. The lifetimes from the two types of precipitates are close to the vacancies associated with dislocations, which make it difficult to be separated. Therefore, the $\tau_2$ values measured in the ARB processed AA6061 sheets are a
combination of the characteristics of micron- and nano-sized precipitates, vacancies associated with dislocations and bulk mono-vacancies. As the lifetimes of the precipitates are smaller or close to the vacancies associated with dislocations, the overall lifetimes of the ARB processed AA6061 sheets appear to be smaller than the sample without precipitates, such as ARB processed AA1050.

As the fractions of AA1050 and AA6061 are approximately equal to each other, the fraction of positrons annihilated in the two layers is supposed to be equal. The positron lifetime values may also apply to the rule of mixture. As a matter of fact, the \( \tau_2 \) values of the ARB processed AA1050/AA6061 composites are generally in between the values of AA1050 and AA6061 sheets, with a small variation for the 1-cycle ARB processed samples. This variation might be because of the measurement errors. The error bars of the lifetimes of the ARB processed samples are larger than that of the ECAP processed samples, as can be compared in the Figs. 4.10, 5.9, 6.11, 7.10 and 8.14. The reason is because the types of defects in the ARB processed samples are more than the ECAP processed samples, especially the refined microstructure in the interfacial areas and the precipitates in the bulk. These defects result in lifetimes close but not equal to the vacancies associated with dislocations, which makes it difficult to resolve but increase the error bars.

**8.7 Summary**

AA1050/AA6061 laminated composites with layered microstructure and mechanical properties distribution were successfully processed by ARB process. The following can be summarised.

(1) Laminated composite materials with strong bonding between the AA1050 and AA6061 layers are obtained and substantial grain refinement is achieved after ARB
processing. The grain sizes of the AA1050 and AA6061 layers in the composites are similar to those in the corresponding monotonic sheets.

(2) During the first 3 ARB cycles, the AA1050 and AA6061 layers in the composite deformed in a nearly identical way. The AA1050 and AA6061 interfaces are straight and both layers have a constant thickness. Afterwards the AA6061 started to neck and eventually fractured at various locations due to the different flow properties of AA1050 and AA6061 which induces plastic instabilities around the interfaces. Severe shear bands are observed throughout the cross-section of the 5-cycle ARB processed composite. The angles of the shear zones with respect to the rolling direction have the highest value close to the thickness centre and decrease when moving towards the surfaces. Shear bands are hardly seen near the two surfaces as the AA6061 layers close to the surfaces of the composite are not fractured.

(3) The areas around the interface of two adjacent layers are observed and it has been found that the microstructure of the bonded interface was quite complex. Two types of interfacial morphologies are observed along the AA1050/AA6061 interfaces: Type I is the area with direct contact between fresh metals and type II is the area with original metals with brittle surface layers in between. Type II interfacial morphologies allow further refinement of the microstructure close to the interface.

(4) The hardness and tensile strength of the 5-cycle ARB processed composite become more than two times greater than the initial values whereas the ductility drops significantly. The tensile strengths of the composite predicted by the rule of mixture are comparable to the measured values. The hardnesses of AA1050 and AA6061 in the composite are almost identical to those in the monotonic material
sheets, which indicates that both materials have accumulated similar strain no matter deformed in the composites or in the monotonic material sheets.

(5) The average texture developed in the composites is similar to the monotonic sheets. The major components are the shear type component Rotated Cube and rolling type components Copper and S. The other components like rolling components Brass and shear components Rotated Copper, E and F are considerably weaker than the major components. The textures developed in the AA1050 and AA6061 layers at the centre of the 5-cycle ARB processed composite are similar to each other and also to those of the monotonic material sheets at the same position.

(6) Vacancies associated with dislocations are the major type of vacancy-type defects in the ARB processed sheets. The positron lifetimes in the ARB processed AA6061 sheets are combinations of characteristic lifetimes of coherent nano-sized precipitates, incoherent micron-sized precipitates, vacancy-associated with dislocations and bulk mono-vacancies. The lifetime values of the ARB processed AA1050/AA6061 composites are generally in between the values of the ARB processed AA1050 and AA6061 sheets.
Chapter 9 Conclusions and suggestions for future work

9.1 Conclusions

In this work, AA1050 was processed by ECAP at both room temperature and cryogenic temperature, and AA1050 sheets, AA6061 sheets and AA1050/AA6061 composites were processed by ARB from AA1050 and AA6061. The evolution of the microstructure, mechanical properties, texture and vacancy-type defects with the number ECAP or ARB passes were investigated. The effect of cryogenic temperature on ECAP, the influence of high roll surface roughness on the heterogeneity of the ARB processed sheets, the properties of AA1050 and AA6061 layers in the AA1050/AA6061 composites and in their monotonic material sheets, and the difference of the properties of AA1050 subjected to ECAP and ARB have been discussed. The following conclusions can be drawn.

(1) Substantial grain refinement was achieved for all the ECAP and ARB deformed samples. The average grain size after CT-ECAP is comparable but slightly smaller than after RT-ECAP due to the suppression of dynamic recovery during CT-ECAP deformation. The ARB processed sheets with good bonding were achieved and the grain refinement of the AA6061 sheets is more pronounced than the AA1050 sheets. The grain sizes of the AA1050 and AA6061 layers in the composites are similar to the corresponding monotonic sheets. Furthermore, it is found that the grain size of the AA1050 after ARB is smaller than those after RT- and CT-ECAP to the same strain, indicating that the ARB process is more efficient in grain refinement.
(2) Severe shear bands were observed throughout the cross-section of the 5-cycle ARB processed composite. The AA6061 layers necked and fractured at various locations due to the different flow properties of AA1050 and AA6061, which induced plastic instability around the interfaces. The areas around the interface of two adjacent layers were observed and it was found that the microstructure of the bonded interface was quite complex. Two types of interfacial morphologies were observed along the AA1050/AA6061 interfaces: Type I is the area with direct contact of fresh metals and type II is the area with original metals with brittle surface layers in between. Type II interfacial morphologies allow further refinement of the microstructure close to the interface.

(3) Significant strengthening was achieved for all the ECAP and ARB deformed samples. The CT-ECAP processed samples have higher hardness and tensile strength than the RT-ECAP processed samples deformed to the same strain. The ductility of the two kinds of samples is similar to each other. The tensile strengths of the ARB processed AA1050/AA6061 composites predicted by the rule of mixture are comparable to the measured values. The hardness of the AA1050 and AA6061 layers in the ARB processed composites are nearly identical to those in the monotonic material sheets, which indicates that both materials accumulated similar strain regardless of whether they were deformed in the composites or the monotonic material sheets. The hardness and tensile strength of AA1050 after ARB were higher than after RT- and CT-ECAP to the same strain, which suggests that the ARB process is more efficient at strengthening compared to ECAP.

(4) The textures developed after RT-ECAP displayed similar features as the theoretical ECAP texture reported in the literature. The texture developed after CT-ECAP is completely different from after RT-ECAP. For the ARB processed samples,
Copper dominated β fibre and Rotated Cube component were observed for all sheets, indicating that these components are the most favourable texture in the ARB process.

(5) The hardness, grain size and texture distribution throughout the thickness of the ARB processed sheets were not uniform due to the redundant shear strain close to the surface areas of the sheets because of the high roll roughness used in this work. For the ARB processed AA1050 and AA6061 sheets, a typical rolling texture was obtained at the sheet centre for the 1-cycle ARB processed sheets, and a combination of rolling texture and Rotated Cube texture was observed for the 3 and 5-cycle ARB processed sheets. The textures at the quarter-thickness were pure shear texture after 1-cycle ARB and a combination of rolling and shear texture after 3 and 5-cycle ARB. Strong Rotated Cube texture developed at the sheet surfaces. Copper dominated β fibre and Rotated Cube component were observed for both the AA1050 and AA6061 layers in the centre of the 5-cycle ARB processed AA1050/AA6061 composite.

(6) A high fraction of vacancy-type defects were detected for all the ECAP and ARB deformed samples. Vacancies associated with dislocations and bulk mono-vacancies are the major vacancy-type defects in the ECAP processed samples. Bulk di-vacancies were also identified in the 4-pass CT-ECAP processed sample. A quantitative relationship between the hardness and the defect concentration and defect diffusion coefficient has been established. The increased hardness of the CT-ECAP processed samples can be attributed to the existence of bulk mono- and di-vacancies in these samples. The positron lifetime of the ARB processed AA1050 is much smaller than for RT- and CT-ECAP processed AA1050 which is due to the elevated deformation temperature during ARB.
Vacancies associated with dislocations are dominant in the ARB processed AA1050 sheets. The positron lifetimes in the ARB processed AA6061 sheets are combinations of characteristic lifetimes of coherent nano-sized precipitates, incoherent micron-sized precipitates, vacancy-associated with dislocations and bulk mono-vacancies. The positron lifetimes in the ARB processed AA1050/AA6061 composites are between the ARB processed AA1050 and AA6061 sheets.

9.2 Suggestions for future work

The following areas are suggested to be conducted to continue the research from this work.

ECAP at cryogenic temperature was conducted in this work and further refinement of the grains and an improvement in the hardness and strength were observed compared with room temperature ECAP. However, the difference in the grain size is not great and the ductility of the samples after RT- and CT-ECAP is similar to each other. It is recommended that aluminium alloys with a higher content of alloy elements such as AA6061 can be processed by CT-ECAP to further restrict the dynamic recovery and refine the microstructure.

The texture developed after CT-ECAP is different from RT-ECAP but the formation mechanism is not completely clear. The CT-ECAP texture is assumed to be consistent with the texture of simple shear at cryogenic temperature but it turned out to be different. The torsion of pure aluminium at cryogenic temperature can be conducted and used to compare with the CT-ECAP texture. In addition, the CT-ECAP textures reported in this work are the global texture and are the textures at a steady state of each pass. EBSD work is recommended at different areas of the sample to investigate the homogeneity of the specimens. The textures of the areas
close to the intersection plane of a halfway extruded specimen can be tested to study the progressive evolution of textures during CT-ECAP deformation.

The interfacial areas of the ARB processed AA1050/AA6061 composites were investigated by OM and TEM in this work. Severe shear bands were observed but cannot be analysed in detail due to the limited resolution of OM and localised observation area of TEM. Detailed EBSD work of the ARB processed AA1050/AA6061 composites is recommended to analyse of shear banding and provide a better understanding of the influence of the interfaces and precipitates.

It is concluded that ARB is a more effective way to achieve grain refinement and sample strengthening, and the smaller positron lifetimes of the ARB processed samples are due to the elevated deformation temperature for ARB. Therefore, PALS tests are recommended for room temperature or lower temperature ARB deformed samples to compare with the results of ECAP. Doppler broadening method is also recommended to be combined with PALS to investigate the effect of the precipitates on the lifetimes.
References


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References


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