

Deformation Mechanism of Twinning- Detwinning of Wrought and Constrained Groove Pressing Processed Magnesium AZ31B Alloy



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Abstract

Magnesium alloys have attracted great interest in the 21st century as ultra lightweight metallic structural materials with outstanding mechanical properties. AZ31B alloy is one of the most widely used magnesium wrought alloys in sheet form for which twinning, a dominant plastic deformation mechanism at room temperature, remains less well understood than crystal slip. The main objective of this thesis is to advance, both experimentally and numerically, the understanding of the mechanism and the conditions of the twinning-detwinning (TDT) process, and the influence of these processes on the mechanical deformation response of magnesium AZ31B alloy in the wrought and constrained groove pressing (CGP) processed conditions.

To achieve this goal in the present study, new applications of Digital Image Correlation (DIC) analysis were developed for two strain evaluation techniques. In the context of the Focused Ion Beam (FIB)-DIC micro-ring-core method of residual stress evaluation, platinum deposition at the sample surface in the Scanning Electron Microscope vacuum chamber led to a dramatic improvement of the correlation accuracy. Another new approach was proposed to track the strain-induced changes in the Debye-Scherrer rings on 2D powder X-ray diffraction (XRD) patterns using DIC (referred to as XRD-DIC) allowing to minimise the error and increase the efficiency of obtaining the full in-plane strain information.

The effect of twinning on the deformation asymmetry was studied by *ex situ* XRD on a bent bar of wrought sample. The asymmetry of twinning was shown to lead to the variation in the plastic strain accommodation efficiency, which affected yielding. Furthermore, wrought and/or CGP processed samples were examined under *in situ* cyclic loading by XRD and electron backscatter diffraction for comparison. The first cycle exhibited a clear difference in the twin volume fraction and activation of TDT compared to the subsequent cycles.

An advanced crystal plasticity finite element model of crystal slip, twinning and detwinning was developed to simulate the behaviour of a wrought sample subjected to compression-tension loading. Information on the activity of deformation systems and the critical resolved shear stress for detwinning was obtained. The model gave new insights into the non-Schmid behaviour (grain neighbour effects) for twinning, and particularly for detwinning. The grain neighbour effect becomes more pronounced with increasing plastic strain level, particularly when detwinning dominates the deformation response.

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Most of the work in this thesis has already been published or submitted as listed below:

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- [4] E. Salvati, **H. Zhang**, K.S. Fong, R.J. Paynter, X. Song, and A.M. Korsunsky, *Fatigue and Fracture Behaviour of AZ31B Mg alloy Plastically Deformed by Constrained Groove Pressing in the Presence of Overloads*. Procedia Structural Integrity, 2016. 2: p. 3772-3781.
- [5] E. Salvati, T. Sui, **H. Zhang**, A.J.G. Lunt, K.S. Fong, X. Song, and A.M. Korsunsky, *Elucidating the Mechanism of Fatigue Crack Acceleration Following the Occurrence of an Underload*. Advanced Engineering Materials, 2016. 18(12): p. 2076-2087.
- [6] E. Salvati, L.R. Brandt, F. Uzun, **H. Zhang**, C. Papadakis, and A.M. Korsunsky, *Multiscale Analysis of Bamboo Deformation Mechanisms Following NaOH Treatment Using X-Ray and Correlative Microscopy*. Acta Biomaterialia, 2018.
- [7] E. Salvati, L.R. Brandt, C. Papadakis, **H. Zhang**, S.M. Mousavi, D. Wermeille, and A.M. Korsunsky, *Nanoscale Structural Damage due to Focused Ion Beam Milling of Silicon with Ga Ions*. Materials Letters, 2018. 213: p. 346-349.
- [8] S. Ying, L. Ma, T. Sui, C. Papadakis, E. Salvati, L.R. Brandt, **H. Zhang**, and A.M. Korsunsky, *Nanoscale Origins of the Size Effect in the Compression Response of Single Crystal Ni-Base Superalloy Micro-Pillars*. Materials, 2018. 11(4).

- [9] T. Sui, K. Titov, S. Ying, **H. Zhang**, I.P. Dolbnya, J.-C. Tan, and A.M. Korsunsky, *Probing the Nano-Scale Architecture of Diamond-Patterned Electrospun Fibre Mats by Synchrotron Small Angle X-Ray Scattering*. RSC Advances, 2017. 7(14): p. 8200-8204.
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- [11] L.R. Brandt, E. Salvati, C. Papadaki, **H. Zhang**, S. Ying, E. Le Bourhis, I.P. Dolbnya, T. Sui, and A.M. Korsunsky, *Probing the Deformation and Fracture Properties of Cu/W Nano-Multilayers by in situ SEM and Synchrotron XRD Strain Microscopy*. Surface and Coatings Technology, 2017. 320: p. 158-167.
- [12] A.J.G. Lunt, N. Baimpas, E. Salvati, I.P. Dolbnya, T. Sui, S. Ying, **H. Zhang**, A.K. Kleppe, J. Dluhoš, and A.M. Korsunsky, *A State-of-the-Art Review of Micronscale Spatially Resolved Residual Stress Analysis by FIB-DIC Ring-Core Milling and Other Techniques*. The Journal of Strain Analysis for Engineering Design, 2015. 50(7): p. 426-444.

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Acronyms

1D	– One-Dimensional
2D	– Two-Dimensional
3D	– Three-Dimensional
BCC	– Body Centred Cubic
CG	– Composite Grain
CGP	– Constrained Groove Pressing
ConFit	– Conversion + Gaussian Fitting
CPFE	– Crystal Plasticity Finite Element
CPFEM	– Crystal Plasticity Finite Element Method
CRSS	– Critical Resolved Shear Stress
CYS	– Compression Yield Stress
DIC	– Digital Image Correlation
EBSD	– Electron Backscatter Diffraction
EBSP	– Electron Backscatter Diffraction Pattern
FCC	– Face Centred Cubic
FIB	– Focused Ion Beam
GIS	– Gas Injection System
HCP	– Hexagonal Close-Packed
HEM	– Homogeneous Equivalent Medium
HR-EBSD	– High-Resolution Electron Backscatter Diffraction
LD	– Loading Direction
ND	– Normal Direction
PTR	– Predominant Twin Reorientation
RD	– Rolling Direction
RE	– Rare Earth metals
ROI	– Region of Interest
RSS	– Resolved Shear Stress
RVE	– Representative Volume Element
SEM	– Scanning Electron Microscope/Microscopy
SF	– Schmid Factor
SPD	– Severe Plastic Deformation
TD	– Transverse Direction

TDT – Twinning-Detwinning

TYS – Tension Yield Stress

VPSC – Visco-Plastic Self-Consistent

WAXS –Wide-Angle X-Ray Scattering

XRD – X-Ray Diffraction

1 Introduction

1.1 Magnesium and Magnesium Alloys

With the ever-increasing demand for reducing the environmental impact of technology, magnesium and its alloys have come into the spotlight as ultra lightweight metallic structural materials [1-3]. Table 1.1 lists the density for commonly used structural materials. Magnesium has density of 1.74 g/cm^3 , making it by far the lightest structural metal. The use of lighter material reduces energy consumption and CO_2 emissions in transportation. Thus, replacing heavier metals with magnesium alloys in transport, packaging and structural engineering can help to address the environmental issue. Magnesium alloys have relatively high specific stiffness and specific strength [2, 4] as illustrated in Figure 1.1. Therefore, magnesium and its alloys offer a favourable choice as energy-efficient materials.

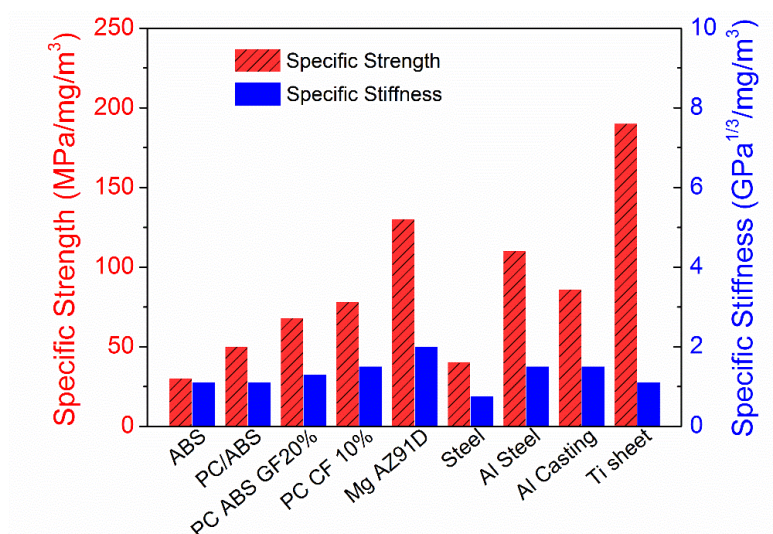


Figure 1.1 Specific strength and specific stiffness of selected structural materials (data extracted from Ref. [4]).

In terms of abundance, magnesium is the 8th most abundant element in the earth crust (2.7% by weight) [5] and comprises 0.13% (or 1.1 kg/m^3) in seawater [1, 6]. It is only found in compounds, such as dolomite ($\text{MgCO}_3 \cdot \text{CaCO}_3$), magnesite (MgCO_3) or carnallite ($\text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in nature [1].

Compared to pure magnesium, magnesium alloys have altered properties compared to pure magnesium due to the addition of various alloying elements that produce different effects. For instance, the most commonly used alloying element aluminium leads to the rise of strength and hardness. Zinc, one main alloying element in Mg AZ31B, is normally used together with aluminium to enhance strength without decreasing ductility and together with nickel and iron impurities to increase the corrosion resistance [6]. Rare earth metals (RE) can improve the high temperature strength, corrosion resistance and creep resistance, but they are primarily used for advanced alloys due to their high cost [6]. The effects of other alloying elements can be found in Ref. [2].

Table 1.1 Density of popular structural materials [2]

Materials	Density (g/cm ³)
Structural polymers	1.0 - 1.7
Magnesium	1.74
Aluminium	2.71
Titanium	4.51
Steel (cast iron)	7.2

Table 1.2 is a list of the main alloying elements and the corresponding abbreviation letters in the designation of magnesium alloys in a universally used alloy classification system introduced by the American Society for Testing and Materials (ASTM). The nomenclature of a magnesium alloy has three parts. Part 1 is the designated letters (refer to Table 1.2) of the two major alloying elements. The two letters are arranged alphabetically if the two alloying elements have the same composition fraction (wt.%). The second part is the respective rounded composition fractions (in wt.%) of the two alloying elements. Part 3 distinguishes between different alloys with the same percentages of the two major alloying elements using a letter of the alphabet assigned in the same order as the registration of the alloy with ASTM. It shows the stage of the development of this alloy and, in most cases, the degree of purity [1]. For example, the material used in this

thesis is magnesium AZ31B which is nominal Mg-3Al-1Zn (composition listed in Table 1.3) and is the second alloy registered with ASTM among other alloys with the same composition.

Table 1.2 Main alloying elements and their abbreviation letters in ASTM designation system of magnesium [2, 4, 6]

Alloying Element	Abbreviation Letter
Aluminium	A
Beryllium	B
Copper	C
Cadmium	D
RE	E
Iron	F
Thorium	H
Zirconium	K
Lithium	L
Manganese	M
Nickel	N
Silver	Q
Silicon	S
Tin	T
Yttrium	W
Zinc	Z

Table 1.3 Chemical Composition of Mg AZ31B

Element	Mg	Al	Zn	C	O	Mn
Content (wt.-%)	88.37	2.4	0.85	7.5	1.07	0.98

Fabrication conditions can be specified by adding temper designation after the three parts mentioned above with a dash in between. Table 1.4 lists the general divisions of temper designations for magnesium alloys from ASTM [7]. See Ref. [2] for subdivisions of ‘H’ and ‘T’.

A range of casting processes can be employed to produce magnesium alloys, such as die casting, sand casting and permanent mould casting. Mg AZ91 is the most widely used

magnesium die-casting alloy, thanks to its high castability [1]. Table 1.5 displays the characteristics of the most important die-casting alloy groups.

Table 1.4 General divisions of temper designation for magnesium alloys [2]

F	As fabricated
O	Annealed, recrystallized (wrought products only)
H	Strain hardened
T	Thermally treated to produce stable tempers other than F, O or H
W	Solution heat treated (unstable temper)

Table 1.5 Characteristics of the most important groups of die-casting alloys [1]

Alloy Group	Characteristics
AZ alloys	- relatively good properties at room temperature - low creep resistance and heat resistance
AM alloys	- improved ductility due to a lower Al percentage and the elimination of Zn - limited castability at room temperature
AS alloys	- considerably higher creep and heat resistance via Mg-RE precipitations - only casting is possible - limited castability

The usage of wrought magnesium alloys is more limited because of their poor cold workability compared to casting ones. The most commonly used wrought alloys belong to Mg/Al series, which are AZ31 (extrusion), AZ61 (extrusion) and AZ80 (forging) [8, 9]. The wrought magnesium AZ31B alloy used in this thesis features relatively good strength, ductility and corrosion resistance at room temperature, excellent weldability and high thermal and electrical conductivity (see Ref. [2] for more mechanical properties).

1.2 Applications

The favourable characteristics of magnesium and its alloys have led to a wide range of applications [10]. Some examples are listed below and illustrated in Figure 1.2.

1) Automotive application

Magnesium parts started to be used in racing cars and commercial vehicles since the 1920s and 1930s, respectively [2]. At present, major car manufacturers worldwide are

using magnesium alloys for parts such as gearbox housing (Figure 1.2(a)) [1], steering wheels (Figure 1.2(b)) [4], fuel tank cover, seat frame, air bag housing and steering column housing [11].

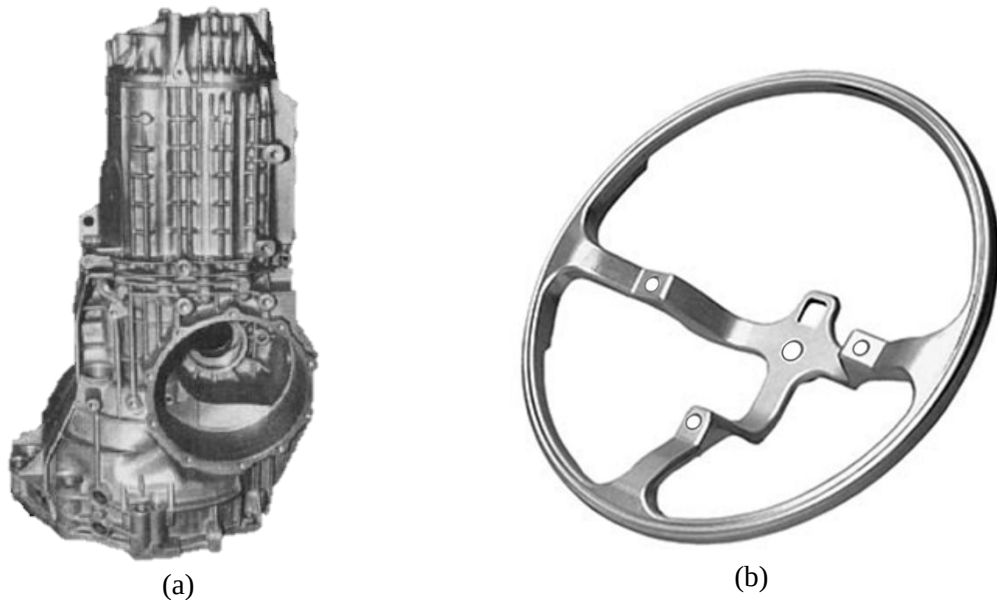


Figure 1.2 Applications of magnesium alloys: (a) gear box housing [12]; (b) steering wheel.

2) Aerospace application

Magnesium alloys have a long history of being used in the aerospace industry as part of some major components, such as engines, helicopter transmission casings and thrust reverser [2].

3) Medical applications

Magnesium alloys have drawn extensive attention as orthopaedic load-bearing implant materials [13-16] due to their biodegradable and biocompatible characteristics. A second surgery for implant removal can be avoided, as magnesium implants can dissolve in a biological environment [17] and no toxic ions are introduced, as the human body naturally has this element. In addition, the elastic modulus of magnesium (41-45 GPa) is very close to that of the bone, which can prevent stress shielding effect [13]. The key issue currently is that magnesium alloy corrosion rate in the human body is too high to match tissue healing rate [16].

4) Electronic applications

Thanks to the fact that magnesium alloys can be formed into complex shapes and demonstrate good heat transfer properties, they are being used for parts of electronic devices, such as mobile phone housing and hard drive reader arms [18].

1.3 Motivation

Extensive attention has been paid to improving the mechanical properties of wrought magnesium alloys and, in particular, their poor ductility [19] arising from the lack of independent deformation slip systems at room temperature due to the Hexagonal Close-packed (HCP) crystal structure [20]. Twinning (and detwinning), the key deformation mechanisms for magnesium, can accommodate plastic strain particularly along the *c*-axis (more details in section 2.1.1), which other easy slip systems fail to do. Twinning has a significant impact on the microstructure by causing large lattice rotation and forming subdivided grains in the twinned region, which increases the amount of grain boundaries and leads to grain refinement [20, 21]. It is well known that the mechanical properties of magnesium alloys are strongly affected by the microstructure (grain size and grain orientation) [22-27]. Therefore, twinning can fundamentally influence many aspects of their mechanical behaviour, such as strain hardening [28, 29], texture evolution [30, 31], formability [21, 32] and fatigue behaviour [33, 34].

Twinning-detwinning (TDT) is being widely studied from various perspectives both experimentally and numerically [35-40]. A detailed literature review can be found in section 2.1.2 for experimental work and in section 2.3.2 for simulation work. It is clear that more effort is needed to improve the limited understanding of the mechanism of the TDT process.

This thesis is mainly aimed to push forward the understanding of the mechanism of the TDT process by employing a combination of powder X-ray diffraction (XRD) and

electron backscatter diffraction (EBSD) techniques, on the one hand, and Crystal Plasticity Finite Element Method (CPFEM) simulation on the other. Two statuses of magnesium AZ31B alloy were studied: as-received wrought and Constrained Groove Pressing (CGP) processed. In particular, the effects of the TDT process on the deformation asymmetry and lattice rotation were investigated in greater depth. Multiple experimental approaches using XRD and EBSD were employed to achieve a more comprehensive examination. Additionally, attention was paid to the change of the twin volume fraction between different loading cycles and to the conditions for activating twinning and detwinning, respectively. An advanced Crystal Plasticity Finite Element (CPFE) model was developed specially in this study in order to obtain a faithful model of twinning and detwinning deformation. With a satisfactory validation of the model against *in situ* compression-tension XRD experimental observations reported in this thesis, the model was utilised to explore the non-Schmid behaviour during twinning and, specially, detwinning. Aside from the experimental and numerical work, method development was sought to improve and widen the usage of digital image correlation (DIC) in strain evaluation techniques, including Focused Ion Beam – Digital Image Correlation (FIB-DIC) micro-ring-core residual stress evaluation and XRD strain mapping. Since intra-granular and inter-granular strains and stresses are directly affected by the TDT process [37], advanced strain evaluation techniques can provide insights into the average and spatially resolved (local) influence of the TDT process on the internal stress-strain state of the material, and its subsequent deformation behaviour.

1.4 Thesis Structure

There are 8 chapters in this thesis. Following Chapter 1 containing the introduction and Chapter 2 containing the scientific background and literature review, Chapter 3

explains the details of all experimental techniques and Chapter 4 describes the framework of crystal plasticity simulation used in the thesis.

Chapter 5, Chapter 6 and Chapter 7 are the three main parts for reporting the results and their discussion of the DIC technique development, experimental study and numerical work, respectively.

Chapter 5 focuses on the development of DIC for two strain evaluation techniques – FIB-DIC ring-core milling and XRD. Firstly, Gas Injection System (GIS) in the Scanning Electron Microscopy (SEM) vacuum chamber was utilised to deposit platinum nano-dots at the sample surface subjected to FIB ring-core milling and SEM imaging. The objective is to increase the surface contrast to facilitate image correlation and, hence, the accuracy of DIC analysis. Secondly, XRD-DIC was proposed to utilise DIC for tracking the radial shift of Debye-Scherrer rings on the powder XRD two-dimensional (2D) patterns, which is directly linked to elastic lattice strain. By omitting the conventional multi-step conversion from 2D pattern to one-dimensional (1D) profile, XRD-DIC is aimed to minimise the error source with reduced procedures and to increase the efficiency of obtaining full in-plane strains, which requires only one DIC analysis, compared to multiple analyses for the conventional approach.

Chapter 6 explores the influence of the TDT process on deformation asymmetry and lattice rotation of as received wrought and/or CGP processed materials via *in/ex situ* XRD and *in situ* EBSD experiments. In particular, the first part investigates the difference in plastic behaviour of grains with different orientations via *ex situ* XRD on the central line of a bent bar of wrought material, which reveals the effect of twinning on the deformation asymmetry. The second part is concentrated on the observation of lattice rotation indirectly through diffraction peak intensity change during *in situ* XRD and directly through *in situ* EBSD, which informs the beginning and the end of twinning and detwinning, respectively.

Comparison of twin volume fraction and activation of twinning and detwinning was made between XRD and EBSD experiments. Additionally, different cycles were compared to shed light on the different behaviour of the first cycle.

Chapter 7 is devoted to multi-scale CPFE modelling. First, details are elucidated regarding implementing the detwinning system into crystal plasticity constitutive framework alongside with the finite element setup. The second part concerns the calibration of the starting texture and some newly introduced parameters for detwinning against the *in situ* compression-tension XRD experiment and the validation of the model through examining the macroscopic response (stress-strain curve). The final part reports the simulation results from three aspects: (a) activation of four slip systems and, particularly, twinning and detwinning systems; (b) TDT-induced rotation; (c) non-Schmid behaviour during the TDT process.

Lastly, Chapter 8 concludes the main contributions of this thesis and discusses some limits encountered in the experimental and the numerical work. Additionally, possible future work is proposed including further applications of the XRD-DIC method and more simulation work to compare with some of the current experiments in the thesis.

2 Scientific Background

This chapter includes the scientific background and the literature review. The first part is about the basics of magnesium with HCP structure as well as the plastic deformation mechanism of magnesium and its alloys. Secondly, the measurement of residual stresses will be discussed by specifically focusing on XRD and FIB-DIC techniques. Aside from experiment, the general background of crystal plasticity simulation is introduced in the third part.

2.1 Plastic Deformation Mechanism of Magnesium Alloys

2.1.1 Magnesium as HCP Metal

A crystal is a solid material in which the arrangement of atoms is regular [41]. The arrangement can be described by choosing a small unit with a certain arrangement of atoms and bonds so that the crystal can be obtained by repeating the unit to fill all space. The unit chosen is called unit cell. Therefore, only describing the unit cell is sufficient to describe the crystal symmetry and the crystal structure [41]. A unit cell represented by lattice parameters, such as the lengths of the principal axes and the angles between them. Magnesium alloy AZ31B has HCP crystal structure with the unit cell shown in Figure 2.1. It features a hexad symmetry with the lattice parameters $a_1=a_2$, $a_3 \neq c$ and $\alpha=\beta=90^\circ$, $\gamma=120^\circ$ for its hexagonal unit cell. Close packing is considered as a way of packing spheres with the same size together and stacking atomic planes with the highest efficiency. For HCP structure, plane B with 3 atoms lies in the middle of plane A with 6 atoms, giving the stacking series of ‘...ABABABAB...’.

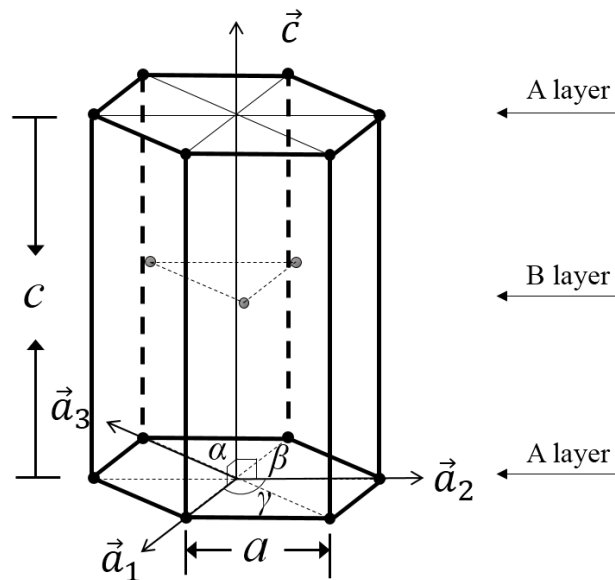


Figure 2.1 Unit cell of HCP crystal structure.

Magnesium has the c/a ratio of 1.623 [42] which is very close to the ideal value 1.633 [43]. Bravais-Miller indices can be used to describe directions and planes in HCP lattice

[44]. The four-index ($h\ k\ i\ l$) notation scheme is related to three basal vectors $\mathbf{a}_1$, $\mathbf{a}_2$, $\mathbf{a}_3$ and the vertical axis $\mathbf{c}$ (Figure 2.1), and obeys the constraint in Equation (2.1):

$$h + k + i = 0 \quad (2.1)$$

For convenience, ($h\ k\ i\ l$) can be shortened as ($h\ k.\ l$) and a period here is used to replace the third redundant index $\mathbf{a}_3$. Finding a vector with four-index coordinates in a unit cell can start with determining the vector in the basal plane represented by the first three indexes [45]. Vectors $\langle \bar{1}010 \rangle$ and $\langle 11\bar{2}0 \rangle$ are given as examples in Figure 2.2. The dashed grey lines are parallel to either one of the three coordinate axes to serve as guidelines when counting coordinates. To find $\langle 11\bar{2}0 \rangle$, we move one grid from the origin (centre of the hexagon) along the direction of $\mathbf{a}_1$, then move one grid along the direction of $\mathbf{a}_2$ followed by another two grids opposite to the direction of $\mathbf{a}_3$. Eventually the vector $\langle 11\bar{2}0 \rangle$ is from the origin to the end of this ‘journey’. The ‘journey’ is shown as three dashed yellow arrows and the final vector as solid blue arrow. The vector in the basal plane added by the purely vertical vector represented by the forth index is the final vector wanted.

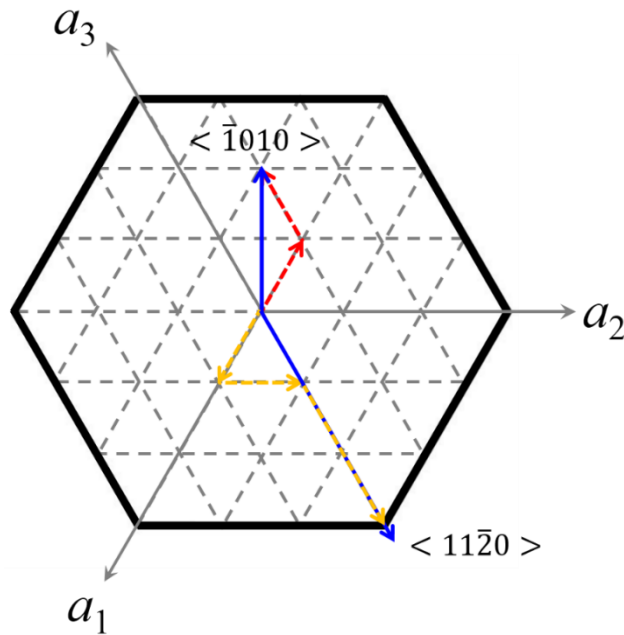


Figure 2.2 Identifying vector on the basal plane with 4-index coordinates in an HCP unit cell.

Magnesium alloys suffer from poor ductility at ambient temperature [6] due to a lack of independent slip systems. Critical resolved shear stress (CRSS) represents the critical value of shear stress required for a deformation system to be activated on a given crystallographic plane [46, 47]. Basal $\langle a \rangle$ slip has the lowest CRSS and is the primary slip system at ambient temperature. Moreover, other slip systems observed include $\langle a \rangle$ slip on non-basal planes, such as prismatic $(1\bar{1}00)$ plane [48, 49] and pyramidal $(1\bar{1}01)$ plane [50, 51] (shown in Figure 2.3) [52]. Non-basal slip systems were observed only at elevated temperatures ($>180^\circ\text{C}$) [52]. All the slip systems mentioned above can provide four independent slip systems, which is insufficient to satisfy the von Mises criterion that five independent deformation systems are required for homogeneous and general deformation [36, 52, 53]. In addition, none of them can accommodate strain along c -axis. Therefore, mechanical twinning plays a crucial role in the plastic deformation of magnesium alloys [52].

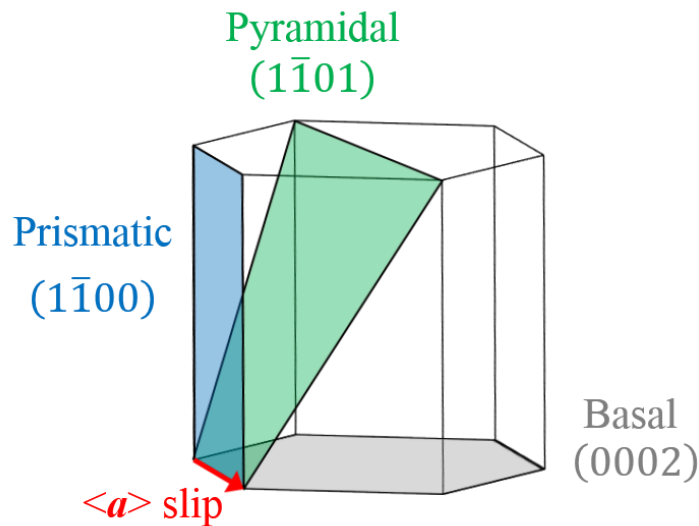


Figure 2.3 $\langle a \rangle$ slip systems observed in magnesium alloys including basal slip, prismatic slip and pyramidal slip.

2.1.2 Twinning and Detwinning

Twinning acts as an additional deformation mode that can accommodate inelastic tensile strain along the c -axis. $(10\bar{1}2) < \bar{1}011 >$ extension twin system is easily

activated in magnesium AZ31B alloy at room temperature [54, 55] under tension parallel or compression perpendicular to c -axis [56, 57]. $(10\bar{1}2)$ is the twinning plane and $\langle \bar{1}011 \rangle$ is the twinning direction. Figure 2.4 is the schematic illustration of $(10\bar{1}2) \langle \bar{1}011 \rangle$ extension twin where the blue plane is the twinning plane and the white arrow shows the twinning direction.

Twinning and detwinning are considered to be activated by resolved shear stress (RSS) similar to slip [58]. However, unlike slip which involves incremental shear and gradual local change of lattice orientation, twinning (also detwinning) induces an abrupt large change in the lattice orientation in the twinned region [59, 60]. $(10\bar{1}2) \langle \bar{1}011 \rangle$ extension twin features a lattice reorientation angle of 86.3° [61, 62] leading to the characteristic shear strain of 0.129 [40, 63]. As shown in Figure 2.4, the twinned region of the crystal (in red lines) assumes an arrangement that represents a mirror symmetry of the remaining parent matrix with respect to the twinning plane [64] and undergoes reverse reorientation during detwinning. Moreover, twinning can only accommodate a limited amount of strain that is proportional to the fraction of the twinned volume. In the case of $(10\bar{1}2) \langle \bar{1}011 \rangle$ extension twin, it can accommodate 0.065 in tensile strain at maximum [52, 65].

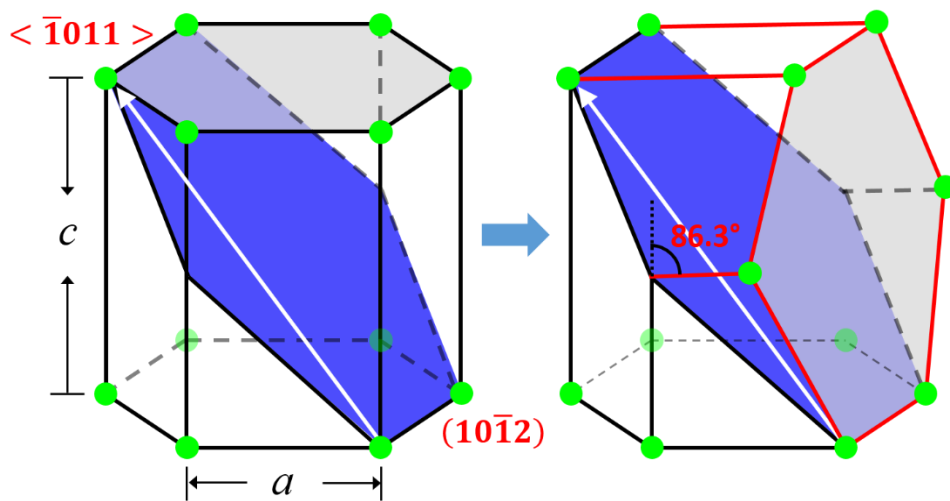


Figure 2.4 Schematic demonstration of $(10\bar{1}2) \langle \bar{1}011 \rangle$ extension twin.

The whole twinning process involves four stages: nucleation, formation, growth and propagation [60]. In the first stage, nucleation leads to embryo at the twin zone where dislocations for twinning accumulate, which will possibly become a twin. Formation of twins in the second stage is a result of twinning-related dislocations reaching a critical value. In the third stage, a twin that has formed in a small local area in a grain grows across the whole grain into a lenticular needle shape. Lastly, twins thicken through propagation sideways into the rest of the grain at a near-sonic speed [66].

Detwinning is the reverse process of twinning in terms of causing lattice reorientation and accommodating strain. Nevertheless, it is easier to activate detwinning compared to twinning, meaning that detwinning has a lower CRSS [35, 36]. This is mainly attributed to two reasons. Firstly, nucleation process is already finished during twinning, so it is skipped during detwinning [35]. Additionally, backstress generated during twinning can act as part of the driving force that facilitates detwinning [36].

It should be recognised that twinning and detwinning are polar processes meaning that they only allow one-direction simple shear instead of both-direction shear induced by slip (forward and backward) [44, 52, 63, 67, 68]. In the case of magnesium, twinning is activated under tension along the *c*-axis not compression. Consequently, different response is expected for grains with different orientation under the same loading [44, 67]. This is the main reason for the widely observed tension-compression yield asymmetry demonstrated by wrought magnesium alloys (see more details in section 2.1.3) [36].

Currently, experimental work regarding the TDT process in magnesium alloys mainly concentrates on two perspectives: 1) observing the TDT process and its influence on mechanical properties to understand its mechanism; 2) improving the mechanical properties via controlling the TDT process.

Wu *et al.* [37] studied the TDT process during cyclic loading using *in situ* neutron diffraction and observed obvious accompanying internal stress relaxation and load redistribution. *In situ* three-dimensional (3D) XRD was utilised to reveal the variation of sub-micron strains across the twin boundary, which was further confirmed by theoretical calculation [69]. Twin-twin interactions were investigated in magnesium by Yu *et al.* [70] and, depending on the twin-twin boundary, three types of twin-twin structures were observed experimentally, while two types were obtained from the crystallographic theory. Yin *et al.* [71] discovered that twinning led to the prolific lamellar structure at crack initiation and in the crack stable propagation zone, and its formation mechanism was proposed based on the TDT process. The role of the initial twin was revealed to deteriorate the fatigue resistance of Mg AZ31, since detwinning can fully accommodate compressive deformation, which considerably reduced compressive flow stress and increased mean stress [34]. The difference in activation sequences of TDT systems was shown to result in different low-cycle behaviours between through-thickness and in-plane loadings in Mg AZ31B alloy [72]. Knezevic *et al.* [30] described the effect of twinning on anisotropic strain-hardening rates, tension-compression yield asymmetry and texture evolution. Additionally, the hypothesis that thin contraction twins are effective in strain hardening was successful in explaining the observations in the experiment. Three stages were characterised by Sarker *et al.* [73] to describe the TDT process of Mg AM30 alloy: stage A featured twinning and a dropping strain-hardening rate; stage B saw the activation of detwinning due to twin-dislocation interactions with an ascending strain-hardening rate; stage C experienced a descending strain-hardening rate because of less resistance for dislocation slip from fewer twins.

In the last few years, equipped with more knowledge about the TDT process, some efforts have been made to utilise pre-twinning [21] for the purpose of improving

mechanical properties and performance of magnesium alloys. Twin boundaries introduced by pre-twinning exhibited the capability of strengthening Mg AZ31 alloy via grain refinement [74]. Song *et al.* [75] demonstrated that pre-twinning in combination with aging treatments could effectively enhance the yield strength of Mg ZK60 alloy. Xin *et al.* [32] discovered that the rolling capacity at 300 °C of rolled Mg AZ31 plates was significantly increased by pre-twinning. Park *et al.* [76] showed that roughly 24% volume fraction of pre-twinning could improve the stretch formability of rolled Mg AZ31 alloy by approximately 65% at room temperature. This improvement was attributed to the activation of basal slip and detwinning in the twinned region that could provide effective accommodation of a thickness-direction strain. Pre-compression was utilised by Hong *et al.* [33] to control the TDT characteristics of rolled Mg AZ31 alloy in order to reduce the tensile mean stress during fatigue deformation, which resulted in a jump in the fatigue resistance.

2.1.3 Deformation Asymmetry

Wrought magnesium alloys commonly show a tension-compression asymmetry [6, 68, 77-79], meaning mechanical responses (e.g. yield stress [68, 80]) are different under tensile and compressive loading. The extent of tension-compression yield asymmetry is estimated by the fraction between the compression yield stress (CYS) and the tension yield stress (TYS) along the rolling direction (RD) [68]. Wrought magnesium alloys feature a strong rod texture where most of the grains have their basal pole (*c*-axis) perpendicular to the RD [9, 81, 82]. $(10\bar{1}2) < \bar{1}011 >$ extension twin can only happen when RSS along the $< \bar{1}011 >$ direction on the $(10\bar{1}2)$ plane is larger than the CRSS of this twinning system under tension along *c*-axis or compression perpendicular to *c*-axis. In combination with rod texture, it is much easier to activate extension twin under compression along the

RD or along the transverse direction (TD) but not under tension along the same directions. Therefore, wrought magnesium alloys normally display a lower CYS than TYS [6, 83, 84].

Loading direction (LD) and grain size have significant influence on tension-compression asymmetry [68]. It was examined by Yin *et al.* that the CYS/TYS ratio was 1.02 when the LD was 45 ° relative to the RD, meaning there was nearly no asymmetry [68]. However, loading along or perpendicular to the RD led to strong asymmetry. Barnett *et al.* discovered that grain size could dramatically change twin fraction, strain hardening rate and CYS [27]. Yin *et al.* investigated the influence of grain size on asymmetry of as-extruded, as-cast and equal channel angular pressed magnesium AZ31B samples and observed that asymmetry can be reduced via refining grain size or weakening texture [85]. Moreover, 0.8 μm was estimated to be the critical grain size below which there is no asymmetry. Various efforts have been made to reduce asymmetry [86, 87]. The relationship between tension-compression asymmetry in Mg-8Al-0.5Zn and precipitation state was studied by Jain *et al.* [86] and it was illustrated that the existence of precipitates could more significantly reduce asymmetry than solution treatment. Heat treatment was also found to be far more effective than solution treatment by Lv *et al.* [87]. Rate sensitivity of tension-compression asymmetry in AZ31B was investigated by Kurukuri *et al.* [88]. The sample under compression along the normal direction (ND) exhibited conventional positive sensitivity to strain rate while no sensitivity was shown during tension until twinning was finished and slip started to be dominant.

2.2 Residual Stress Measurement

The definition of residual stresses is the self-equilibrating stresses that remain in a material after manufacturing processes when any external force or thermal gradient is removed [89, 90].

Residual stresses can result from various processes, such as casting, forming (e.g. rolling, extrusion, forging), cutting (e.g. grinding, machining, turning), joining (e.g. soldering, welding), external treatment (e.g. shock laser treatment, shot peening) and thermal treatment [91-93]. Regardless of how different the processes are, the origin of residual stresses can be considered to follow the same manner. An inelastic deformation (normally referred to as *eigenstrain* [94-96]) is induced by those processes which causes accommodating elastic strains to rise in the surroundings to rebuild equilibrium and continuity [92]. Hence, residual elastic stresses are introduced.

It is critically important to develop accurate and reliable methods for residual stress measurement in order to evaluate and predict the boundary between safe deformation behaviour and failure of engineering components [97, 98]. Residual stresses are classified into three types based on the scale on which variation of stresses is shown: type I, type II and type III [90, 99]. Figure 2.5 demonstrates the separation of these three types of residual stresses. Type I residual stresses are macroscopic residual stresses which are of the concern of engineers. Type II and type III are both microscopic residual stresses and they are respectively inter-granular and intra-granular residual stresses. In other words, type II residual stresses vary between different grains while type III vary within a grain. The total residual stress is the sum of the residual stresses of all the three types. The knowledge of the three types of residual stresses can allow better understanding of mechanical failure which usually begins on a grain level [99].

Residual stresses have a significant influence on the mechanical properties of components and materials, such as fatigue behaviours [98]. Generally, compressive residual stresses are favoured at the surface of the material to increase the mechanical performance by preventing crack formation and propagation [91, 99].

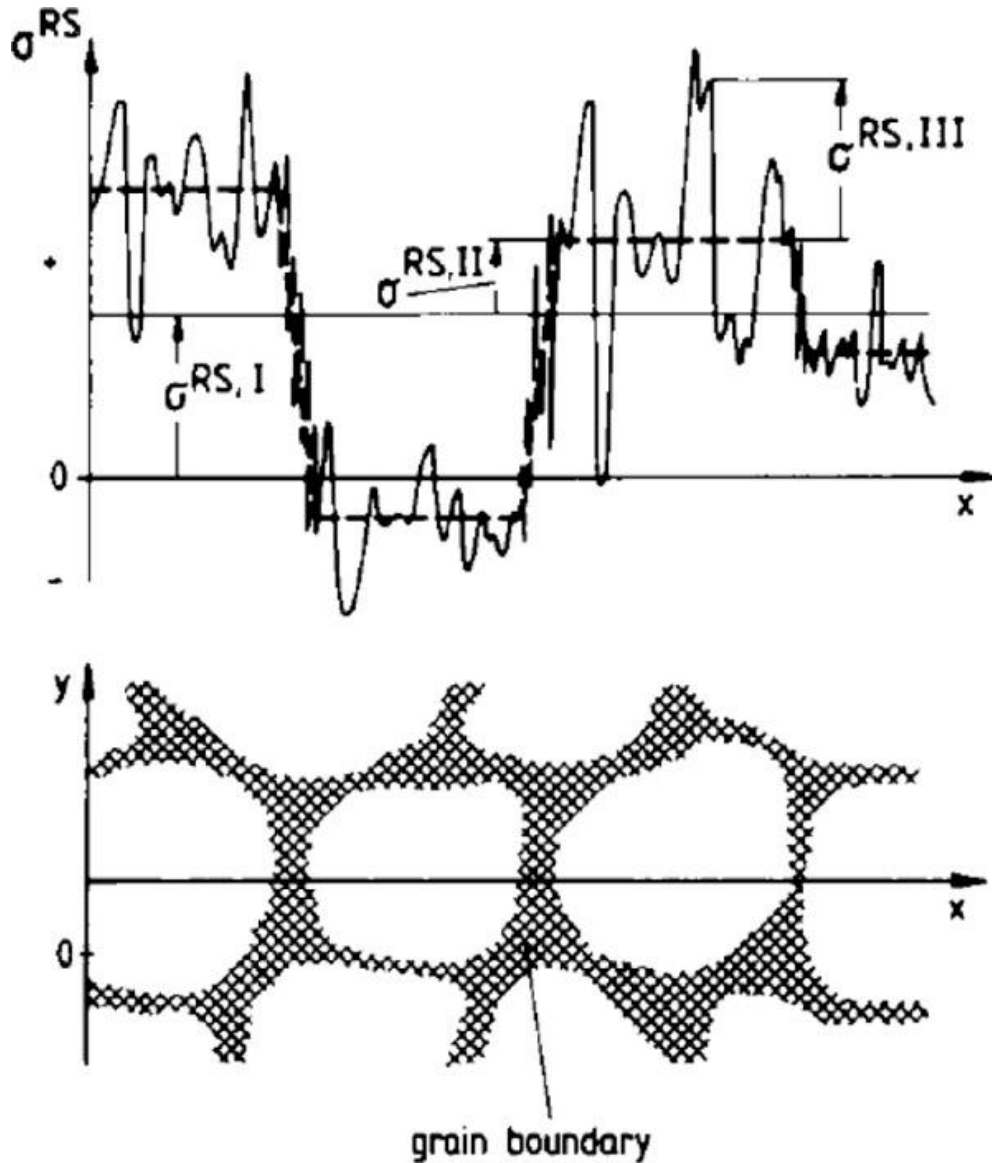


Figure 2.5 Distribution of total residual stresses along several neighbouring grains with variation in type I, type II and type III residual stresses (RS stands for residual stress) [90].

2.2.1 Classification of Measurement Techniques

Based on the manner of material removal, there are three categories of techniques for measuring residual stresses: non-destructive, semi-destructive and destructive [92, 97].

Non-destructive methods evaluate residual stresses through qualifying small variations in structural or physical parameters without bringing much interference to the material itself [97]. For instance, diffraction techniques evaluate the change of interplanar atomic lattice spacing, which leads to elastic strain measurement. Ultra-high resolution (tens of nano-meters) has proven to be achievable using those methods [100]. There are a

wide range of techniques in this category including high-resolution EBSD (HR-EBSD) [101, 102], diffraction methods (X-ray [103, 104] and neutron [105, 106]) , Raman spectroscopy [107] and high-resolution optical techniques [108, 109]. All the methods mentioned above can provide relative elastic strain/stress values in comparison with a reference value representing strain-free status. Therefore, the limitation of measurement accuracy lies in the precision of determining reliable reference values.

Semi-destructive methods rely on measuring the localised stress relief during hole drilling and ring coring [110, 111] to quantify the magnitude of residual stress on the sample surface at a specific location [97, 112]. It involves only minimal interference with the overall structure. Other examples of semi-destructive methods are slitting [113] and layer removal [114]. Strain change during hole drilling is typically evaluated by strain gauges or DIC and it can be used to allow back-calculation of stresses present before drilling. Conventional semi-destructive methods can resolve stress at a depth resolution of ~ 0.03 mm and a lateral resolution of ~ 1 mm [97]. The need to investigate microscopic residual stresses drove the exploration of finer tools to conduct material removal [115, 116]. The newly proposed FIB-DIC ring-core milling method [117, 118] employs nano-scale FIB for material removal, SEM for sample surface imaging and DIC for strain evaluation. Using FIB-DIC, material removal can be accurately defined in a micro-scale volume, which enables the evaluation of residual stress on the scale from several microns to sub-microns.

Destructive methods involve major material removal and introduce traction-free surfaces through sample sectioning which leads to stress redistribution and strain relief in the surrounding area in the material. Strain change quantified in the process together with simulation can be used to back-calculate the stresses existing before sectioning. Two

common techniques using this approach are slitting and contour method [113, 119, 120] and the usual resolution achieved is in the range of fractions of a millimetre [97].

Additionally, depending on the length scale interrogated, residual stress measurements may range from macroscopic down to mesoscopic and microscopic levels. The following two sections will be focused on two techniques that are of concern in this thesis: XRD and FIB-DIC ring-core methods.

2.2.2 XRD

XRD finds a wide range of applications in materials engineering [121-128]. It can be used for the quantification of phase composition and preferred grain orientation (i.e., texture). In addition, it offers a powerful non-destructive way to measure lattice strain in a variety of crystalline materials [129-132]. The relationship between residual stresses and lattice strains has been the subject of extensive study over the last century [133-135], which has led to the appreciation of the multi-scale nature of these phenomena [90] and the refinement of modelling tools that assist the conversion from XRD measurements to strain evaluation and residual stress calculation. The presence of residual strain changes the interplanar lattice spacing with respect to the strain-free state and affects the XRD pattern by causing a systematic shift of the peak centre. Hence, careful analysis of diffraction pattern changes can be used to quantify lattice strain [136].

One advantage of XRD is its non-destructive nature, which means no material removal is involved in the experiment. XRD is also eminently suitable for *in situ* strain measurement to observe and quantify the strain evolution during thermal and deformation processes. An additional advantage of the high-energy synchrotron XRD method is that it can penetrate the sample and provide information about the average bulk strain, as opposed to other methods, such as strain gauge analysis [137] or laboratory XRD [138] which can only evaluate strains at the sample surface.

It is convenient to distinguish between synchrotron-based and conventional laboratory XRD due to the vast difference in flux and resolution. Although lab-based X-ray equipment is more commonly available, it has significant limitations in terms of penetrable sample thickness, spatial resolution and data collection time. High energy synchrotron X-ray transmission configuration relies on high flux, brightness, energy and penetration ability, making it possible to use micro-scale beam sizes to achieve better spatial resolution [139].

XRD is intensively used in this thesis for lattice strain measurement. Developing reliable and efficient methods for 2D XRD pattern interpretation is one of the key remaining challenges in the area of XRD strain analysis. Therefore, XRD-DIC is newly proposed in section 5.2 that employs DIC to track the radial shift of Debye-Scherrer rings on XRD patterns in order to measure the lattice strain.

2.2.3 FIB-DIC Ring-Core Milling

FIB-DIC ring-core milling is versatile and adaptable as a semi-destructive method for residual stress measurement on the surface of materials [92, 97, 115-117, 140, 141]. FIB is utilised to perform milling to remove material while SEM is adopted to record the sample surface during milling which results in a sequence of high-resolution SEM images. DIC analysis on the sequence of images provides displacement field which can be used to calculate strain relief. Multiple geometries have been proposed for FIB milling to meet specific needs or to measure a particular stress state [115], such as single micro-slot [142], double micro-slit [143], four-slot [144], micro-hole [145], ring-core [116] and H-bar [146], as shown in Figure 2.6. The current work focuses on ring-core geometry. More details of the principle of this method and milling geometries can be found in [97, 115, 147].

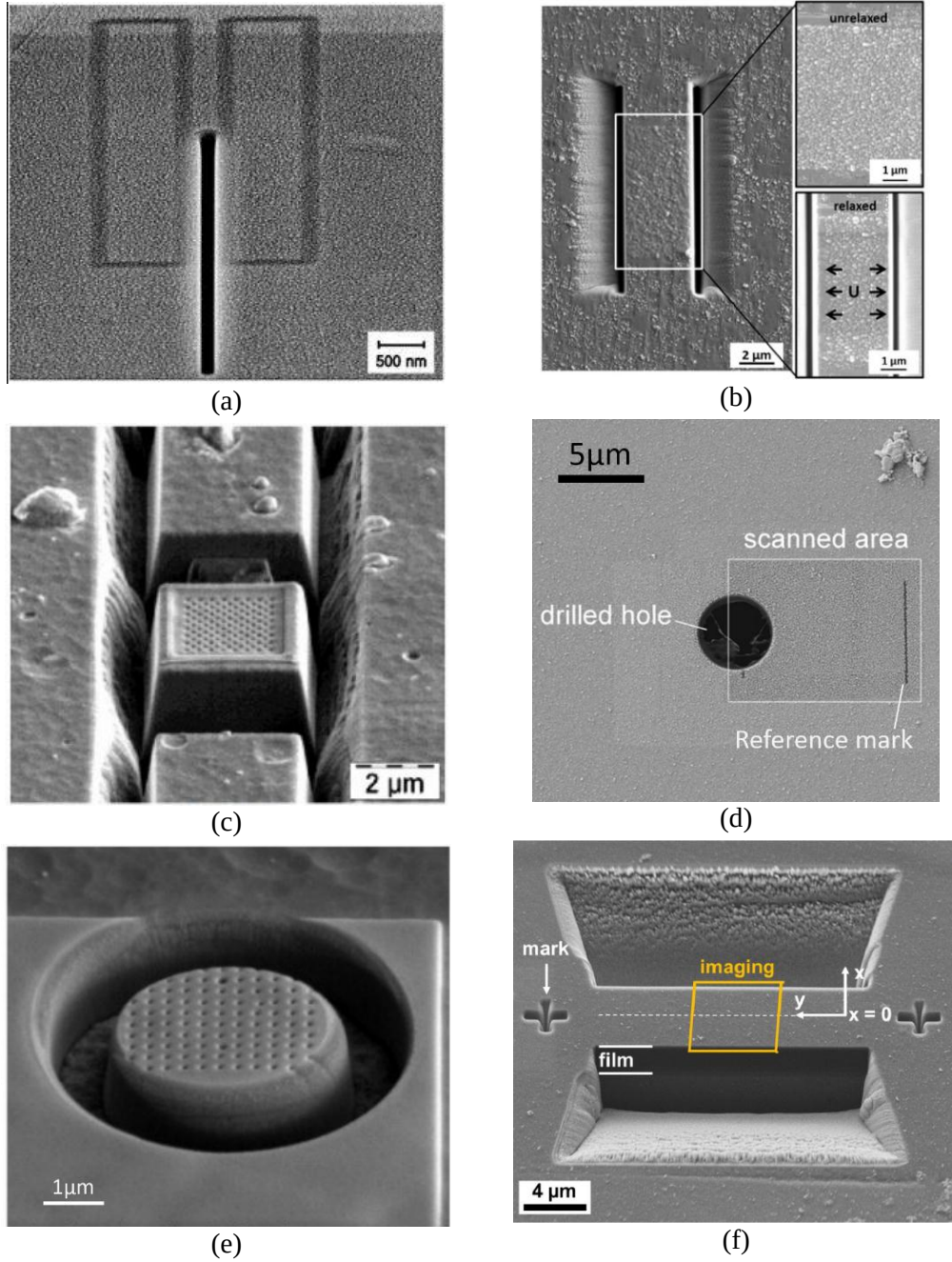


Figure 2.6 Geometries used for FIB milling in FIB-DIC method: (a) single micro-slot [142]; (b) double micro-slit [143]; (c) four-slot [144]; (d) micro-hole [145]; (e) ring-core [116]; (f) H-bar [146].

The FIB-DIC ring-core milling method enjoys various advantages. Firstly, it is able to provide evaluation of a full 2D in-plane stress tensor [115]. Additionally, an improved gauge volume resolution can be achieved compared to XRD which only gives average result of the whole gauge volume illuminated by X-ray. The feature of having an accurately defined gauge volume also allows spatially resolved residual stress analysis [115]. Since it measures microscopic strains, it has the potential of investigating type II and type III

residual stresses in a particular micro-scale area or even within a specific grain. Moreover, FIB-DIC provides absolute measurements via comparing the deformed surface with the stress-free one (achieved at the end of strain relief process), while XRD can only evaluate relative strains, which is highly limited by the reliability of the reference strain value used. In addition, FIB-DIC can be equally suitable to study non-crystalline materials, such as amorphous systems. For comparison, XRD cannot conventionally investigate materials without crystalline structure or materials with too severe lattice distortion [147].

However, FIB-DIC ring-core milling also suffers from some disadvantages. Aside from intrinsic limitations, the main error source is from imaging and sample surface preparation [147]. SEM images are required to be carefully focused and free from any distortion or drift to ensure a satisfying image correlation accuracy. Sample surface contrast is another key factor for the reliability of the DIC results. A random pattern with high contrast is crucial for DIC to achieve satisfactory accuracy during image correlation. Normally, this can be addressed by pre-depositing some pattern onto the surface using Pt or even sputter coating. Aside from having contrast, the sample surface should also be free from residual strain introduced during sample preparation, such as grinding and polishing. Therefore, techniques with gentle material removal are preferred to minimise the influence of machining. Electropolishing is optimal to achieve this [147]. In section 5.1, the effect of Pt deposition on DIC analysis will be studied and discussed in details.

2.3 Crystal Plasticity Modelling

2.3.1 Overview

Sophisticated and advanced equipment and effort-intensive sample preparation are usually required to experimentally study the micro-scale mechanical behaviour and the mechanism of plastic deformation, which is not always accessible and normally expensive

[59]. Hence, numerical simulation is of great interest as an approach to assist the experimental investigations.

Constitutive modelling is currently under the spotlight since it enjoys the capability of relatively accurately simulating macroscopic response and microscopic behaviour (e.g., texture evolution) of polycrystalline materials [148] while not having the disadvantage of extremely long calculation duration or limited simulation scale compared to other simulation methods, such as atomistic simulation [149-151] and dislocation dynamics [149, 152-154]. Constitutive models essentially define constitutive equations which provide the relationship between stresses and strains [155]. It is simply a linear relation (Hooke's law) when it comes to elastic deformation. In the case of inelastic deformation, however, it is a much more complicated matter.

Developing constitutive models to study the deformation of materials with HCP crystal structure is far more complex compared to that with Body Centred Cubic (BCC) or Face Centred Cubic (FCC) structures. The dominating deformation mechanism in BCC and FCC materials is normally slip, whereas that in HCP materials is both slip and twinning. In addition, the HCP crystallographic structure features low symmetry, leading to the existence of various types of slip systems. Moreover, the mechanical responses of grains in HCP materials show low symmetry and evident anisotropy, which leads to another demanding fact that the effect of grain interaction is too significant to neglect when simulating polycrystalline materials so detailed interaction between grains has to be taken into consideration [156].

There are three main crystal plasticity constitutive models of polycrystalline materials that have been developed and are discussed below.

1) Taylor Model

Taylor model [157] is also named ‘full constrain theory’ [60, 148, 156]. In this model all grains are assumed to experience the same strain which is equal to the macroscopic strain applied, leading to different stresses in different grains [60, 156]. Consequently, force equilibrium is not satisfied between neighbouring grains. In the meantime, grain interaction, the shape of the grains [158] and the kinematic coupling between grains are all ignored [148]. Hence, the rotation of one grain has no influence on the rotation of its neighbouring grains. More details and applications of this model can be found in literature [159-167].

2) Viscoplastic Self-Consistent Model

VPSC was initially introduced by Kröner [168] to deal with elastic deformation and later extended by Hutchinson [169] to viscoplastic cases [156]. VPSC models are more favourable when it comes to simulating large deformation in HCP polycrystalline materials [35, 52, 156, 170-175], especially after been developed to consider anisotropy [173, 176, 177]. The interaction of one grain with its neighbouring grains is included by having this grain to interact with a homogeneous equivalent medium (HEM) which effectively represents the average of the surrounding neighbours [60, 148, 156]. Different response of strain in different grains is allowed and decided between the grain and its HEM, which is different from the Taylor model. Meanwhile, the average response of all the grains is required to be the same as the imposed macroscopic one. Equilibrium is not completely satisfied between neighbours, neither is compatibility [164]. Moreover, similar to the Taylor model, grain shape is not considered and the rotation of one grain is not linked to the rotation of its surrounding grains [148].

3) Crystal Plasticity Finite Element Method

CPFEM has the most realistic grain interaction since geometry is allowed both in terms of the shape of the sample [148] and the shape of the grain [156] (as well as grain

shape change [178]). Each grain directly interacts with its neighbours and its rotation has direct effect on the rotation of the surrounding grains. In the finite element mesh, an element represents either a complete grain or part of a grain and at each integration point the constitutive model for single crystal is used to simulate the response of the elements individually [156]. Additionally, equilibrium and compatibility between grains are both satisfied at the same time in the weak form [60, 156, 164, 178-181]. Thanks to those features mentioned above, CPFEM enjoys the potential of modelling local behaviour and variation in deformation at grain level [182-184].

CPFEM finds applications in various aspects [185], such as grain interaction [186, 187], grain size effects [188], phase transformation [189, 190], texture evolution [191, 192] and recrystallization [193, 194]. Extensive use of CPFEM can be found for FCC and BCC materials [195-201]. For HCP materials, twinning is more significantly present as a plastic deformation mechanism compared to FCC and BCC materials. The mechanism of twinning such as its interaction with slip is not well understood [40, 150, 151, 202]. Therefore, CPFEM is currently being intensively developed for HCP materials and, particularly in the interest of this work, for magnesium and its alloys. A rate-independent non-hardening model for Mg AZ31B alloy including both slip and twinning was developed by Staroselsky and Anand [203, 204]. Through comparing experimental data with numerical results, the model revealed information about dominant activated slip and twinning systems at ambient temperature and calibrated some parameters for slip and twinning systems in single crystals. The effect of twinning deformation and consequent lattice rotation in pure magnesium was simulated by Mayama *et al.* and significant twinning-induced strength differential effect (yielding behaviour dependent on the loading direction [205, 206]) was demonstrated [207]. Grain shape change and texture evolution

in Mg AZ31B were investigated numerically by Prakash *et al.* with a model which recognised internal stresses and explored strain localization [178]. However, mechanisms of different cross-hardening were not differentiated. A similar model was introduced by Choi *et al.* for the case of deformation at high temperature (200 °C) where deformation mechanisms were activated in a different sequence compared to room temperature [208, 209].

2.3.2 Twinning and Detwinning in Crystal Plasticity Modelling

Twinning and detwinning have proven to be indispensable in plastic deformation of HCP materials in crystal plasticity models [30, 35, 38, 205, 207, 210, 211].

Twinning is usually introduced as an additional slip system since they have similar shearing modes. This is often referred to as pseudo-slip scheme and has been exploited by many investigators [203, 212-215]. In terms of texture evolution, implementing twinning and detwinning into continuum framework requires special consideration since a sudden lattice rotation of 86.3° (in the case of magnesium AZ31B) is introduced to the crystal lattice of the twinned region [216]. This will lead to too large of a number of crystal orientations in the twinned regions for simulation as demonstrated in Ref. [213]. Major breakthroughs regarding this problem were firstly made in the 1970s to the 1990s [213, 217, 218]. Van Houtte proposed a solution where the number of grain orientations stayed the same throughout the entire simulation [217]. From one deformation step to the next, one crystal orientation will be reorientated due to slip if it is not twinned or replaced by the new orientation of the activated twin. The replacement happens at *a priori* probability corresponding to the twin volume fraction that is calculated as the ratio of the twinning shear strain and its characteristic strain (0.129 for magnesium AZ31B). This approach, however, requires that the model must have a substantial number of crystals to ensure the statistical criterion is meaningful. Another problem is that it is possible that the orientation

of the most dominant twin system is not chosen to replace the previous one. To tackle the latter issue, Tome *et al.* introduced a scheme where the crystal will only be reorientated to the dominant twin orientation. This is the so called a predominant twin reorientation (PTR) scheme. Kalidindi [213] developed a constitutive model with efficient time integration in which all calculations were based on intermediate related configuration and the crystal orientations of untwinned or twinned regions were defined *a priori* using the initial crystal orientation. Staroselsky and Anand [203, 204] included ‘a grain boundary layer’ to surround each grain with a very small volume fraction in order to simulate grain boundary deformation. This is realised by adding a component of isotropic plasticity in the flow rule to account for the effects from grain boundary deformation. The model used in the thesis is based on the one developed by Ana *et al.* who extended Staroselsky and Anand’s model by differentiating self-hardening and cross-hardening mechanisms between slip and twinning and calibrating them individually against uniaxial compression experimental data [38, 40]. Furthermore, grain boundary deformation was not considered since it is not significant given the grain size of the material used.

Detwinning is not considered in the twinning models mentioned above which are mainly focused on twinning-induced lattice reorientation and hardening mechanisms. Given the indispensable role that detwinning plays in the mechanical properties of crystalline materials [36, 37, 219-221], it is necessary to understand and describe correctly its underlying mechanism. Alongside intensive experimental investigation in the recent decades [71, 220, 222-227], simulation models are being developed for modelling the detwinning process [35, 58, 211, 228-232]. Most of them utilise self-consistent crystal plasticity models [35, 58, 228-230] and choose to ignore the difference in the value of CRSS between twinning and detwinning [58, 211, 228, 229]. So far, to our knowledge, Proust *et al.* were the first to propose a formulation including both twinning and

detwinning that can be implemented into a crystal plasticity model for polycrystalline materials – the composite grain (CG) model [35, 171]. VPSC was the platform for the CG model. To model the process of detwinning, rather than introducing a separate detwinning system, the model [35] favoured the predominant twin system that had already twinned during loading by giving other twin systems in the twinned region higher CRSS values upon reloading. Even though the detwinning CRSS was practically smaller than twinning CRSS during simulation, the model failed to give a specific value for detwinning CRSS. In the meanwhile, some features and mechanisms of the CG model remained difficult to characterise [58]. Additionally, a nucleation model [233] had since been developed to replace the empirical model of twin nucleation and propagation used in the CG model. Wang *et al.* employed an elastic VPSC model to include TDT mechanisms for polycrystalline HCP materials [58, 229]. The TDT model treated a newly formed twin band (child) as a new grain and was validated against experimental data of magnesium AZ31B alloy under cyclic loading and under changed strain paths. Nevertheless, no additional parameters for detwinning were introduced into the model. As a result, the CRSS values for twinning and detwinning were not separated. Based on Wang *et al.*'s model, Qiao *et al.* [230] used the same CRSS values for twin growth and twin shrinkage (similar to partial detwinning) in elasto-viscoplastic self-consistent model, but discriminated between the stresses needed for twin initiation (56 MPa) and twin growth (32 MPa), which essentially supported different CRSS values for twinning and detwinning. CPFEM was developed to include twinning and detwinning by Hama *et al.* [211] in order to investigate the deformation response of commercial pure titanium Grade 1 sheet under various strain paths. Both $(10\bar{1}2)$ extension twin and $(11\bar{2}2)$ contraction twinning systems were considered alongside with another five slip systems. $(10\bar{1}2)$ extension detwinning was set to be activated when the positive sign of RSS was inverted to be negative. Similarly,

to activate $(11\bar{2}2)$ contraction detwinning, the sign of RSS needed to be inverted from negative to positive. Again, the CRSS of detwinning was not separately identified as no additional parameter was assigned for it.

3 Experimental Methods

This chapter lists all the experiments, including an overview of the experimental techniques, the detailed process and the data analysis procedures. There are two experimental techniques involved: *in/ex situ* XRD and *in situ* EBSD. Commercially available magnesium AZ31B alloy was used with two conditions: as-received wrought material with coarse grain (averagely $\sim 20\ \mu\text{m}$) and CGP processed material with fine grain (averagely $3\text{-}5\ \mu\text{m}$). Additionally, general information of DIC is given in this chapter to prepare for further development in Chapter 5.

3.1 Constrained Groove Pressing

CGP is a version of Severe Plastic Deformation (SPD). For convenience, if a sample is processed with CGP, it is referred to as CGPed sample in the following discussion.

A schematic explanation of a CGP sequence is shown in Figure 3.1. Note that the figure is only showing the cross-section of the sheet. Referring to Figure 3.1(a), the pitch is the period length of the die that equals to $4t$, where t is the equal unit horizontal length spacing. All inclined and horizontal surfaces have the same t . The lower die is fixed and the upper die is movable. The sample is placed between the dies and deformed to the shape of the dies at the first pressing (Figure 3.1(b)). At this stage, regions of the sample adjacent to the inclined parts of the dies are subjected to shear deformation with the shear strain of 1 and the nominal effective strain ε_{eff} of 0.58 when θ is 45° ; while the rest of the sample remains approximately undeformed. During the second pressing (Figure 3.1(c)), a pair of flat dies are used to perform straightening, which leads to reverse plastic deformation in the previously deformed regions, accommodating cumulative effective strain ε_{eff} of 1.16

[5]. Yet the remaining parts are still unaffected. The sample is then rotated by 180 °(Figure 3.1(d)), which causes a horizontal shift of the sample by t so that the previously unaffected parts are deformed in the following pressings, including groove pressing and straightening pressing (Figure 3.1(e), (f)). This leads to the effective strain of 1.16 distributed approximately uniformly over the entire sample.

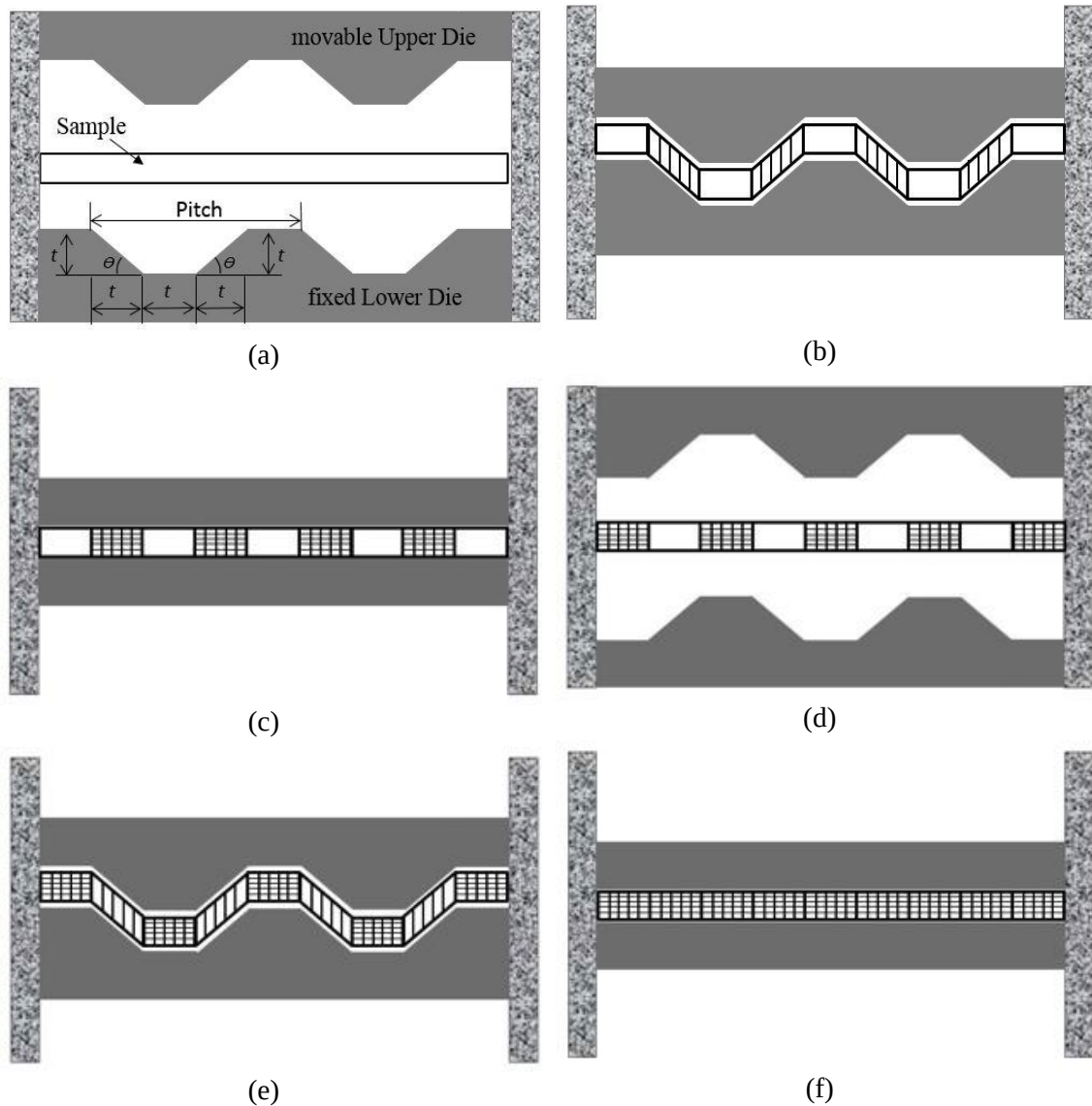


Figure 3.1 Schematic illustration of a CGP circle: (a) dimension of groove dies and setup of experiment; (b) groove pressing; (c) straightening pressing; (d) 180 °rotated sample; (e) groove pressing after rotation; (f) straightening pressing after rotation.

In summary, a complete cycle of CGP consists of four pressings (Figure 3.1(b), (c), (e) and (f)). The initial dimensions of the sample remain almost unaffected after one cycle. In the meanwhile, plastic strain is introduced and grains are refined. Dies can be heated up

to a certain temperature. Before each pressing the sample can be placed between the dies to allow uniform temperature in the sample [234].

To prepare the sample studied here, the dies were heated to a set temperature at the beginning of each cycle and maintained for 5 minutes to allow homogenization. Each cycle involved constrained groove pressings and straightening pressings. Before each subsequent pressing, the sample was placed between upper and lower dies to allow heating for 2 minutes. After each pressing, the sample was removed immediately to cool down in the air. The pressing speed was 5.2 mm/s, corresponding to the initial strain rate of 2.3 s^{-1} approximately. Die dimension t was 2 mm. Four cycles of CGP were applied with the temperature of 250 °C for the 1st cycle, 200 °C for the 2nd, and 150 °C for the 3rd and 4th cycles.

3.2 Digital Image Correlation Analysis

3.2.1 Overview

DIC provides a non-destructive measurement of the displacement field on the sample surface and estimation of the strain field by comparing sequential images taken at different stage of deformation [235, 236]. DIC was first developed in the 1980s [237-240]. With the development of imaging techniques and the enhancement of the processing power of computers, DIC has been dramatically improved to allow detection of sub-pixel displacement and micro-strains [235]. Note that DIC discussed and used in this study is 2D DIC which deals with the measurement of in-plane displacement.

3.2.2 Applications

There is currently a broad range of applications of DIC in science and engineering for measuring displacements and evaluating strains at both the microscopic and macroscopic level. The application of DIC in combination with FIB ring-core milling allows the evaluation of residual strain at the microscopic scale [97, 116-118]. In macroscopic

mechanical experiments, such as crack opening experiments [241, 242], DIC is used to locate cracks and perform precise crack opening measurements [236]. Moreover, DIC can be utilised to monitor displacements of large engineering structures [243], such as bridges, roads or rails for detecting crack opening [236].

3.2.3 Advantages and Disadvantages

Being non-destructive, DIC has a number of advantages when compared to some other optical techniques and conventional non-destructive techniques used for the same purpose [235, 236, 244]:

1) Low requirement in experimental setup or environment

Only one fixed camera is required to record the deformation history by taking digital images of the sample surface. No anti-vibration tables or laser source is needed, hence it can be utilised for both laboratory-based work and field experiments. In some cases of field applications when other techniques are not suitable, DIC opens the possibility of combining digital photography and techniques for civil engineering surveying to supply accurate monitoring of structures.

2) Broad measurement sensitivity and resolution

DIC deals with digital images. Therefore, DIC can be used for processing data from a wide range of imaging techniques, such as optical microscope, SEM, laser scanning microscopy and atomic force microscopy. Deformation of different scales (even down to micro- or nano- scale) can be obtained depending on the resolution of the images.

3) Real-time measurement

Automated image collection and DIC processing can be easily achieved with computer-aided techniques. In the meantime, real-time measurement becomes possible by simultaneously comparing the current image with the one before deformation [245].

Nevertheless, DIC has some disadvantages [235, 236]. Firstly, 2D DIC used in this work is limited to measuring in-plane deformation, which also requires that the sample must have a planer surface. To extend the current technique to 3D deformation measurement, 3D DIC is developed based on binocular stereovision [246-249]. Additionally, the accuracy of measurement heavily depends on the quality of the imaging system and the status of the sample surface. The surface must have features with a random distribution of grey scale for correlation. If the surface is too smooth (in the case of polished samples) or has a repetitive pattern, image correlation will very likely fail to provide satisfactory accuracy. Therefore, techniques have been developed to create suitable surface features e.g. using Pt nano-dots deposition on the sample surface in the SEM chamber for image correlation [97, 250]. More details about the effect of Pt deposition will be discussed in section 5.1.

3.3 Synchrotron XRD for Lattice Strain Evaluation

3.3.1 Overview

The diffraction of X-ray by crystalline materials is described by Bragg's Law [129] shown in Equation (3.1):

$$2d\sin\theta = n\lambda \quad (3.1)$$

where λ is the wavelength of X-ray beam, d is the lattice spacing, θ is half of the angle between the incident and reflected beams and n is a positive integer (=1,2,3,...) that describes the order of reflection.

Figure 3.2 demonstrates diffraction of the X-ray of two groups of lattice planes with different orientations. These two orientations can represent two grains and they will be called grain A and grain B in the following discussion for convenience. Note that A and B are drawn at different positions along X-ray in order to show them more clearly. Accordingly, DO and CO are different in length. In a real experiment, all the grains in one

sample are considered to have the same sample-detector distance if the sample thickness is small enough to be neglected. Insets with higher magnification of A and B explain the geometry relationship between the grain's orientation and the X-ray beam, which satisfies Bragg's law. The two diffracted X-ray beams AD and BC are collected by the 2D detector as diffraction spot C and D on the 2D diffraction pattern.

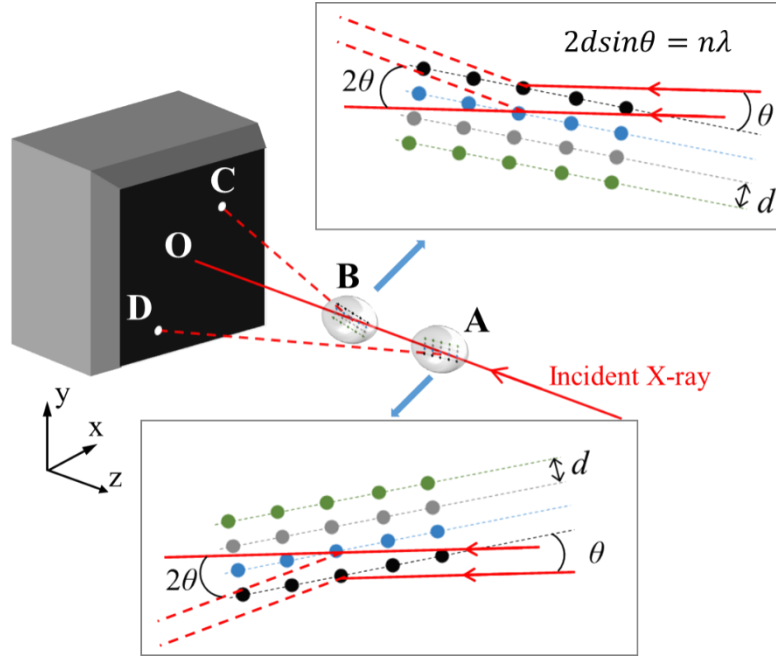


Figure 3.2 Schematic explanation of X-ray diffraction and Bragg's law.

Using grain A as an example, the d -spacing is related to DO by Bragg's Law and Equation (3.2):

$$\tan 2\theta = DO/AO \quad (3.2)$$

Similarly, $\tan 2\theta = CO/BO$ applies for grain B. Therefore, the change of d -spacing can be obtained by tracking the radial change of the diffraction spot position on the pattern. When the bulk of material is non-textured polycrystalline, the diffraction pattern of the grains will become a ring (Debye-Scherrer ring) with uniform intensity. With the existence of texture, the ring will have non-uniform intensity. Since only polycrystalline material is of interest in the current work, the following discussion will be focused on Debye-Scherrer rings. In this case, DO in Equation (3.2) becomes the radius of the diffraction ring and

the radius change can be used to calculate d -spacing change and hence the elastic lattice strain embedded using Equation (3.3):

$$\varepsilon = \frac{d - d_0}{d_0} \quad (3.3)$$

where ε is the elastic strain, d is the d -spacing of the processed sample and d_0 is the value of d -spacing when the sample is in a strain-free status [251].

In order to obtain θ , it is essential to know some key values: sample-detector distance (AO in Figure 3.2), beam energy (λ in Equation (3.1)) and image centre (position of O in Figure 3.2). Those can be calibrated using a standard calibration sample which was lanthanum hexaboride (LaB₆) in this thesis for wide-angle X-ray scattering (WAXS) experiment. A typical powder XRD pattern is shown in Figure 3.3. The conventional way of analysing powder XRD patterns for strain evaluation is to convert the 2D pattern into the 1D 2θ -intensity profile, shown as top left inset in Figure 3.3. A 1D profile has bell-shaped peaks representing different sets of lattice planes. The 2D-1D conversion is performed through a combination of radial binning and azimuthal integration. When the azimuthal integral is restricted between certain angles, strain information along a certain direction can be extracted. This approach is often informally referred to as ‘caking’ [252], literally meaning integral within a sector (similar to a piece of cake). For instance, as the white sector demonstrates in Figure 3.3, ‘caking’ starts at -15° and ends at 15° . In this thesis, the angle range of the section will be called ‘caking angle’ and the centre of the caking sector will be referred to as caking orientation, which is also the direction of information (e.g. lattice strain) obtained from ‘caking’. Hence, the caking angle is 30° and the caking orientation is 0° (refer to the coordinate system at the bottom left of Figure 3.3 for the definition of 0°). In addition, lower and upper bounds can be applied to radial binning to extract information for selected rings. For example, radial binning in Figure 3.3 is restricted in between the red curves so that only the information of the first three rings

is shown on the 1D profile. With the help of different caking orientations, strains along different directions can be extracted from one diffraction pattern. Note that ‘peak’ and ‘ring’ in this thesis have different meanings. A ring is the original Debye-Scherrer diffraction ring on the diffraction pattern, whereas a peak is from a 1D profile which is produced through ‘caking’. Therefore, a peak only contains specified information of the ring depending on how ‘caking’ is performed. For instance, the 1st peak here only has the average information between -15 ° to 15 ° of the 1st ring.

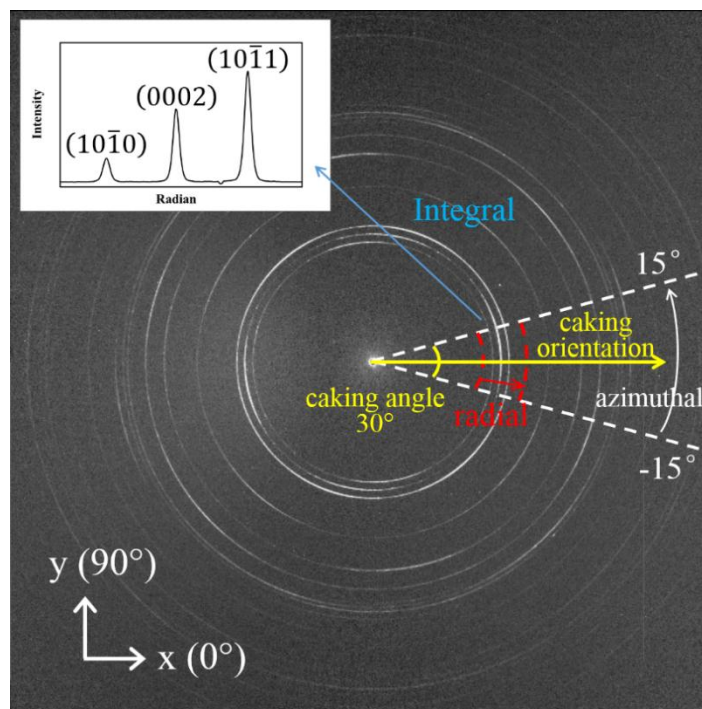


Figure 3.3 Conversion of 2D diffraction pattern into 1D profile with ‘caking’.

As shown in the top left inset of Figure 3.3, each peak/ring has its *hkil* miller index, which corresponds to the normal of the series of successive lattice planes that are diffracting X-ray. A visual illustration can be seen in Figure 3.4 where one unit cell represents one grain with the corresponding orientation. For instance, (10 $\bar{1}$ 0) planes of the grain with current orientation satisfy Bragg’s Law and their diffraction gives one spot on the 1st ring. In polycrystalline materials, there are a group of grains with the same orientation (referred to as grain group). Therefore, one diffraction spot is a consequence of a grain group. Note that a full ring on the pattern corresponds to grains with the same

orientation, but rotating around 360° with respect to the X-ray beam, which forms a diffraction cone. The same principle applies to the 2nd and the 3rd rings.

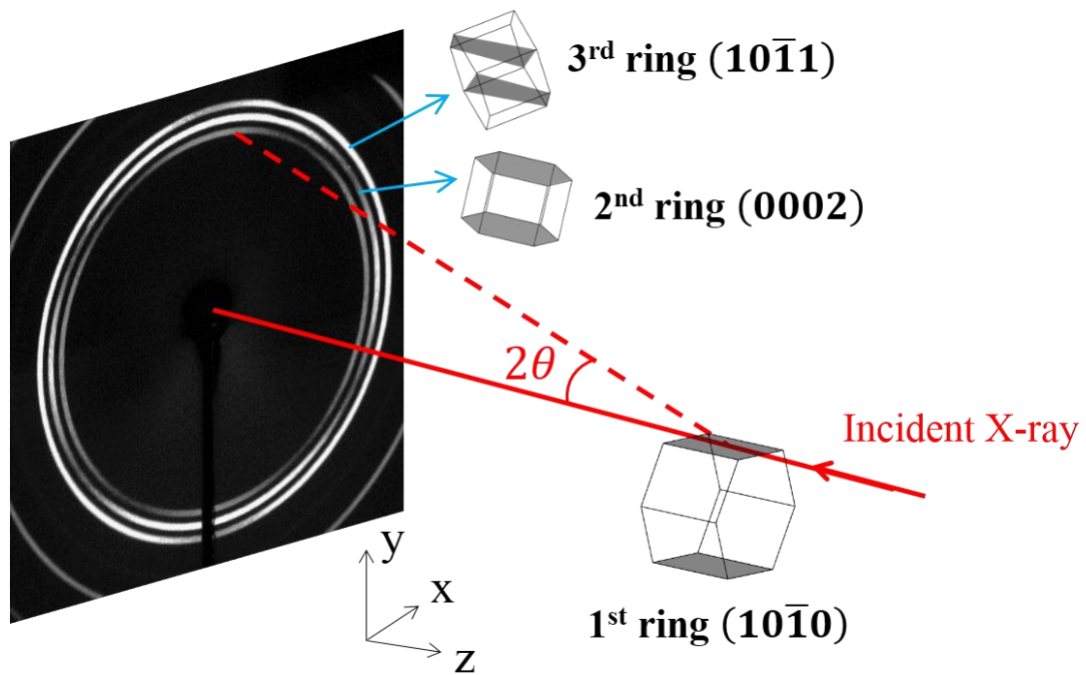


Figure 3.4 Orientations of grain groups for different diffraction rings.

3.3.2 XRD of a Bent Bar

3.3.2.1 Bending Test

Figure 3.5 is the cross-section of part of the bending sample (dimension $23 \times 1.8 \times 2.1$ mm, cut from the CGPed sheet) before and after bending test which was imaged using an Alicona Infinite Focus 3D Profilometer. The cross-section has a periodic wavy shape resulting from incomplete straightening after CGP processing (referring to the ‘half period’ in Figure 3.5), with the period length of 8 mm. A Deben loading rig (5 kN Extended Tensile Tester, MT10223) was used to carry out the 3-point bending test. The setup of the pins for the 3-point bending is shown in left figure in Figure 3.5. The black circles marked with numbers represent the pins for 3-point bending, and the spacing used is 3/3 mm (the distance between pin 1 and pin 2, pin 1 and pin 3). The centre pin (pin 1) was aligned with the trough of the periodic wave surface profile and the loading force was parallel to the ND. Maximum force $F=400$ N was reached to ensure sufficient plastic deformation was

introduced in the sample, resulting in a curvature change of 86.75 m^{-1} . Then the load was removed (hence the dashed empty circles on the right image of Figure 3.5) and the sample was examined using XRD.

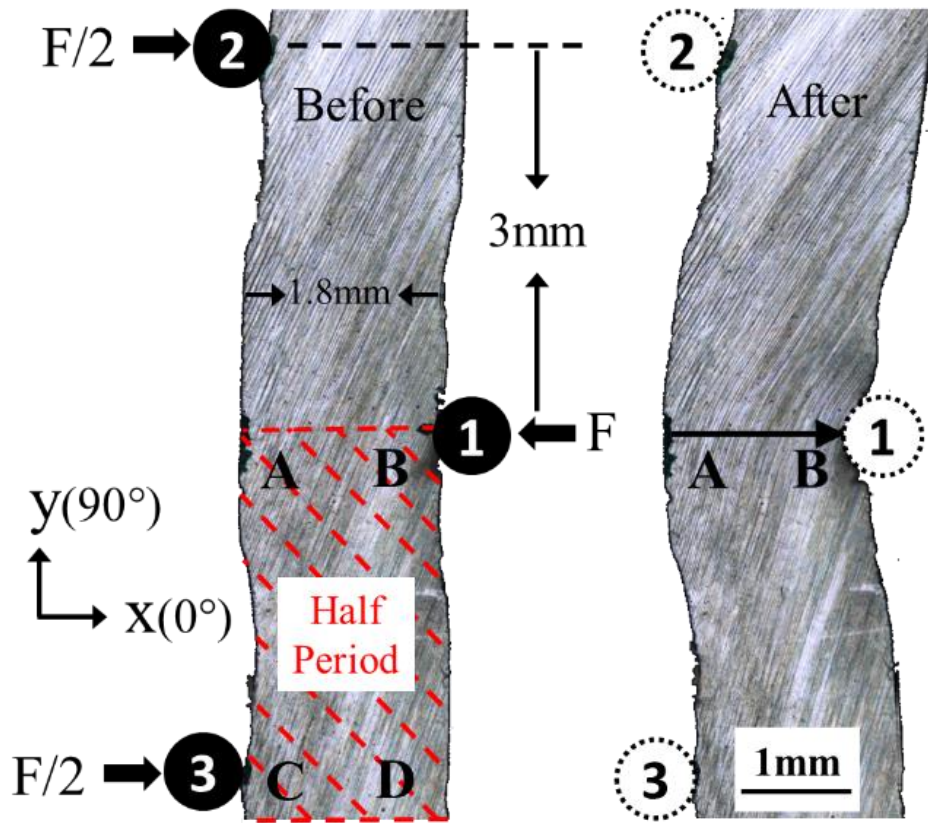


Figure 3.5 Setup of the bending test and comparison of the sample before and after bending, indicating a change of 86.75 m^{-1} in curvature.

3.3.2.2 XRD Experiment

The high Energy *ex situ* XRD experiment was carried out at Beamline I15, Diamond Light Source (Harwell Campus, UK). The layout of the experiment is illustrated in Figure 3.6. Note that the bending load was already removed during the XRD experiment. The beam size of the X-ray beam used was $0.02 \text{ mm} \times 0.02 \text{ mm}$ to ensure that there were a sufficient number of grains within the gauge volume to provide enough powder averaging and good statistics. The resulting Debye-Scherrer rings were mostly continuous, which ensured that after the conversion of 2D data to 1D plots, the bell-shaped diffraction peaks had sufficient resolution to facilitate high precision Gaussian fitting. A monochromatic

beam with an energy of 72 keV was scanned along the central line of the 3-point bending sample from points A to B, as shown in Figure 3.5. The step size of the line scan was 0.01 mm and 180 data points were collected (a sequence of 180 2D diffraction patterns were acquired), covering the entire width of the bar. The beam size was twice the step size and, therefore, there was 50% overlap between successive steps. A Perkin-Elmer flat panel detector 1621 EN (Wiesbaden, Germany) with 0.2 mm×0.2 mm pixel size was placed 1125.5 mm away from the sample for pattern collection. The matrix of the detector used in this study was 2048×2048 pixels.

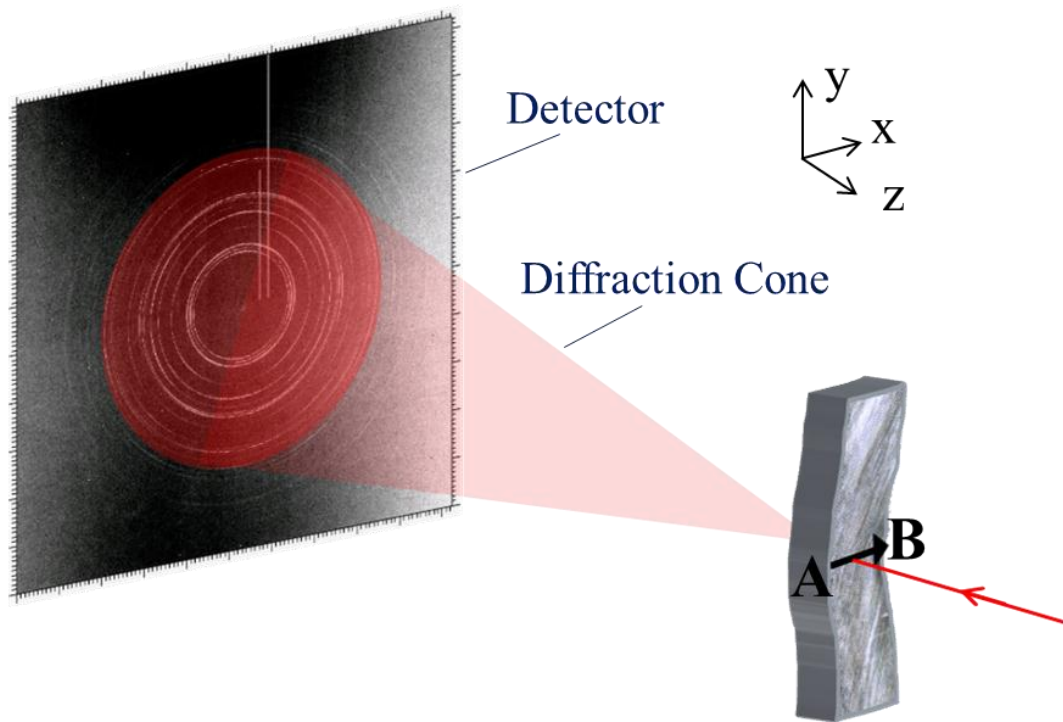


Figure 3.6 Setup of *ex situ* XRD experiment with the sample after 3-point bending.

3.3.3 XRD with *in situ* Compression-Tension Loading

The material used in this experiment was as-received wrought magnesium alloy AZ31B with average grain size at approximately 20 μm . Typical rod texture was seen in the material from both XRD pattern (Figure 3.7) and pole figures from EBSD mapping (Figure 7.4), meaning that basal poles are mainly parallel to ND. Twinning is, therefore, preferably activated under compression along TD or RD [3]. Dog-bone-shaped samples

used were machined using electrical discharge machining and their cross-section was 1 mm wide and 2 mm thick. The LD of compression-tension test was parallel to TD.

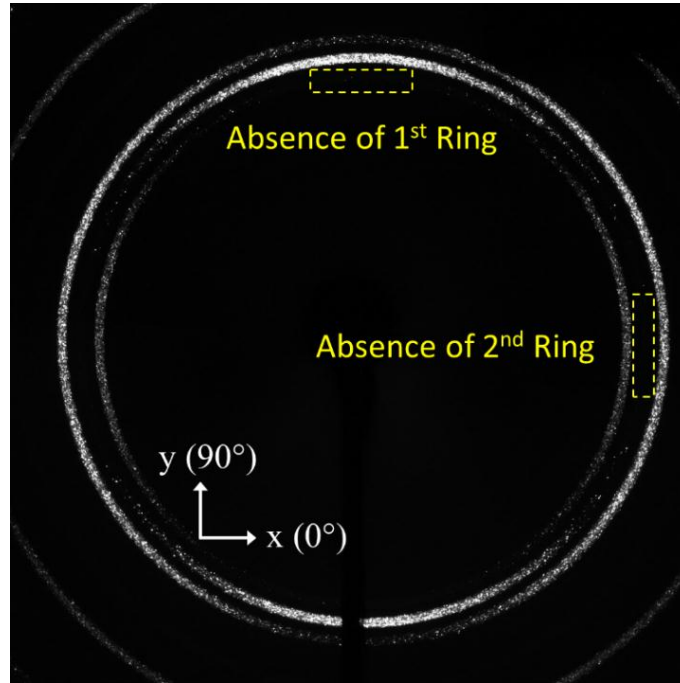


Figure 3.7 Powder XRD pattern of as-received wrought sample.

The *in situ* XRD experiment was conducted at Beamline B16, Diamond Light Source (Harwell Campus, UK). The monochromatic X-ray beam was set to 18 keV and shaped to size 1 mm × 1 mm by two sets of slits with the first set determining the beam size and the second set cleaning the X-ray scattering from the first set. A 5 kN Deben (5 kN Extended Tensile Tester, MT10223) loading rig was placed in the beam for *in situ* compression-tension test (see Figure 3.8). The sample-detector (Photonic Science X-ray Image Star 9000, 3056 × 3056 pixels, 31 μm pixel size) distance was roughly 130 mm so that only the first three Debye-Scherrer rings could appear on the diffraction pattern in order to achieve a higher spatial resolution. Loading rate was 0.2 mm/min for both compression and tension. Max compressive engineering stress 112.7 MPa (250 N) was reached followed by unloading and tension until fracture at nearly 560 N. XRD patterns were collected at every 10 N (loading force was kept constant during collection). The loading was held at a constant force (force control) during XRD pattern collection here since the

activation of twinning and detwinning is directly decided by stress. Exposure time of every pattern was optimised to 20 s to ensure enough intensity for peak fitting. It turned out that creeping is negligible in 20 s, so choosing stress control or strain control makes little difference in this case.

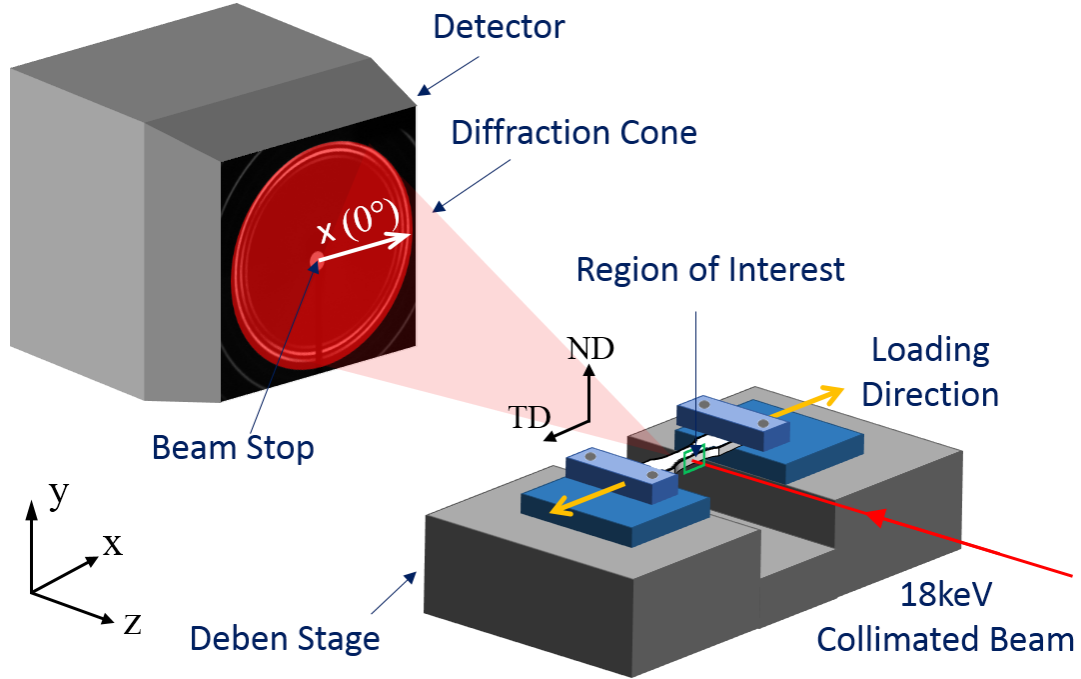


Figure 3.8 Setup of in situ XRD experiment.

3.3.4 XRD with *in situ* Cyclic Loading

The material used in this experiment was CGPed magnesium AZ31B. The same setup as shown in Figure 3.8 was employed. Figure 3.9 is the XRD pattern of the sample prior to applying any loading. Compared with the wrought sample (see Figure 3.7), CGPed sample has less grainy patterns thanks to its microstructure with finer grains. In addition, it features less degree of rod texture since the 2nd ring and the 1st ring do appear, even though with less intensity, respectively along 0 ° and 90 ° direction instead of being totally absent as in the case of wrought material.

Cyclic T-C-T-C-T-C (T: tension; C: compression) loading followed by unloading to 0 MPa was performed with the loading rate of 0.2 mm/min. The maximum loading reached

corresponded to 283 MPa in engineering stress for both directions. The XRD patterns were taken at the exposure time of 30 s.

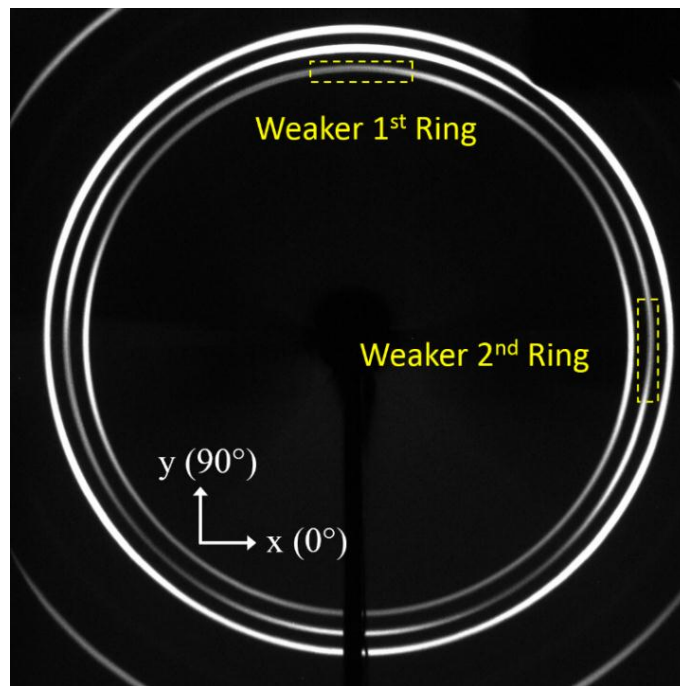


Figure 3.9 XRD pattern of CGPed magnesium AZ31B sample.

3.4 Electron Backscatter Diffraction (EBSD)

3.4.1 Overview

EBSD, a microstructure characterisation technique, allows acquisition of crystallographic information [253, 254] from thin films and bulk materials [255-258]. EBSD mapping is carried out by going through an area on the sample surface (normally approximately 70 °tilted) point by point with a focused electron beam. The sample surface at each point backscatters some electrons which are then captured by a detector. The detector has a scintillator screen which is coupled to a photon-sensitive imaging detector to collect the electron backscatter diffraction patterns (EBSPs). In each pattern, there are various bands with higher intensity (Figure 3.10), which are the Kikuchi bands. The edges of the bands seem to be straight and parallel but are actually hyperbola-shaped as a consequence of the intersection between the detector plane and the Kossel diffraction cones [255, 259]. However, each band centre is a straight line that indicates effectively the

crystal plane that forms the band. A tilt or rotation of the crystal will be reflected respectively by a shift or rotation on the EBSP [258]. Figure 3.10 illustrates an example of FCC crystal with different orientations and the corresponding Kikuchi patterns which are indexed in software AZtec[®] developed by Oxford Instruments for EBSD and EDS detectors [260].

Indexing the EBSP provides the crystallographic information, such as crystal orientation, crystal type and pattern quality. Provided the knowledge of the current experimental geometrical setup, the modern EBSD systems can perform automatic indexing relatively fast [255].

EBSD finds applications in various aspects of material characterisation among which crystal orientation measurement is the focus in this study. It enables characterisation of texture, observation of texture evolution [261-265] and examination of recrystallization [266-268] during deformation. Additionally, it is used to identify phases and explore phase transformation [269-272]. Aside from all the applications mentioned above, there are two aspects that are of great interest and experiencing significant development: HR-EBSD for strain mapping and 3D EBSD characterisation. HR-EBSD is based on cross-correlation analysis of EBSPs to extract local deformation and, therefore, local stress as well as dislocation density distribution [255, 256, 273]. Strain accuracy of $\sim 10^{-4}$ can be reached by this method [256]. 3D EBSD relies on successive removing of the sample surface layer by layer (usually using FIB) and mapping on the newly revealed surface [255]. Instead of only 2D information that needs assumptions to understand the whole structure, 3D characterisation provides a complete microstructure including real grain shape, grain size and twin morphology. [274-278]

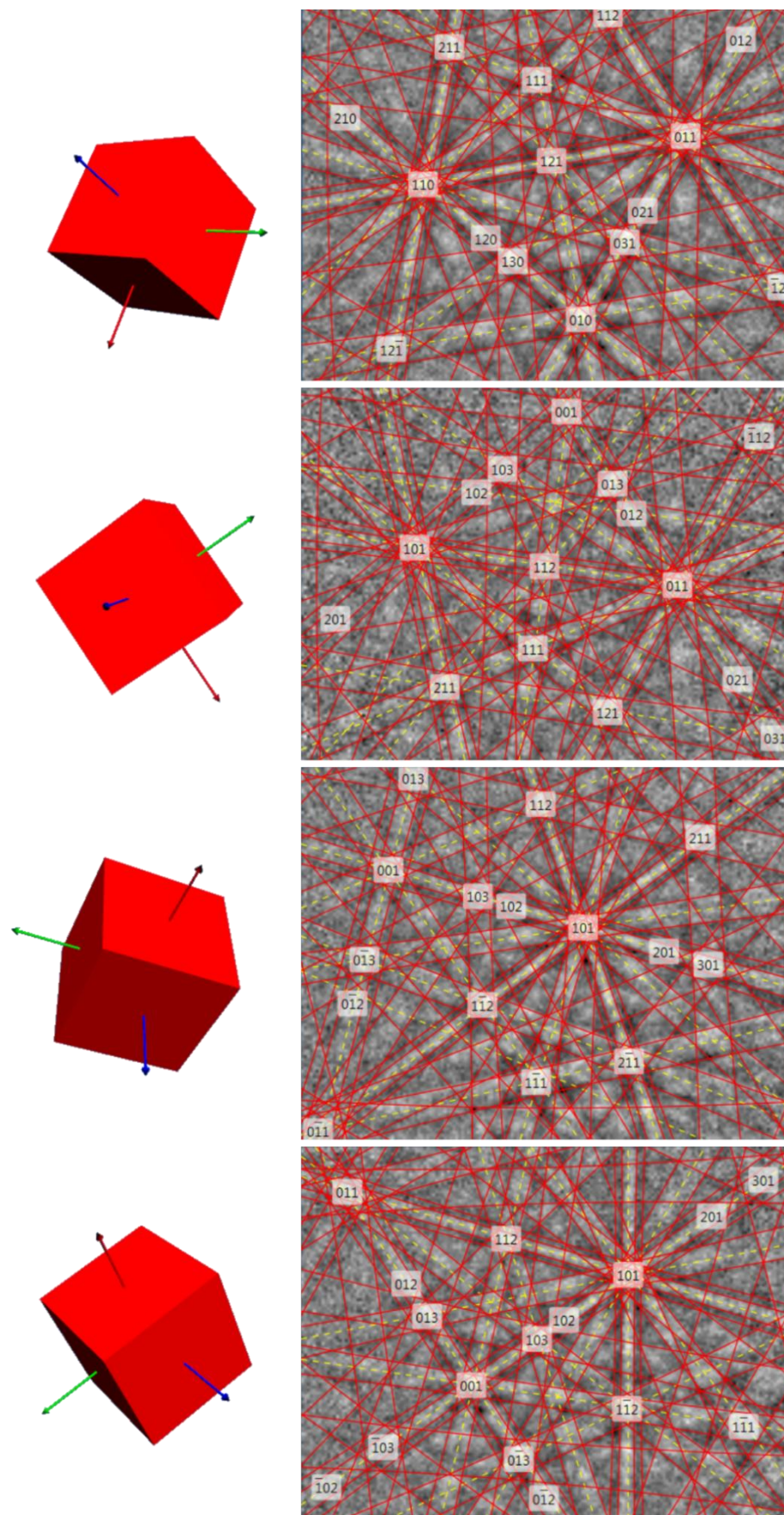


Figure 3.10 FCC unit cells with different crystal orientations and the corresponding indexed Kikuchi patterns.

One disadvantage of using EBSD is that the sample surface must have high quality. Standard sectioning-grinding-polishing processes only marks the general sample preparation. For EBSD samples, additional preparation for final polish is critical to ensure that the finishing surface is free from any defect since EBSP is only generated from a very thin layer (roughly 40 nm [255]) on the surface.). Depending on the materials, common methods for final polishing include colloidal silica polishing, electro polishing, chemical etching and ion polishing etc. Improving the quality of sample surface reduces the time needed to take an indexable EBSP, which consequently reduces the time needed for EBSD mapping.

3.4.2 EBSD with *in situ* Cyclic Loading

In situ EBSD was performed in the Multi-Beam Laboratory for Engineering Microscopy (MBLEM), Oxford. The same dog-bone samples from section 3.3.3 were used. They were polished in the sequence of 1200 grit SiC paper (until plane), 3 μm diamond suspension with MD-Mol cloth (20 min), 1 μm diamond suspension with MD-Nap cloth (15 min) followed by 0.04 μm colloidal silica with MD-Chem cloth (15 min). All the polishing consumables used above (diamond suspension, colloidal silica and polishing cloths) were from Struers[®]. Throughout the whole polishing process, ethanol was used for lubrication and cleaning instead of water to avoid oxidation. A pre-tilted grip was specially designed for the Deben loading rig to satisfy the geometry where the sample surface is required to be 70 °tilted, see Figure 3.11. EBSPs were captured with an Oxford Instruments NordlysNano detector. A two-cycle compression-tension *in situ* EBSD was carried out with the 1st cycle the same as the loading test carried out for *in situ* compression-tension XRD (described in section 3.3.3).

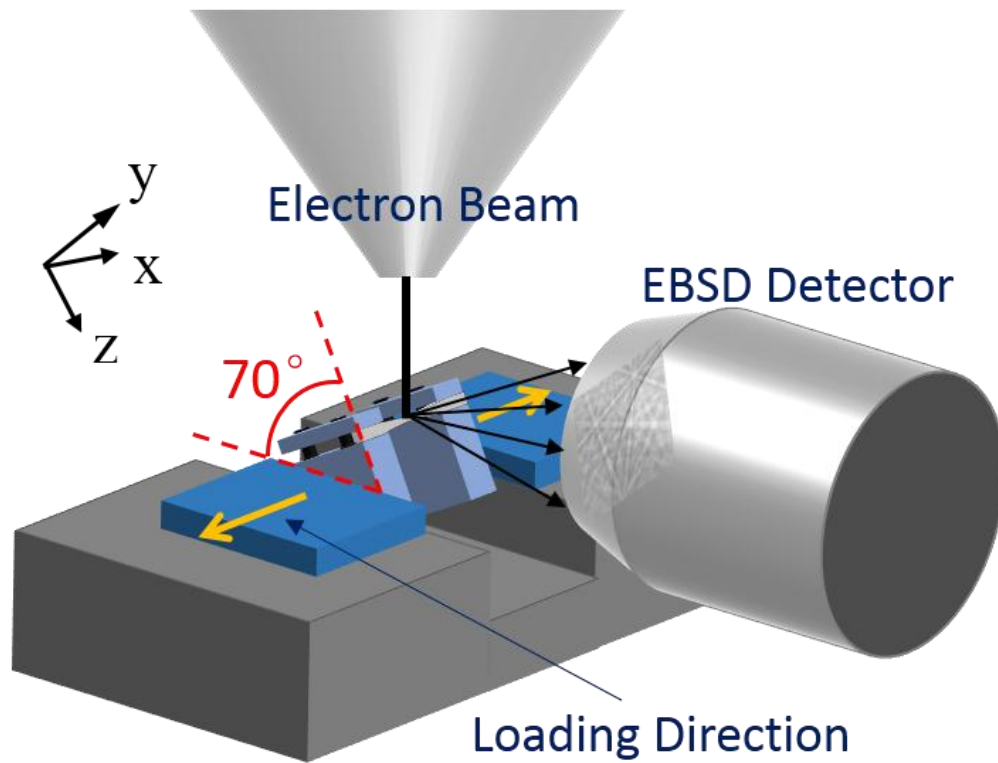


Figure 3.11 Setup of in situ EBSD experiment.

Informed by XRD experiments about the beginning and the end of twinning and detwinning, four measure points (see points B, C, E and F in Figure 6.5 and Figure 6.6) were chosen for EBSD mapping in the 1st cycle. The four points were respectively sitting right before the beginning of twinning, at the maximum compression force, in the middle of detwinning and at the end of detwinning. For the 2nd cycle, EBSD maps were taken at another four measure points at the same stresses, as those in the 1st cycle. Therefore, in total, eight EBSD maps were taken. EBSD mapping was performed at 25 kV with the spatial resolution of 0.8 μm . To make sure that mapping was carried out on the same area, four features were deposited at the four corners of the rectangular mapping area using GIS for Pt sputtering, referring to Figure 3.12. Different shapes of markers were used so that the orientation of the mapping area could be recognised under SEM. Due to the surface deformation during loading, the size of maps slightly varied. The loading cell was kept at a fixed position during mapping. Post-processing was performed using software Tango from CHANNEL 5 system (developed by Oxford Instruments [279]) for noise reduction

and inverse pole figure colouring map plotting. Additionally, MTEX [280] was used for plotting pole figures. The EBSD maps are in the form of a matrix of index points that are similar to pixels of an image. The size of index point is the resolution of the map which was set to be $0.8\ \mu\text{m} \times 0.8\ \mu\text{m}$ in this experiment. One index point has a set of Euler angles automatically solved from its Kikuchi pattern.

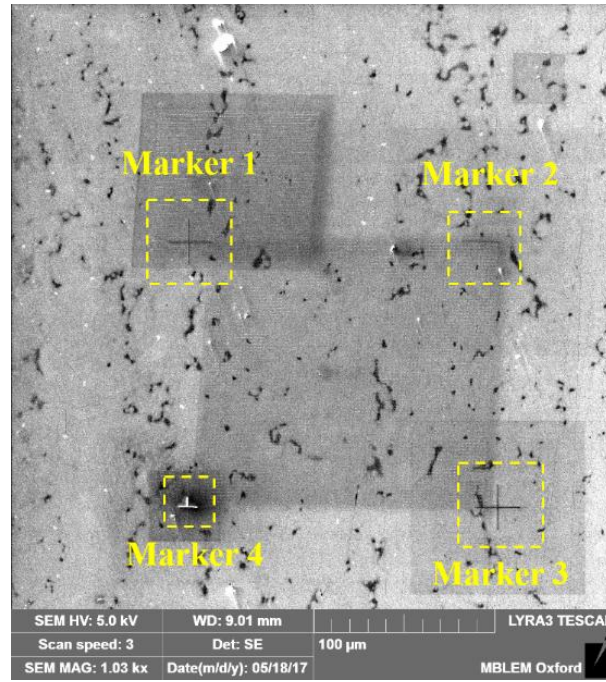


Figure 3.12 Features created with platinum deposition at the four corners of the rectangular area for in situ EBSD mapping.

4 Crystal Plasticity Constitutive Framework

This chapter introduces the continuum formulation and the implementation of lattice rotation for the crystal plasticity simulation. The calculation procedure is listed in steps for a better understanding of the whole program.

4.1 Continuum Formulation

The constitutive framework developed in this thesis accounts for detwinning. It is based on the model proposed by Fernández *et al.* [38, 40] which followed the study of Staroselsky and Anand [203, 204].

Deformation can be completely described by the deformation gradient $\mathbf{F}$. It includes stretch and rigid body rotation but excludes translation. Following the classical multiplicative decomposition from Lee [281], as shown in Figure 4.1, a unique decomposition of deformation gradient can be found to separate elastic and plastic parts [282] following Equation (4.1):

$$\mathbf{F} = \mathbf{F}^e \mathbf{F}^p \quad (4.1)$$

Therefore, $\mathbf{F}^e$ can be calculated using Equation (4.2):

$$\mathbf{F}^e = \mathbf{F} \mathbf{F}^{p-1} \quad (4.2)$$

In theory, elastic deformation gradient $\mathbf{F}^e$ and plastic deformation gradient $\mathbf{F}^p$ can both contain stretch and rigid body rotation at the same time. However, conventionally, plastic deformation gradient contain all of the rigid body rotation, leaving all the stretch to be included into elastic deformation gradient [282]. In the case of crystal plasticity, elastic deformation includes elastic crystal lattice distortion, while plastic deformation involves dislocation motion (i.e., slip) and shear introduced by twinning [59, 60]. The distortion in $\mathbf{F}^e$ increases the Cauchy stress σ with $\det(\mathbf{F}^e) > 0$ and $\det(\mathbf{F}^p) = 1$.

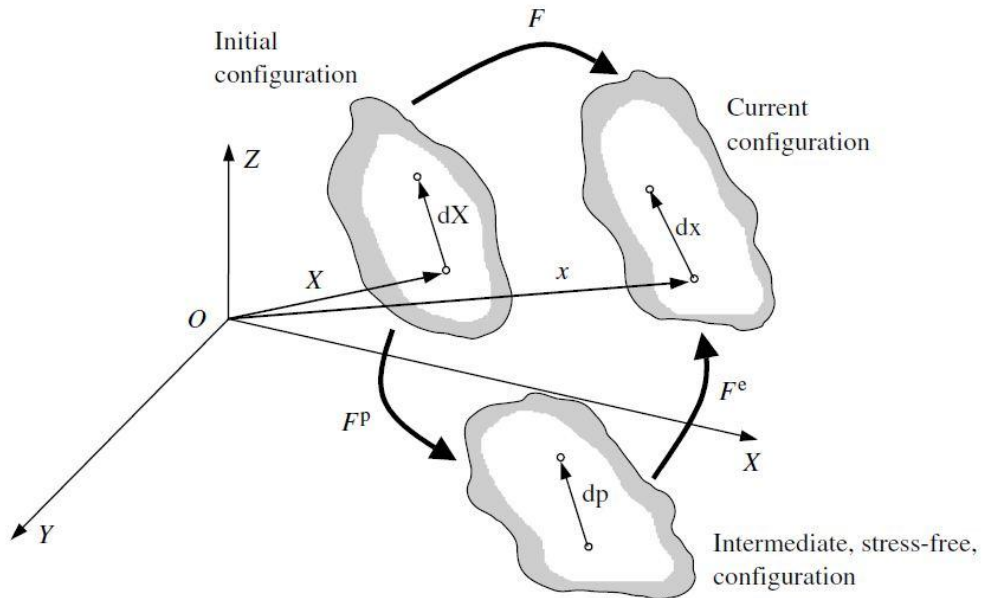


Figure 4.1 Multiplicative decomposition of deformation gradient into elastic and plastic parts [282].

Note that time is not taken into consideration when separating rotation and stretch. Nevertheless, many plasticity formations have rate-dependent quantities. Therefore, it is necessary to consider a rate form. The velocity gradient $\mathbf{L}$ depicts the spatial rate of the change of velocity and can be obtained by Equation (4.3).

$$\mathbf{L} = \dot{\mathbf{F}}\mathbf{F}^{-1} \quad (4.3)$$

One unit reference volume has deformation power p defined in Equation (4.4).

$$p = \mathbf{P} : \dot{\mathbf{F}} = \mathbf{S}^e : \dot{\mathbf{E}}^e + (\mathbf{C}^e \cdot \mathbf{S}^e) : \mathbf{L}^p \quad (4.4)$$

$\mathbf{L}^p$ is the plastic velocity gradient tensor calculated by Equation (4.5).

$$\mathbf{L}^p = \dot{\mathbf{F}}^p \cdot \mathbf{F}^{p-1} \quad (4.5)$$

$\mathbf{C}^e$ is the right Cauchy-Green elastic deformation tensor.

$$\mathbf{C}^e = \mathbf{F}^{eT} \cdot \mathbf{F}^e \quad (4.6)$$

$\mathbf{E}^e$ is the Green-Lagrange elastic strain tensor.

$$\mathbf{E}^e = \frac{1}{2}(\mathbf{C}^e - \mathbf{I}) \quad (4.7)$$

$\mathbf{S}^e$ is the second Piola-Kirchhoff stress tensor. For metallic materials a linear relation can be used to describe the constitutive equation [203]. Therefore,

$$\mathbf{S}^e = \mathbb{C} : \mathbf{E}^e \quad (4.8)$$

where $\mathbb{C}$ is a fourth order anisotropic elasticity tensor. Therefore, Cauchy stress can be obtained following Equation (4.9).

$$\begin{aligned} \mathbf{S}^e &= \det(\mathbf{F}^e) \mathbf{F}^{e-1} \cdot \boldsymbol{\sigma} \cdot \mathbf{F}^{e-1T} \\ \boldsymbol{\sigma} &= \mathbf{F}^e \{ [\det(\mathbf{F}^e)]^{-1} \mathbf{S}^e \} \mathbf{F}^{eT} \end{aligned} \quad (4.9)$$

In addition, $\mathbf{P}$ in Equation (4.4) is the first Piola-Kirchhoff stress tensor and its relationship with Cauchy stress is defined by Equation (4.10).

$$\mathbf{P} = \det(\mathbf{F}) \boldsymbol{\sigma} \cdot \mathbf{F}^{-1T} \quad (4.10)$$

For simplification, the superscript i (and j) will refer to slip systems, u (and v) to all the deformation systems of both twinning and detwinning. The only difference between twinning and detwinning in the model lies in the detailed activation rule that will be given in section 7.2.2. Therefore, no differentiation is needed between twinning and detwinning to introduce the general formulation in this section.

Putting together Equation (4.4) to (4.9) leads to the knowledge that as long as $\mathbf{L}^p$ is known, the Cauchy stress can be calculated. $\mathbf{L}^p$ can be extended from previous work by Taylor [157] and Rice [283] to include twinning/detwinning expressed as follows:

$$\mathbf{L}^p = \sum_i \text{sign}(\tau^i) \dot{\gamma}^i \mathbf{S}_0^i + \sum_u \text{sign}(\tau^u) \dot{\gamma}^u \mathbf{S}_0^u \quad (4.11)$$

where $\dot{\gamma}$ is the shear strain rate on one deformation system. Note that twinning and detwinning do not happen in one element at the same time according to the activation rule (section 7.2.2) so $\mathbf{L}^p$ either contains twinning or detwinning and using superscript s in Equation (4.11) does not cause any confusion. The key point of calculating $\mathbf{L}^p$ here becomes calculating $\dot{\gamma}$.

A deformation system is characterised by a unit vector $\mathbf{n}_0$ denoting deformation plane normal and a unit vector $\mathbf{m}_0$ denoting deformation direction. Projecting $\mathbf{S}^e$ onto the slip, twin or detwin systems following Equation (4.12) gives the RSS value τ on the system.

$$\begin{cases} \tau^i = (\mathbf{C}^e \cdot \mathbf{S}^e) : \mathbf{S}_0^i \\ \tau^u = (\mathbf{C}^e \cdot \mathbf{S}^e) : \mathbf{S}_0^u \end{cases} \quad (4.12)$$

$\mathbf{S}_0$ is the Schmid tensor that is related to the direction and the plane normal of a deformation system via Equation (4.13).

$$\begin{cases} \mathbf{S}_0^i = \mathbf{m}_0^i \otimes \mathbf{n}_0^i \\ \mathbf{S}_0^u = \mathbf{m}_0^u \otimes \mathbf{n}_0^u \end{cases} \quad (4.13)$$

When calculating RSS of infinitesimal elastic stretches, Equation (4.12) can be approximated into Equation (4.14).

$$\begin{cases} \tau^i = \mathbf{S}^e \cdot \mathbf{S}_0^i \\ \tau^u = \mathbf{S}^e \cdot \mathbf{S}_0^u \end{cases} \quad (4.14)$$

The evolution of CRSS s of slip, twin and detwin systems is also related to the shear strain rates $\dot{\gamma}$ through a generalised linear hardening rule in Equation (4.15).

$$\begin{cases} \dot{s}^i = \sum_j h^{ij} \dot{\gamma}^j + \sum_v h^{iv} \dot{\gamma}^v \\ \dot{s}^u = \sum_v h^{uv} \dot{\gamma}^v + \sum_j h^{uj} \dot{\gamma}^j \end{cases} \quad (4.15)$$

Note that the expression ‘twin/detwin’ (or ‘tw/detw’) in text or equations means either twin or detwin rather than both twin and detwin. In Equation (4.15), h^{ij} , h^{iv} , h^{uv} and h^{uj} are hardening moduli respectively for the interactions of slip-slip, twin/detwin-slip, twin-twin (or detwin-detwin) and slip-twin/detwin. They can be obtained from power law type relations specified in Equation (4.16):

$$\begin{cases} h^{ij} = q^{ij} h^j, \text{ with } \begin{cases} q^{ij} = q_{sl} + (1 - q_{sl}) \delta^{ij} \\ h^j = h_{0;sl} \left(1 - \frac{s^j}{\tilde{s}_{sl}}\right)^{a_{sl}} \end{cases} \\ h^{uv} = q^{uv} h^v, \text{ with } \begin{cases} q^{uv} = q_{tw/detw} + (1 - q_{tw/detw}) \delta^{ij} \\ h^v = h_{0;tw/detw} \left(1 - \frac{s^v}{\tilde{s}_{tw/detw}}\right)^{a_{tw/detw}} \end{cases} \\ h^{iv} = h_{0;tw/detw-sl} \left(1 - \frac{s^v}{\tilde{s}_{tw/detw-sl}}\right)^{a_{tw/detw-sl}} \\ h^{uj} = h_{0;sl-tw/detw} \left(1 - \frac{s^j}{\tilde{s}_{sl-tw/detw}}\right)^{a_{sl-tw/detw}} \end{cases} \quad (4.16)$$

where δ^{ij} is the Kronecker symbol defined as below:

$$\delta^{ij} = \begin{cases} 1 & i = j \\ 0 & i \neq j \end{cases} \quad (4.17)$$

In Equation (4.16), $h_{0;sl}$, $h_{0;tw/detw}$, $h_{0;tw/detw-sl}$, $h_{0;sl-tw/detw}$, a_{sl} , $a_{tw/detw}$, $a_{tw/detw-sl}$ and $a_{sl-tw/detw}$ are the reference hardening parameters and hardening exponents for slip-slip, twin-twin (or detwin-detwin), twin/detwin-slip and slip-twin/detwin interactions. $\tilde{s}_{sl}$, $\tilde{s}_{tw/detw}$, $\tilde{s}_{tw/detw-sl}$ and $\tilde{s}_{sl-tw/detw}$ are the saturation stresses. q_{sl} and $q_{tw/detw}$ are the ratios of the

reference cross-hardening parameter to the reference self-hardening parameter of slip, twin and detwin systems, respectively.

4.2 Lattice Rotation

Twinning-induced rotation is essentially a local lattice rotation. Note that detwinning experiences a reverse rotation process to twinning so they share the same lattice rotation scheme. For convenience, only twinning will be talked about in the following discussion and the same rotation scheme applies to detwinning. The transformation can be defined by a rotation matrix for HCP materials [203]:

$$\mathbf{R}^\alpha = 2\mathbf{n}^\alpha \otimes \mathbf{n}^\alpha - \mathbf{I} \quad (4.18)$$

where $\mathbf{n}^\alpha$ is the plane normal of the current twin system α . The transformation rotates the lattice in a way that the rotated orientation mirrors the original one with respect to the twin plane. The new orientation after rotation might be unfavourable for twinning. Therefore, the main deformation mechanism in the twinned region will switch from twin to slip.

A twin is treated as a pseudo-slip at first [217]. Only when the volume fraction of the twin exceeds a certain threshold does the twinning-induced rotation happen. During the pseudo-slip phase, the volume fraction f^α is characterised as follows:

$$\begin{cases} f^\alpha = \int_0^{t1} \dot{f}^\alpha dt \\ \dot{f}^\alpha = \frac{\dot{\gamma}^\alpha}{\gamma_0} \end{cases} \quad (4.19)$$

where γ_0 is the characteristic shear strain of 0.129. The maximum twin fraction $f_{\max} = \max\{f^\alpha\}$ is compared with a random number $\zeta \in [\zeta_{\min}, \zeta_{\max}]$ picked for every increment. $\zeta_{\min}$ and $\zeta_{\max}$ respectively represent the lower and upper bounds of the extend of twinning process. If $f_{\max} > \zeta$, rotation is triggered by imposing $\mathbf{R}$ from Equation (4.18) (calculated with the twin system with the maximum f) on the rotation matrix defined by the Euler angles (ZXZ convention). Specifically, if the Euler angles of the original orientation define

the rotation matrix $\mathbf{Q}$ that links the fixed global coordinate system to the co-rotational crystal coordinate system, the rotation matrix becomes $\mathbf{QR}$ after twin-induced rotation is triggered. This means all the directions and plane normals of the slip and twin systems are rotated. After rotation, the accumulated volume fraction is set to zero.

4.3 Calculation Procedure

Now let us consider deformation over an infinitesimal time gap $\Delta t = t_1 - t_0$. Before explaining how to calculate $\dot{\gamma}$, it is worth mentioning that $\mathbf{F}^p(t_0)$ at every increment is known from the last increment. At the beginning, $\mathbf{F}^p$ is a unit matrix meaning that there is no plastic deformation yet. The general process of getting $\dot{\gamma}$ is that a trial RSS is calculated based on the knowledge of $\mathbf{F}^p(t_0)$ and the difference between RSS and CRSS contributes to plastic deformation (i.e., $\dot{\gamma}$) if RSS is larger than CRSS. The steps in the process are described in details as below. Note that when no time is given it always means a quantity at the current increment (t_1) while a quantity with (t_0) refers to one from the increment before.

Step 1 - Calculate the trial second Piola-Kirchhoff stress tensor $\mathbf{S}_{\text{tr}}^e$ from $\mathbf{F}^p$ using Equation (4.2), (4.6), (4.7) and (4.8).

$$\begin{aligned}\mathbf{F}_{\text{tr}}^e &= \mathbf{F}\mathbf{F}^{p-1}(t_0) \\ \mathbf{C}_{\text{tr}}^e &= \mathbf{F}_{\text{tr}}^{eT} \cdot \mathbf{F}_{\text{tr}}^e \\ \mathbf{E}_{\text{tr}}^e &= \frac{1}{2}(\mathbf{C}_{\text{tr}}^e - \mathbf{I}) \\ \mathbf{S}_{\text{tr}}^e &= \mathbb{C}:\mathbf{E}_{\text{tr}}^e\end{aligned}\tag{ 4.20 }$$

Step 2 – Calculate the trail RSS τ_{tr} on each system.

$$\begin{cases} \tau_{\text{tr}}^i = \mathbf{S}_{\text{tr}}^e \cdot \mathbf{S}_0^i(t_0) \\ \tau_{\text{tr}}^u = \mathbf{S}_{\text{tr}}^e \cdot \mathbf{S}_0^u(t_0) \end{cases}\tag{ 4.21 }$$

Step 3 – Deciding the group $\mathcal{R}$ of the potentially activated slip and twin/detwin systems.

They have to satisfy the following conditions to be potentially activated.

$$\begin{cases} |\tau_{\text{tr}}^i| > s^i(t_0) \\ \tau_{\text{tr}}^u > s^u(t_0) \end{cases} \quad (4.22)$$

The polar nature of twinning and detwinning is accounted for by not having absolute value condition on τ_{tr}^u . Note that Equation (4.22) only states the general activation rule of slip, twinning and detwinning based on whether RSS is larger than CRSS. A more detailed activation rule will be given in Equation (7.2) (section 7.2.2) with differentiation between twinning and detwinning.

Step 4 – Calculate the shear strain increment from the consistency condition.

The consistency condition for the activated deformation systems during plastic flow is

$$|\tau| = s(t_0) \quad (4.23)$$

The relationship between $\mathbf{F}^p$ and $\mathbf{F}^p(t_0)$ is

$$\mathbf{F}^p = (1 + \mathbf{L}^p)\mathbf{F}^p(t_0) \quad (4.24)$$

Equations (4.2), (4.6), (4.7), (4.8), (4.14) and (4.20) together give

$$|\tau^q| = |\tau_{\text{tr}}^q| - A^{pq}x^q, p \text{ and } q \in \mathcal{R} \quad (4.25)$$

where A^{pq} is the matrix defined over the whole potentially activated slip and twin/detwin systems involving known quantities, such as $\mathbf{S}_0$ and $\text{sign}(\tau_{\text{tr}}^q)$ and $x^q \equiv \Delta \gamma^q \geq 0$. The superscripts p and q refer to activated slip and twin/detwin systems belonging to the group $\mathcal{R}$. τ and τ_{tr} are always positive for activated twin/detwin system so adding absolute value condition in Equation (4.25) also suits twin/detwin systems. Equation (4.25) in combination with consistency condition in Equation (4.23) gives a system of linear equations:

$$Ax = b \quad (4.26)$$

where $b^p = |\tau_{tr}^p| - s^p(t_0)$.

Step 5 – Update $\mathbf{F}^p$ using Equation (4.24). Normalise $\mathbf{F}^p$ if $\det(\mathbf{F}^p) \neq 1$.

Step 6 – Update $\mathbf{F}^e$ and $\mathbf{S}^e$ using Equation (4.2) and (4.8), respectively.

Step 7 – Update Cauchy stress $\boldsymbol{\sigma}$ and CRSS s using Equation (4.9) and (4.15), respectively.

Step 8 – Update the twin/detwin volume fraction f^q for the activated twin/detwin systems.

Step 9 – Perform twin/detwin-induced lattice rotation if $f_{\max} > \zeta$.

5 DIC Development

5.1 Effect of Pt Deposition on DIC Analysis

5.1.1 Introduction

With the rapid development of applications in areas such as micro- and nano-electromechanical systems, residual stress measurement at the level of individual grains is becoming more important for studying miniature systems [118]. However, despite the growing demand, reliable residual stress measurement at microscopic resolution remains a major challenge [284]. Korsunsky *et al.* proposed a method that combines incremental ring-core milling by FIB with SEM high-resolution imaging and DIC analysis [116-118]. FIB-DIC ring-core milling allows minimally destructive measurement of residual stress down to the sub-micron scale [97, 118, 285]. The relatively wide availability of FIB and SEM means that the technique benefits from easy access compared with synchrotron X-ray and neutron diffraction instruments.

Stress evaluation relies on strain relief quantification and the latter depends on reliable tracking of surface displacement. Hence, it is essential to ensure that the sample surface has high contrast for tracking. There are several ways to increase the sample surface contrast, such as creating roughness, depositing precipitates, sputter coating and etching.

A popular way to ‘decorate’ the sample surface is to deposit a pattern of platinum nano-dots as contrast features [97, 116-118, 284-287].

This section presents a study of the effect of platinum deposition at the sample surface on the quality and robustness of DIC analysis by comparing the tracking quality respectively for unpatterned and Pt-patterned ring-cores on a polycrystalline CGPed Mg AZ31B alloy.

5.1.2 Experiment

5.1.2.1 Ring-Core Milling

The sample was mechanically polished using SiC paper to grit 1200/4000 finish to remove the oxidized layer and to introduce some scratches on the surface to improve the contrast.

Figure 5.1 demonstrates the ring-core milling configuration. By convention, the SEM column is aligned vertically and the secondary FIB column is tilted at an angle (55° in our case) from the SEM column. The sample was also tilted by 55° to present its surface to the FIB at normal incidence [288]. The SEM and the FIB columns were both focused and aligned to point to the same location at the sample surface to ensure that high resolution images were taken by SEM at the same position where FIB was performing incremental milling. Since the images were acquired by the SEM at the oblique angle of 35° , dynamic focusing and tilting correction were both set at 55° during the image acquisition.

Figure 5.2 illustrates a sequence of images collected from the two ring-cores studied. The sample surface area within the inner circle is referred to as island in the following discussion. Figure 5.2(a) demonstrates the ring-core with Pt deposition on the island and Figure 5.2(b) is the one without. Pt deposition was accomplished in the SEM vacuum chamber using the GIS which delivered a local flow of volatile organic compound containing Pt that was then deposited onto the sample surface by the electron beam. The

nano-dots were arranged in a pattern of ‘sunflower seeds’ [286] as it does not contain any repeated motifs translated (shifted) with respect to each other. Images labelled II and III are intermediate stages during milling whilst the images labelled IV are the final ones.

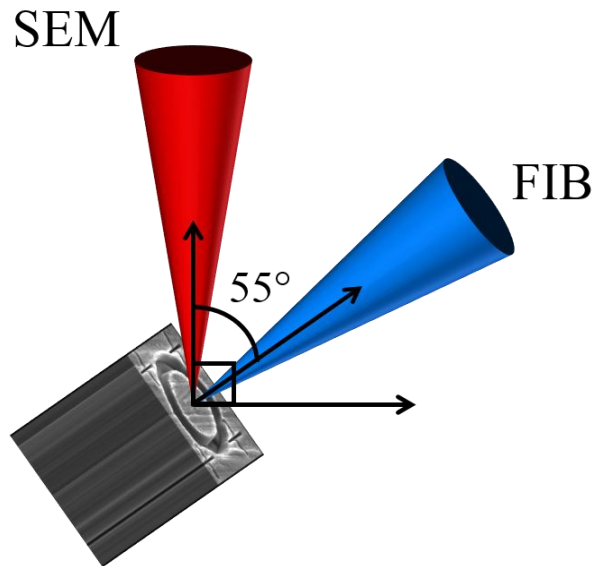


Figure 5.1 Configuration of SEM and FIB columns for ring-core milling.

The inner diameter and annular trench width (marked in IV in Figure 5.2(a)) of both ring-cores were $5\text{ }\mu\text{m}$ and $1\text{ }\mu\text{m}$. The two ring-cores were located reasonably far away from each other so the interference can be neglected. Four crosses were milled outside the outer circle of each ring-core as references to aid alignment and drift correction during the DIC analysis. Note that this is not recommended when FIB-DIC ring-core approach is utilised for residual strain evaluation since the crosses will affect the residual strain status within the ring-core island. The cross milling was adopted here to further eliminate the influence of drift correction so that the difference in the image correlation procedure is mainly a consequence of Pt deposition, which serves better the purpose of investigating the effect of Pt deposition in this section.

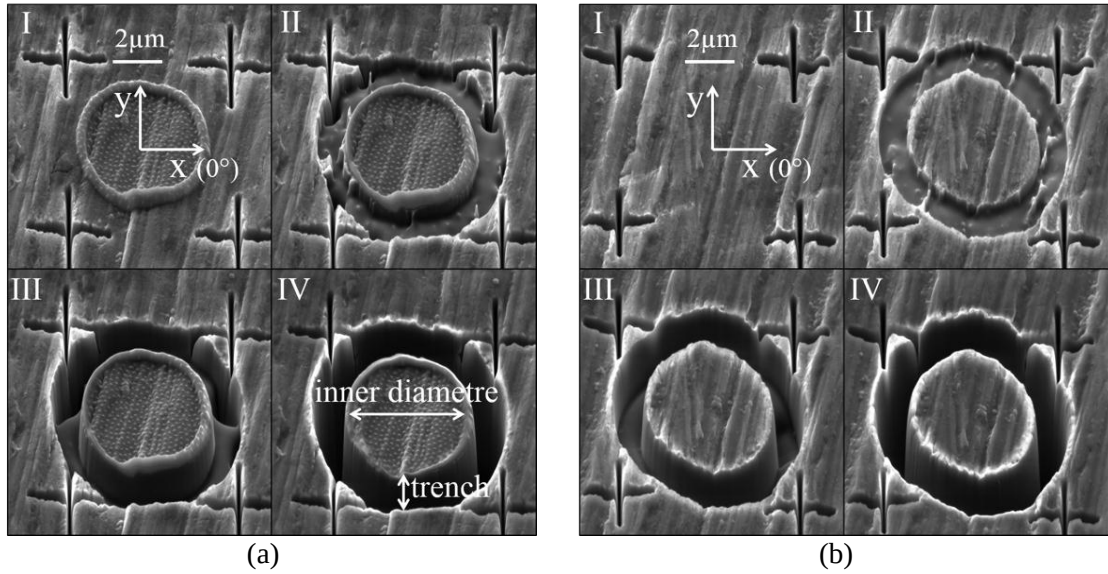


Figure 5.2 Process of ring-core milling: (a) Pt-deposited ring-core; (b) non-deposited ring-core.

The island area corresponds to the gauge volume within which strain relief takes place and, therefore, is the region of interest (ROI) for DIC analysis. The milling process for both ring-cores were identical. FIB was used to conduct the gradual material removal within the trench incrementally. Milling step size was 100 nm (in depth) and 50 steps were conducted in total. In the end, the total depth of the trench milled was $\sim 5 \mu\text{m}$ for both ring-cores. Between every milling step, FIB was paused for image taking. 10 images were taken before milling started as well as for each milling step so that image averaging could be performed during post-processing. Image averaging gives one averaged image out of 10 images to reduce noise, which improves the reliability of DIC analysis. A sequence of 51 averaged images (1 image from initial state and 50 images during FIB milling) were obtained for each ring-core.

5.1.2.2 DIC Analysis

DIC analysis consists of mainly two steps: processing and post-processing. More details will be explained in section 5.2.2.1. The processing step includes two main parts: drift correction and displacement tracking. Drift correction is to correct the SEM image drift through aligning the sequence of images using the unchanging area outside the trench. The four crosses milled provide distinctive features for image drift correction. During

displacement tracking, ROI is examined to decide displacement field from which a strain profile can be obtained. Both of these steps are performed by tracking the displacement of ‘virtual’ DIC markers (markers, for short) located within the ROI. In this experiment there were in total 4488 markers within ROI for each ring-core. Post-processing involved ‘cleaning out’ poorly tracked markers by checking each marker’s relative displacement with respect to its neighbours. When the displacement of a marker is significantly larger than that of its surrounding markers, it normally means that its tracking is somehow deficient. Therefore, ‘cleaning’ is carried out by deleting markers whose relative displacement is larger than a specific value. This value mentioned is referred to as the threshold of marker ‘cleaning’ in the following discussion. Note that displacement and the threshold both have the unit of pixel. Displacement of a marker contains two values that are along x- and y-direction, respectively.

The quality of image matching during tracking is evaluated by the correlation coefficient (*cc*) which is equal to the maximum magnitude of the cross-correlation function. It ranges from 0 (no preferred correlation) to 1 (strong correlation). For each tracked marker, the *cc* value is calculated by comparing the current (deformed) image to the original image of the undisturbed sample surface. To examine the reliability and accuracy of DIC tracking, the *cc* value distribution was studied carefully.

5.1.3 Results and Discussion

Figure 5.3 depicts the relative displacement of markers along x-direction (defined in Figure 5.2) before and after the ‘cleaning’ operation. The threshold for ‘cleaning’ was set to be 2 pixels. The horizontal axis in Figure 5.3 corresponds to the milling step number.

It can be seen that the relative displacement of tracked markers on the non-patterned ring-core surface is as large as 15 pixels, whereas for the Pt-patterned ring-core the distribution of relative displacement is much narrower (mostly lying within 2 pixels). With

the cleaning threshold value of 2 pixels, the fraction of markers cleaned (named as marker cleaning ratio) for the Pt-patterned and the non-patterned ring-core was 0.33% and 40.05%, respectively. The overwhelming difference indicates that the tracking of a significant larger proportion of the markers from the non-deposited ring-core was far less reliable.

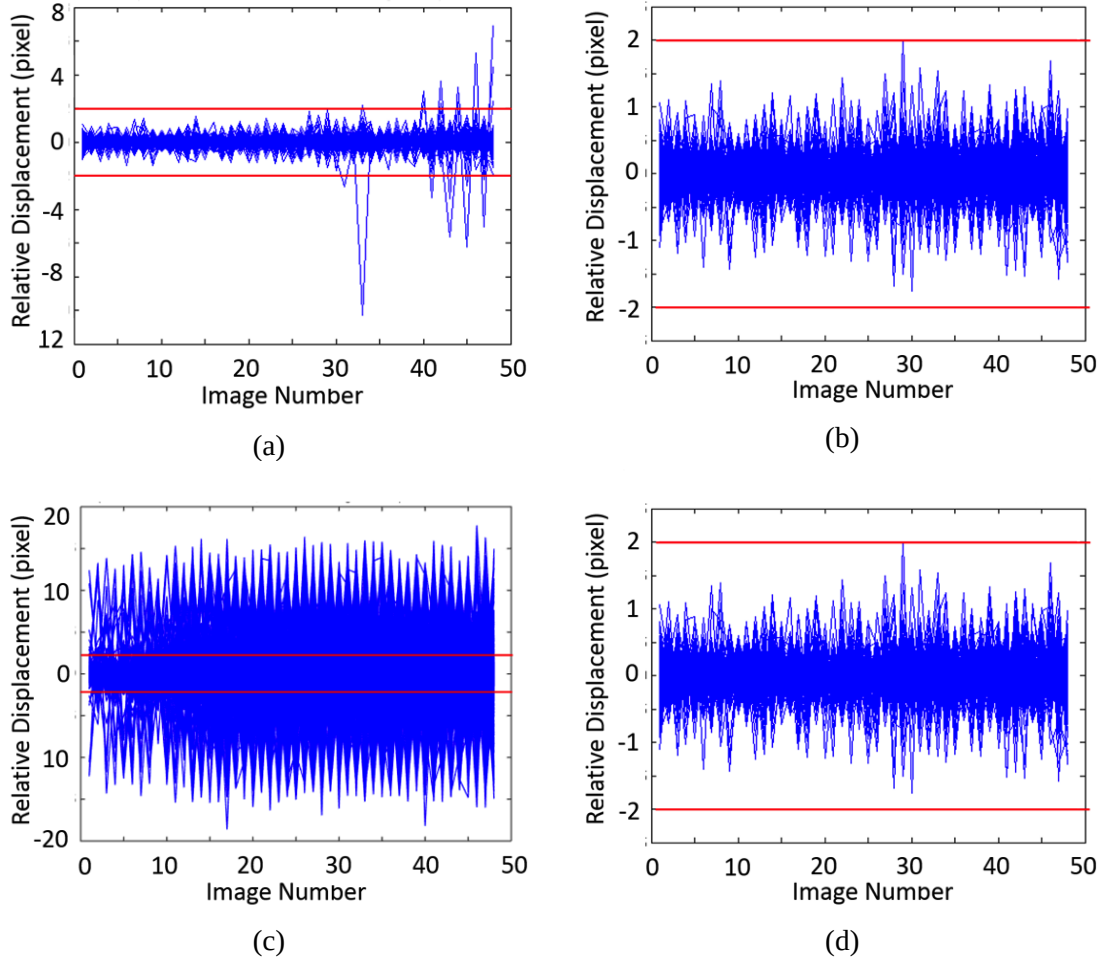


Figure 5.3 Relative displacement of each marker with respect to its neighbours and cleaning process with threshold value of 2 pixels: (a) Pt-patterned ring-core feature before marker ‘cleaning’; (b) Pt-patterned ring-core after ‘cleaning’; (c) non-patterned ring-core before ‘cleaning’; (d) non-patterned ring-core after ‘cleaning’.

To investigate further the marker tracking quality, a histogram plot of the cc for all the markers from both ring-cores is shown in Figure 5.4. The cc of the non-patterned ring-core displays a much wider distribution starting from approximately 0.6 while the distribution for the Pt-patterned ring-core is concentrated within a far narrower range

starting from ~ 0.95 . It is evident that the DIC analysis of the non-patterned ring-core had a much poorer marker tracking quality.

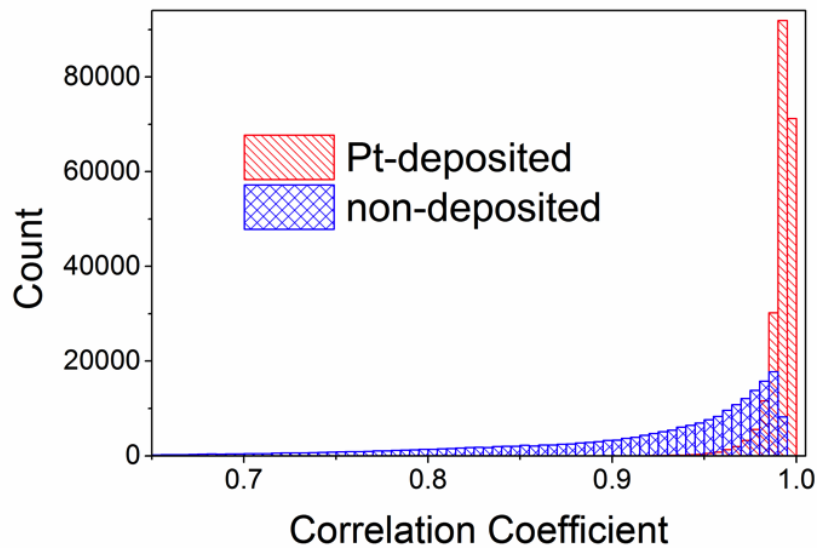


Figure 5.4 Histogram of correlation coefficient of all the markers on both of the ring-cores.

The quality of tracking is dependent on various factors, such as the accuracy of drift correction, the size of subset window and the correlation size (see more detailed definition in section 5.2.2.1 and Figure 5.6). However, the most important factor is the contrast of the sample surface: it is usually the lack of contrast (unique and ‘recognisable’ features) when the sample surface has not been suitably prepared that leads to poor tracking. To study the link between poor tracking and the lack of contrast in this case, a comparison of the distribution of remained markers after ‘cleaning’ for both ring-cores was made (Figure 5.5). For the Pt-patterned ring-core shown in Figure 5.5(a), only a small fraction of markers distributed at several small domains pointed by arrows were ‘cleaned out’. On the contrary, several large regions on the non-patterned ring-core surface indicated by the black meandering boundary in Figure 5.5(b) were removed in the ‘cleaning’ process. Figure 5.5(c) illustrates a high-magnification image of the central island of the non-patterned ring-core with the irregular outlines of poorly tracked regions. It is easily noticeable that all of these domains correspond to the locations where the sample surface is relatively ‘smooth’ and featureless (low contrast). At the position where the arrow is pointing in Figure 5.5(b)

and (c), no markers were removed, which is very different from its surrounding area where most of the markers were ‘cleaned out’. This is due to the presence of small features at that position that are shown at a higher magnification in the bottom right inset in Figure 5.5(c) indicated by the arrow. The existence of these small features increased the contrast and, therefore, allowed a better tracking quality of the markers placed at the position.

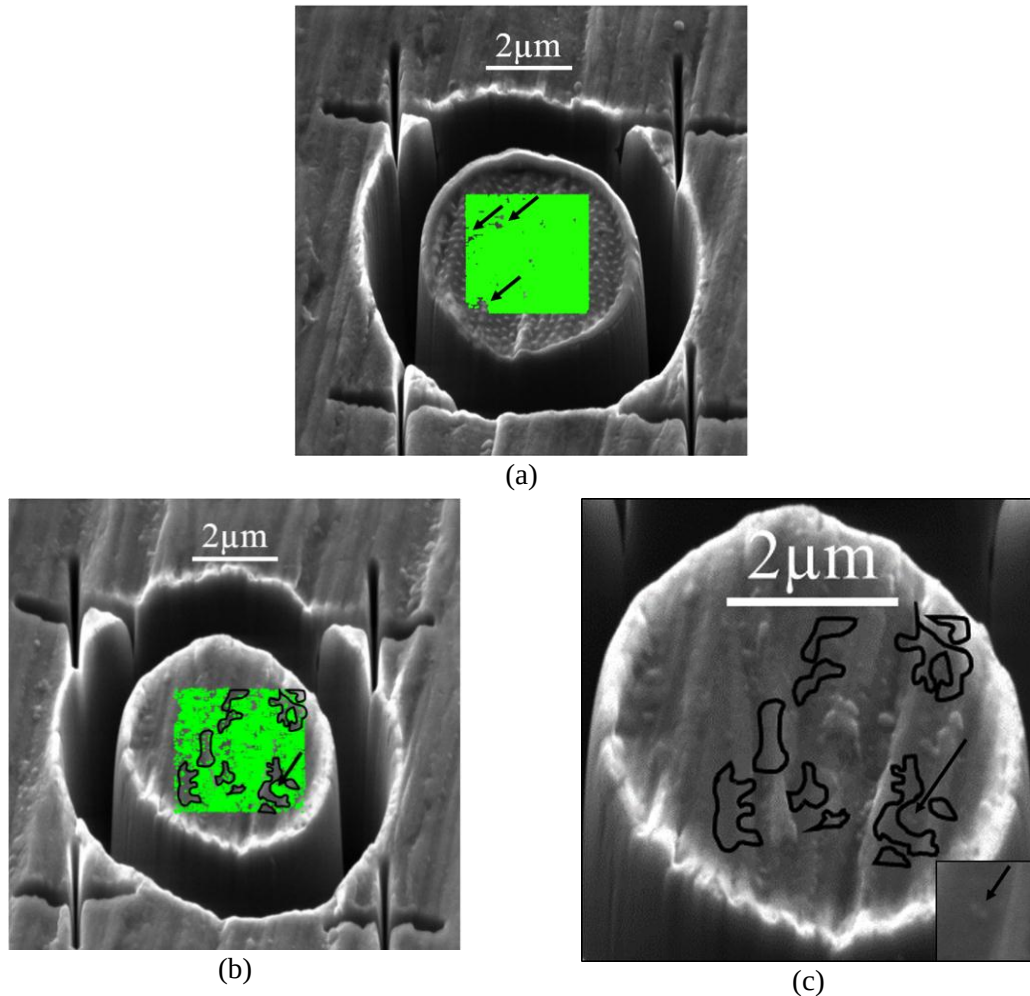


Figure 5.5 Comparison of the distribution of remained markers after ‘cleaning’ on the two ring-cores: (a) patterned ring-core; (b) ring-core without patterning; (c) a close-up of the region of poor tracking shown in (b).

Figure 5.5 is a direct evidence of the fact that the lack or presence of contrast is the main distinguishing aspect between poor and good tracking quality in DIC analysis. Pt nano-dot patterning can clearly play an important role in ensuring the quality of DIC analysis.

5.1.4 Conclusions

Two options for FIB-DIC ring-core method implementation were considered – with and without Pt nano-dot deposition on the sample surface within the ring-core island. FIB milling and SEM image acquisition of the sample surface were followed by DIC analysis. The quality of the analysis was evaluated. The results show that DIC analysis of images taken for the Pt-patterned ring-core resulted in a dramatically smaller distribution of relative displacement of markers, a significantly smaller marker cleaning ratio, an overwhelmingly narrower distribution of cc with much higher values. In contrast, many of the markers tracked at the surface of non-patterned ring-core have anomalously larger relative displacements, a larger marker cleaning ratio and smaller cc with a wider distribution.

Therefore, it is concluded that the contrast of the sample surface is the main reason that affects the marker tracking quality. Pt nano-dot deposition dramatically enhanced the surface contrast as well as the quality and reliability of the DIC analysis. It is suggested that Pt deposition can be utilised especially for samples with smooth surface to ensure the reliability and accuracy of DIC analysis.

5.2 XRD-DIC Development for Lattice Strain Evaluation

High-energy 2D powder XRD experiments are widely used for lattice strain evaluation. The 2D to 1D conversion of diffraction patterns is a necessary step used to prepare the data for full pattern refinement, but is inefficient when only the information of peak centre position is required for lattice strain evaluation. The multi-step conversion process is likely to lead to increased errors associated with 2D-1D conversion or fitting procedures. A new method is proposed in this section that relies on direct DIC analysis of 2D powder XRD patterns (XRD-DIC, for short). As an example of using XRD-DIC, residual strain values along the central line in a Mg AZ31B alloy bar after 3-point bending

(experiment described in section 3.3.2.1) are evaluated by using both XRD-DIC and the conventional ‘caking’ with fitting procedures. Comparison of the results for strain values in different azimuthal angles demonstrates excellent agreement between the two methods. The principal strains and directions are calculated using multiple direction strain data, leading to full in-plane strain evaluation. Therefore, it is concluded that XRD-DIC provides a reliable and robust method for strain evaluation from 2D powder diffraction data. The XRD-DIC approach simplifies the analysis process by skipping 2D to 1D conversion and opens new possibilities for robust 2D powder XRD data analysis for full in-plane strain evaluation.

5.2.1 Introduction

As mentioned in section 2.2.2, XRD is a powerful tool for lattice strain evaluation. In the present study we leave aside the single crystal Laue XRD configuration [289] and concentrate on polycrystalline powder XRD. The interaction between an incident collimated monochromatic X-ray beam and a polycrystalline aggregate or powder leads to a series of scattering cones that produce concentric Debye-Scherrer rings on a 2D detector mounted coaxially with the beam. Obtaining precise strain values requires careful interpretation of the 2D XRD data so that the radial peak position is determined with the accuracy approaching 10^{-4} or better. Developing reliable and efficient methods for 2D XRD pattern interpretation is one of the key remaining challenges in the area of XRD strain analysis.

Conventional data analysis is a multi-stage process that involves three main steps: calibration, conversion of the 2D pattern(s) into 1D profile(s) and Gaussian fitting for peak centre determination to calculate lattice strains.

In the first step, calibration is performed for sample-detector distance, image centre of diffraction pattern as well as corrections for the geometrical distortion due to detector's

orientation. Errors are inevitable during calibration and interpretation. They can arise when collecting diffraction pattern from the positioning of a standard calibration sample or in the subsequent calculation that involves data reduction and manipulation. This is the first error source. Microstrains normally feature sub-pixel ring movement. Therefore, it is very likely that errors become dominant and cover up the real value of residual strain when the magnitude of micro-level residual strain itself is not large enough.

For the second step, 2D diffraction patterns are converted into 1D intensity- 2θ profiles via ‘caking’ (see details in section 3.2.1 and Figure 3.3). Nevertheless, the ‘caking’ approach is not entirely straightforward as it requires defining sectors for azimuthal integration range. On top of this, the results from this analysis are based on an average of the whole section, which is imprecise in the case of textured sample (whose ring intensity is not uniform). This is the second error source.

Step three is to calculate strain values from peak position change on the 1D intensity- 2θ plot. Peak position is attained by Gaussian fitting of integrated profiles. Note that peak centre obtained here is in 2θ value. d -spacing, the distance between adjacent lattice planes, is related to θ according to Bragg’s law in Equation (3.1). The relationship between residual strain and d -spacing is given by Equation (3.3). Useful Gaussian peak fitting demands continuity and symmetry of peaks, which requires high quality Debye-Scherrer rings with high intensity and good uniformity. However, this is not always possible due to the existence of texture introduced in the processed sample or the lack of sufficient grain sampling by the beam (within the gauge volume). For instance, a 1D plot converted from a grainy diffraction pattern has poor quality peaks and this leads to inaccurate or even unsuccessful Gaussian fitting. This is the third error source.

For convenience, the 3-step method described above is referred to as ConFit (conversion + Gaussian fitting) method in the following text. The determination of the

absolute peak position involves errors from the three sources discussed above. The problems and limitations of the ConFit method come from the fact that conversion from the 2D diffraction pattern to 1D plot followed by fitting is required. The interpretation of each 1D plot provides complete information including peak position, peak width, peak shape and relative intensity. However, complete information is only sought in the more complicated full pattern analysis that is used to evaluate phase composition, texture, grain size and strains. In contrast, strain evaluation only relies on the relative change in the lattice parameter that corresponds to the radial shift of the ring segment in the diffraction pattern. Therefore, tracking the ring shift only is sufficient for strain evaluation and may be accomplished without the 2D to 1D conversion.

DIC can offer a means of determining the relative displacement between two or more 2D images. In recent years, DIC has been newly adopted for the treatment of Laue diffraction data. Petita *et al.* [290] presented Laue-DIC as a new method which is able to determine the Laue spot motion field and obtain the local elastic strain increment and the relative lattice rotation. Later on, enhanced Laue-DIC was developed by Zhang *et al.* [291]. The improvement of enhanced Laue-DIC is mainly reflected on two aspects: the capability of calculating a) the deviatoric elastic strain and orientation of the lattice and b) the calibration parameters. Nonetheless, to the best of our knowledge, so far no work has been reported that utilises DIC to analyse 2D XRD patterns for strain evaluation. In this section, XRD-DIC is proposed as a method of analysing 2D XRD pattern directly for full in-plane strain measurement using the DIC technique without 2D to 1D conversion. It has the advantages of being robust, easy to use and accurate. As an illustration, XRD-DIC is used here to analyse high-energy synchrotron 2D XRD data attained from an X-ray line scan along the central line of a CGPed magnesium alloy AZ31B bar after 3-point bending as described in section 3.3.2.1.

5.2.2 XRD-DIC Approach

5.2.2.1 DIC Implementation

DIC determines the displacement field of the ROI in a deformed image by comparing it with the reference image. A variety of DIC packages are available, such as Opencv [292], Ncorr [293] and DaVis [294]. The DIC package used herein is a Matlab compiled version developed by C. Eberl and M. Senn [295] which includes pre-processing, tracking and post-processing.

5.2.2.1.1 Pre-processing

In this compiled version, a sequence of images are saved into a file named ‘filelist’ allowing analysing multiple images in one DIC analysis. The first image in the sequence is called the base image, which acts as the reference image. The arrangement (e.g. evenly distributed in a rectangle area or on a line) and the position of markers can be defined by choosing ROI on the base image. The positions of each marker are then written in files named ‘gridx’ and ‘gridy’ with respectively x and y coordinates (unit: pixel) as its initial position. Figure 5.6 demonstrates some definitions related to markers in DIC. There are two markers (green dots) in Figure 5.6. Each marker occupies one pixel and is associated with a particular subset window. In the current analysis subset window was a square area with size 3×3 pixels.

Aside from placing markers, a parameter ‘Correlation Size’ (*corrsize*) needs to be set for DIC. It has a unit of pixel and limits the size of the ‘searching area’ (blue squares in Figure 5.6). Searching area is a square region around a marker within which the marker is being tracked. The size of the searching area is $2 \times \text{corrsize} + 1$, where 1 pixel comes from the pixel occupied by the marker itself. In Figure 5.6, *corrsize* is 10 pixels and the searching area is 21×21 pixels.

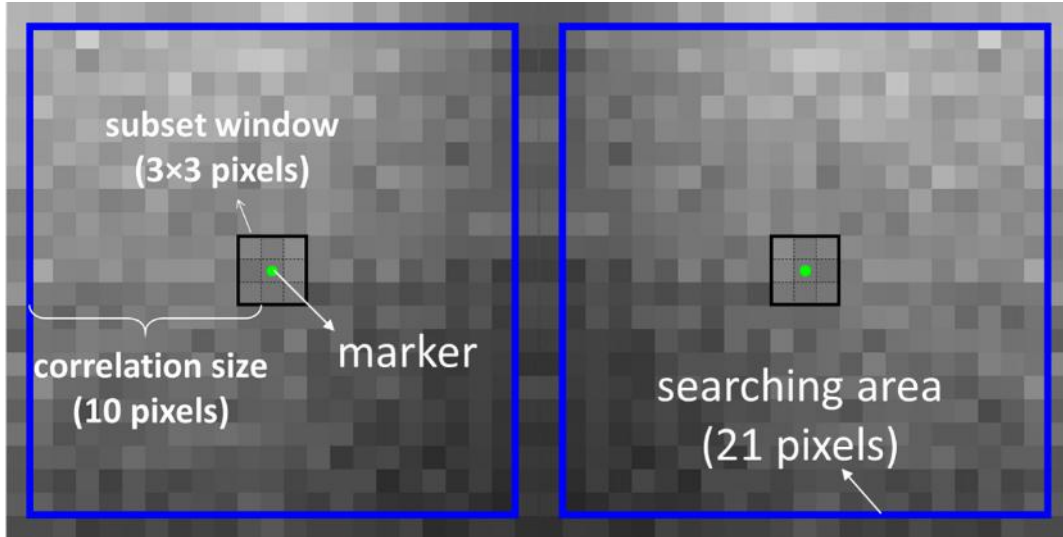


Figure 5.6 Schematic demonstration of definitions of subset window, marker, correlation size and searching area in the DIC package used.

5.2.2.1.2 Tracking and Post-Processing

In the tracking process, subsequent images are sequentially compared with the base image in order to determine the displacement field of the deformed ROI in each image. Displacement field is described with the displacement of each marker distributed in the ROI.

Each marker corresponds to its own 3×3 subset window. By moving the subset window of a marker around its searching area, the cross-correlation surface can be calculated, as shown in Figure 5.7. The details of cross-correlation surface calculation are explained in Ref. [115]. The new position of the marker is the peak position of the cross-correlation surface. Integer pixel accuracy can be determined by simply locating the coordinates that have the maximum value in the cross correlation surface, whilst sub-pixel accuracy is achieved by peak fitting of the cross-correlation surface. In practice the local shape of the correlation function landscape in the vicinity of the peak can be sufficiently well approximated by a low (e.g. second) order polynomial. This facilitates the determination of the peak position to a resolution typically better than 0.05 pixel [115]. The quality of the peak is expressed by the correlation coefficient as explained in section 5.1.2.2. Another important measure of the marker tracking effectiveness is the standard

deviation (sd) which gives a measure of displacement precision (more details can be found in Ref. [115]). Images with good contrast and low noise lead to sharp peaks on the cross-correlation surface with cc magnitude close to 1 and small peak width (a low sd), which results in small errors in the evaluation of the relative displacement between images. The new position of each marker on a series of deformed images is recorded and its relative displacements (both in the x - and y - direction) is obtained by comparing the marker to its initial position in the base image.

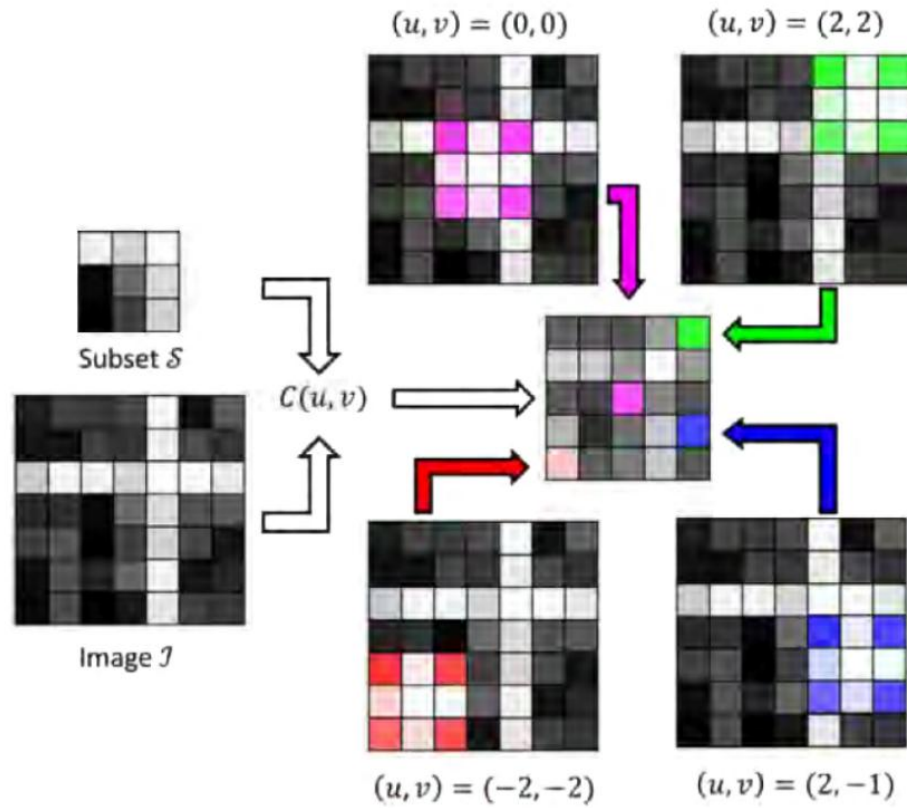


Figure 5.7 Schematic illustration of the process of obtaining 2D cross-correlation surface $C(u, v)$ between image J and subset S : correlating the subset S around within the image J gives the cross-correlation surface $C(u, v)$ (shown as the 5×5 matrix on the right side). Each pixel on $C(u, v)$ has a different (u, v) recording the relative offset of the subset window with respect to its original position.[115].

If a marker has a relatively large sd , small (near zero) cc or large displacement with respect to its neighbours, it means the quality of tracking is poor. For this reason, marker ‘cleaning’ is performed to eliminate those exceeding a particular threshold. In the current

case, only 13 out of 279 markers (4.66%) were removed ('cleaned out') due to ineffective DIC tracking. This also indicates that the DIC results have good robustness.

The application of DIC on 2D XRD patterns (XRD-DIC) is outlined in detail below via an example of evaluating residual strains along the central line of a sample after 3-point bending.

5.2.2.2 Advantages of XRD-DIC

The major advantage of using DIC to analyse 2D powder XRD patterns (Debye-Scherrer rings) is that it can avoid 2D-1D conversion and the succeeding Gaussian fitting. Therefore, errors due to calibration, conversion and fitting can be minimised. Moreover, the effort-intensive radial-azimuthal binning exercise 'caking' is no longer needed to attain strains in different directions. Instead, in the XRD-DIC approach, the lattice strain along a specific azimuthal direction can be evaluated by tracking the ring segment in this direction, extracting the radial component of displacement and having it divided by the original ring radius to quantify lattice strain. XRD-DIC is more convenient, more straightforward and even potentially more precise since 'caking' typically performs binning and averaging within an entire sector whilst XRD-DIC only seeks to interpret information about the immediate vicinity of the selected markers. Furthermore, full in-plane strain analysis can be readily accomplished [296] in one DIC analysis by selecting as many ring segments as necessary and calculating strains of different directions from which principal directions and principal strains can be determined.

5.2.3 XRD-DIC Analysis

In this section, data from XRD experiment of a bent bar (described in section 3.3.2) are analysed both using the conventional ConFit method and the XRD-DIC approach. Comparison is made to investigate the reliability of the XRD-DIC approach. The detector used for the experiment has a matrix of 2048×2048 pixels. With the smallest reliably

detectable ring shift of 0.05 (typical for sub-pixel accuracy DIC), the accuracy of lattice strain evaluation can reach $\sim 10^{-5}$.

Image processing was applied before automated tracking is performed in order to optimise image quality for the subsequent DIC analysis. This is necessary as the DIC process relies on comparing the intensity of different images, which puts a high demand on maintaining the background intensity throughout the XRD experiment. In this study, it was evident that electronic noise (dark field) of the detector had impact on the results obtained and, therefore, was necessary to be dealt with during the image processing stage. A single dark field image was subtracted from each pattern of the XRD line scan in order to correct for variations in detector sensitivity. Furthermore, image filtering was performed using '2D Gaussian' and 'Contrast' filters. Both of the filters are available in the MathWorks Image Processing Toolbox [297] and were embedded in the DIC code used [295].

DIC was performed on the 3rd ring of the diffraction pattern that corresponds to crystallographic plane normal (10 $\bar{1}$ 1). The first image of the sequence of 180 diffraction patterns was used as the base image from which all marker displacements were calculated. The markers were 'manually' placed by calculating their initial coordinates using the ring radius and azimuthal angle (β) and saving them into 'gridx' and 'gridy' files. Figure 5.8 illustrates the positioning of the markers on the base image. Eight ring segments were chosen, corresponding to the azimuthal angles 0 °; 45 °; 90 °; 135 °; 180 °; 225 °; 270 ° and 315 °; respectively. For each ring segment, 31 markers were uniformly placed within the central angle range of 1 °. A image with higher magnification of the markers positioned at azimuthal angle 90 ° is shown in the top right inset of Figure 5.8 as an example. The shift along x- and y- direction of each marker was determined using DIC and converted into radial shift and then lattice strains based on Equations (3.1) and (3.3) (more details will

be given below). The complete range of strain values served as input for the computation of the full in-plane strain tensor.

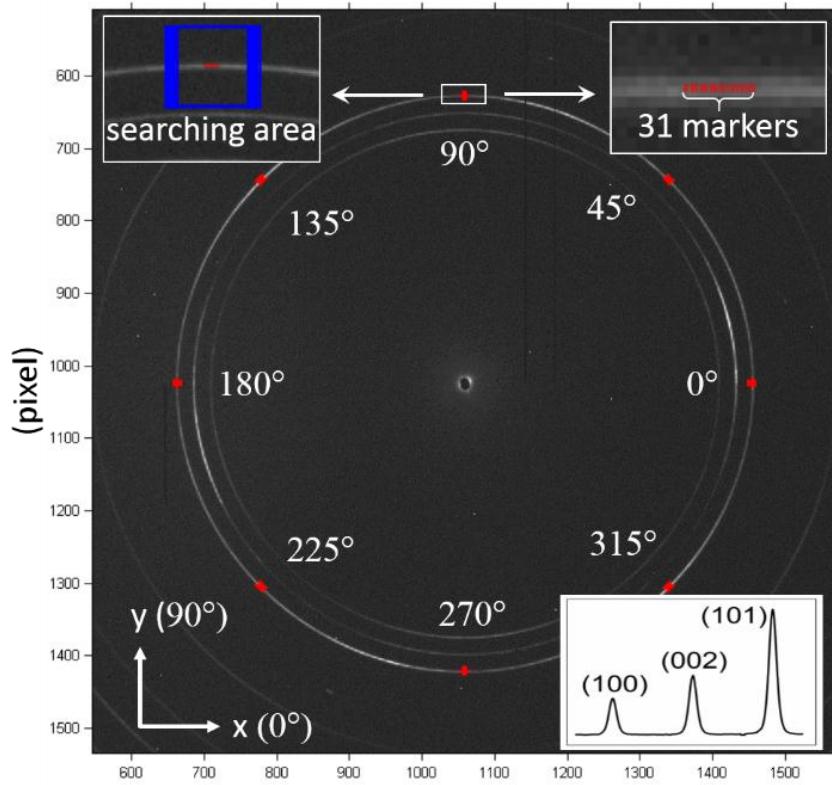


Figure 5.8 DIC setup illustrated for the base image with markers placed at different azimuthal angles on the $(10\bar{1}1)$ diffraction ring. The markers and the searching area for the 90° azimuthal angle are shown at a higher magnification in the inserts as an example.

In the DIC analysis performed, the *corrsz* was set to 20 pixels, which corresponds to a square searching area of 41×41 pixels. The principle of choosing *corrsz* for XRD-DIC analysis is to ensure that each marker could be tracked in a large enough area while avoiding aliasing (false matches) that arise from other high intensity features (other rings in this case) within the searching area. The searching area for the markers located at 90° is illustrated in the top left inset of Figure 5.8. It can be seen that the searching area covers all the ‘empty’ space between the 2nd ring and the 3rd ring while excluding the 2nd ring.

The output of the XRD-DIC analysis is the new matched shifted coordinates of each marker. By comparing a marker’s new position with its initial position, the radial displacement can be derived. The radial displacement of the 3rd ring on each image $\overline{dp}$ is calculated by averaging the radial displacement of markers:

$$\overline{dp} = \frac{1}{n} \sum_{i=1}^n \sqrt{(p'_i - p_i)^2} \quad (5.1)$$

where p'_i and p_i denote the marker radial position in the current image and the base image, respectively.

According to Equation (3.1) and (3.3), the equation for elastic lattice strain calculation is as follows:

$$\varepsilon = -\frac{1}{2} \frac{\left(\frac{p_0}{D}\right)^2}{\sqrt{1 + \left(\frac{p_0}{D}\right)^2} - 1} \cos^2(2\theta) \frac{dp}{p_0} \approx -\frac{dp}{p_0} \quad (5.2)$$

where p_0 (mm) is the radial distance between the ring and the image centre in the strain-free condition (similar to the role of d_0 in Equation (3.3)), D (mm) is the sample-detector distance and 2θ is the angle between the incident and the diffracted X-ray beams. Note that the pixel area is equal to $0.2 \text{ mm} \times 0.2 \text{ mm}$, so p_0 has mm as unit. In the current experimental configuration, θ is as small as $\sim 1.8^\circ$. Therefore, small angle approximation $\cos 2\theta \approx 1$ can be used to simplify strain calculation. Note that in this section p_0 and d_0 were both decided by placing the strain of the central position of the line scan to zero since the middle point on the sample is likely to be close to strain-free state.

The strain uncertainty for each azimuthal angle was estimated based on the sd value which was obtained using the approach described in Ref. [115]. These values are used to generate the error bars shown in the strain profile plot in Figure 5.9 for different azimuthal angles.

5.2.4 Results and Discussion

The accuracy of the XRD-DIC technique can be validated by comparison with the conventional ConFit analysis. Figure 5.9 illustrates the comparison between strain results obtained using both methods at the azimuthal angles 0° , 45° , 90° , 135° , 180° , 225° , 270° and 315° respectively. The horizontal axis is the distance across the sample. The results

from ConFit and XRD-DIC are shown as black solid line and red dot markers with the associated error bars, respectively. Fit2D [298, 299] and Matlab were used for the conversion from 2D to 1D patterns and Gaussian fitting respectively for the ConFit method.

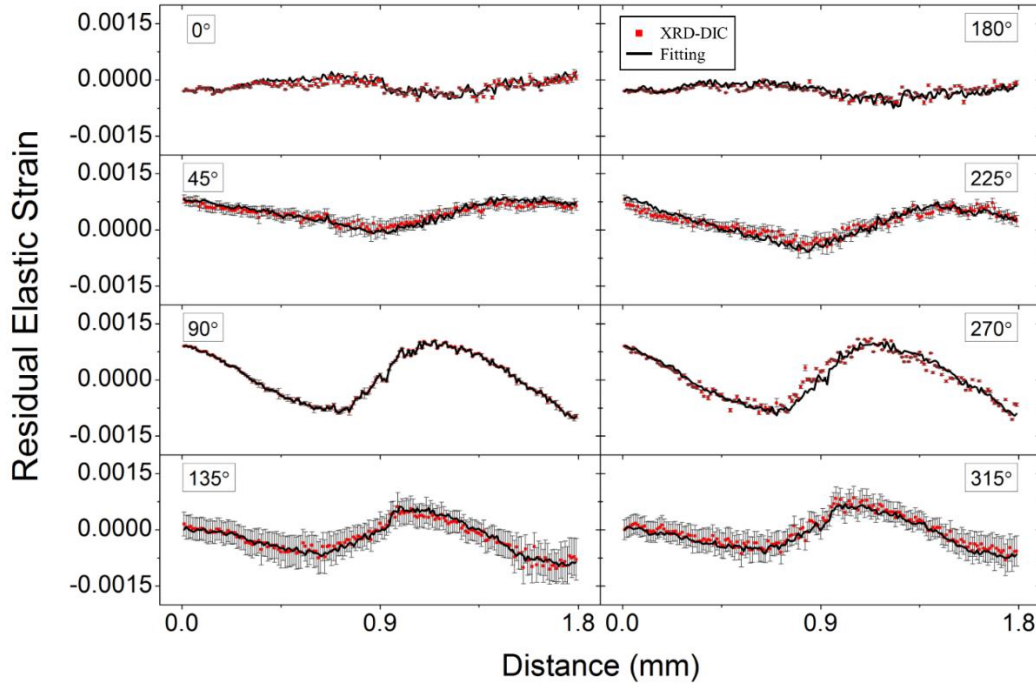


Figure 5.9 Comparison of residual elastic strains obtained from XRD-DIC (red dots with error bars) and ConFit method (solid black curve) at the azimuthal angles of 0 °, 45 °, 90 °, 135 °, 180 °, 225 °, 270 ° and 315 °, respectively.

The XRD-DIC results clearly show good agreement with the results from the ConFit method even when the absolute value of strain is relatively small as is the case for the angles of 0 ° and 180 ° where strains fall below 5×10^{-4} . It is interesting to observe that the error of strain determination varies for different angles. For example, the error bars are larger at azimuthal angles 45°, 135°, 225° and 315° than that of the other (‘square’) angles. This effect may be associated with the nature of the DIC analysis process, but may also be affected by the presence of texture within the sample.

Whilst only a 1D line profile is obtained for the scattering vector located in the sample normal plane in the conventional lab XRD, high-energy transmission XRD with 2D detector gives the scattering vector directions form a conical surface with the half angle very close to 90 °. This corresponds to scanning the scattering direction around 360 ° in the

plane of the sample including the LD (horizontal), TD (vertical) and all the intermediate directions, which are all recorded in one 2D Debye-Scherrer ring pattern. In principle, the knowledge of three non-collinear strain values is enough to determine the in-plane principal strains and directions through fitting using Equation (5.3). However, fitting here relied on data from 8 azimuthal angles to improve the robustness of interpretation.

$$\varepsilon(\beta) = \frac{\varepsilon_1 - \varepsilon_2}{2} \cos[2(\beta - \beta_0)] + \frac{\varepsilon_1 + \varepsilon_2}{2} \quad (5.3)$$

In Equation (5.3), β is the azimuthal angle and ε_1 and ε_2 are the unknown principal strains. In an ideal 3-point bending experiment, 0° and 90° in the current coordinate system should be the principal directions. However, this is not always true in reality because of reasons such as the misalignment of the sample during the XRD experiment. To account for this potential offset, the variable β_0 was introduced into Equation (5.3) as the offset angle and least squares fitting was used to determine an estimation.

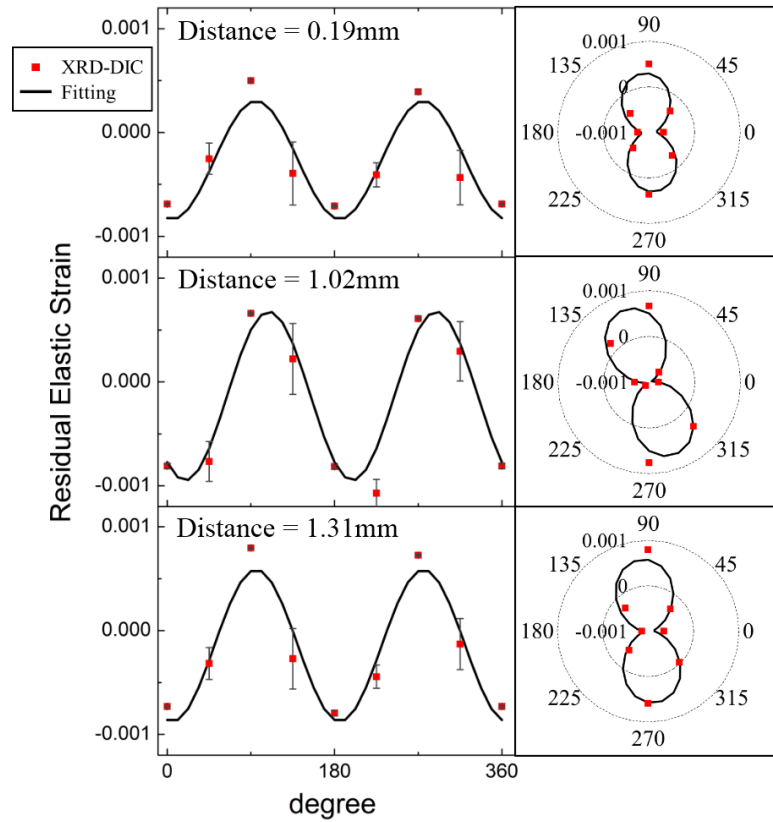


Figure 5.10 The azimuthal variation of residual elastic strains obtained from XRD-DIC from distance 0.19 mm, 1.02 mm and 1.31 mm as examples, showing the results of least squares fitting with the corresponding polar plots.

Figure 5.10 demonstrates the XRD-DIC strain estimates (red dots) and fitting result (black solid line) as a function of azimuthal angle for distance 0.19 mm, 1.02 mm and 1.31 mm (corresponding to images 19, 102 and 131) as examples, along with the corresponding radar plot. The values of β_0 for distance 0.19 mm, 1.02 mm and 1.31 mm are 5.6°, 19.2° and 5.6°, respectively.

Figure 5.11 shows the ‘quiver plot’ of XRD-DIC residual strain results including the principal strain magnitude and direction. The black curve indicates the residual elastic strain along the 90° azimuthal direction calculated by XRD-DIC. One of the principal strains is represented by red (ε_2) arrows, whose length and direction indicate the magnitude and direction of the principal strains. The other principal strain ε_1 is always perpendicular to ε_2 . ε_1 is not shown since the vectors are almost parallel to the horizontal axis and cannot be seen clearly.

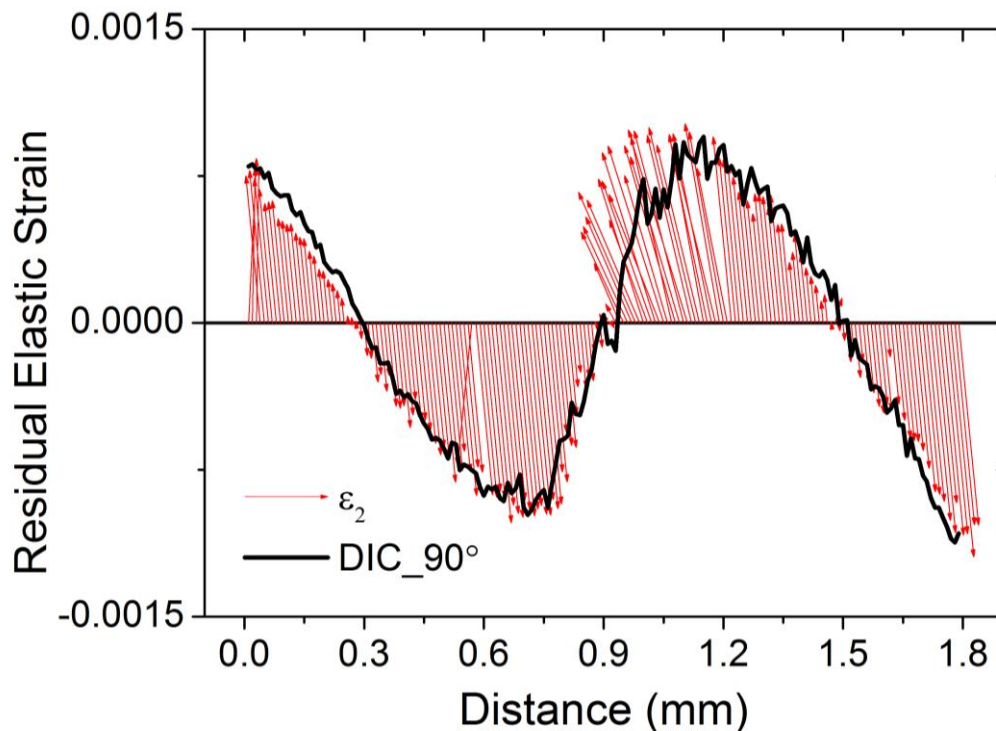


Figure 5.11 Quiver plot of one of the in-plane principal strains (red arrows showing the direction and the magnitude) across the central line in the bent bar plotted together with the residual elastic strain at 90° calculated using XRD-DIC (black curve).

5.2.5 Conclusions

A new approach for the analysis of 2D XRD data has been proposed, developed and implemented, which is referred to as XRD-DIC. It offers a convenient and robust way of analysing XRD data for lattice strain evaluation. The advantages of XRD-DIC stem from the fact that the conversion of 2D patterns to the equivalent 1D intensity- 2θ profile and fitting for peak position determination are no longer necessary. Instead, 2D patterns are analysed directly to monitor the radial shift of the Debye-Scherrer rings by correlation image tracking. As a result, errors are minimised. Meanwhile, ‘caking’ operation for obtaining the strains at different azimuthal angles that is required in the conventional ConFit approach is obviated when using XRD-DIC. Lattice strains can be obtained at as many angles as needed using this approach in one DIC analysis, preparing the data input for full in-plane strain evaluation. The strain results attained from XRD-DIC and ConFit approaches demonstrated good agreement, even when low magnitude (5×10^{-4}) strains were examined. The principal directions of strain in a 3-point-bent bar were aligned with the axial and TD with a small angular difference. The full in-plane strain state at each point of the scan line was obtained by fitting the complete strain data extracted to the azimuthal strain variation equation that corresponds to Mohr’s circle construction.

It is concluded that XRD-DIC offers a reliable and accurate approach for the analysis of 2D powder XRD data for the purpose of strain evaluation. The approach is beneficial in several aspects: it a) simplifies the analysis process by omitting the ‘caking’ conversion between 2D and equivalent 1D data, saving effort and minimising possible error sources associated with ‘caking’; b) allows evaluation of the full in-plane strain state efficiently.

6 Experimental Study of Twinning-Detwinning Mechanism

6.1 Deformation Asymmetry in CGPed Magnesium AZ31B Alloy

6.1.1 Objectives

As introduced in section 2.1.2, wrought magnesium alloys demonstrate a significant tension-compression asymmetry, particularly in yield stress. This is mainly a consequence of the polar nature of twinning deformation and the existence of rod texture after various stages of metal processing. The material used in this section is CGPed magnesium AZ31B alloy. The focus is mainly on probing the strain evolution in a bent bar of this material and analysing the difference in mechanical responses of grain groups aligned with different orientations in order to reveal the fundamentals of deformation asymmetry induced by twinning.

6.1.2 Results and Discussion

Data used in this section are from the XRD experiment of a bent bar described in section 3.3.2. A series of 180 diffraction patterns collected from X-ray line-scanning the sample were ‘caked’ with caking angle 30° along -0° and 90° -direction (shown in Figure 3.3 Conversion of 2D diffraction pattern into 1D profile with ‘caking’). Only the 1st and the 2nd rings were studied in this section.

Referring to Figure 6.1, using the 1st ring as an example, one unit cell is used to represent a grain group (the concept of ‘grain group’ was introduced in section 3.2.1 and in Figure 3.4) that is contributing to the intensity around 90° on the 1st ring. They are oriented in a way that their $(10\bar{1}0)$ plane normal is parallel to the direction of stress introduced by 3-point bending, meaning their basal planes are parallel to the LD and straining is along a-axis. Therefore, they will be referred to as the ‘a-group’ in the following discussion. In a similar way, the grains for the 2nd ring will be called ‘c-group’ since their (0002) plane normal (also c-axis) is parallel to the stress direction.

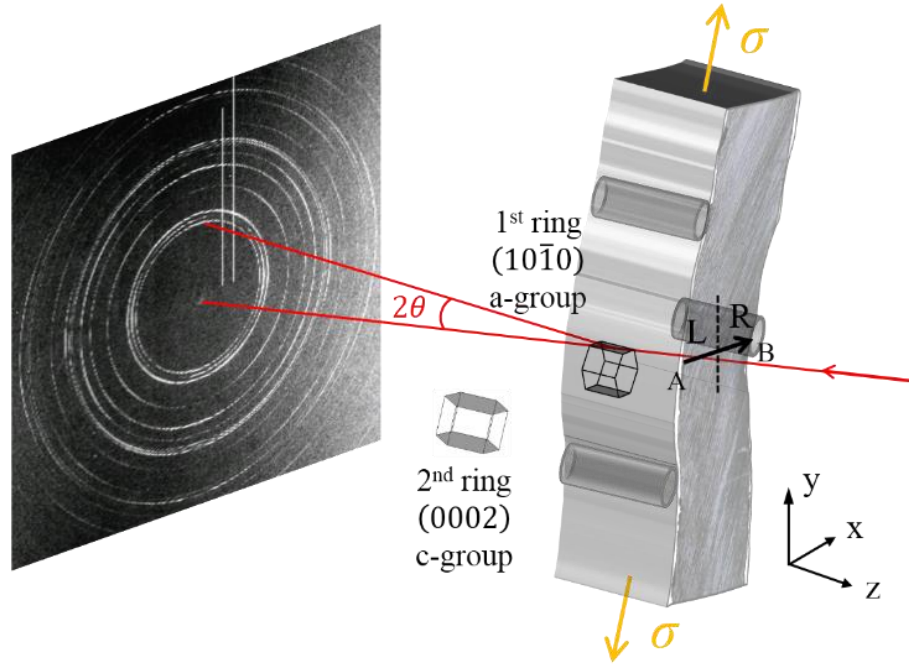


Figure 6.1 Schematic explanation of different behaviours of grains for a-group and c-group in XRD experiment

It was verified in section 5.2.4 that the principal axes of strain are aligned with laboratory x and y axes through plotting residual strains obtained from a sequence of caking orientations (0° , 45° , 90° , ..., 270° , 315° as shown in Figure 5.8) and fitting the plot with the strain transformation equation. Therefore, only the strains and stresses along 0° and 90° direction are studied here in detail. In the following discussion, for convenience, the sample (bent bar) is divided into two parts according to the deformation status: the left side is where tensile plastic deformation took place and compressive residual strain was accommodated afterwards (the side where the load was applied using two pins). The right hand side is the other half of the bar that was subjected to compressive plastic deformation during loading and was left with tensile residual strain after loading (the side with one pin). They are respectively marked as L and R in Figure 6.1.

Figure 6.2 indicates the residual elastic strains of both grain groups along 90° direction. Equation (3.3) was used to calculate strain from the diffraction pattern. d_0 was decided by placing the strain of the central position of the line scan to zero, since the middle point

on the sample is likely to be nearly strain-free state (same as how p_0 and d_0 were decided in section 5.2).

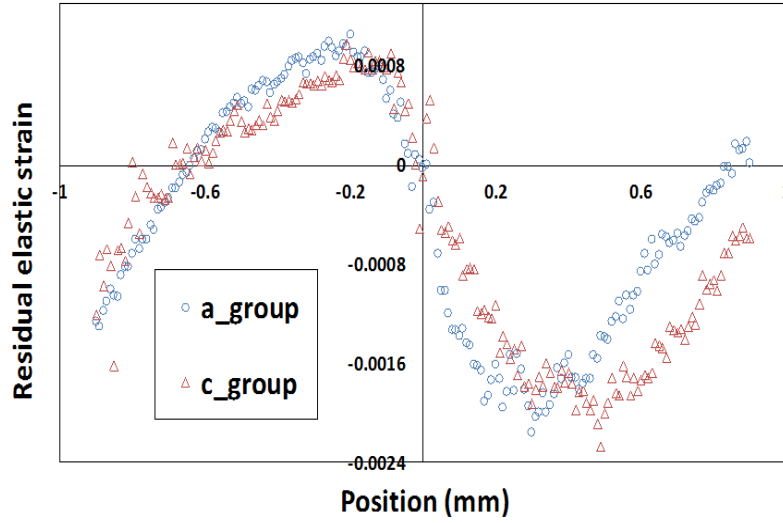


Figure 6.2 Residual elastic strains after the removal of loading of a -group and c -group across the beam measured via XRD.

Slip is the principal deformation mode for accommodating a -axis strain, whereas the activation of twinning is required to introduce strain along c -axis. On the left side the two curves exhibit a fairly good agreement, indicating that the grains in these two groups experienced little or no difference in plastic deformation when the sample was subjected to tensile loading. Therefore, slip and twinning demonstrate similar deformation behaviour under tension. On the right side, however, noticeable difference can be observed between the a - and c - groups. Asymmetry is also observed from c -group due to the polar nature of twinning system that plastic deformation is precluded under compression. Therefore, the most prominent difference in response is under compressive loading. This is consistent with the fact that twinning is preferably activated under tensile loading perpendicular to the basal plan or compressive loading parallel to the basal plan [300].

According to the Kirchhoff assumption of plane sections remaining plane, the total strain ($\epsilon_{\text{tol}} = \epsilon_{\text{el}} + \epsilon_{\text{pl}}$) should be linear across the beam [301, 302]. The linear variation of total strain can be deduced from linearly fitting the central region of the sample that remains elastic throughout the deformation and therefore can be found in the entire sample

by extrapolation. Subtraction of the residual elastic strain from the total strain gives the plastic strain shown in Figure 6.3(a). Noticeably, the behaviour of the plastic strain in the *a*-group and *c*-group on the right side differs, with larger amount of plastic strain accommodated in the *a*-group grains compared to the *c*-group. This is a consequence of the fact that the mechanism of plastic deformation in the *a*-group grains is slip, especially $\langle a \rangle$ basal slip, which is activated most easily. In contrast, in the *c*-group plastic strain is accommodated readily under tension through extension twinning, but inhibited in compression. The fact that plastic strain nevertheless is not zero on the right side is associated with grain rotation during deformation, which enables basal slip to cause plastic strain that has a projected component on the chosen direction, albeit smaller than that in the *a*-group.

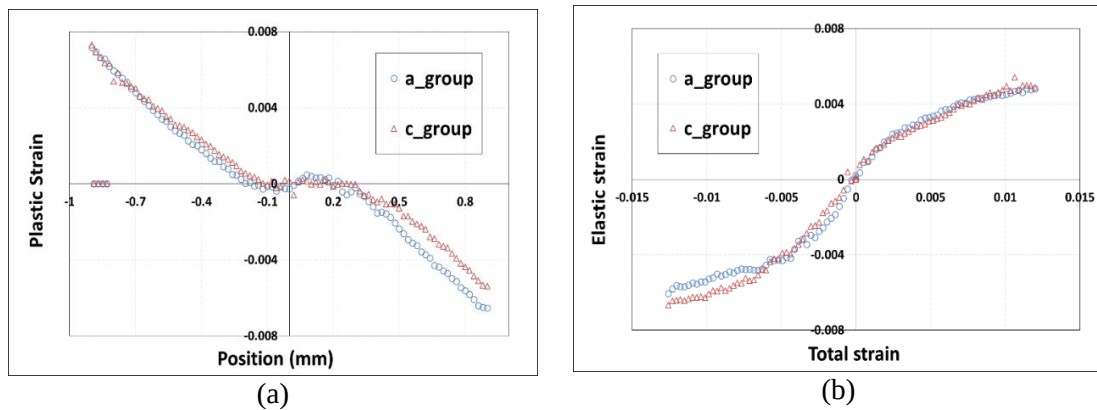


Figure 6.3 (a) Plastic strains of *a*-group and *c*-group along the central scanning line of the sample obtained through linear fitting of residual elastic strains and subtraction. (b) elastic strains vs total strains of *a*-group and *c*-group during loading.

When calculating the total elastic strain during loading, the strains introduced by bending moment should be taken into consideration, which is linear throughout the beam. Figure 6.3(b) demonstrates the plots of elastic strain vs total strain. Asymmetry of response can be observed here. It can also be seen that under compressive loading, these two groups are characterised by different ease of yielding: *a*-group grains exhibit easier yielding than *c*-group, which shows apparent hardening. This can be explained considering the ease of activation of slip and twinning in the system: slip is activated more easily than twinning

so it is easier for *a*-group to yield. In other words, grains that experienced loading parallel to the basal plane are more ready to deform plastically compared with those whose *c*-axis is parallel to the loading.

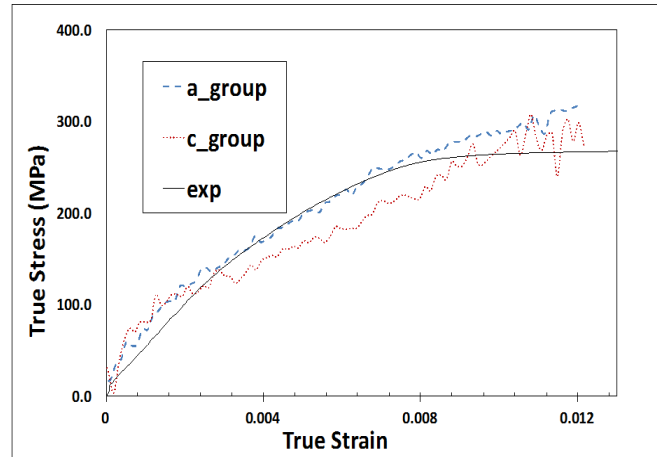


Figure 6.4 Stress-strain profiles of *a*-group and *c*-group with the comparison to the macroscopic curve.

In order to calculate the true stress along 90° direction across the beam using plane-strain assumption, the total strain along 0° direction is needed. Residual strain for 0° direction was obtained from XRD data. The corresponding stress was calculated using the sum of residual strain and the strain introduced due to Poisson effect. Comparison between the XRD results and the experimental stress-strain curve is given in Figure 6.4. The XRD results show satisfactory agreement with the macroscopic tensile test results.

6.1.3 Conclusions

By studying the residual elastic lattice strain introduced in a 3-point bending test, the asymmetry of tensile-compressive behaviour of *c*-group grains was revealed in CGPed magnesium AZ31B alloy. It was demonstrated that the activation of twinning under tensile loading along the *c*-axis results in efficient accommodation of tensile plastic strain, whilst this mechanism is inhibited under compression. This important distinction in the deformation mode affects the course of yielding and determines the subsequent residual stresses. For each grain group, the ease of yielding depends on the activity of specific deformation modes of slip or twinning, with grains most readily deforming plastically if

the LD is parallel to the basal plane. The residual and ‘live’ stresses were calculated using the plane-strain assumption and were compared with the stress-strain curve from conventional macroscopic mechanical test, showing good agreement.

6.2 Lattice Rotation Induced by Twinning-Detwinning

6.2.1 Introduction

This section focuses on the exploration of lattice rotation induced by TDT during plastic deformation under compression-tension and cyclic loading. Both *in situ* XRD experiments described in section 3.3.3 and 3.3.4 and *in situ* EBSD experiment in 3.4.2 are utilised to study lattice rotation and twin/detwin fraction. This study provides an insight into the reorientation process of TDT under different levels of external macroscopic loading.

6.2.2 Results and Discussion

6.2.2.1 Lattice Rotation

Figure 6.5 demonstrates the results from the *in situ* compression-tension XRD experiment that provides information about lattice rotation in a bulk material. Figure 6.5(a) is the loading history. A series of 2D XRD patterns were converted into 1-dimensional intensity- 2θ profiles through ‘caking’ between -15° to 15° , referring to the inset in Figure 6.5(a). Hence, information along 0° direction is extracted for Figure 6.5(b).

The height of the peaks was obtained from Gaussian fitting and taken as the intensity value. Figure 6.5(b) illustrates the intensity change of the 1st, the 2nd and the 3rd peaks respectively representing lattice planes $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$ (see Figure 3.4 for lattice plane miller index) as well as denoting different grain groups. For example, grains contributing to the 1st peak are those with the $(10\bar{1}0)$ plane normal parallel to the LD (Figure 3.8) and basal plane normal (c -axis in the unit cell) perpendicular to the LD. In a similar way, grains for the 2nd peak have their basal plane normal parallel to the LD. For a

better understanding of the following discussion, it is worth noting that ‘peak’ and ‘ring’ have different meanings in this thesis (referring to the explanation in section 3.2.1). According to the intensity change, the loading history is divided into stages I-V (marked at the top). Six loading steps are marked out as A-F (at the bottom). EBSD maps were taken at loading steps B, C, E and F (underlined).

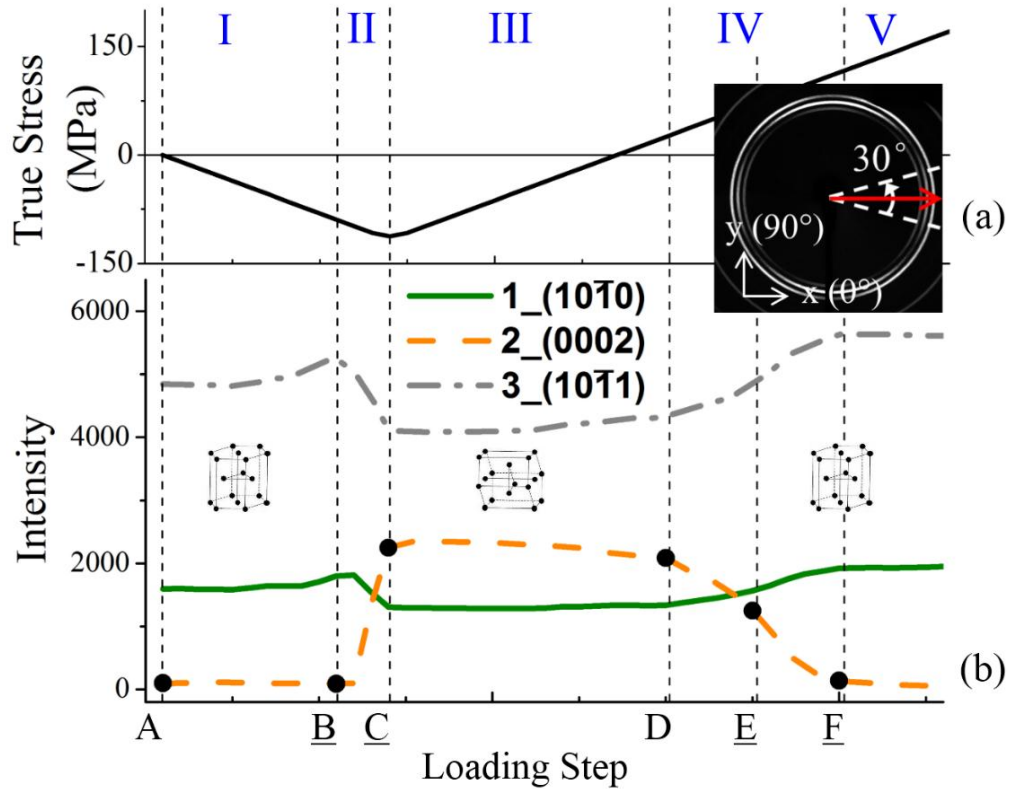


Figure 6.5 Demonstration of lattice rotation by in situ compression-tension XRD experiment results: (a) true stress from external loading; (b) intensity change of three diffraction peaks.

It is evident that the starting texture is rod texture (referring to the explanation in section 3.3.3) since the intensity of 2nd peak is nearly zero at the beginning of the loading. Considering that (10 $\bar{1}$ 2) extension twin is preferably activated under tension parallel or under compression perpendicular to *c*-axis, most of the grains are in favourable orientation for twinning when the sample is subjected to compression. Therefore, a sudden change in the intensity in segment II and IV in Figure 6.5(b) is very likely to be caused by twinning or detwinning. In segment II in Figure 6.5(b), the intensity of the 1st peak drops while that

of the 2nd peak ascends, demonstrating that some of the grains originally with their *c*-axis perpendicular to the LD are experiencing approximately 90 ° lattice rotation and end up with *c*-axis parallel to the LD. This is in line with the rotation behaviour that twinning has under compressive loading in this stage. Similarly, the intensity change in segment IV can be explained as a consequence of detwinning under tension since detwinning undergoes a reverse rotation compared to twinning. Since the intensity of the 2nd peak was almost zero at the beginning, it can be considered that the intensity of the 2nd peak in segment II, III and IV are fully from twins. It is worth noting that the intensity of the 2nd peak during unloading (segment III) remains almost the same and starts dropping when it reaches a certain level of reverse loading. This shows that once twinning has happened it will not detwin merely with the removal of external loading. This opens the possibility of *ex situ* characterisation of twinning when conditions for *in situ* experiment cannot be met.

The four measure points for EBSD mapping were decided according to the results in Figure 6.5(b). The inverse pole figure colouring maps given in Figure 6.6 were plotted from the perspective of x-axis (Figure 3.11) and the four maps from the 1st cycle can be compared with results from the *in situ* XRD experiment. Capitalised letters ‘B’, ‘C’, ‘E’ and ‘F’ refer to the measure points in Figure 6.5. Compressive loading is indicated as arrows in map C1_1. Arrows shown in map C1_2 indicate an example of the orientations of a parent matrix and its twinned region. In the following discussion a twin that persists on complete load reversal is called residual twin [36]. Obvious twinning and detwinning processes can be observed and a few residual twins remain in map C1_4. It is interesting to note that the vast majority of the twined regions are across the parent grains horizontally which is perpendicular to LD in the 2D map. However, it is not enough to conclude the geometric relationship between the LD and twined region without 3D EBSD. Additionally,

cross-boundary twins [303, 304] can be observed aside from twinning in an individual grain.

6.2.2.2 Twin/Detwin Fraction

The next step is to confirm the assumption that the intensity change is caused by twinning and detwinning, as well as to compare the XRD and the EBSD data. It is reasonable to assume that the absolute intensity of the peak is proportional to the volume of the grains that are contributing to it. Following this assumption, the twin fraction in the XRD experiment η_{XRD} is defined as the ratio of the intensity of the 2nd peak and the sum of the intensity values of all the three peaks in Equation (6.1):

$$\eta_{XRD} = \frac{I_2}{I_1 + I_2 + I_3} \quad (6.1)$$

where I_1 , I_2 and I_3 are the absolute intensity value of the 1st, the 2nd and the 3rd peaks. To compare with XRD experiment, it is imagined that the sample in the EBSD experiment is also being illuminated by an X-ray beam. Since the same coordinate system is used for both XRD and EBSD experiments, the imaginary X-ray beam should come from the z-axis (Figure 3.11) in the EBSD experiment. An EBSD map is a matrix of index points as mentioned in section 3.4.2 and analysing a map is essentially checking index points individually. If an index point has Euler angles which make its (0002) lattice planes satisfy Bragg's law, it is considered to 'contribute' to the intensity of the 2nd peak should there be an X-ray beam. The index points that could 'diffract' for the 1st and the 3rd peaks can be identified in the same way based on their Euler angles. The twin fraction in the EBSD experiment η_{EBSD} can be calculated as the ratio of the area of the index points for the 2nd peak and the sum area of the index points for the 1st, the 2nd and the 3rd peaks. The area of index points can be replaced by the number of index points as shown in Equation (6.2) since all the index points have the same area of 0.64 μm^2 :

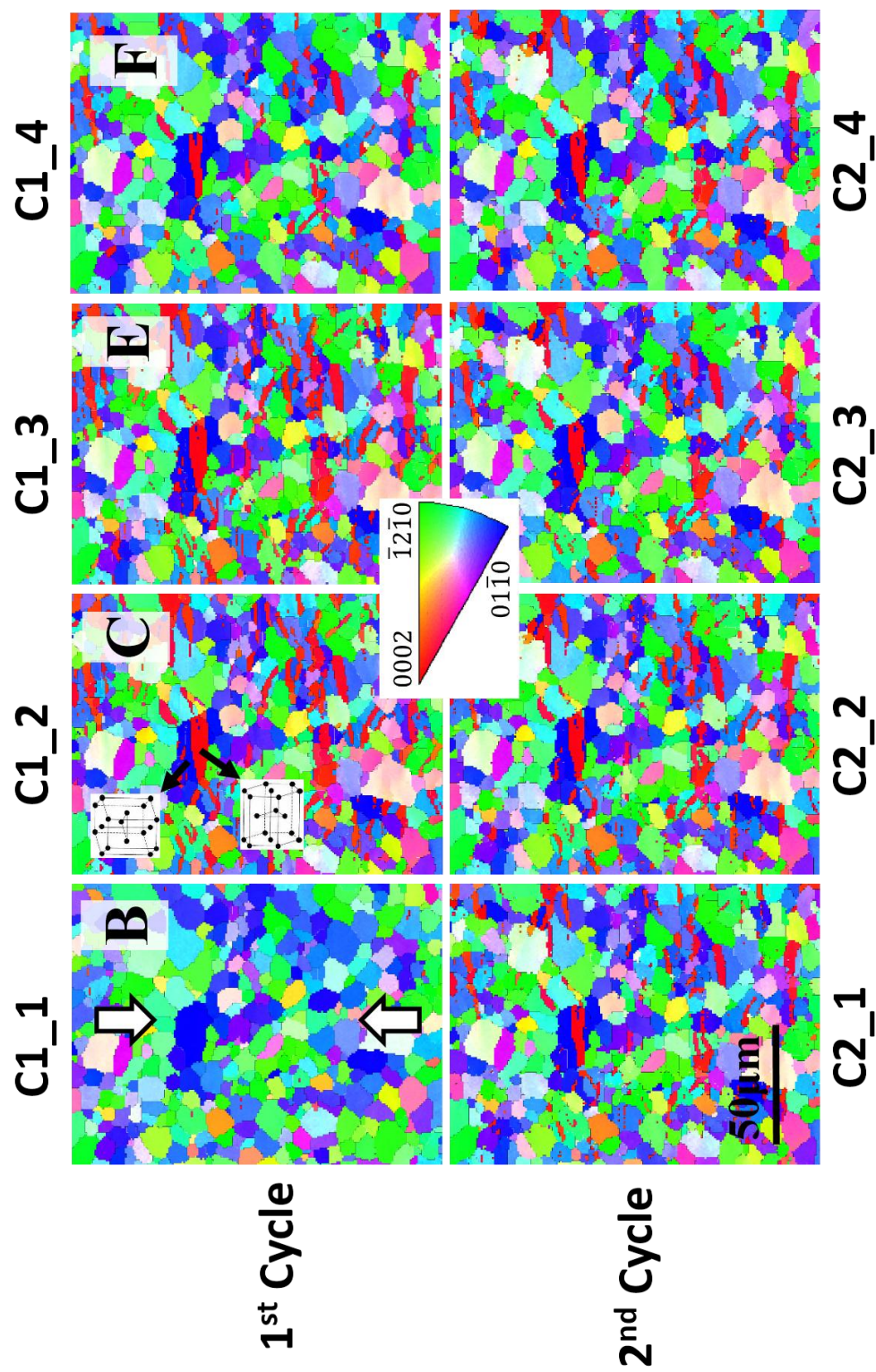


Figure 6.6 Inverse pole figure colouring maps obtained from cyclic in situ EBSD mapping.

$$\eta_{EBSD} = \frac{N_2}{N_1 + N_2 + N_3} \quad (6.2)$$

where N_1 , N_2 and N_3 are the number of index points ‘contributing’ to the 1st, the 2nd and the 3rd peaks, respectively. It is described below how to judge whether an index point is ‘contributing’ to the 2nd peak as an example. Demonstrated in Figure 6.7 is the geometrical relationship between the orientation of an index point (represented by a unit cell) and the imaginary X-ray (parallel to the z-axis). The coordinate system in Figure 6.7 is the same as that in Figure 3.11 for consistency, which is the sample coordinate system. The dog-bone sample is included in the figure to demonstrate the relationship between sample, mapping and the coordinate system. φ_1 is the angle between imaginary X-ray and the normal of (0002) planes of the unit cell (under sample coordinate system) while φ_1 is the angle between x-axis and the project of the normal of (0002) planes on the x-y plane. φ_1 decides whether the index point satisfies Bragg’s law. If it does, φ_1 should be the difference between 90 ° and θ in Bragg’s Law for the 2nd ring which is approximately 7.59 ° in this material. Error of ± 1.14 ° (15% of 7.59 °) is allowed when making judgement. φ_2 determines whether the diffraction spot sits on the ring segment for ‘caking’. In this case, the absolute value of φ_2 should be no more than 15 ° due to the caking angle of 30 °. If the two conditions above are both met, this index point will be counted as one that is ‘contributing’ to the 2nd peak. For the other two peaks, only the plane normal and θ value need to be changed accordingly. It is worth mentioning that $N_1 + N_2 + N_3$ is not the total number of the index points in the map as only those satisfying Bragg’s Law are counted in. Since the 2nd peak is mainly from twins, η_{XRD} and η_{EBSD} can be considered as twin fraction.

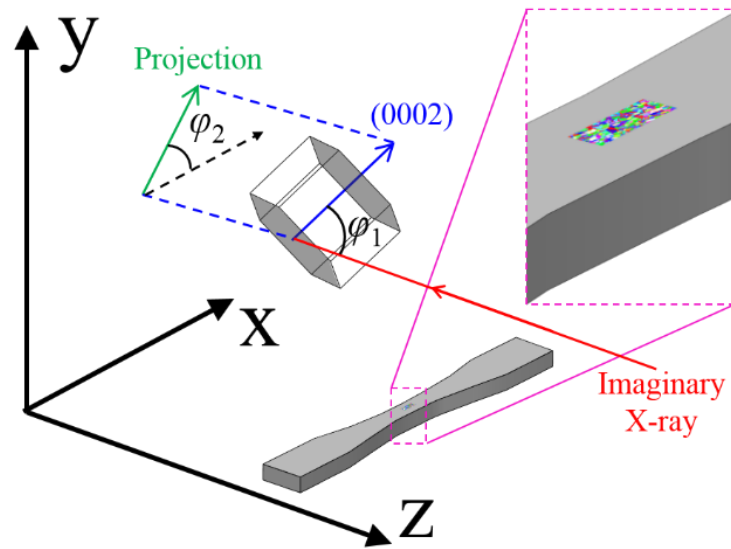


Figure 6.7 Geometrical relationship between the in situ EBSD experiment and the imaginary X-ray beam

Figure 6.8 is the comparison of twin fraction values from two experiments. A good agreement is achieved, which confirms that the abrupt change of intensity in Figure 6.8 is from the TDT process. Additionally, the agreement shows that the sample is homogeneous along the ND in terms of grain distribution and texture since the twin fraction shows little variation between XRD experiment for bulk material study and EBSD experiment for 2D sample surface examination.

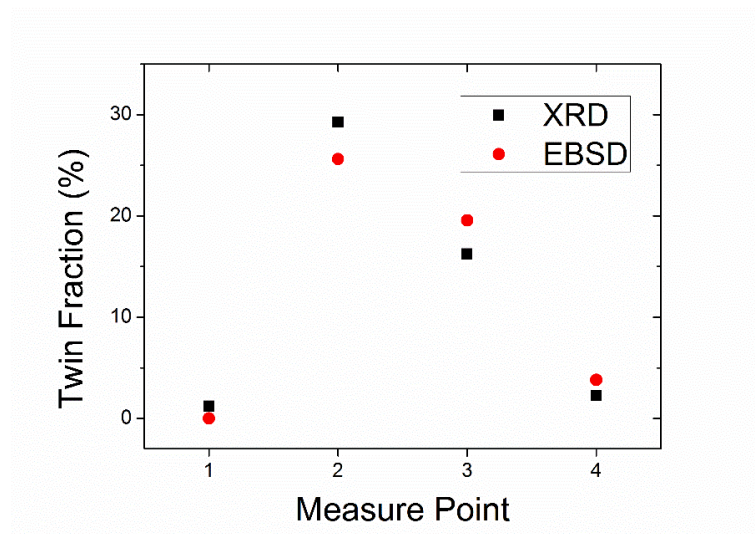


Figure 6.8 Comparison of twin fraction respectively obtained from XRD and EBSD

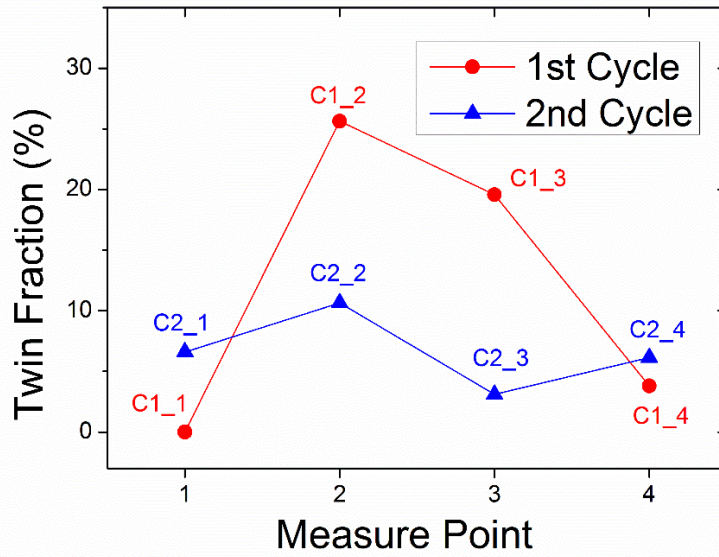


Figure 6.9 Comparison of twin fraction obtained from the 1st and the 2nd cycle of in situ EBSD.

Figure 6.9 demonstrates the twin fraction values for eight measure points from two cycles. The same twin fraction calculation approach used for Figure 6.8 is adopted here. It must be noted that the areas that eight EBSD maps cover are with subtle variations at the edges due to the distortion of the sample surface during cyclic loading. Therefore, the difference in twin fraction values between the eight maps can be to some extent affected. This might have also contributed to the difference between C1_4 and C2_1 which, in theory, should have the same twin fraction because there should be barely any twinning happening when unloading from maximum tension force to before the inception of twinning. However, it cannot be denied that a small quantity of twinning might still happen locally due to strain concentration. It is evident that the twin fraction is significantly smaller in the 2nd cycle compared to the 1st cycle meaning that less twins are activated in the 2nd cycle. One possible reason for this could be the hardening of twinning CRSS, which makes activating twinning more difficult. It is surprising yet interesting to observe that C2_3 has a smaller twin fraction than C2_4. This might be an ‘unfortunate’ coincidence that the sample at C2_3 actually has a larger proportion of twins compared to that at C2_4

but less of them ‘satisfy’ the ‘diffraction’ condition (Figure 6.7) at the sample surface so are not included in twin fraction calculation. This demonstrates that twins seem to have a more unpredictable rotation behaviour during the 2nd cycle, which leads to the less smooth transition in the twin fraction compared to the 1st cycle. This gives a hint on a greater deal of localised strain concentration in the 2nd cycle.

6.2.2.3 Cyclic Loading

The same data analysis approach regarding caking and Gaussian fitting used for the *in situ* compression-tension XRD was employed to analyse patterns from the *in situ* cyclic loading XRD experiment and the results are shown in Figure 6.10. The numbers marked in Figure 6.10(a) are the true stresses (unit: MPa).

Figure 6.10(b) shows the intensity change of the three peaks. Similar to section 6.2.2.1, the intensity in Figure 6.10(b) is the peak height from Gaussian fitting. Figure 6.10(d) is a more sophisticated contour plot with both intensity (refer to colour bar for intensity value) and peak position (2θ value, vertical axis) change. The sample has a certain number of grains with their *c*-axis parallel to the LD judging from the existence of the intensity of 2nd peak at the beginning. For convenience, those grains will be referred to as P group grains. Tension along the LD is able to activate twinning in P group grains and the order of tension followed by compression was adopted to provide variation in the experiment for more discoveries. Simply judging the absolute intensity value for the TDT process is not entirely accurate in this case as it is noticed that the intensity of 2nd peak is still changing when it should have maintained (e.g. from **f** to **g**). Therefore, the fractions of the 1st peak intensity and the 2nd peak intensity to the sum intensity of all the three peaks are shown in Figure 6.10(c) (similar to the way of calculating twin fraction in section 6.2.2.2). When tensile loading reached point **b**, twinning was triggered in P group grains and they started to rotate ending up with their *c*-axis perpendicular to the LD and to contribute to the intensity of the

1st (and 3rd) peak. The degree of twinning continues to rise until reaching the maximum loading force at point **c**. Intensity values maintain at almost the same level during unloading and have a slight change at point **d**. It is very interesting to observe the subtle, yet noticeable, intensity change between **d** and **e**. It is very likely that this is caused by detwinning in P group grains which have already twinned between **b** and **c** as it is well known that detwinning has lower CRSS than twinning. Large amount of rotation happens right after point **e** until point **f**. This is mainly due to twinning in grains originally with their c-axis perpendicular to the LD. In the meantime, the possibility cannot be excluded that a small amount of detwinning is still going at this stage. Again, between **f** and **g**, intensity change is neglectable. The intensity change in the second and the third cycles of tension-compression loading can be interpreted to understand lattice rotation in the same way. However, the difference is that in the second and the third cycles only re-twinning and re-detwinning are happening.

Comparing **h-i** with **c-e**, a subtle change as **d-e** is not observed. Additionally, rotation triggered at point **i** has almost the same true stress level (-94.4 MPa) as point **d** (-94.4 MPa) instead of point **e** with a stress value of -200.6 MPa. This evidence strongly supports the speculation about detwinning happening in the twinned grains in P group between **d** and **e** because detwinning has a lower CRSS compared to twinning (explained in section 2.1.2). On the other hand, -200.6 MPa at point **e** theoretically should be almost the same as 236 MPa at point **b** in terms of absolute value as they both correspond to the beginning of twinning. However, there is a 15% difference between them, which might be a consequence of the ongoing detwinning. Hence, it is reasonable to consider that the absolute values of true stress for twinning and detwinning to happen for the first time are 236 MPa and 94.4 MPa, respectively. Note that the values of true stresses here for activating twinning and detwinning are only an estimation because there was a gap in

loading force between loading steps. For example, the true stress was 217.1 MPa for the step before 236 MPa and was 264.3 MPa for the step after, meaning twinning was activated between 217.1 MPa and 264.3 MPa.

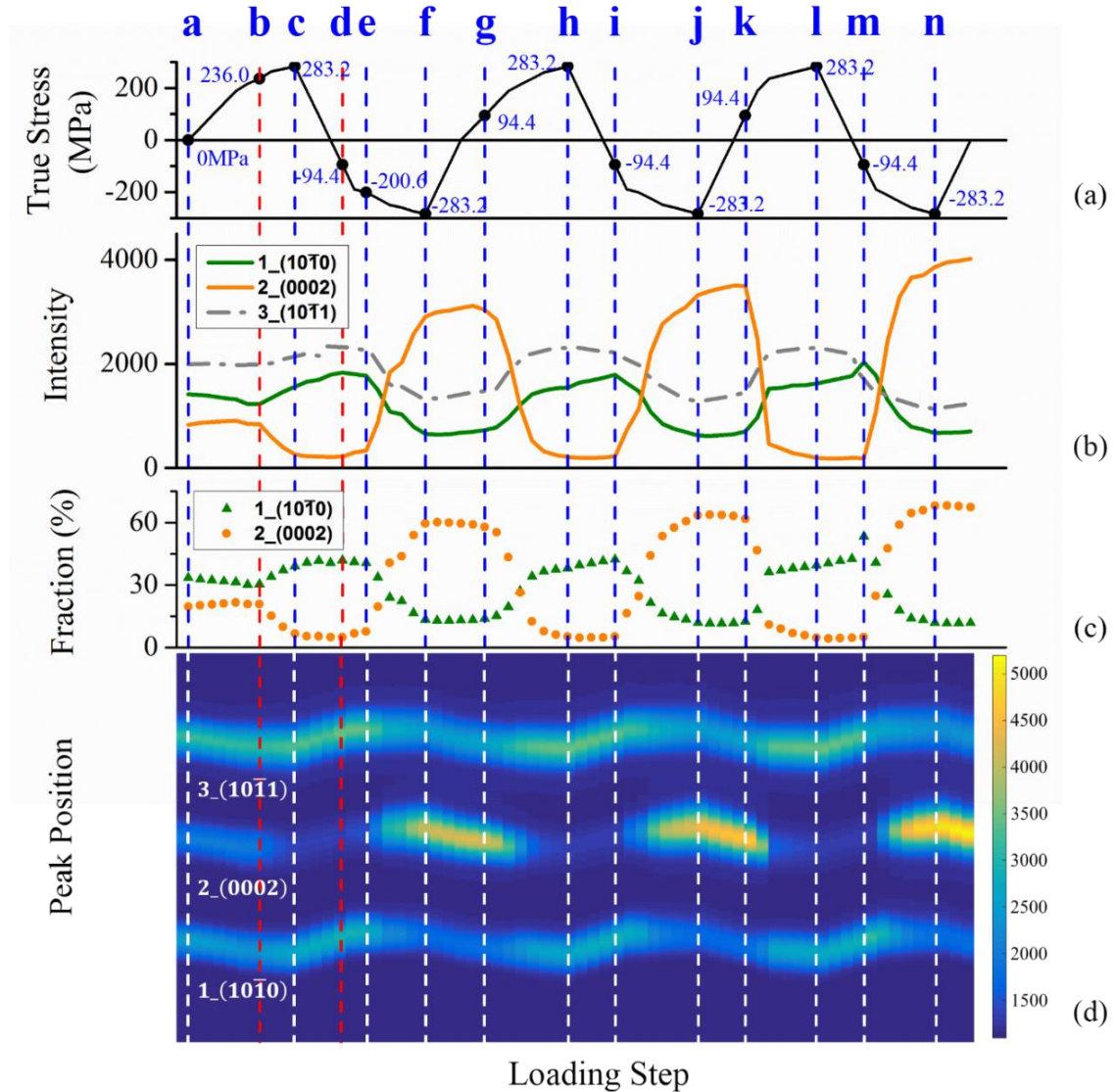


Figure 6.10 Observation of lattice rotation from in situ cyclic loading XRD experiment results: (a) true stress from external loading; (b) intensity change of three diffraction peaks; (c) fraction of intensity for the first two peaks; (d) contour plot with peak position (2θ) as the vertical axis and the colour showing the peak intensity (refer to colour bar for intensity value).

In the second and the third cycles, the absolute true stress value for triggering rotation is 94.4 MPa for both tension and compression loadings, which is the same as the stress for detwinning (at point **d**). This indicates that once twinning is triggered (at a higher stress which is 236 MPa in this case) the following detwinning, re-twinning and re-detwinning

all have similar activation mechanism in terms of activating stress. Therefore, the only difference in activation mechanism lies in the stage when twinning happens for the first time. For all the loading processes afterwards, only detwinning needs to be considered.

6.2.3 Conclusions

In situ compression-tension experiments were performed respectively with XRD and EBSD to study lattice rotation caused by the TDT process. Sudden change in diffraction peak intensity from XRD and evident the TDT process in EBSD maps were both observed. The twin fraction was calculated in both XRD experiments by using absolute intensity value and in EBSD experiment by summing up the area of index points that satisfy Bragg's law with imaginary X-ray. The twin fraction matched well between the two experiments. Therefore, it is confirmed that TDT led to the abrupt change in the intensity of the diffraction peaks. In the meantime, the grain distribution and texture of the sample was homogeneous along the ND. It was observed that detwinning was only largely activated when the loading was reversed rather than removed.

The twin fraction was extracted from both compression-tension cycles of the *in situ* EBSD experiments and comparison exhibited obvious difference between the 1st cycle and the 2nd cycle. Far less twins can be activated during the 2nd cycle, which was possibly attributed to hardening during plastic deformation. In the meanwhile, rotation behaviour is less predictable in the 2nd cycle showing that there was more localised strain concentration.

Investigation of intensity change of three peaks from *in situ* cyclic loading XRD experiment discovered that twinning and detwinning were respectively activated for the first time at the true stress level of 236 MPa and 94.4 MPa, respectively. It was also concluded that the activation mechanism of twinning only applies when it first happens

and the successive detwinning, re-twinning and re-detwinning follow similar activation mechanism in terms of activating stress.

7 Multi-Scale CPFÉ Modelling of Twinning-Detwinning

7.1 Introduction

Extensive experimental studies have been carried out to understand the mechanism of twinning in magnesium alloys [37, 57, 205, 305-310]. The existing literature covers a wide range of aspects relevant to twinning, such as evolution of texture and microstructure [30, 311-313], deformation anisotropy and asymmetry [68, 86, 308, 310, 314] and non-Schmid behaviour [315-319]. It is intuitively expected that twin variants which are selected to successfully form twins should have relatively high Schmid factors (SFs) and, hence, high RSS [319]. Nevertheless, twinning has demonstrated significant non-Schmid behaviour [315-319]. Exploration of SF leads to the understanding of twinning variant selection and strain accommodation that twinning requires from its surroundings. Almost half of the contraction twins that appeared in AM30 and AZ31 magnesium alloys belonged to ‘low’ SF type (0.15-0.3) and nearly 5% had even lower SFs [319]. According to the authors, when nucleation of a twin variant is triggered, shear in the matrix happens and the adjacent surrounding must also be sheared to accommodate the shear in the matrix [319], which caused the observed non-Schmid behaviour. As a result, whether a twin variant can be activated is not solely dependent on the SF but also on how ‘easy’ the accommodation is in the surrounding. Pei *et al.* [320] focused on extension twin in magnesium AZ31B and explained that the theory of accommodation strains can also be used to explain twin variant selection for secondary and tertiary twins. Aside from non-Schmid behaviour, a linear relationship was discovered between a grain’s SF and the maximum twin fraction that a twin variant can achieve in the grain. Shi *et al.* differentiated between $(10\bar{1}2)$ extension twins formed only at grain boundaries (group 1) and pairs of twins that crossed a grain

boundary (group 2) in magnesium AZ31 [315]. Based on the statistics of a large EBSD data set, the difference between these two groups was illustrated to mainly lie in the type of slip systems they required to accommodate the twinning-induced shear. In the meanwhile, a large number of numerical studies of twinning have been carried out [203, 212-215, 233, 319, 321]. Some of them explored the SF effect and twin variant selection of twinning with crystal plasticity models [233, 319, 321]. They all reported non-Schmid behaviour of twinning and attributed it to the localized influence from the surrounding stress. Barrett *et al.* employed molecular dynamic simulation to investigate the correlation between non-Schmid stresses and non-Schmid behaviour of twinning [322].

Detwinning has an indispensable effect on the mechanical properties of crystalline materials [36, 37, 219-221] so it is necessary to understand and describe correctly its underlying mechanism. Both experimental and numerical studies have been carried out recently [35, 58, 71, 211, 220, 222-232]. A detailed background introduction and literature review can be found in section 2.3.2. Most of the work mentioned in section 2.3.2 concentrates on developing CPFE model to include detwinning systems and correctly match the simulation with experimental results. To our best knowledge, the SF effect of detwinning has only been experimentally studied by Chapuis *et al.* [323] and no numerical investigation has been conducted.

In this chapter, a CPFE model is extended from a previous model to include detwinning systems. An independent parameter denoting the CRSS of detwinning is introduced and calibrated against the experimental results. The model is then used to investigate the activation of deformation systems by direct comparison with experimental observations. To this end, the onset and the end of the TDT process during compression-tension loading are determined experimentally by the *in situ* XRD experiment and verified

by *in situ* EBSD mapping. Finally, detwinning SF effect is investigated based on the insight into micro-scale deformation that the model provides.

7.2 FEM Implementation

7.2.1 Finite Element Setup

Two finite element models are used in this work which were built based on the formulation reported in previous papers [38, 40]. The starting texture was imposed upon the initial grain orientation distribution in the models. Both models had 2592 grains each characterized by specific Euler angles.

The model used in Ref. [38] was simplified to one element representing one grain. This approach will be referred to as Model 1 in the following discussion. Figure 7.1 shows the meshed cuboid representative volume element (RVE) of model 1. Element type is C3D8R. One element is highlighted in red and the orientation of the grain it represents is shown with a unit cell. The notations ‘Y0’ and ‘Y1’ are introduced for the two surfaces perpendicular to Y-axis in Figure 7.1. Y1 was set to have zero displacement in all directions while Y0 had zero displacement along both X-axis and Z-axis. A displacement in the Y-axis was applied to conduct compression-tension loading.

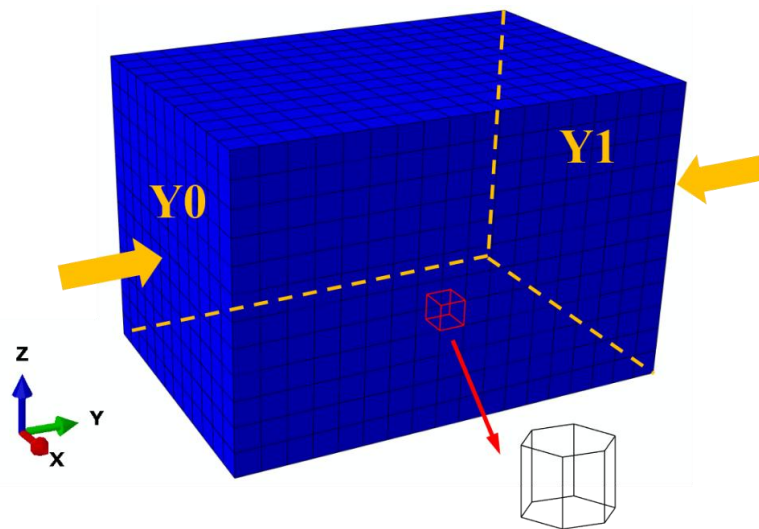


Figure 7.1 Cuboid RVE of Model 1: Y1 has zero displacement in all directions while Y0 has zero displacement along X- and Z- axis but has displacement along Y-axis for compression-tension loading.

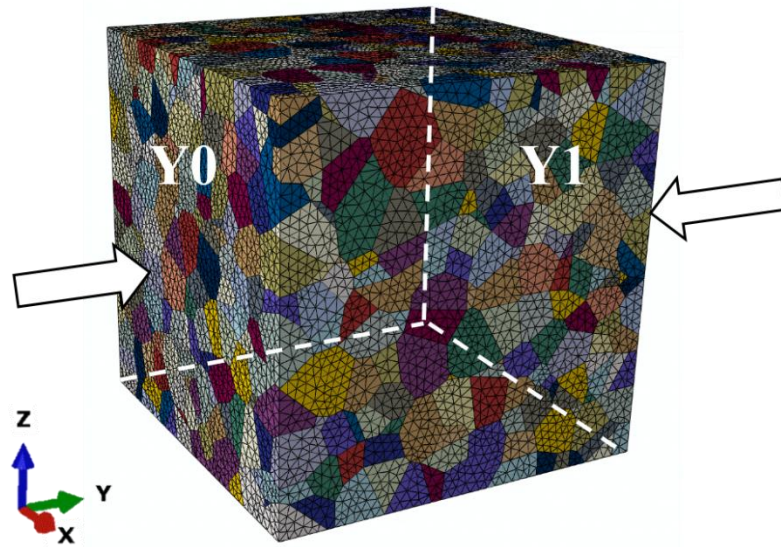


Figure 7.2 Cubit polycrystalline bulk of Model 2.

The model from Ref. [40] (referred to as Model 2) has hundreds of elements for each grain to allow investigation of intra-granular deformation. As a result, the calculation time is increased by more than 900%. 3D Vorono itessellations were generated to represent the polycrystalline aggregate. The cubic polycrystalline RVE (shown in Figure 7.2) had a volume of 1 mm^3 . The software package Neper [324] was used for generating the polycrystal and meshing a total number of 475,947 tetrahedral elements. Notations ‘Y0’ and ‘Y1’ in Figure 7.2 are also two surfaces marked in the same way as that in Figure 7.1. Boundary conditions for Y0 and Y1 in Model 2 were the same as model 1.

A user subroutine VUMAT of the commercial software ABAQUS/Explicit was used to implement the crystal plasticity constitutive law including slip, twinning and detwinning.

7.2.2 Constitutive Framework

The model used in this work is extended from the previously developed CPFE model for Mg AZ31 alloy detailed in Ref. [38, 40] by including the contribution of detwinning in order to simulate a complete set of mechanisms present in the compression-tension loading process. In the following, only the detwinning constitutive behaviour and its implementation in the model is presented, the reader being referred to Chapter 4 and Ref.

[38, 40] for the underlying constitutive model. Here only the $(10\bar{1}2) < \bar{1}011 >$ extension twin system and its corresponding detwin system are considered. Twin and detwin each has six variants. However, detwin variants have two sets: set 1 has exactly the same deformation plane normal and direction as its corresponding twin while set 2 is obtained by preserving the twin plane normal but reversing the twin direction (Table 7.1).

Table 7.1 Twinning and detwinning systems

	Twin		Detwin (set 1)		Detwin (set 2)	
	Normal	Direction	Normal	Direction	Normal	Direction
Variant 1	$(10\bar{1}2)$	$< \bar{1}011 >$	$(10\bar{1}2)$	$< \bar{1}011 >$	$(10\bar{1}2)$	$< 10\bar{1}\bar{1} >$
Variant 2	$(01\bar{1}2)$	$< 0\bar{1}11 >$	$(01\bar{1}2)$	$< 0\bar{1}11 >$	$(01\bar{1}2)$	$< 01\bar{1}\bar{1} >$
Variant 3	$(\bar{1}102)$	$< 1\bar{1}01 >$	$(\bar{1}102)$	$< 1\bar{1}01 >$	$(\bar{1}102)$	$< \bar{1}10\bar{1} >$
Variant 4	$(\bar{1}012)$	$< 10\bar{1}\bar{1} >$	$(\bar{1}012)$	$< 10\bar{1}\bar{1} >$	$(\bar{1}012)$	$< \bar{1}01\bar{1} >$
Variant 5	$(0\bar{1}12)$	$< 01\bar{1}\bar{1} >$	$(0\bar{1}12)$	$< 01\bar{1}\bar{1} >$	$(0\bar{1}12)$	$< 0\bar{1}1\bar{1} >$
Variant 6	$(1\bar{1}02)$	$< \bar{1}101 >$	$(1\bar{1}02)$	$< \bar{1}101 >$	$(1\bar{1}02)$	$< 1\bar{1}0\bar{1} >$

Two sets of detwinning were needed to suit the special nature of lattice rotation in the model. In reality, the lattice rotation (almost 90°) takes place upon twinning under compression perpendicular to c -axis, resulting in a lattice orientation ending up with its c -axis parallel to compression loading which is unfavourable for further twinning. The reverse loading applies tension parallel to c -axis, which meets the geometry requirement and leads to reverse twinning (detwinning). In the formulation used in the present simulation, however, lattice rotation in a twin system α only happens when the twin volume fraction f^α reaches a threshold ζ , which is a random number set to lie between 0.75 and 0.8 in the model. More details are provided in the section 4.2 and Ref. [38]. As a result, twinning deformation occurs and the associated shear strain begins to accumulate long before the finite element lattice rotation happens. It is possible that the external compression loading is ended after twinning starts, but before lattice rotation happens in

the model, which will lead to a situation where an element has already experienced some twinning shear strain but still remains in almost the same orientation as the starting one (subtle orientation change caused by slip is not considered here). In this case, detwinning cannot be activated by reversed loading if using set 1 as detwin variants. In order to allow detwinning in those unrotated but twinned elements, detwin variants from set 2 are used. Making detwin direction negative has the same effect as rotating lattice during twinning when it comes to satisfying the twinning/detwinning geometry. For simplification, the superscript i (and j) will refer to a slip system, α (and β) to a twin system, m (and n) to a detwin system (set 1) and k (and l) to a detwin system (set 2).

Table 7.2 Twinning and detwinning systems expressed in Cartesian coordinate systems used in subroutine

	Twin		Detwin (set 2)	
	Normal	Direction	Normal	Direction
Variant 1	0.4705, 0, 0.8824	-0.7295, 0, 0.6840	0.4705, 0, 0.8824	0.7295, 0, -0.6840
Variant 2	0.2353, 0.4075, 0.8824	-0.3647, -0.6318, 0.6840	0.2353, 0.4075, 0.8824	0.3647, 0.6318, -0.6840
Variant 3	-0.2353, 0.4075, 0.8824	0.3647, -0.6318, 0.6840	-0.2353, 0.4075, 0.8824	-0.3647, 0.6318, -0.6840
Variant 4	-0.4705, 0, 0.8824	0.7295, 0, 0.6840	-0.4705, 0, 0.8824	-0.7295, 0, -0.6840
Variant 5	-0.2353, -0.4075, 0.8824	0.3647, 0.6318, 0.6840	-0.2353, -0.4075, 0.8824	-0.3647, -0.6318, -0.6840
Variant 6	0.2353, -0.4075, 0.8824	-0.3647, 0.6318, 0.6840	0.2353, -0.4075, 0.8824	0.3647, -0.6318, -0.6840

To facilitate the calculation of RSS and rotation transformation, a Cartesian coordinate system was used to express slip and twin/detwin systems in the VUMAT subroutine. A transformation was performed to change Bravais-Miller four-index coordinates into orthogonal coordinates. The detailed transformation can be found in Appendix I. The directions and the plane normals of the twin and detwin systems used in the subroutine are normalised vectors expressed in Cartesian coordinate systems as listed in Table 7.2. Note that only set 2 is listed since set 1 is exactly the same as twin systems.

To track the lattice rotation caused by twinning/detwinning, a variable *twincounter* was introduced in the model. This variable is increased by 1 every time rotation happens. Rotation can be triggered under two conditions stated in Equation (7.1). At the start of the simulation, *twincounter* is set to 0 for all the elements. If the f^α of a twin system α of one element reaches the threshold ζ , twinning-induced rotation occurs (*condition1*) and *twincounter* is incremented to 1. During reverse loading, rotation from detwinning (set1) is triggered when the detwin fraction f^m reaches the threshold and *twincounter* is increased from 1 to 2.

$$\begin{cases} \text{condition1: } twincounter = 0 \text{ and } f^\alpha \geq \zeta \\ \quad \quad \quad \text{rotation due to twinning} \\ \text{condition2: } twincounter = 1 \text{ and } f^m \geq \zeta \\ \quad \quad \quad \text{rotation due to detwinning(set 1)} \end{cases} \quad (7.1)$$

Let γ denote the shear strain of the system. Using $s_{\alpha/m/k}$ to denote the CRSS of twinning/detwinning systems and $\tau_{\alpha/m/k}$ the corresponding RSS, the condition for activating twinning/detwinning is given as below:

$$\begin{cases} \text{twinning: } \tau_\alpha - s_\alpha > 0, twincounter = 0 \\ \text{detwinning(set 1): } \tau_m - s_m > 0, \gamma^\alpha - \gamma^m \geq 0.15\gamma^\alpha, twincounter = 1 \\ \text{detwinning(set 2): } \tau_k - s_k > 0, \gamma^\alpha - \gamma^k \geq 0.15\gamma^\alpha, twincounter = 0 \end{cases} \quad (7.2)$$

The value 0.15 in Equation (7.2) comes from the observation that only approximately 85% of twins are able to detwin (i.e., 15% of twins persist after full reverse loading). This

is modelled here by retaining 0.15 of a twin's maximum shear strain during detwinning. The algorithm is fully presented in Figure 7.3.

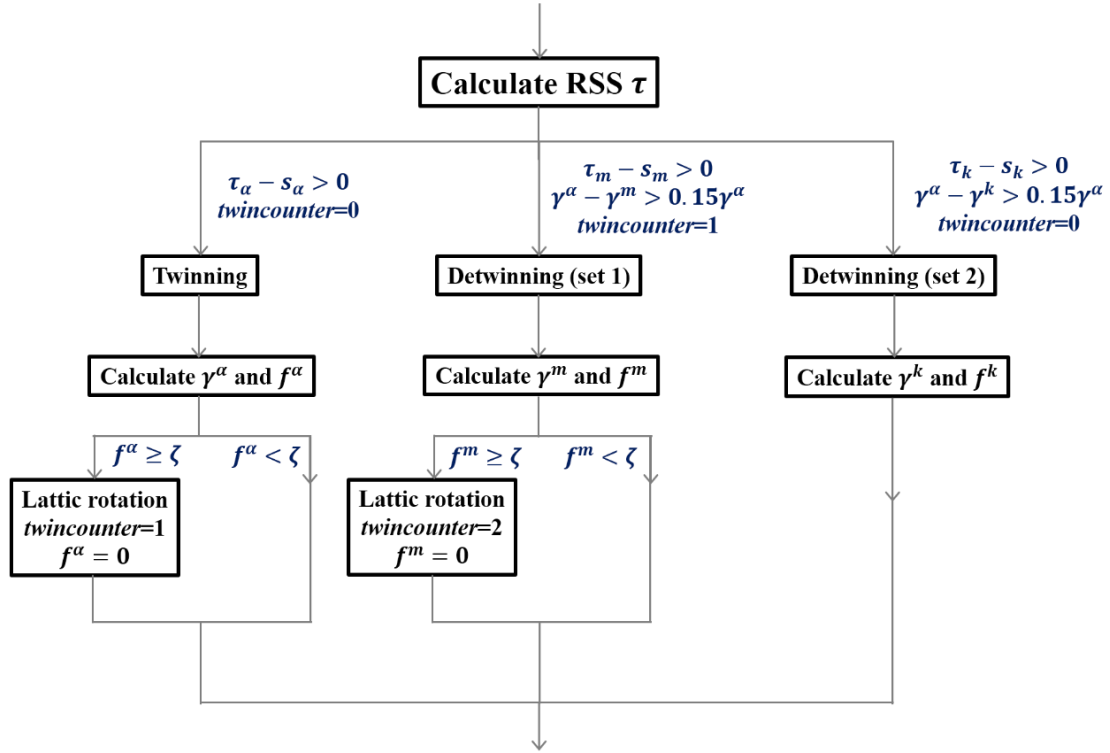


Figure 7.3 Flow chart of part of the algorithm concerning TDT activation and lattice rotation.

7.2.3 Model Calibration

It has been confirmed in Ref. [40] that the two FEM models introduced in section 7.2.1 give almost the same macroscopic response when using the same parameters. This is important since it reflects the fact that capturing the correct shear strain in the simpler lighter model (Model 1) is sufficient to simulate the overall deformation behaviour of the material, while the distinction at the microscopic scale persists. Therefore, Model 2 is further pursued here to ensure that micro-scale deformation phenomena are captured correctly. To improve computational efficiency, the stress-strain (thus macroscopic in nature) calibration carried out in the present study for Model 2 was started by using Model 1, the parameters of which were then passed on to Model 2 for further study. The detwinning parameters were calibrated so as to match the stress-strain curve while the other parameters were taken from the literature [38, 40] [325], see Table 7.3. Following

the notation in Ref. [38], $S_{0;\text{basal}}$, $S_{0;\text{prism}}$, $S_{0;\text{pyr}\langle a \rangle}$, $S_{0;\text{pyr}\langle c+a \rangle}$, $S_{0;\text{tw}}$ and $S_{0;\text{detw}}$ are the CRSS for basal slip, prismatic slip, pyramidal $\langle a \rangle$ slip, pyramidal $\langle c+a \rangle$ slip, twin and detwin respectively. $h_{0;\text{sl}}$, $h_{0;\text{tw/detw}}$, $h_{0;\text{sl-tw/detw}}$, $h_{0;\text{tw/detw-sl}}$, a_{sl} , $a_{\text{tw/detw}}$, $a_{\text{sl-tw/detw}}$ and $a_{\text{tw/detw-sl}}$ are the reference hardening parameters and hardening exponents for slip-slip, twin-twin, detwin-detwin, slip-twin, slip-detwin, twin-slip and detwin-slip interactions. $\tilde{S}_{\text{basal}}$, $\tilde{S}_{\text{prism}}$, $\tilde{S}_{\text{pyr}\langle a \rangle}$, $\tilde{S}_{\text{pyr}\langle c+a \rangle}$, $\tilde{S}_{\text{tw}}$ and $\tilde{S}_{\text{detw}}$ are the saturation stresses for deformation systems. q_{sl} , q_{tw} and q_{detw} are the ratios of the reference cross-hardening parameter to the reference self-hardening parameter of slip, twin and detwin systems, respectively. γ_0 is the shear strain of twin and detwin. C_{11} , C_{12} , C_{13} , C_{33} and C_{55} are the crystal stiffness elastic constants for Mg AZ31B.

Table 7.3 Parameters for crystal plasticity model

$S_{0;\text{basal}}$	$S_{0;\text{prism}}$	$S_{0;\text{pyr}\langle a \rangle}$	$S_{0;\text{pyr}\langle c+a \rangle}$	$S_{0;\text{tw}}$	$S_{0;\text{detw}}$
12.0 MPa	60.0 MPa	100.0 MPa	100.0 MPa	46.5 MPa	23.0 MPa
$h_{0;\text{sl}}$	$h_{0;\text{tw/detw}}$	$h_{0;\text{sl-tw/detw}}$	$h_{0;\text{tw/detw-sl}}$	q_{sl}	$q_{\text{tw/detw}}$
600.0 MPa	1000.0 MPa	0.0 MPa	600.0 MPa	2	1
a_{sl}	$a_{\text{tw/detw}}$	$a_{\text{sl-tw/detw}}$	$a_{\text{tw/detw-sl}}$	$\tilde{S}_{\text{tw}}$	$\tilde{S}_{\text{detw}}$
0.6	2	N/A	2	100.0 MPa	300.0 MPa
$\tilde{S}_{\text{basal}}$	$\tilde{S}_{\text{prism}}$	$\tilde{S}_{\text{pyr}\langle a \rangle}$	$\tilde{S}_{\text{pyr}\langle c+a \rangle}$	$\tilde{S}_{\text{sl-tw/detw}}$	$\tilde{S}_{\text{tw/detw-sl}}$
15.0 MPa	150.0 MPa	190.0 MPa	190.0 MPa	n/a	100.0 MPa
$\gamma_{0,\text{tw/detw}}$	C_{11}	C_{12}	C_{13}	C_{33}	C_{55}
0.129	58.0 GPa	25.0 GPa	20.8 GPa	61.2 GPa	16.6 GPa

The Euler angles for the 2592 grains were generated by randomly distributing basal poles within 20 ° (distributing angle) around the north pole, so as to fit the experimental pole figures, as illustrated in Figure 7.4 (the pole figures are plotted with MTEX [280]). It was verified that changing the distributing angle by $\pm 20\%$ had negligible effect on the stress-strain curves of the simulations. The same starting texture was used for both Model 1 and Model 2.

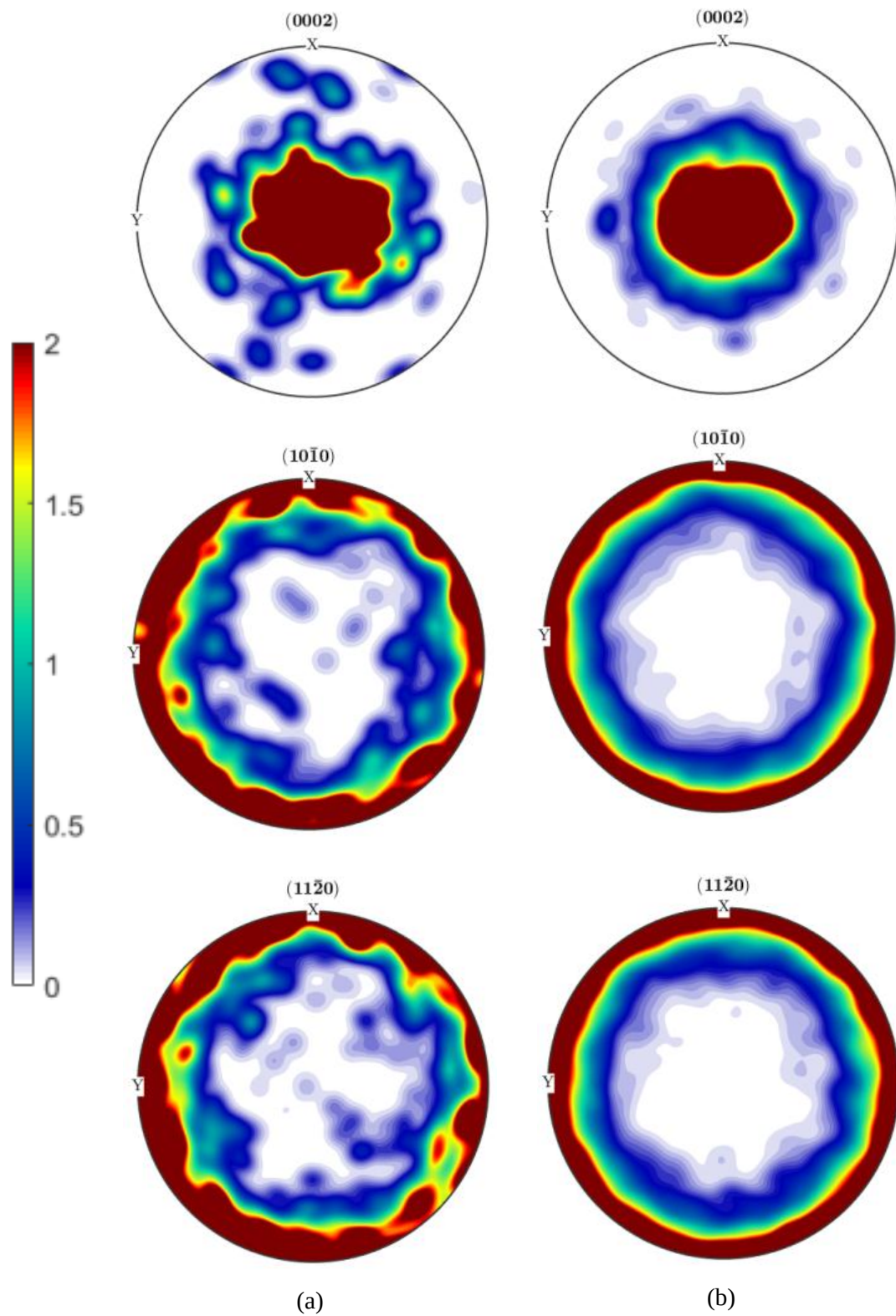


Figure 7.4 Initial texture of material in: (a) experiment; (b) simulation.

7.3 Post-Processing

All the variables which need to be passed on from last increment to carry out the calculation of the current increment were saved as state variables in the subroutine. There were in total 208 state variables for one element (e.g. current CRSS and accumulative shear strains of deformation systems) which were output to a binary result file with expansion '.fil' by ABAQUS. In the case of Model 1, this file could be converted into a ASCII file with expansion '.fin' and be processed by Matlab post-processing package Abaqus2Matlab [326]. However, for Model 2 if a sufficient number of increments were output, this would give a rather huge file with dozens of gigabytes. Therefore, it was more efficient to access the binary file directly with an ABAQUS utility routine DBFILE. The code for post-processing can be found in Appendix II.

7.4 Results and Discussion

7.4.1 Activation of Twinning and Detwinning

The experimental and numerical results (Model 2) for the compression-tension loading are given in Figure 7.5. The detailed elaboration of XRD data in Figure 7.5(b) can be found in section 6.2.2. It has been verified that the abrupt change in the intensity of the three rings indicates the beginning and ending of twinning and detwinning through comparing data from *in situ* XRD and *in situ* EBSD experiments. Twinning and detwinning was activated from B to C and from D to F, respectively. The average shear strain of deformation systems in Figure 7.5(c) is the mean value of the accumulative shear strain (i.e., equivalent strain) of all the twinned grains at one loading step as predicted by the simulation. Accumulative shear strain was output as one of the state variables in ABAQUS. The model demonstrates a good match with both of the experiments in terms of the activity of deformation systems. Basal slip and twinning were the most active deformation systems from point B to C, followed by detwinning from D to F. After

detwinning was no longer available, prismatic slip started, which is in agreement with the conclusions of the previous study [327].

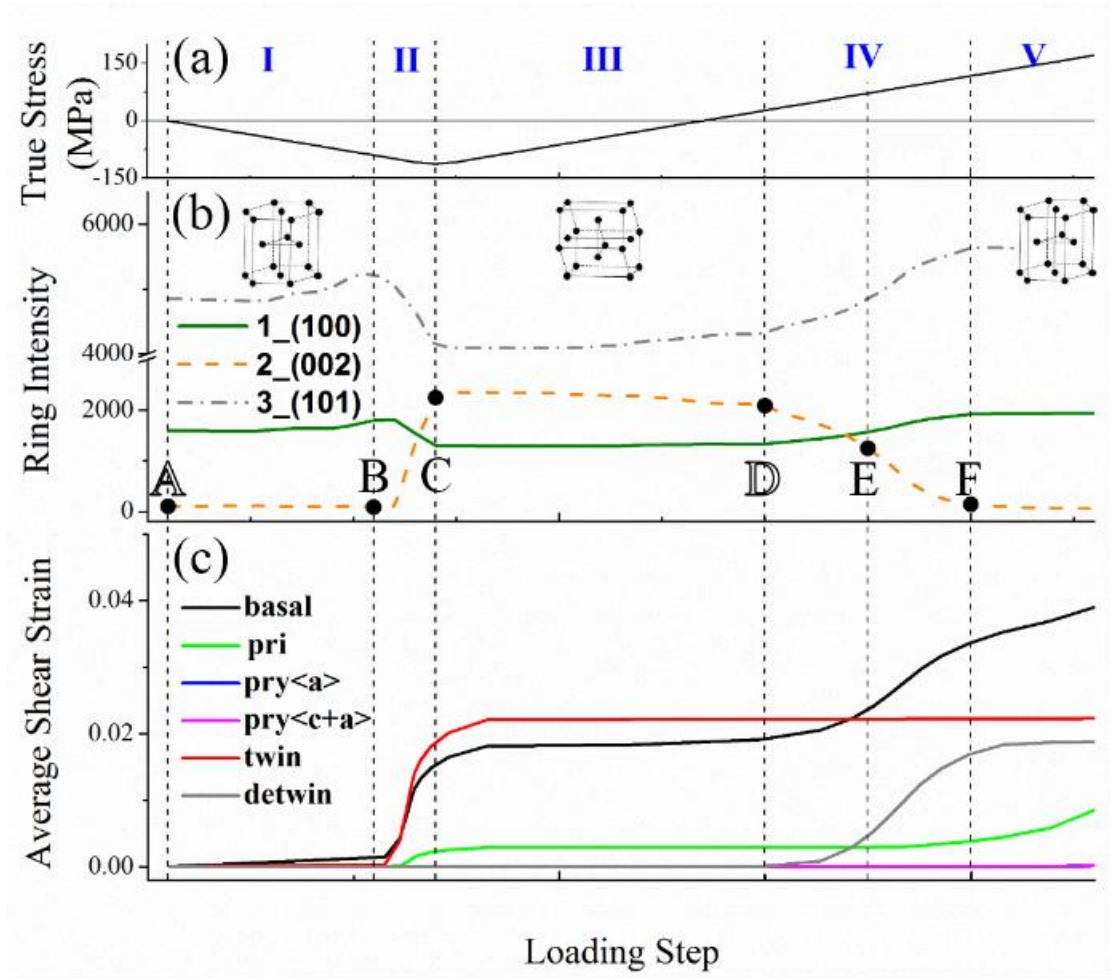


Figure 7.5 Change of quantities with loading step: (a) true stress of compression-tension loading; (b) the intensity of the three Debye-Scherrer rings in XRD experiment; (c) the average shear strain of deformation systems in simulation.

Figure 7.6 is the contour plot of the difference between the twinning shear strain and the detwinning shear strain at different true strain levels. Figure 7.6(I) and (II) depict the RVE as a 3D volume. Figure 7.6(a)-(i) are the cross-sections at the middle of the material RVE indicated by the red plane in Figure 7.6(II). Twinning is increasing from Figure 7.6(a)-(d) under compressive loading. Following that, under increasing tension, detwinning begins to happen and the amount of twinned regions decreases. Twinning reaches its maximum at the end of compression in Figure 7.6(e). It is noted that there are several grains in the central area in Figure 7.6(e) which have little twinning unlike their

surrounding grains. This might be attributed to their unfavourable orientations for twinning. Additionally, intra-granular variation in shear strain is evident, as pointed out by red arrows in Figure 7.6(e).

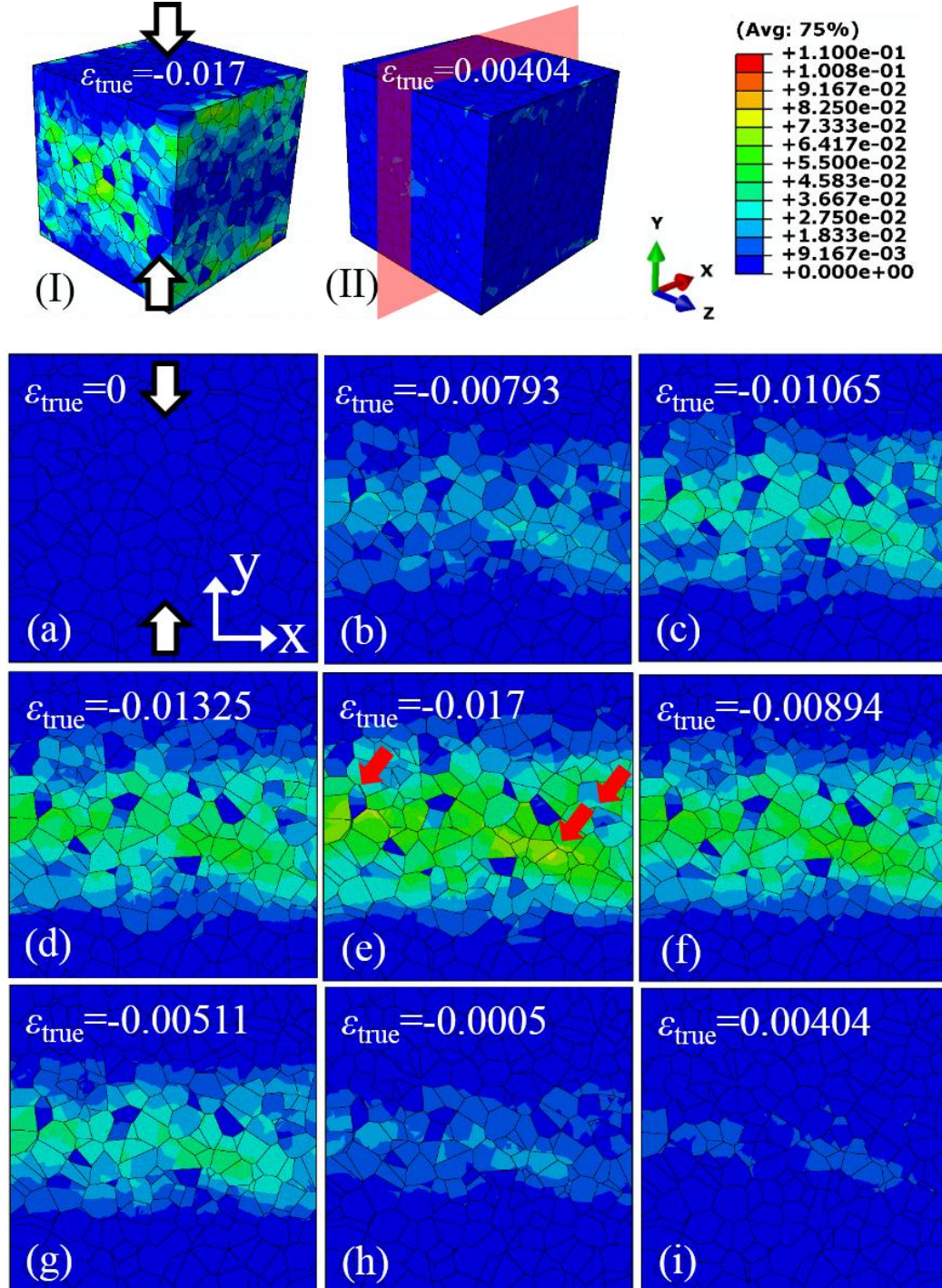


Figure 7.6 Contour plot of the difference between the twinning shear strain and the detwinning shear strain at different true strain values, which is essentially the residual twin: (I) and (II) the RVE as a 3D volume; (a)-(i) the cross-sections at the middle of the RVE indicated by the red plane in (II).

The earlier calibration of the CRSS values for twin and detwin led to 46.5 MPa and 23.0 MPa, respectively. It has already been reported that detwinning happens at lower CRSS compared to twinning [20]. One reason for this is that no nucleation is required during detwinning. The second reason is that the backstress introduced by twinning promotes the reverse process of detwinning. It is worth mentioning that the CRSS is considered to be the property of the deformation system itself, unchanged by the orientation of the grain and its neighbouring grains. Therefore, CRSS values should not be affected by lattice rotation. 56 MPa and 32 MPa have been reported in Ref. [230] to be the CRSS values for twin initiation and twin growth which can be essentially considered as CRSS for twin and detwin, respectively. Even though the absolute values vary slightly, the difference between these two values is almost the same, being 23.5 MPa in our work and 24 MPa in Ref. [230].

7.4.2 Macroscopic Response

Figure 7.7 illustrates the stress-strain curves obtained via experiment and simulation. The six black dots correspond to the points A-F in the XRD and the EBSD experiments with B, C, D and F indicating the activation of twinning/detwinning, whereas the four red stars mark the beginning and the end of twinning and detwinning predicted by the simulation. EBSD maps were not taken at points A and D, which is indicated by making the letters hollow in Figure 7.7. The start and the end of twinning are well matched between the simulation and experiment, whereas detwinning occurs over a somewhat longer range of strain in the simulation. To understand more fully the effect of deformation systems on the macroscopic curve, it is worth comparing these results with Figure 7.5(c). Evidently, the section II during compression was mainly a result of twinning. For reverse loading, the first ‘dip’ in IV was caused by detwinning. The second jump that appears in V was the consequence of the activation of prismatic slip. It can be concluded that the validated CPFE

model captured correctly not only the macroscopic stress-strain curve, but also the transition between different plastic deformation modes, namely, different slip systems, twinning and detwinning.

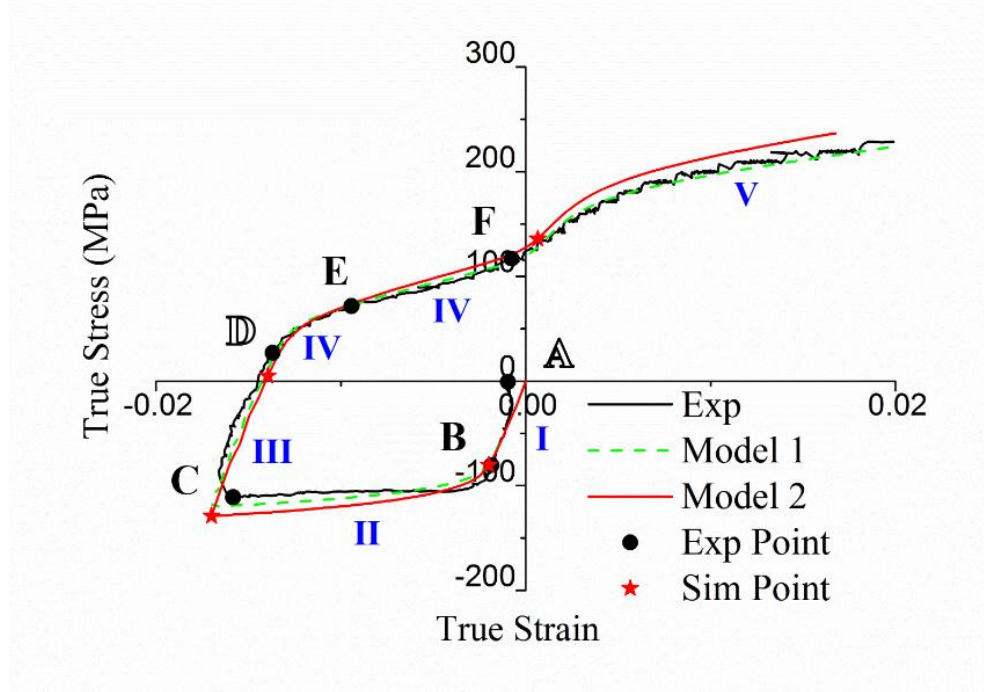


Figure 7.7 Comparison of stress-strain curves from experiment and simulation: black dots B, C, D and F: the beginning and the end of twinning and detwinning observed in the XRD and the EBSD experiments; black dot A: the beginning of the loading; black dot E: the intermediate stage of detwinning (A and D are made hollow to indicate that EBSD maps were not taken there); red stars: the beginning and the end of twinning and detwinning predicted by the simulation.

7.4.3 Twin/Detwin-Induced Lattice Rotation

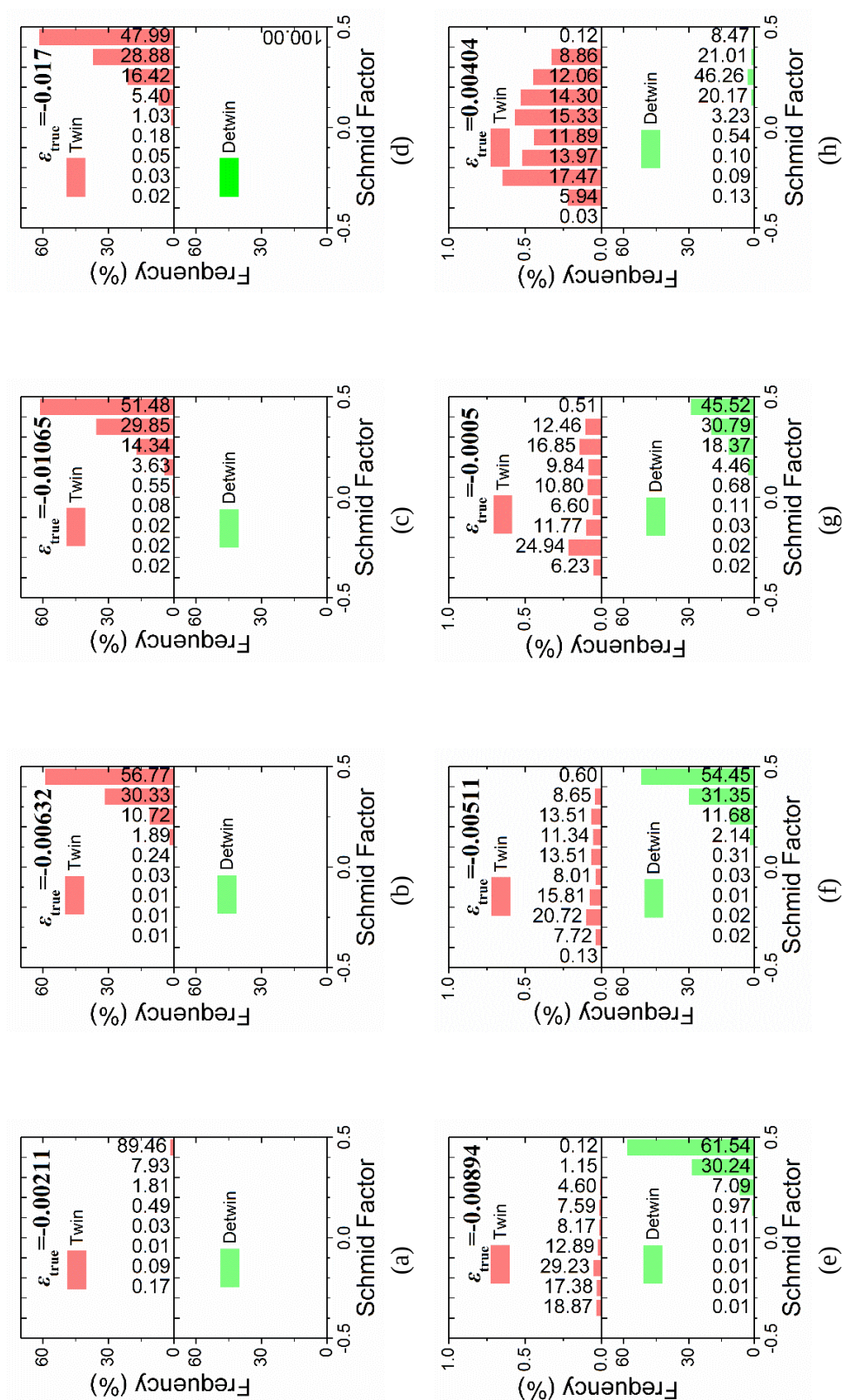
The simulation using Model 1 did not exhibit any element lattice rotation, whereas a small volume of elements went through twinning-induced lattice rotation. Lattice rotation is ‘suppressed’ in simulation due to the binary nature of the element lattice rotation characteristic of the formulation of this constitutive model as explained in the Ref. [38]. It was also discussed that there is a delay of lattice rotation in the simulation compared to experiment and Model 2 has a smaller delay compared to Model 1 [40]. This is mainly because the more detailed model allows intergranular variation which leads to local strain concentration in turn causing lattice rotation. The compression loading in the current

simulation did not reach large enough true strain to activate a significant amount of lattice rotation.

In spite of the fact that the actual lattice rotation of element is delayed with respect to the experimentally observed lattice rotation, the macroscopic behaviour of the material is adequately captured. As already discussed in previous work [40], this apparent discrepancy can be rationalised by the fact that rotation is a microscale localised phenomenon at the onset, hence its influence on the activation of deformation systems is insignificant. The model thus assumes that the element rotation is only needed when the lattice twin rotation is starting to alter the lattice plasticity at the grain level. Overall, the prediction of the macroscopic behaviour by the model was observed to be trustworthy [40]. We thus follow the same approach for the detwinning rotation here while accounting for the detwinning occurrence in the absence of element lattice rotation (see detwinning set 1 in Figure 7.3).

7.4.4 Schmid Factor Effect

Figure 7.8 illustrates the spread of SFs at various true strain levels in the form of histogram. Figure 7.8(a)-(d) correspond to compression and (e)-(h) to tension. The calculation of SFs was based on individual elements and only elements with $\dot{\gamma} > 0$ in the current simulation increment were included in the statistics, meaning that all the elements included in the histograms were undergoing twinning or detwinning at that moment. There are two percentage values shown for each plot. One is the vertical axis ‘Frequency (%)’ which is the ratio of the number of active twinning/detwinning variants to the total number of elements in the RVE (475,947). The numbers on each column represents the relative frequency (i.e., the ratio of the number of active twinning/detwinning variants within the specific SF range to the total number of active twinning/detwinning variants at the current strain level).



An increase of non-Schmid behaviors with strain can be observed for both twinning and detwinning, indicating a gradual rise of the local influence. Higher loading strain leads to a larger frequency of localized deformation mostly arising from stress localization at the grain boundaries, see Figure 7.6. These effects arise from the mismatch of orientation between neighbors: when surrounding grains do not have suitable slip systems to accommodate the sudden change brought by twinning/detwinning, other non-Schmid systems with more accommodating surroundings are activated.

The increased non-Schmid behaviour of twinning during tension compared to compression is also noteworthy. In the meanwhile, a more even distribution of both negative and positive SFs is present in twinning during tension compared to mainly positive during compression. The discussion given above provides a clear evidence of the importance of local grain interaction for the deformation behaviour of a polycrystalline material.

7.5 Conclusions

This section investigated the activation of different plastic deformation systems during mechanical loading with *in situ* XRD and *in situ* EBSD. In the *in situ* compression-tension XRD experiment, the change of the intensity of the Debye-Scherrer rings for different grain groups was considered as an indication of the TDT process. This was verified in a spatially resolved fashion by *in situ* EBSD through direct observation of crystal rotation induced by twinning and detwinning. A CPFE model was developed to account for the activity of four slip systems, $(10\bar{1}2) < \bar{1}011 >$ extension twinning and detwinning. The information on deformation system activity gathered from experiments was essential for calibrating the parameters for the models. The investigation adopted two finite element models (Model 1 and Model 2) which have different calculation efficiency but give almost the same stress-strain curve when using the same parameters. The one-element-one-grain

Since Model 1 has a much shorter calculation duration, it was used to calibrate the parameters against the experimental stress-strain curve. CRSS of detwinning (23 MPa) was found to be significantly lower, compared to the CRSS of 46.5 MPa for twinning. The calibrated parameters were then passed on to the multiple-element-one-grain Model 2 which demonstrated the capability of correctly predicting the microscopic activity of plastic deformation systems. A small amount of lattice rotation was observed in Model 2 compared to no lattice rotation in Model 1, meaning that microscale lattice rotation had little influence on the macroscopic behaviour predicted by both Model 1 and Model 2. Moreover, the SFs of individual elements were investigated using Model 2. It was concluded that non-Schmid behaviour revealed a growing trend with the increasing plastic strain level. When detwinning was the dominant mechanism under tension, a small portion of twinning was activated with small SFs evenly distributed between positive and negative values. A clear evidence of the influence of local grain interaction was shown on the deformation behaviour of a polycrystalline material.

8 Conclusions and Future Work

This chapter draws conclusions and gives discussion regarding the method development and the experimental and the numerical results presented in the previous chapters. This thesis has mainly contributed in three areas:

- 1) Development of DIC application for strain evaluation techniques: FIB-DIC and newly proposed XRD-DIC;
- 2) Experimental study of deformation mechanism of TDT in magnesium AZ31B particularly concerning its influence on deformation asymmetry and TDT-induced lattice rotation;

3) Extended CPFE model to include detwinning mechanism which was used to conduct multi-scale numerical CPFE investigation of TDT deformation process and the non-Schmid behaviour during twinning and, for the first time, detwinning.

Special attention is paid here on the discussion of the limits encountered during DIC development, experiments and simulation work. Furthermore, future work based on the current discoveries and limitations will be proposed to improve the understanding of the topic.

8.1 XRD-DIC Development

The XRD-DIC approach proposed in section 5.2 demonstrated satisfactory reliability and accuracy with enhanced convenience and minimised error sources. However, it is considerably affected by the background of the image since DIC tracking entirely relies on the intensity of the ring. Therefore, conducting background reduction for all the patterns is of critical importance. Ideally, one diffraction pattern should have one background image taken right before or after it because the background is changing all the time especially for long-duration experiment. In addition, the inherent electronic noise generated by the detector is also likely to change. The factors mentioned above both have unneglectable influence on the DIC analysis. Nevertheless, synchrotron experiments are costly and taking one background image for each XRD pattern is time-consuming and not practical. It is most efficient and hence desirable if one background image is taken at the very beginning of the experiment and used for the background subtraction for all the images. To make this work as effectively, it requires maintaining the background level throughout the whole experiment via careful design of experiment and commissioning of the beamline equipment. Alternatively, an image filter can be potentially developed to tackle the problem if only one background image is taken and the background level varies too much that DIC shows poor precision. The image filter has to be able to recognise the

change in background level and correlate it to the same level. Generally, more image filters are needed to post-process the XRD patterns so that the influence from experiment can be minimised and the accuracy of DIC analysis can be more independent from the experimental condition.

In terms of the outlook for the future use of the XRD-DIC approach proposed herein, it is worthwhile considering the effect of grain statistics within the sampling volume on the quality of the 2D diffraction pattern and its interpretation. Grainy diffraction patterns arise when the number of grains that satisfy Bragg's scattering condition within the sampling volume is low. This is known to cause problems for the evaluation of average lattice strain using the ConFit approach because grainy Debye-Scherrer rings give rise to poor quality peaks on the 1D plot, leading to large scatter in the Gaussian peak fitting results. An interesting potential scope for future XRD-DIC analysis is to interpret diffraction patterns consisting of grainy rings in order to extract strain information from them. This approach may be combined with grain tracking and with sample tilting in order to obtain full 3D information in the reciprocal space. However, some difficulties can be predicted. First of all, the *in situ* loading experiment might cause significant lattice rotation that will lead to the diffraction spots where markers are placed to experience dramatic intensity change or even to disappear. This could very likely lead to unstable tracking and, therefore, misinterpretation for strain evaluation. Subset window (refer to Figure 5.6) works as a mechanism of averaging intensity in a small area for correlation. Therefore, it can be used to average a certain size of area on the diffraction pattern so that the influence of intensity change of individual diffraction spots is minimised. To make this work, the size of subset window needs to be decided with care so that the intensity change or even disappearance of a diffraction spot can be somehow made up via averaging in the subset window.

8.2 Limitations of EBSD Experiments

The *in situ* EBSD experiment is a versatile technique which can provide many aspects for the TDT process investigation, such as dependence of twinning activity on the strain path [309], localised twin bands during bending [328], evolution of twin morphology [329] and activation and evolution of secondary and tertiary twins [330]. The *in situ* EBSD experiment conducted (section 3.3) was a lengthy process. 8 maps were collected and each one took roughly 12 hours. Nevertheless, it was only currently used to verify TDT-induced rotation and twin fraction in this thesis. It seems to have underused the *in situ* EBSD results but actually is a sensible choice since the index rate at grain boundaries is not high enough to carry out similar localised observations in the papers mentioned above and to draw reliable conclusions.

The main limit lies in the surface quality of the sample for EBSD. The polishing process is finished with colloidal silica. However, it is highly recommended and proven largely effective if electro-polishing is employed as the last step of polishing [313, 331-336]. In addition, more dislocations in the sample lead to a lower chance of successful indexing. That is the reason why index rate is much lower at boundaries where two regions with different lattice orientation meet, such as grain boundary or twin boundary. Moreover, the index rate gets progressively lower with the loading process. Figure 8.1 illustrates the original EBSD maps at loading point C1_1 and C2_2 (refer to Figure 6.6) without noise reduction. CGPed samples have a higher percentage of grain boundaries due to their fine grains. Therefore, the results from EBSD with CGPed samples were not satisfactory. On the other hand, as-received wrought samples, thanks to their coarse grains, demonstrated results that were relatively presentable. Nevertheless, the un-indexed points still account for a considerable percentage so that it is not reliable for observation at twin boundary or grain boundary.

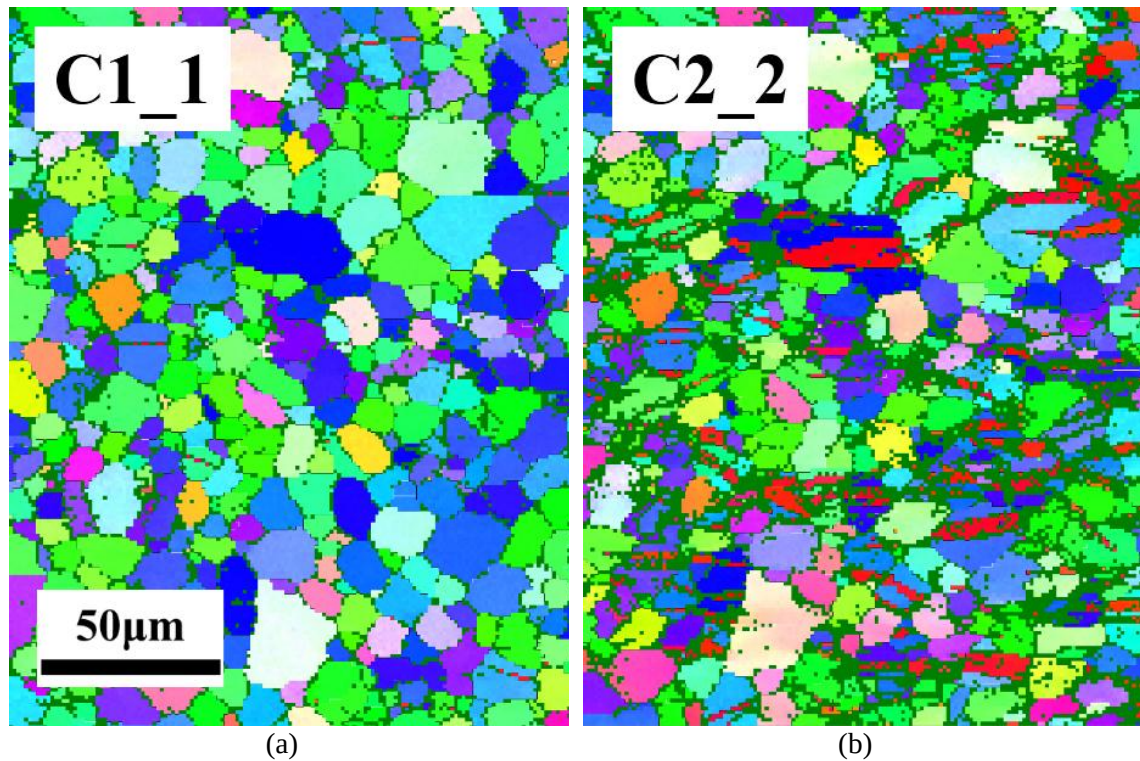


Figure 8.1 Original EBSD maps without noise reduction (the dark green points are un-indexed ones): (a) C1-1, the first map in the first cycle; (b) C2_2, the second map in the second cycle. (referring to the labels of Figure 6.6)

Several solutions can be adopted to improve the index rate:

1) Ion beam polishing: this can give almost perfect surface finish for EBSD but it is extremely time-consuming if a large area of sample is to be examined.

2) Electro-polishing: various recipes of electrolytes can be found in the literature. The most popular two are Struers AC2 solution and 3:5 (volume fraction) of phosphoric acid and ethanol. However, temperatures below 0 °C are generally required for the process.

3) Use material with even coarser grains: index rate can be increased with less percentage of boundaries. Most of the samples presented in previous studies [309, 337, 338] that were prepared only with colloidal silica finish and led to successful observation of individual twins have average grain size at more than 100 µm.

3D-EBSD enjoys an increasing popularity for twinning studies because of its capability of exploring a bulk of materials instead of only a 2D surface [336]. This can

give comprehensive insights into the real shape of grains and twins. Moreover, index rate can be maintained or even increased because material removal is carried out by FIB, which gives an optimal surface for EBSD. Therefore, for the next step, 3D-EBSD is considered to obtain the microstructure for a bulk of material that has twinned and then detwinned. Information such as the influence of detwinning on the surrounding materials can be obtained from this.

8.3 CPFEM Simulation

In this thesis, the first step of implementing detwinning into CPFEM was finished and the model was then used to simulate compression-tension as a comparison to both the *in situ* XRD and the *in situ* EBSD experiments. The next step is to simulate the other experiments involving detwinning presented in this thesis. Firstly, simulation of the cyclic loading used for *in situ* XRD (section 3.3.4) can be used as a comparison to confirm the observed TDT behaviour. In the meanwhile, non-Schmid behaviour in the second and the third cycles can also be further studied. Additionally, if 3D-EBSD is successful, the RVE in Model 2 can be assigned with the microstructure of the bulk examined by 3D-EBSD. This can be used to study the twin boundary propagation and shrinkage (corresponding to detwinning). Comparison between experimental and numerical results can shine light on the activation mechanism of twinning and detwinning.

Appendix I – Transformation between Coordinate Systems

i_1 , i_2 and i_3 are unit coordinate vectors for Cartesian coordinate system. 4-index coordinate vectors a_1 , a_2 , a_3 and c can be expressed under Cartesian coordinate system as below

$$\begin{bmatrix} a_1 \\ a_2 \\ a_3 \\ c \end{bmatrix} = \begin{bmatrix} \frac{\sqrt{3}}{2} & -\frac{1}{2} & 0 \\ 0 & 1 & 0 \\ -\frac{\sqrt{3}}{2} & -\frac{1}{2} & 0 \\ 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} i_1 \\ i_2 \\ i_3 \end{bmatrix} = \mathbf{M} \begin{bmatrix} i_1 \\ i_2 \\ i_3 \end{bmatrix} \quad (\text{A.1})$$

Any vector d with coordinates (d_1, d_2, d_3, d_4) in the unit cell under 4-index system is

$$d = d_1 a_1 + d_2 a_2 + d_3 a_3 + d_4 c = [d_1 \quad d_2 \quad d_3 \quad d_4] \begin{bmatrix} a_1 \\ a_2 \\ a_3 \\ c \end{bmatrix} \quad (\text{A.2})$$

Use Equation (A.1) in Equation (A.2) gives

$$d = [d_1 \quad d_2 \quad d_3 \quad d_4] \begin{bmatrix} a_1 \\ a_2 \\ a_3 \\ c \end{bmatrix} = [d_1 \quad d_2 \quad d_3 \quad d_4] \mathbf{M} \begin{bmatrix} i_1 \\ i_2 \\ i_3 \end{bmatrix} \quad (\text{A.3})$$

Therefore, the coordinates (d'_1, d'_2, d'_3) of vector d under Cartesian coordinate system is

$$[d'_1 \quad d'_2 \quad d'_3] = [d_1 \quad d_2 \quad d_3 \quad d_4] \begin{bmatrix} \frac{\sqrt{3}}{2} & -\frac{1}{2} & 0 \\ 0 & 1 & 0 \\ -\frac{\sqrt{3}}{2} & -\frac{1}{2} & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad (\text{A.4})$$

Equation (A.4) gives the transformation from 4-index coordinate system to Cartesian coordinate system.

All the slip systems used in the simulation including 3 basal slip systems, 3 prismatic slip systems, 6 pyramidal $\langle a \rangle$ slip systems and 6 pyramidal $\langle c+a \rangle$ systems are listed in Table A.1 under both 4-index and Cartesian coordinate systems.

Table A.1 Slip systems both in 4-index coordinate system and Cartesian coordinate system

	4-index		Cartesian	
	Normal	Direction	Normal	Direction
Basal	(0002)	$\langle 2\bar{1}\bar{1}0 \rangle$	0, 0, 1	0.866, -0.5, 0
	(0002)	$\langle \bar{1}2\bar{1}0 \rangle$	0, 0, 1	0, 1, 0
	(0002)	$\langle \bar{1}\bar{1}20 \rangle$	0, 0, 1	-0.866, -0.5, 0
Prismatic	(10 $\bar{1}$ 0)	$\langle \bar{1}2\bar{1}0 \rangle$	1, 0, 0	0, 1, 0
	(0 $\bar{1}$ 10)	$\langle 2\bar{1}\bar{1}0 \rangle$	-0.5, -0.866, 0	0.866, -0.5, 0
	($\bar{1}$ 100)	$\langle \bar{1}\bar{1}20 \rangle$	-0.5, 0.866, 0	-0.866, -0.5, 0
Pyramidal $\langle a \rangle$	(01 $\bar{1}$ 1)	$\langle 2\bar{1}\bar{1}0 \rangle$	-0.4412, -0.7642, 0.4705	0.866, -0.5, 0
	($\bar{1}$ 101)	$\langle 11\bar{2}0 \rangle$	0.4412, -0.7642, 0.4705	0.866, 0.5, 0
	(10 $\bar{1}$ 1)	$\langle \bar{1}2\bar{1}0 \rangle$	0.8824, 0, 0.4705	0, 1, 0
	(0 $\bar{1}$ 11)	$\langle \bar{2}110 \rangle$	0.4412, 0.7642, 0.4705	-0.866, 0.5, 0
	($\bar{1}\bar{1}$ 01)	$\langle \bar{1}\bar{1}20 \rangle$	-0.4412, 0.7642, 0.4705	-0.866, -0.5, 0
	(10 $\bar{1}$ 1)	$\langle 1\bar{2}10 \rangle$	-0.8824, 0, 0.4705	0, -1, 0
Pyramidal $\langle c+a \rangle$	(1 $\bar{2}$ 12)	$\langle \bar{1}2\bar{1}3 \rangle$	0, -0.8515, 0.5243	0, 0.5243, 0.8515
	(2 $\bar{1}\bar{1}$ 2)	$\langle \bar{2}113 \rangle$	0.7375, -0.4257, 0.5243	-0.4541, 0.2622, 0.8515
	(11 $\bar{2}$ 2)	$\langle \bar{1}\bar{1}23 \rangle$	0.7375, 0.4257, 0.5243	-0.4541, -0.2622, 0.8515
	($\bar{1}2\bar{1}$ 2)	$\langle 1\bar{2}13 \rangle$	0, 0.8515, 0.5243	0, -0.5243, 0.8515
	($\bar{2}11$ 2)	$\langle 2\bar{1}\bar{1}3 \rangle$	-0.7375, 0.4257, 0.5243	0.4541, -0.2622, 0.8515
	($\bar{1}\bar{1}$ 22)	$\langle 11\bar{2}3 \rangle$	-0.7375, -0.4257, 0.5243	0.4541, 0.2622, 0.8515

Appendix II – Post-Processing ABAQUS Binary Output File

The command lines in the input file (‘.inp’) for ABAQUS to output state variables into the binary ‘fil’ file is as below:

```
*FILE OUTPUT, number interval=19
*EL FILE
SDV
```

where ‘number interval’ is used to decide how many increments are output.

The following is the code that uses subroutine ABQMAIN along with utility routine DBFILE.

```
      SUBROUTINE ABQMAIN
C
  INCLUDE 'aba_param.inc'
  CHARACTER*80 FNAME
  DIMENSION ARRAY(513),JRRAY(NPRECD,513),LRUNIT(2,1)
  EQUIVALENCE (ARRAY(1),JRRAY(1,1))
  Integer*8 KK, K1, K2, ct5, ct2000, ct11
  Integer*8 eleno, loop
  Real*8 TTime(1000)
  Real*8 rotation1, rotation2
  Real*8 temp_real
C
C  maximum times of calling DBFILE
  loop=160000000
C  Parameter initialization
  eleno=475947
  TTime=0.d0
  rotation1=0.d0
  rotation2=0.d0
  temp_real=0.d0
C  File initialization
  FNAME='name of the job'
  NRU=1
  LRUNIT(1,1)=8
  LRUNIT(2,1)=2
  LOUTF=0
  CALL INITPF(FNAME,NRU,LRUNIT,LOUTF)
  JUNIT=8
  CALL DBRNU(JUNIT)
C
  open (unit=24,file="directory\\SDV.txt")
  open (unit=25,file="directory\\time.txt")
```

```

open (unit=26,file="directory\\twincounter.txt")
open (unit=27, file="directory\\twinned_detwinned.txt")
open (unit=28,file="directory\\stress.txt")
C   Initialise all counting numbers
ct2000=0
ct5=0
ct11=0
C   Loop on all records in results file
DO 100 KK=1,loop
CALL DBFILE(0,ARRAY,JRCD)
C   End of result file
IF(JRCD.NE.0) GO TO 110
KEY=JRRAY(1,2)
C
IF(KEY.EQ.2000) THEN
ct2000=ct2000+1
TTIME(ct2000)=ARRAY(3)
WRITE(25,120, advance='no') ARRAY(3)

ELSE IF(KEY.EQ.5) THEN
ct5=ct5+1

C   SDV.txt - output all SDVs,208columns, step*eleno lines
DO 1000 K1=1,208
IF(K1.NE.208) THEN
WRITE(24,120, advance='no') ARRAY(K1+2)
ELSE
WRITE(24,120) ARRAY(K1+2)
ENDIF
1000 CONTINUE

C   twincounter.txt - output all twincounters
IF(mod(ct5,eleno).LT.0.2) THEN
WRITE(26,120) ARRAY(6)
ELSE
WRITE(26,120, advance='no') ARRAY(6)
ENDIF

C   ____sum up rotation_twin and rotation_detwin
IF((ARRAY(6).GT.0.5) .AND. (ARRAY(6).LT.1.5)) THEN
rotation1=rotation1+1
WRITE(27,*) '1',ct5
ELSE IF(ARRAY(6).GT.1.5) THEN
rotation2=rotation2+1
WRITE(27,*) '2',ct5
ENDIF
C

```

```

ELSEIF(KEY.EQ.11) THEN
  ct11=ct11+1
  DO 1020 K1=1,6
    IF(K1.NE.6)THEN
      WRITE(28,120, advance='no') ARRAY(K1+2)
    ELSE
      WRITE(28,120, advance='yes') ARRAY(K1+2)
    ENDIF
1020  CONTINUE

  ENDIF
C
100 CONTINUE
110 CONTINUE

  WRITE(*,*) 'Complete JOB=', FNAME
C
120 FORMAT (E16.7)
  STOP
  END

```

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