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**Investigation of microstructural evolution of aluminium
alloy 7050 at hot forging conditions**

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Abstract

Accurately predicting the grain dynamic recrystallisation (DRX) behaviour during hot forging aluminium alloy AA7050 poses a challenge to the aerospace industry, since the final grain structure and distribution in hot forged components determines many vital mechanical properties, such as yield strength, fatigue life and fracture toughness. So far, little research has been devoted to studying the dynamic recrystallisation of AA7050, even aluminium alloys as a whole, during typical hot forging thermal-mechanical processes due to the limited statistically meaningful microstructure characterisation technique and nature of its complexity involving strain, strain rate, temperature as well as microscopic dislocation density, low-angle grain boundaries (subgrain) and high-angle grain boundaries (grain).

Therefore, the aim of this work is to develop a physically-based hot forging material model and associated finite element (FE) simulation to accurately predict the thermomechanical behaviour and microstructural evolution as well as the geometry change in hot forging AA7050 parts. The thermomechanical behaviour is studied using thermal-mechanical simulator Gleeble 3800 under various hot forging conditions, including different strain levels (0 – 0.58), strain rates (0.0005 to 5 s⁻¹) and temperatures (380 - 460 °C).

Electron backscatter diffraction (EBSD) reveals the underlying dynamic recrystallisation mechanism. The GND density, LAGB and flow stress exhibit near-linear relationships with the Zener-Hollomon parameter. High dislocation density and more LAGBs are formed at lower temperature and higher strain rate. The dislocations in GND density and the LAGB area are found to be the dominating factor governing the flow stress in the tested ranges of strain rates and temperatures, regardless of the change of HAGBs. The relationship between HAGB area per unit volume and the Zener-Hollomon parameter is complex, comprising three regions: namely DRV, CDRX and DDRX. The largest HAGB area per unit volume within the CDRX region is found at the $\ln(Z)$ value of 22.5 (0.05 s⁻¹, 420 °C), where a relatively homogenous grain structure is observed due to the balance of the grain boundary growth and the transformation from LAGBs. These findings advanced understanding of dynamic recrystallisation is then mathematically described in a viscoplastic constitutive framework, which is subsequently embedded into a commercial FE software, ABAQUS, as a subroutine to simulate the open die hot forging process. The FE hot forging model for AA7050

demonstrates high accuracy in predicting material flow, dislocation density, low-angle grain boundary (LAGB) and high-angle grain boundary (HAGB), which are validated and evaluated through tests and characterisation.

This pilot study considerably advances our fundamental understandings of DRX in the thermal-mechanical processes of aluminium AA7050. It establishes a FE modelling framework to enable the stress-strain and DRX process to be accurately predicted in hot forging processes, which could potentially be used to significantly lower the trial and testing time and cost for hot forging large and extra-large aerospace components.

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6. Q Lu, C Zhang, W Wang, **S Jiang**, L Aucott, J Jiang, **Reveal the Viscoplastic Behaviour and Microstructure Evolution of Stainless Steel 316L**, Journal of material science (2022).

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Nomenclature

BCC	Body centred cubic
BE	Backscattered electron
CDRX	Continuous dynamic recrystallisation
DDRX	Discontinuous dynamic recrystallisation
DRX	Dynamic recrystallisation
EBSD	Electron backscatter diffraction
FCC	Face centred cubic
FE	Finite element
GDRX	Geometric dynamic recrystallisation
GND	Geometric necessary dislocation
HAGB	High angle grain boundary
HCP	Hexagonal close-packed
LAGB	Low angle grain boundary
SEM	Scanning electron microscopy
SFE	Stacking fault energy
SSD	Statistically stored dislocations

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Chapter 1

Introduction

1.1 Background and motivation

In the aerospace sector, as safety is a vital factor to be considered in aircraft design, excellent and known mechanical properties of its structural components are required. A large number of these components are manufactured through the hot forging process [1], which compresses the raw materials into their final designed shapes at elevated temperatures, deriving the benefit of homogenous, fine microstructure, which provides excellent and predictable yield strength and fatigue resistance [2]. Traditionally, designing a hot forging process for a component is mainly based on the empirical and user-experience method (*i.e.* trial & error). A few trials have to be undertaken in order to identify the acceptable, may not be the optimal, forging parameters such as forging temperature, speed and die design for material flow, such that forged parts have the acceptable geometries, grain structure and hence mechanical properties.

However, this trial-error-based approach results in a long lead time, high material wastage and manufacturing costs to the aircraft companies, especially for large or extra-large components, for which the die design & manufacturing, forging, and product inspection and testing are very costly.

Therefore, the aim of this project was to establish a reliable processing model to accurately simulate both material flow and microstructure evolution in the hot forging process. Consequently, the optimal forging condition can be efficiently determined through simulation, which will significantly shorten the lead time, lower the manufacturing cost and materials wastage.

Aluminium alloy, AA7050, an Al-Zn-Mg-Cu heat treatable alloy, has an excellent combination of high yield strength, fracture toughness, fatigue and corrosion resistance [3]; AA7050 has been widely employed in most current aircraft fuselages and rib structural

components, which are conventionally manufactured through hot forging process [4]. Hence, AA7050 is selected as the studying material in this thesis work.

During the hot forging, dynamic recrystallisation (DRX) is the main microstructure evolution mechanism, which is driven by stored plastic deformation energy, *i.e.* dislocation density, thermal energy, and time. It has been extensively studied in steel [5]. However, the detailed mechanism (DRX) in AA7050 remains still unclear, due to the high stack fault energy (SFE) of aluminium. Materials with high SFE at high temperatures undergo a very rapid recovery process, in which dislocations rapidly cross slip and jump to annihilate each other or rearrange themselves to form low-energy dislocation structures, *i.e.* low-angle grain boundaries (LAGB) with misorientation less than 15 degrees or so-called subgrains [6]. These LAGBs later transform into HAGBs as the process proceeds. This type of DRX is so-called continuous dynamic recrystallisation (CDRX). Although the general CDRX mechanism has been explored, there is still a debate on the detailed LAGB to HAGB transformation process, for which some research found the aluminium alloys undergo a gradual LAGB-HAGB transformation process with LAGB misorientation angle continuously increases to become HAGBs, which has the misorientation angle larger than 15 degrees. At the same time, some evidence [6] suggest that LAGBs pinch off to form HAGB without a gradual misorientation increase process. This LAGB pinching-off transformation process is defined as geometric dynamic recrystallisation (GDRX). So far, no study regarding the DRX mechanism in AA7050 has been found in the literature. Moreover, the majority of the current material science research has been devoted to advancing the understanding of underlying the underlying DRX mechanism of aluminium alloys at a given temperature and strain rate. The effects of deformation temperature and strain rate on DRX of aluminium alloys have been rarely explored and reported [6].

1.2 Structure of the thesis

This thesis consists of 8 chapters. First, Chapter 1 provides the background and challenges of hot forging aluminium alloy in the aerospace industry and introduces the overall aim of this study and the specific objectives to be achieved. Then, Chapter 2, the literature review chapter, gives an overview of research findings for the current forging techniques, followed by a detailed review of the DRX mechanisms in aluminium alloys and available material

constitutive models to describe the stress-strain behaviour in hot forming conditions. The research gaps are identified and discussed in each section. Chapter 3 is the experimental methodology chapter, which describes the as-received AA7050 samples, thermomechanical tests and microstructural characterisation conducted by Gleebe 3800 and EBSD, respectively.

Following on, Chapter 4 and Chapter 5 are the main experimental results chapters, reporting the thermomechanical behaviour and providing an in-depth discussion regarding the relationship between the stress-strain relationship and the DRX as strain level increased. Additionally, the effects of temperature and strain rate on the dynamic recrystallisation during hot compression are presented in Chapter 5. The microstructural results, including the dislocation density, GND density, LAGB and HAGB evolution and distributions, are used to develop constitutive relations with the material's viscoplastic responses (stress-strain relation).

Chapters 6 and 7 present the modelling work, including the establishment of a new set of DRX constitutive equations based on the experimental findings obtained in Chapters 4 and 5. This dynamic recrystallisation constitutive model is embedded into FE software, ABAQUS, to simulate an isothermal open die forging. The FE hot forging simulation results are compared and validated with experimental data at macroscopic (stress-strain and shape change) and microscopic (GND density, LAGB & HAGB) levels.

Finally, the conclusions drawn from Chapters 4-7 and suggested future work are presented in Chapter 8.

Chapter 2

Literature review

2.1 Introduction

This review focuses on the advancement of the studies on the AA7050 forging processes, their underlying scientific issues and constitutive model development. It mainly consists of four parts:

- (i) Introduce forging techniques and review the research on forging and forging process of AA7050.
- (ii) Introduce the background knowledge and physical properties of the aluminium alloy AA7050; the industry requirements and applications of hot forged AA7050 structural components.
- (iii) Comprehensively review the current state of DRX mechanisms in aluminium alloys and their resulted macroscopic stress strain responses; The effect of temperature and strain rate on the kinetics of dislocation density, GND density, low-angle grain boundary (subgrain) and high-angle grain boundary (grain) evolution is also reviewed.
- (iv) In-depth review materials constitutive models: the current research status of the phenomenological constitutive models, the physically-based models, emphasising on the development of DRX within physically-based constitutive models.

2.2 Forging process

Forging process eliminates internal flaws and refines the microstructure through plastic deformation [7]. The forging process could give good thermomechanical properties and corrosion resistance, compared with other manufacturing processes such as rolling, casting, drawing and extrusion. In addition, the cost of forging process is much higher than other as well. Thus, how to choose a right manufacturing method would be balanced with the desired properties and cost. Forging products have been widely used in the structural materials of

transportation industries: typically, around 85% of aircraft components, 60%~70% of automotive components are made by forging products [8].

Forging can be classified as open die forging, close die forging and flashless forging, as displayed in Fig. 2.1. Open die forging, also named as upset forging, is the simplest one which formed by two flat dies with a work piece in the middle. Close-die forging is the process which squeezes work piece within its cavity to deform to a desired shape during compression process. Flashless forging is similar to close die forging without any flash.

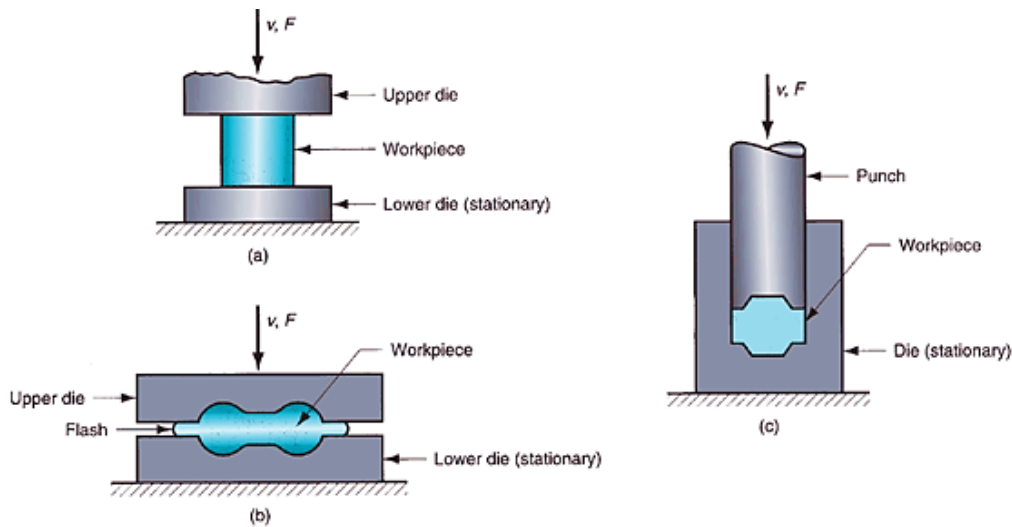


Fig. 2. 1 (a) Open die forging, (b) impression-die forging, and (c) flashless forging [9].

Depending on the forging temperatures, forging can also be categorised as: cold forging $T < 0.3 T_m$ (typically near room temperature), where T is the temperature in Kelvin and T_m is the melting temperature of material; warm forging $T = 0.3-0.5 T_m$ (above room temperature and below recrystallisation temperature); and hot forging $T > 0.6 T_m$ (above recrystallisation temperature) [10]. The advantages and disadvantages of cold, warm and hot forging are listed in Table 2.1.

There are plenty of parameters during forging process need to be considered: temperature, strain rate (compressive force and speed), deformation magnitude (True strain/deformation level). Meanwhile other forging operating additions and facilities also need to be optimised: preferable lubricants, design the shape of the mould, *etc.*

Table 2.1 The advantages and disadvantages of cold, warm and hot forging

Type of the Process	Pros	Cons
Cold forging	• High precision • Good surface finish • Low wastage • Improved parts integrity and strength	• High forming force requirements • Low formability • Several preform and annealing processes needed
Warm forging	• Combined merits of cold and hot forging • No annealing needed	• Tooling withstanding high forging-force and high temperature. High Tooling Cost
Hot forging	• Good formability • Low forming force requirement • Further elimination of chemical incongruities	• Formation of scales • Less accuracy • Difficult microstructural control • Poor final surface finish

2.3 Aluminium alloys 7050 and applications

2.3.1 Aluminium alloy 7050

In this study, AA7050 is selected as the studying material due to its industrial significance. The recommended forging temperature range for AA7050 is from 405 to 434 °C, as shown in Fig. 2.2. With the forging temperature increasing, the forge ability of aluminium alloys improves [11]. The temperature for heat treatment should below overheat temperature 485 °C. Therefore, to cover a slightly large range of temperatures, 350 to 450 °C is selected in this project. Its main thermal and physical properties are reported in Table 2.2.

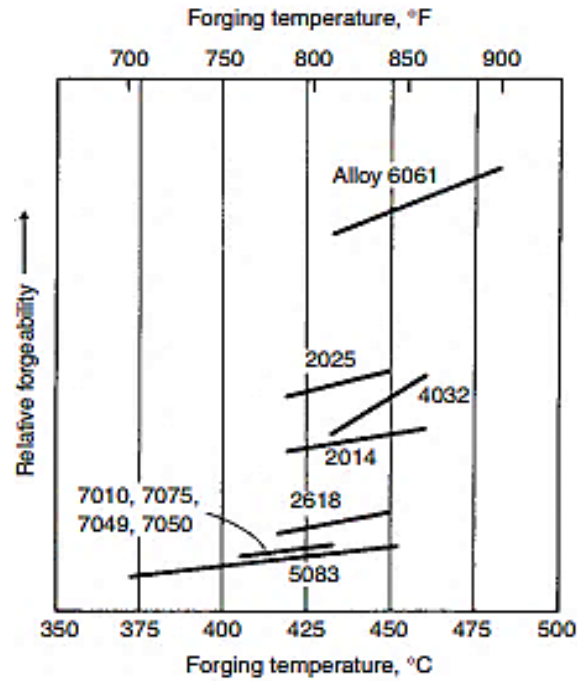


Fig. 2. 2 Different forging temperature and forgeability of aluminium alloys [11].

Table 2.2 Related physical and thermal properties of AA7050 [12-13] .

Physical properties	Value	Thermal properties	Temperature (°C)
Young modulus	72000 MPa	Annealing	415
Yield point	435 MPa	Solid heat treatment	475
Ultimate tensile strength	524 MPa	Solidus	488
Density	2.83 g/cm ³	Melting	629
Poisson ratio	0.33	Overheat	485

AA7050 material is received in plate form. It is important to understand its thermal mechanical history to interpret its initial microstructure. The manufacture process history mainly comprises casting, homogenising, hot rolling, solution heat treatment, quenching and age hardening stages, as displayed in Fig. 2.3 [14].

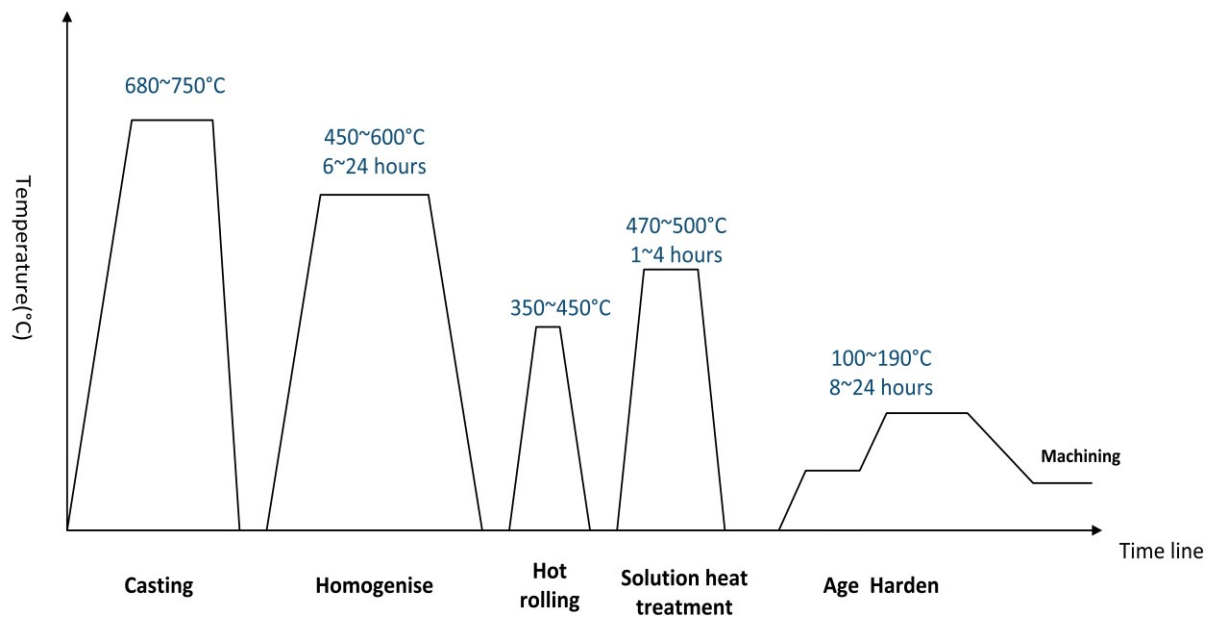


Fig. 2. 3 The thermal-mechanical history of AA7050 plate.

The casting temperature is around 680-750 °C. In this stage, aluminium and other addition elements are melted together with grain refiner added. The grain refiner forms particles in the melt which act as nucleation sites during solidification leading to a finer grain structure [14]. After casting process, the alloying elements are very unevenly distributed in the ingot (segregation). To tackle the segregation problem, homogenising stage (at 450-600 °C for 6-24 hours) is added to redistribute soluble alloying elements, dissolve large intermetallic particles and precipitate sub-micron dispersoid particles [15]. Subsequently, multi-pass hot rolling (at 350-450 °C) is applied to develop fibrous grain structure and breaks up large particles [14]. Then, solution heat treatment with controlled quenching is undertaken to dissolve all precipitates. Finally, the plate undergoes an aging process (100-190 °C, 8-24 hours) to form fine uniformly distributed precipitates to maximums its strength [16].

2.3.2 AA7050 applications

In spite of growing use of composite materials in airframe construction nowadays, aluminium alloy still remains important as structural materials. As shown in Fig. 2.4, aluminium alloy used in new generation of Airbus 380 is still more than 60%. The percentage of aluminium alloy used in Boeings 747, 757, 767 and 777 are more than 70%.

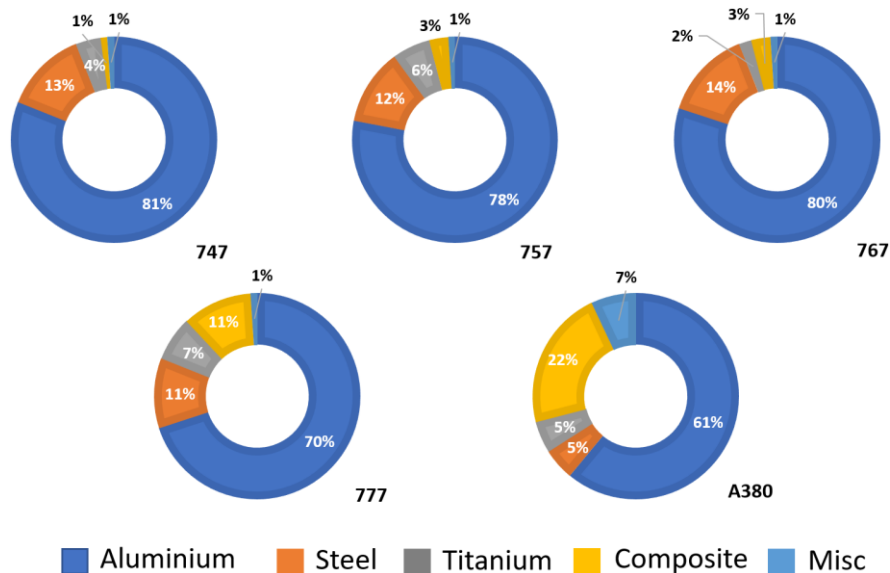


Fig. 2. 4 The percentage of the materials used in aircraft [4, 17-18].

AA7050 has been widely used as aircraft parts such as wing skins, fuselage frames, bulkheads, aerospace structure, and commercial and military aircraft applications [4]. For example, AA7050 used in several structural components of the aeroplane (Boeing 777): upper and lower wing skins, fuselage frames and wing spars and ribs are shown in Fig. 2.5.

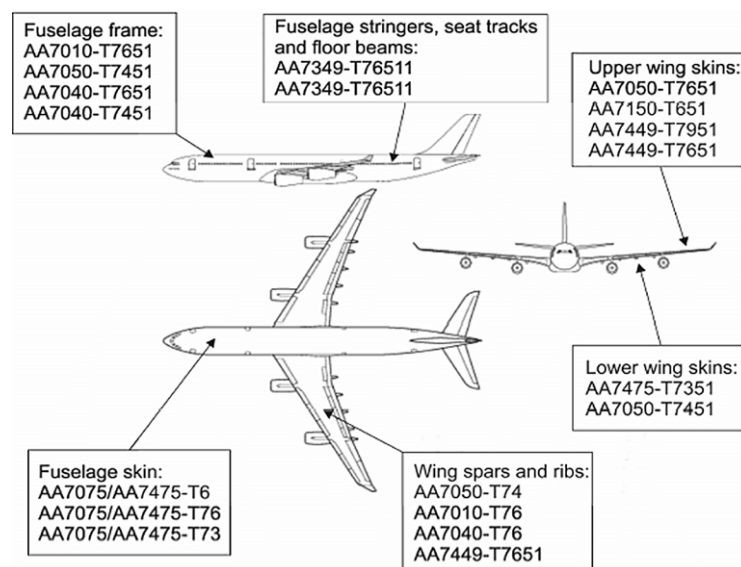


Fig. 2. 5 A schematic diagram of Boeing 777 which is formed by different 7xxx series aluminium alloys [19].

2.4 Dynamic recrystallisation mechanisms

Dynamic recrystallisation is a vital process for metallic alloys to achieve the desired grain structure, morphology and texture, determining the materials' mechanical properties [20]. It typically occurs in alloys at elevated temperatures under imposed plastic deformation. During such a process, the initial grain orientation, morphology, size, and the contained dislocation density are gradually replaced with a completely new set of grains. The grain refinement and hence the improved strength, fracture toughness, fatigue resistance and sometimes ductility can usually be achieved [21].

Differing from static recrystallisation, where the pre-deformed materials are heat-treated for the plastic deformation-induced dislocations to recover, rearrange, and form new grains and grow as a function of time, dynamic recrystallisation has more complex and mixed characters, because of the concurrent plastic deformation and heat treatment, where dislocation generation and annihilation, and grain formation and growth occur at the same time [20].

Dynamic recrystallisation occurs frequently and sometimes predominates the flow stress and microstructure evolution in various metal-forming processes at elevated temperature, such as forging, rolling and extrusion. Because warm/hot forming can lower the required flow stress and hence the tonnage of the required equipment to form the components, the formability and final quality of the parts are generally better than room temperature-formed ones [20]. Thus, an understanding of dynamic recrystallisation is crucial to enable the accurate control and prediction of the materials' flow, microstructure evolution during the forming and the final mechanical properties of the post-formed parts.

2.4.1 SFE and CDRX and DDRX

Primarily, depending on the stacking fault energy (SFE) of the studied alloys, the materials exhibit two distinct dynamic recrystallisation behaviours. SFE is defined as the energy required for an individual dislocation to dissociate into partial dislocations [22]. If the SFE of the material is low, such as nickel, copper and stainless steel, it means that, in these materials, the dislocations are energetically favourable to be dissociated into partial dislocations. These partial dislocations are energetically too stable to cross-slip or climb at elevated temperatures [23]. Thus, high dislocation density, *i.e.* the stored energy, can be effectively accumulated, leading to the formation of nuclei for new grains to be generated. Accordingly, clear dynamic

recrystallised grain formation and growth with relatively random orientation are often seen adjacent to the grain boundary regions, where dislocation densities tend to be the highest. This dynamic recrystallisation is defined as discontinuous dynamic recrystallisation (DDRX).

On the other hand, if the SFE of the material is high, *e.g.* aluminium alloys, the dislocation dissociation will be difficult to occur. At elevated temperatures, dislocations are more mobile in the crystals across different slip systems through cross-slipping and climbing. These processes enable the dislocations to be accumulated as well as annihilated through the cancellation of oppositely signed dislocations. The remaining dislocations tend to rearrange themselves into a low energetic form, so-called subgrains, consisting of low-angle grain boundaries (LAGBs), formed by a group of dislocations resulting in geometrical misorientation within crystal lattices, *i.e.* geometrically necessary dislocations (GNDs). As the dynamic recrystallisation proceeds, these LAGBs are progressively rotated and their misorientation angles are increased and eventually transformed into high-angle grain boundaries (HAGBs) or ordinary grains [23]. Due to the nature of continuous subgrain boundary rotation without recognisable nucleation and growth of recrystallised grains, this dynamic recrystallisation process is termed as continuous dynamic recrystallisation (CDRX) [23].

2.4.2 Outstanding dynamic recrystallisation problems

Dynamic recrystallisation phenomena have been extensively studied for decades. The existing knowledge framework obtained from experimental and numerical studies can already provide valuable guidance to control the microstructure during forming processes. Recrystallised grain nucleation and grain growth through grain boundaries migration in various materials and conditions have been thoroughly investigated: underlying mechanisms and numerical models have been observed and developed, respectively. This is particularly true for the DDRX process in steels [21, 23].

Compared to DDRX, some signs of progress have been made in CDRX, however, the underlying dynamic recrystallisation mechanisms, especially the new grain formation process, are still under debate [21, 23]. The transformation from subgrains to grains is unclear in some cases. For example, under severe plastic deformation, in equal channel angular pressing and accumulative roll bonding, ultra-fine grains can be achieved [24]. Humphreys and Hatherly [6] and McQueen et al. [25] proposed that there is another dynamic recrystallisation

mechanism, called continuous dynamic recrystallisation (CDRX), in which the subgrain LAGB saturates at 2° rather than continuously increasing to form HAGBs, the new grains are formed from the grains' geometrical changes. As pointed out by the recent reviews on dynamic recrystallisation [21, 23], the distribution and evolution of GND density, and the length and misorientation angles of LAGB and HAGB during dynamic recrystallisation are still elusive, so systematic studies are required. Because of this lack of clear understanding of CDRX mechanisms, there are few models to accurately describe the CDRX process and capture the microstructure evolution [23].

2.4.3 Practical limitations

There are a few practical issues hindering us from clarifying the detailed CDRX mechanisms. Firstly, accurately controlling and measuring the thermomechanical process is non-trivial because a large thermal gradient often exists within the samples. This thermal gradient affects the dynamic recrystallisation kinetics and final recrystallised grain structure distribution. Also, post-deformation recrystallisation may occur if the testing samples are not quenched quickly enough. The quenching process is practically difficult to quantify and control and would affect the observed relationship.

Secondly, statistical sampling is not easy and only qualitative observation rather than statistically quantitative results can be obtained. Although macroscopically uniform plastic strain is imposed onto the samples, considerably inhomogeneous plastic deformation is distributed at meso/microscopic scales [26] because of the anisotropic plasticity associated with grain orientations and the geometrical compatibility requirement [27], which ensures that during the plastic deformation a grain and its neighbouring grains have to be deformed together. If only a limited number of grains were characterised, the proposed mechanisms and relationship could be questionable and inconclusive. This may explain why the current CDRX results are very scattered and some of them are confusing and controversial [23].

2.4.3.1 Limited representative data

A CDRX process involves three key variables, namely dislocation density, LAGBs and HAGBs. Acquiring statistically meaningful maps for these three variables was unfeasible. For example, transmission electron microscopy (TEM) is probably the most widely used technique for dislocations and LAGBs characterisation [28]. However, its sampling size is only a few or tens of μm^2 , which is far too small to represent the general behaviour of

dislocations or LAGBs, as different grains, even with the same orientation but different neighbouring grains, would have completely different dislocation and LAGB structures [29].

As the sample preparation is very time-consuming and labour-intensive, acquiring a sufficient number of TEM images to be statistically meaningful is unfeasible. Optical microscopy (OM) is another commonly used grain size and morphology characterisation technique, through chemically etching the free surfaces that grain boundaries with a large number of vacancies are preferentially etched out and showing high contrast to the grain interior [22]. However, the HAGBs and LAGBs are indistinguishable under OM. Also, the stored dislocations are insensitive to the etching process, remaining invisible under OM.

2.4.3.2 The emergence of EBSD

The emergence of electron backscatter diffraction (EBSD) around the early 21st century, which automatically generates crystal orientation/misorientation maps by scanning and measuring the crystal orientation of each probed point, is very promising. The orientation is determined through indexing the formed Kikuchi patterns on a phosphor screen, generated from backscattered electrons in an SEM [30].

EBSD provides valuable information about the misorientation, LAGBs and HAGBs. Much earlier and recent experimental CDRX work used the EBSD technique to identify LAGB and HAGB behaviours [6, 23]. In earlier studies, though EBSD did provide significantly more grain orientation information at a larger map size than TEM, acquiring statistically meaningful grain orientation maps, containing > 1000 grains, was still challenging because the EBSD acquisition rate (10 scanning points per second) was relatively low, especially in the largely deformed materials, for which longer camera exposure time was required [30]. The recent improvement of EBSD hardware allows cameras to achieve 1000-3000 scanning points per second [31]. Also, little information about how the stored dislocations behave was known, which is probably the most important information for determining the flow stress behaviour and recrystallised grain nucleation/subgrain formation.

The estimation of dislocation density using EBSD was premature, as the mathematical framework to convert misorientation to GND density was incomplete and not yet validated until Wolfgang et al. [32] and Wilkinson et al. [33] developed the mathematical framework to estimate GND density tensors from the characterised grain orientation at each probing point and the derived rotation tensors. Also, to what extent GND would represent the total

dislocation density was unknown until more recent research by Jiang et al. [34] on the quantitative study of the relationship between GND density and hardening behaviour during a tensile test and subsequent qualitative comparison between dislocation density characterised using transmission Kikuchi diffraction on steel [35], and etching on polycrystalline silicon [36], which confirmed that GND density estimated by EBSD could indicate the total dislocation density behaviours. These issues were not resolved until recently and hindered us from clarifying the underlying CDRX mechanisms.

2.5 Stress-strain behaviour and microstructure evolution

2.5.1 Constitutive relationships and microstructure evolution

Constitutive relationship describes the stress-strain behaviour of a material [37]. Different from the ambient condition, where the strain is the predominant factor determining the stress responses (*e.g.* hardening, softening or equilibrium), at elevated temperatures, the flow stress (σ) is mainly governed by temperature (T), strain rate ($\dot{\epsilon}$) and strain (ϵ), and the materials become temperature and strain rate sensitive [38]. These fundamentally stem from the activation/increase of the diffusion process at the elevated temperature, which increases the overall thermal energy in the materials. This thermally activated diffusion process gives rise to a number of mechanisms such as dislocation climb/cross-slip, subgrain formation, grain boundary sliding, grain boundary transformation and migration, etc [39]. These mechanisms altered the deformation mechanism from the pure dislocation slips to grain boundary sliding, influencing the flow stress.

As the nature of the diffusion process is time and temperature dependent, the deformation speed (strain rate) and deformation temperature exert significant effects on the flow stress. The dominating mechanism may change from one temperature and strain rate condition to another. Such strain rate and temperature sensitive flow behaviours are so termed as viscoplastic flow in solid mechanics [40]. In the past two decades, there has been increasing attention devoted to studying the mechanical property and microstructure evolution during high temperature deformation, due to its industrial importance, as most metal manufacturing processes are carried out at elevated temperatures [41-43]. Extensively research has been undertaken to explore the thermal-mechanical behaviour of materials. Typically, thermal-

mechanical tests at various temperatures and strain rates are undertaken, and the corresponding stress-strain curves are reported as the properties of the testing materials [44].

The general shapes of the stress-strain curves have been recognised and categorised. Their corresponding governing microstructure mechanisms have been investigated and accepted as schematically shown in Fig. 2.6, in which the main hardening and softening processes are identified as work hardening, dynamic recovery and dynamic recrystallisation (discontinuous dynamic recrystallisation (DDRX), continuous dynamic recrystallisation (CDRX), geometric dynamic recrystallisation (GDRX)) [21].

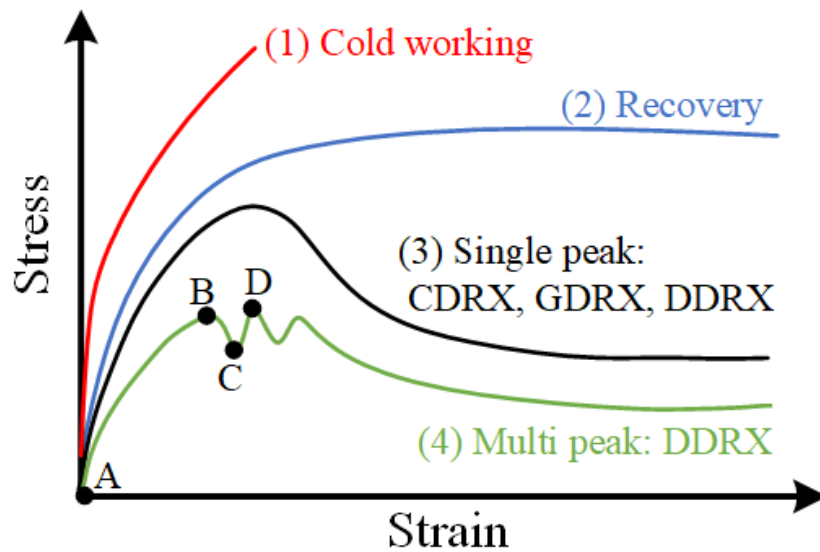


Fig. 2. 6 Schematic illustration of the flow stress behaviour with different mechanisms [21].

Since both strain rate and temperature affect the flow stress and they are all based on the diffusion process, it is convenient to combine these two thermal-mechanical variables into a single parameter - the Zener Hollomon parameter (Z), which is defined as [45]:

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \quad (1)$$

where R is the universal gas constant, $8.31 \text{ J mol}^{-1}\text{K}^{-1}$, Q is a ‘deformation’ activation energy that is close to the bulk diffusion in aluminium alloys. Some thermal-mechanical behaviour studies of AA7050 found that the $Q=160.3 \text{ kJmol}^{-1}$ [46-47].

It is found that there is often a strong positive empirical linear relationship between the flow stress and Z parameter as described by $Z = c_1 \sinh(c_2 \sigma)^n$, where c_1 , c_2 and n are constants,

as proposed by Sellars and Tegart [48]. Therefore, Z is often used to represent the temperature and strain rate effects on the flow stress.

Alternatively, the relationship between strain rate and stress can be expressed explicitly according to Equation 2. This empirical relationship often works well and is able to represent the experimental data. For example, Hu et al. [49] conducted the thermal-mechanical test on the same material as this study, AA7050, their data were analysed using $\ln(\sigma)$, $\ln(\dot{\epsilon})$ and temperature. Linear relationships are exhibited, as shown in Fig. 2.7. It should be pointed out that although these simple empirical relationships can describe the stress, strain rate and temperature well, their underlying physics is unknown, preventing us from predicting microstructure and hence the post-forming mechanical properties.

$$\dot{\epsilon} = k\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (2)$$

where k and n are constants to be determined.

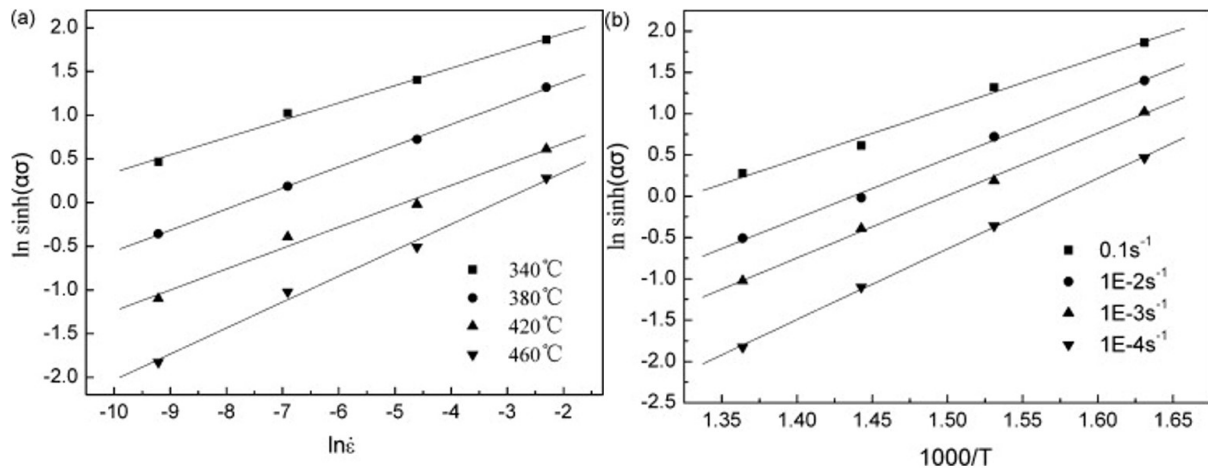


Fig. 2. 7 The relationships of peak stress and strain rate and temperature at ln scale for AA7050 [49].

A general understanding of microstructure evolution during thermal-mechanical testing of aluminium alloys has been established. In aluminium alloys, due to their high stacking fault energy, CDRX/GDRX is found to be the primary DRX mechanism. In short [50], during the plastic deformation, dislocations slip, annihilate, accumulate and pile-up to form a dislocation network and gradually transform into LAGBs. As the deformation proceeds, more dislocations are generated and increase the misorientation angle of LAGBs. Eventually, these LAGBs are transformed into HAGB by increasing their misorientation angle to higher than 15°.

Meanwhile, some of the HAGBs may migrate to eliminate some of the dislocation density, the area of LAGB or even HAGB depending on the migration velocity and driving force (difference in dislocation density across either side of HAGBs). Meanwhile, the grain boundary sliding may occur to accommodate the plastic deformation through grain rotation depending on the deformation conditions ($\dot{\epsilon}$ and T). Under severe plastic deformation ($\epsilon > 2$), in which the thickness of grains is compressed to a level similar to subgrain size, the CDRX is argued to take place by grain boundary impingement to form fine grains [51]. A schematic summary of the overall key mechanisms and their evolution, relationships to flow stress, *e.g.* viscoplastic behaviour, are displayed in Fig. 2.8 [21]. The CDRX in the aluminium alloy is rather a complex process involving many mechanisms concurrence, counterbalancing each other. Therefore, it is vital but challenging to identify the governing mechanism and its kinematic under a selected deformation condition(s), which would ensure the validity and accuracy of the model development and prediction. However, there is still lack of systematic investigation of the CDRX kinetics, as acknowledged in a recent review [23].

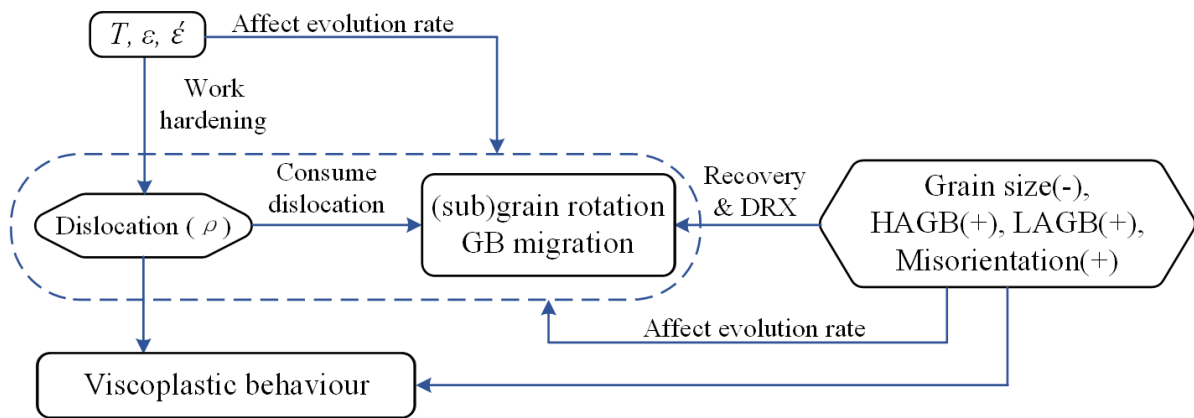


Fig. 2. 8 Relationship between external parameters and microstructural variables [21].

Ample evidence in the literature exists to support the current understanding of microstructure evolution as a function of strain under a selected thermal-mechanical condition. However, little research has been devoted to understanding the roles of temperature and strain rate on the kinetics of these mechanisms, despite the extensive testing data on the thermal-mechanical behaviours of the materials [23]. These are attributed to two main reasons: inappropriate utilisation of characterisation tools and inappropriate data analyses. OM and TEM are inappropriate to investigate the dislocation density, LAGB and HAGB, because of the incapability of distinguishing these three variables (OM), or the statistical nonmeaning nature of TEM. However, many studies [49, 52-53] have adopted these two characterisation

methods to attempt to clarify the relationships between microstructure and viscoplastic behaviour, which led to highly questionable conclusions.

Because of the increasing adoption of the EBSD technique in the past two decades, EBSD has been utilised to shed light on the relationships [46]. However, inappropriate data analyses were performed to clarify the relationships. For example, Zhao et al. [46] studied the strain rate effect on the recrystallisation mechanisms during isothermal compression of 7050, which has similar objectives, the same materials and methodology to the current study. Even more extensive thermal-mechanical testing and EBSD/TEM characterisation were undertaken. However, only the volume fraction of HAGB, the mean grain size and mean subgrain size were investigated as functions of deformation temperature and strain rates, which did provide important information about the underlying microstructure. However, there remain a number of crucial unanswered questions regarding the mechanisms of LAGB and HAGB formation and transformation. Also, despite the available TEM results, the study overlooked the critical dislocation density activities.

The current understanding of the effects of temperature and strain rate on dislocation density, LAGB and HAGB are summarised in the following sections.

2.5.2 Temperature and strain rate effects on dislocation density

The dislocation density evolution is broadly divided into two types, depending on the occurrence of recrystallisation. In the case of no recrystallisation, the dislocation density exhibits a rapid increase by dislocation pile-up through dislocation-dislocation interaction or at dislocation barriers such as grain boundaries. This increasing trend continues until a steady-state is reached, due to the equilibrium between the dislocation accumulation and dislocation recovery through climb and cross-slip [54-56]. This entire trend shifts up with decreasing temperature and increasing strain rate, as schematically illustrated in Fig. 2.9(a). It suggests that there will be a higher dislocation density at the steady-state at lower temperature and fast loading speed due to the suppressed recovery.

On the other hand, if the recrystallisation occurs, instead of reaching a steady-state, there will be a peak during the dislocation density evolution. After the peak, a gradual decrease in dislocation density is shown. The peak of dislocation density suggests the onset of the recrystallisation process. It reduces the dislocation density through either grain boundary migration to eliminate the dislocations and LAGBs, or the HAGB transformation from the

dislocation formed LAGBs [57]. The peak dislocation density values increase with a higher strain rate and lower deformation temperature due to the suppression of dynamic recovery (DRV) and dynamic recrystallisation (DRX) [58-59], as schematically shown in Fig. 2.9(b).

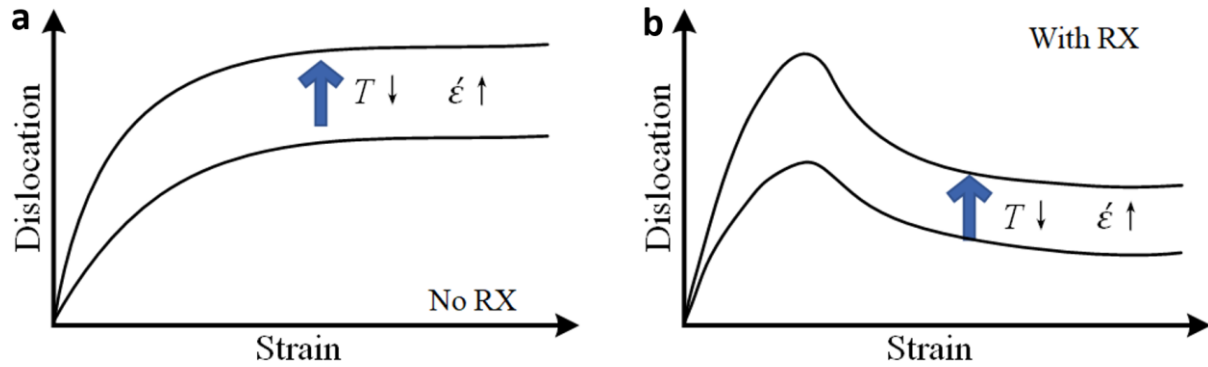


Fig. 2. 9 The dislocation density evolution as a function of plastic strain with (a) no recrystallisation and (b) recrystallisation. The effects of deformation temperature and strain rates are also shown [21, 54, 60].

2.5.3 Temperature and strain rate effects on LAGB, HAGB areas

Regarding the LAGB and HAGB area evolution and their relationships with deformation temperature and strain rates, there are two aspects that should be considered, namely the area of LAGB and HAGB per unit volume, in other words, the sizes of subgrains and grains; secondly, the misorientation angles of LAGB and HAGB since the HAGBs are formed through the transformation of LAGBs with gradually increasing the misorientation angles.

Considering the areas of LAGB and HAGB, the general evolution and exerted influence by the temperature and strain rates are shown in Fig. 2.10(a) and (b). For the LAGB, since they are evolved from the dislocation network, the trends of LAGB as functions of temperature, strain rate and strain, are consistent with the dislocation density evolution trends, in the case of no recrystallisation. The fundamental mechanism is also similar to the dislocation density that the equilibrium state reaches, when the dislocation accumulation and recovery reach an equilibrium state. The higher temperature and lower strain rate would promote dynamic recovery (DRV) and facilitate the dislocation annihilation [50]. Thus, less LAGBs area is formed. It would be expected that dynamic recrystallisation will reduce the steady-state LAGB by HAGB migration.

On the other hand, regarding the HAGB, it is a bit complex as it is formed through a continuous transformation from LAGB in CDRX by further increasing its misorientation angle or grain pinch off from its boundary during CDRX. The HAGB area is larger when there are more LAGBs, *i.e.* under lower temperature and strain rate (dislocation accumulation

overtaken the DRV). However, higher temperature and longer deformation duration (low strain rate) do promote the LAGB to HAGB transformation as sufficient thermal energy and duration are essential to facilitate this transformation process. If the temperature and duration keep increasing, the formed HAGB will start to grow and migrate to lower its surface energy. As a result, the HAGB area will decrease so will LAGB and dislocation density. In practice, identifying the critical condition where the finest grains will be achieved without grain growth is challenging.

It should be mentioned that the LAGB and HAGB areas can represent the subgrain and grain sizes while having inverse relationships. The discussed trends can apply to the subgrain and grain size directly.

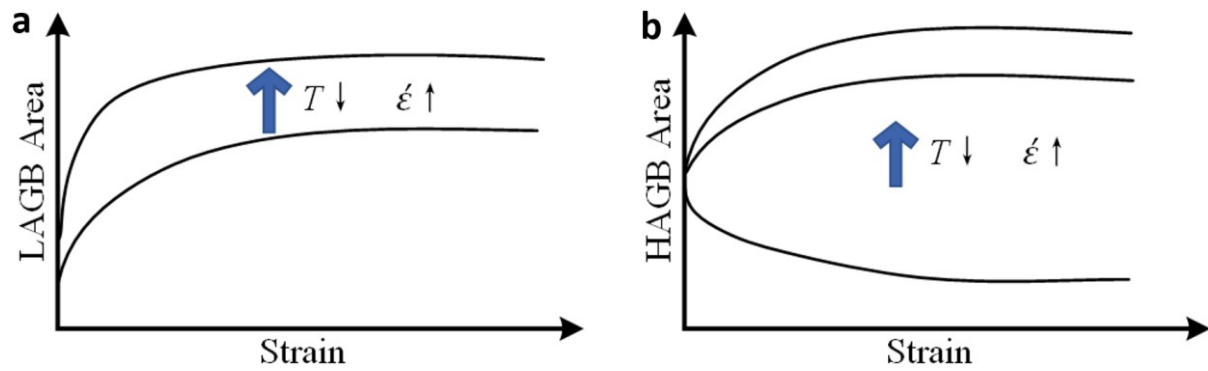


Fig. 2. 10 Schematic illustration of changes in boundary area with strain: (a) LAGB area evolution and (b) HAGB area evolution per unit volume, which have similar trends but different mechanisms depending on whether or not recrystallisation occurs [21].

2.5.4 Temperature and strain rate effects on the misorientation of boundary

The overview of the evolution of misorientation angle as functions of strain, strain rate and temperature are generally accepted in the research community, as schematically illustrated in Fig. 2.11. Regarding the LAGB, it is considered in CDRX and GDRX cases, respectively. If there is no dynamic recrystallisation, the average LAGB misorientation angle increases rapidly and would be saturated at 2° at the strain level of approximately 1 [61]. Note that LAGB in here is defined as the boundaries with the misorientation angle range from 0° to 15° , since geometrically necessary dislocation density is not considered these analyses. Whereas, in a typical CDRX case, the LAGB misorientation angle would rise rapidly and saturated at 6° at the strain level of approximately 1-2 [62-63]. In the case of CDRX, however, $\sim 1/3$ of LAGB would saturate at a higher misorientation angle toward 15° , and the other $2/3$ of LAGB would saturate at 5° [51, 64-65].

Regarding the HAGB misorientation, there is little variation if no dynamic recrystallisation takes place. When dynamic recrystallisation occurs, initially, there is no variation in misorientation angle until the plastic strain level of 1-2 is reached. Then a continuous increase in misorientation angle emerges [66-68]. It should be noted that the onset of HAGB misorientation increase and LAGB misorientation saturation are all subjected to a relatively large strain, *i.e.* larger than 1, which is challenging to achieve in uniaxial compression. It is why severe plastic deformation methods such as equal channel extrusion, accumulative rolling, *etc.*, have been widely used to study the recrystallisation behaviour in aluminium alloys. Severe plastic strain is beneficial to reveal the complete recrystallisation mechanism until the steady-state is reached. Nevertheless, these severe plastic deformation techniques do not represent the industrial process where only moderate plastic strain is imposed, similar to the uniaxial compression.

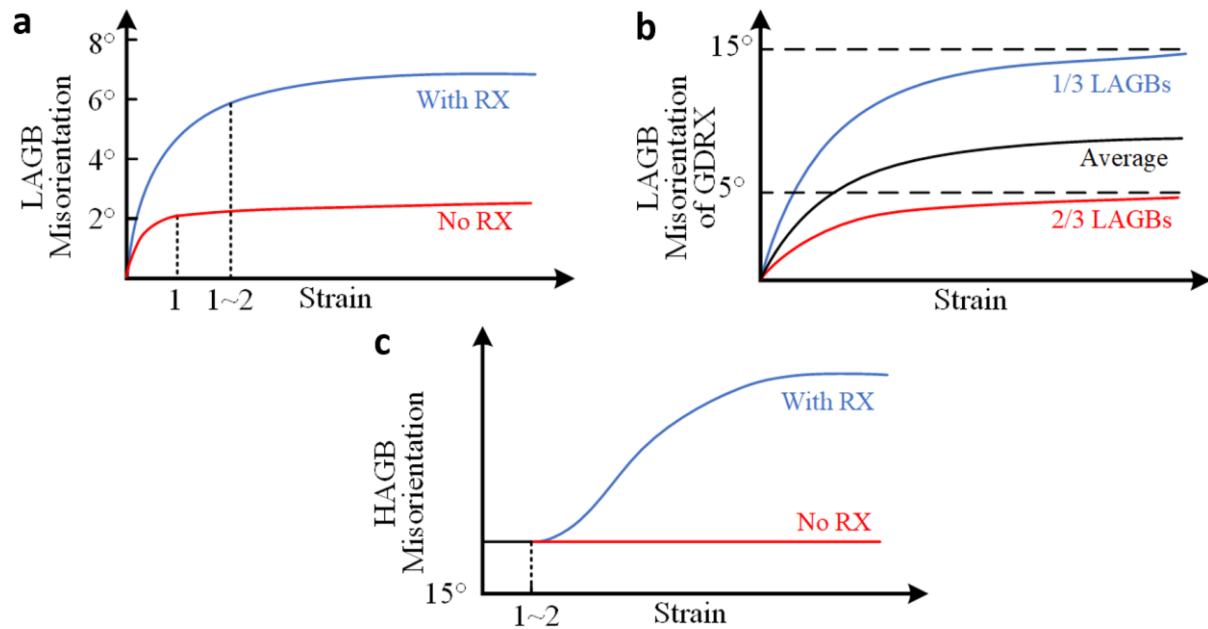


Fig. 2.11 Schematic drawing of the dynamic recrystallisation and no dynamic recrystallisation cases showing how (a) LAGB misorientation evolve in CDRX, (b) LAGB misorientation evolve in CDRX and (c) HAGB misorientation evolve [21].

It should be pointed out that although the general understanding of the dislocation density, LAGB and HAGB evolution as functions of strain, strain rate and temperature has been well accepted, many trends are speculated based on the temperature and strain rate effects observed on a single mechanism or different materials. There is a lack of systematic study on this topic. Probably due to the complex nature of dynamic recrystallisation in aluminium involving DRV, CDRX, GDRX, dislocation accumulation and grain boundary sliding, many existing studies are contradictory. For example, the opposite effects of temperature on LAGB

misorientation evolution have been reported [63]. AA7475 shows an increasing average misorientation with temperature, while the inverse is reported with AA2219 [66, 69-70].

The underlying mechanism is still poorly understood. Despite the decades of research work on dynamic recrystallisation in aluminium, the critical question about the dislocation density evolution as a function of temperature and strain rate is largely overlooked. To date, to the best of my knowledge, no study has been devoted to systematically investigating the roles of temperature and strain rate on the dislocation density, LAGB and HAGB evolution, with the aim to identify the dominating mechanism of flow stress.

2.6 Constitutive modelling of recrystallisation process

Material flow behaviour during hot forming process is very complex. Hot forming temperature, strain rate and magnitude of strain would influence the hardening and softening mechanisms within metals. Based on the experimental results, a number of constitutive models were used to accurately predict the material properties which include temperature dependence, strain rate and strain dependence, and the hot deformation behaviours of metals. There are three different groups of constitutive models: phenomenological constitutive model, physical-based model and artificial neural network.

Understanding and modelling the flow stress responses as functions of strain, strain rate and deformation temperature has great scientific and engineering importance. Scientifically, how materials behave under various strain, strain rate and temperature is critical to understanding the material mechanical properties related to their strength, ductility, fracture toughness and creep performance [71]. The underlying mechanisms causing the changes in flow stress behaviour are important to further understanding of material science and its association with materials' mechanical properties. Engineering-wise, exploring and accurately modelling the materials' flow stress behaviour is vital for manufacturing companies to effectively design and optimise their manufacturing processes, such as rolling, forging, extrusion, *etc.*, where the materials are subjected to a wide range of deformation conditions [71].

Thus, as earlier as 1914, Andrade et al. [2] proposed the first phenomenological model to mathematically describe the viscous flow stress of lead and steels under constant loading, in which power or exponential function was used [2]. Since then, in the past 100 years, tremendous research efforts have been devoted to investigating the constitutive flow stress

behaviour in various metals under various conditions. The experimental results have been further processed and analysed such that underlying trends and relationships between the flow stress and other variables can be found and developed into various forms of constitutive equations, describing the observed flow stress behaviour and making a prediction of other unexplored conditions, which result in a significant saving on the experimental time and effort [21].

A large number of developed constitutive equations [71] exist in the literature, but broadly they can be categorised into two groups, namely phenomenological constitutive models and physically-based models. The aim of this literature review is to provide an overview of current phenomenological and physically-based constitutive models, their general pros and cons. In addition, the two most widely used models in each group will be selected and described in more detail. Due to the extensive literature for this research topic, this review focuses on aluminium alloys, considering only the dislocation, subgrain (LAGBs) and grain (HAGBs) evolution. At the end of this review, the research gaps will be identified.

2.6.1 Phenomenological constitutive model

The phenomenological models only concern the stress-strain curves under testing conditions. The underlying mechanisms are neglected. These models attempt to find and develop appropriate mathematical equations to accurately fit the experimentally obtained data. In this approach, the encountered difficulties should not be underestimated, as most of the relationships between the flow stress and testing conditions are non-linear and mathematically challenging. Furthermore, various materials, *e.g.* face centred cubic (FCC), body centred cubic (BCC) or hexagonal close-packed (HCP), exhibit very distinct behaviours with respect to their flow stresses.

Developing a general phenomenological constitutive model, which can relatively accurately describe the flow stress behaviour under a wide range of testing conditions suitable for a variety of materials, is non-trivial. In the early days, the underlying mechanisms and fundamental physics causing the flow stress changes were unavailable and unknown, because of the fundamental framework of metallurgical science and dislocation theories, driven by the development of microscope to unlock the structure at the micro or nanoscale, were not established until the 1950s [5, 72-74]. The main reason why phenomenological models are still popular and widely used by most the finite element (FE) analysis software is their

mathematical simplicity, high accuracy and robustness. Only a small number of equations and constants is required to be determined and fit well to a wide range of testing conditions. This is particularly desirable for solving engineering problems, *e.g.* FE simulation of metal forging process, since the computational time can be reduced by several orders, compared to the physically-based models.

2.6.1.1 Johnson-Cook constitutive model

Johnson-Cook (JC) model [75], initially proposed in 1983 by Johnson and Cook, probably is one of the most widely used phenomenological models, owing to its simplicity with a small number of materials constants and the availability of parameters for various materials [71]. It has been successfully embedded in various commercial FE software, such as ABAQUS and DEFORM, to describe the flow stress behaviour during deformation under various strain, temperature and strain rates [76].

The original JC model can be expressed as:

$$\sigma = (A + B\varepsilon^n) \left(1 + C \ln \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \right) \left[1 - \left(\frac{T - T_r}{T_m - T_r} \right)^m \right] \quad (3)$$

where σ is the equivalent flow stress and $\varepsilon, \dot{\varepsilon}$ and T are the equivalent plastic strain, testing strain rate and the absolute temperature, respectively. The first bracket describes the strain hardening effect, where A is the initial yield stress without strain hardening at a reference temperature and $B\varepsilon^n$ describes the strain hardening contribution, where B is the coefficient and n is the strain hardening exponent, which can be determined by $\partial \ln \sigma / \partial \ln \varepsilon$. The second bracket describes the strain rate hardening effect, C is a material constant representing the strain rate hardening. The strain rate is normalised as a ratio with respect to a reference strain rate. The term, $\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0}$, determines the ratio of the current strain rate ($\dot{\varepsilon}$) with respect to a reference strain rate ($\dot{\varepsilon}_0$). The third bracket in a square bracket form represents the temperature effect, in which the temperature is also normalised as a ratio of the temperature difference of $(T - T_r)$, *i.e.* the testing and a reference temperature (T_r), to the temperature range of $(T_m - T_r)$, *i.e.* from the melting temperature (T_m) to the reference temperature (T_r). The power term m is the thermal softening exponent.

This original JC model accounts for the observed strain, strain rate and temperature influence on flow stress. It is simple to implement in FE simulation and easy to calculate the few

unknown constants using a small number of thermal-mechanical tests. However, one assumption made in its original form is the independency of strain, strain rate and temperature from each other [71]. These three variables are treated as isolated terms without any interwoven effects among them. This assumption is invalid in some cases when the coupling effect of temperature and strain rate is significant. As fundamentally, the extent of the diffusion process governs the viscoplastic behaviour, and it is the combined effect of strain rate and temperature that affects the diffusion rate. Therefore, they should not be considered as independent terms.

The relationship of strain rate and temperature to flow stress are in Log_e (ln) and power, which is inappropriate for high strain rate and temperature conditions. Flow stress rises steeply with increasing strain rate or becomes steady at high temperature. Furthermore, the JC model did not consider the crystal structure of the materials, for which different crystal structures, *e.g.* FCC, BCC and HCP would display distinct flow stress behaviour due to the activation of completely different mechanisms, *e.g.* twinning (in HCP) versus dislocation slip (in FCC or BCC). These assumptions limit the accuracy and applicability JC model in many materials, such as AA2139 T8 aluminium alloy [77], beryllium, uranium and steels [78]. As a result, many further modifications to the original JC model have been undertaken to account for the intertwining between the temperature, strain rate [77] and strain [79] and crystal structure [80]. These modifications led to a more complex form with an increasing number of constants, while providing more accurate results for a wider range of materials. One example, using a modified JC model to fit the AA2139-T8 stress-strain curves under various thermal-mechanical conditions, is presented in Fig. 2.12, and good consistency is reached between the experiment and model.

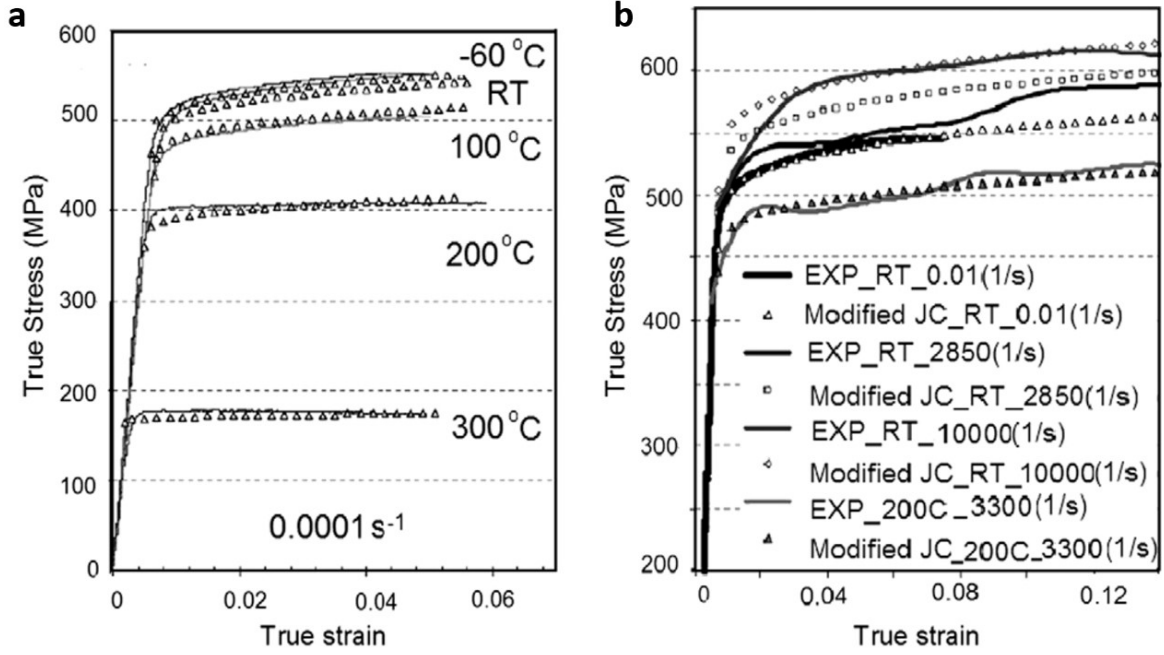


Fig. 2. 12 The AA2139-T8 aluminium alloys stress-strain curves obtained at various temperature (a), and strain rate (b). The stress-strain lines are experimentally tested while the discrete points are predicted using the modified JC model [77].

2.6.1.2 Arrhenius model

Since the viscoplasticity is fundamentally driven by the diffusion process, the Arrhenius equation, which was proposed by Arrhenius in 1889 [81] and used widely in the physical chemistry to describe the temperature dependence of reaction (*e.g.* diffusion) rate, was first adopted by Zenner & Hollomon in 1944 in steel [45], further by Garofalo in 1963 [82] to empirically fit the thermal-mechanical stress-strain behaviour, and later popularised by Sellars and Tegart [48, 83]. In this model, the relationship between the flow stress, strain rate and testing temperature is established, as shown in Equation 4.

$$\dot{\epsilon} = A [\sinh(\alpha\sigma)]^n e^{(-\frac{Q}{RT})} \quad (4)$$

where A , α and n are material constants to be calculated through fitting and regressing experimental data. Q is the activation energy (kJ mol^{-1}), normally close to the bulk diffusion activation energy in aluminium 7050, 160 kJ mol^{-1} [47] and R_g is the universal gas constant ($8.31 \text{ J mol}^{-1} \text{ K}^{-1}$).

Often, it is convenient to express the strain rate and temperature effect using the Zenner-Hollomon parameter, $Z = \dot{\epsilon} e^{(\frac{Q}{RT})}$. Low value of Z means the deformation condition under high temperature and low strain rate, while high Z suggests low testing temperature and high

strain rate. Z can substitute the strain rate and temperature terms in Equation 4 and express the stress in the form below.

$$\sigma = \frac{1}{\alpha} \ln \left\{ \left(\frac{Z}{A} \right)^{1/n} + \left[\left(\frac{Z}{A} \right)^{2/n} + 1 \right]^{1/2} \right\} \quad (5)$$

This Arrhenius equation based constitutive model works well to mathematically describe the coupling effect from strain rate and temperature on the flow stress. However, the main shortcoming of this model is the neglect of the strain effect, which prohibits it from describing the flow stress variation as a function of strain under a given temperature and strain rate [79]. A few researchers have modified the original Arrhenius constitutive model by considering the materials constants, *i.e.* Q , A , α and n as variables, which can be determined through the polynomial functions of strain [84]. This modification is physically reasonable because, under a given strain rate and temperature, the flow stress generally varies with plastic strain due to changes in dislocation density through accumulation, dynamic recovery or dynamic recrystallisation. A good agreement was reached between the experimental results and the modified Arrhenius constitutive model, for example, in the study of strain rate and temperature effect on hot compressed AA7050 aluminium alloys [84], as shown in Fig. 2.13.

Only the two most widely used models are discussed here. However, many other applicable phenomenological models are developed in the field, such as the Voce-Kocks model, Molinari–Ravichandran model, Fields–Backofen model, Khan–Huang, Khan–Huang–Liang, Khan–Liang–Farrokh models and etc. [71]. More details can be referred to these relevant references.

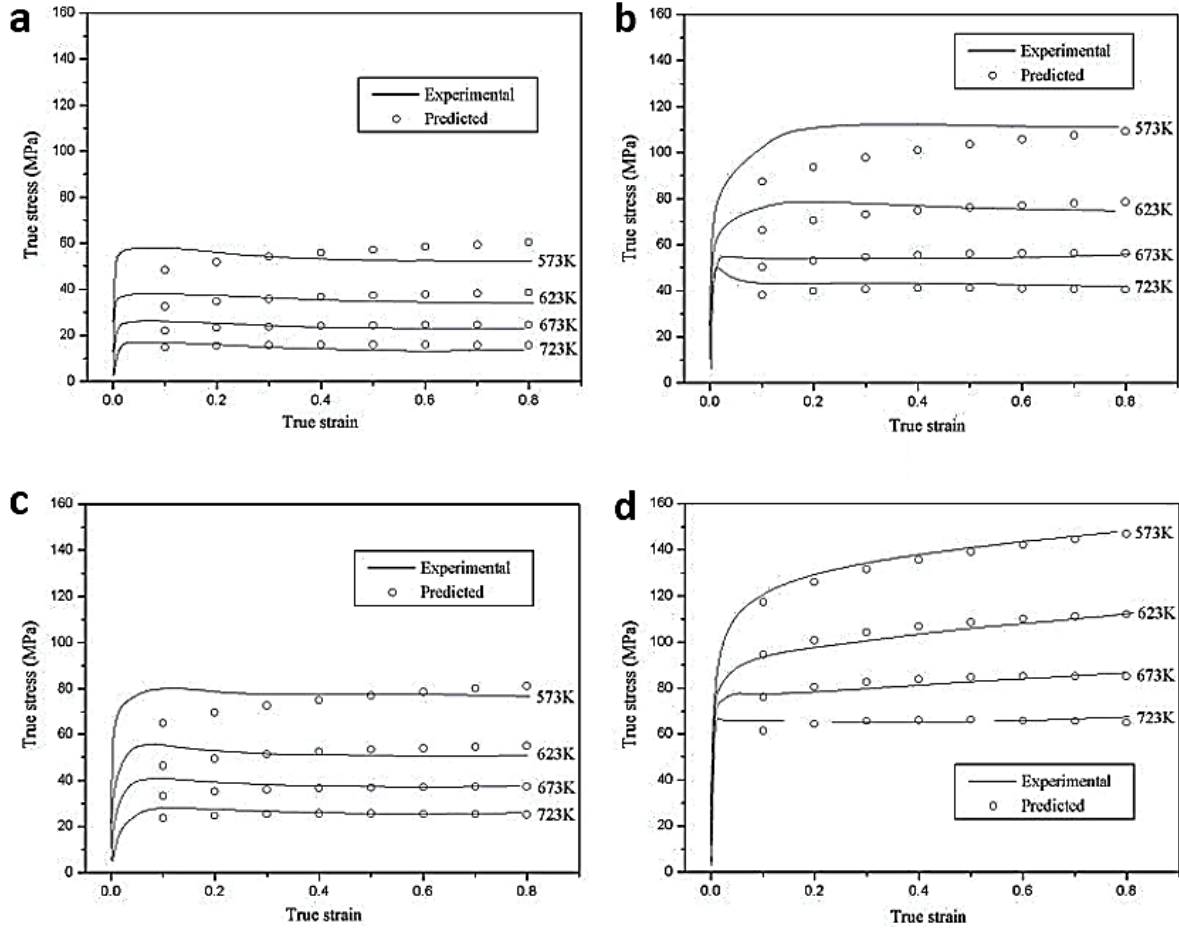


Fig. 2.13 The comparison of the experimental measured and the modified Arrhenius constitutive model predicted strain-strain curves at the strain rate of (a) 0.001 s⁻¹, (b) 0.01 s⁻¹, (c) 0.1 s⁻¹ and (d) 1 s⁻¹ respectively [84].

2.6.2 Physically-based modelling

For the physically-based constitutive models [71], researchers sought to embed the discovered fundamental mechanisms causing flow stress changes, *e.g.* dislocation evolution, dynamic recovery, dynamic recrystallisation, or grain boundary sliding, into their models. This enables their constitutive models to have sound scientific basis, obtained from the fundamental microstructure evolution, to explain the observed flow stress responding phenomena. Furthermore, another significant merit of this physically-based approach is the availability of microstructure information (*e.g.* dislocation density [85-87], secondary precipitations [88]) during the plastic deformation, which allows the corresponding mechanical properties to be predicted.

There are numerous so-called “physically-based ” constitutive models in the literature [71]. In this review, only the models explicitly modelling the physical terms, *e.g.* dislocation density and/or grain size evolution, are considered as physically-based one. Furthermore,

since this thesis is devoted to modelling the thermal-mechanical viscoplastic behaviour of aluminium alloy, we only consider the constitutive models predicting dynamic recovery and dynamic recrystallisation for aluminium alloys. Therefore, as in the previous phenomenological model section, two characteristic examples, *i.e.* dislocation-grain size based constitutive model and Lin's model are selected, described and discussed in-depth. Other models will be summarised and briefly discussed.

2.6.2.1 Dislocation-grain based constitutive model

In physically-based constitutive models, the stress-strain constitutive relationship is modelled using microstructure evolution. Instead of establishing the equation between flow stress and strain, the flow stress (σ) is explicitly determined by the material's initial intrinsic flow stress (k), dislocation density (ρ) and grain size (d), respectively, as displayed in the following:

$$\sigma = k + \alpha M G b \sqrt{\rho} + k_d / \sqrt{d} \quad (6)$$

where the first term k is the initial intrinsic threshold stress of the material causing dislocation slip, excluding any dislocation barriers effect, *e.g.* dislocation network, LAGB, HAGB and etc. The second term, $\alpha M G b \sqrt{\rho}$, describes the dislocation hardening, *i.e.* strain hardening, originally proposed by Mecking and Kocks [86-87], where α is a temperature-dependent material constant; M is the Taylor's factor allowing the dislocation slip in single crystal to be generalised onto polycrystalline materials and accounting for any anisotropic effect caused by texture; G and b are respectively the shear modulus and Burgers vector of the material. The final term, $k_d / \sqrt{d}$, represents the grain boundary effect, *i.e.* the Hall-Petch effect, which k_d is also a temperature-dependent material constant to describe the grain boundary effect on the flow stress. When it is positive, the grain boundary hardens the material by pinning dislocation slip and increases the flow stress. This k_d value will be negative, corresponding to the grain boundary softening due to grain boundary sliding active at relatively high temperature and low strain rate.

As the plastic deformation proceeds, the dislocation density evolution relies on three main mechanisms, namely, strain hardening, dynamic recovery and dynamic recrystallisation. The plastic deformation leads to dislocation accumulation causing work hardening, whereas the dynamic recovery and dynamic recrystallisation result in dislocation density reduction. These are mathematically described in Equation 7 [89].

$$\dot{\rho} = \frac{M}{b} \left(\frac{\sqrt{\rho}}{f_w} + \frac{1}{d} \right) \dot{\epsilon}_p - f_v \rho \dot{\epsilon}_p - H(\rho - \rho_{cr}) f_x S \dot{\epsilon}_p \quad (7)$$

where the first term, $\frac{M}{b} \left(\frac{\sqrt{\rho}}{f_w} + \frac{1}{d} \right) \dot{\epsilon}_p$, represents the strain hardening process, in which the mobile dislocations are pinned by obstacles. According to the Orowan equation [90], the pinning rate of dislocation is proportional to strain rate ($\dot{\epsilon}_p$) and inversely proportional to the dislocation mean path due to either the existing dislocation network ($f_w/\sqrt{\rho}$), or the grain boundary (d). f_w is the work hardening coefficient, depending on the initial deformation conditions. The M is the Taylor factor to account for the grain orientation effect and b is the Burger vector of the material.

The second term $-f_v \rho \dot{\epsilon}_p$, introduces the dislocation density reduction caused by dynamic recovery. Since the dislocation recovery and annihilation are mainly due to the dislocation cross-slip and jump promoted by the elevated temperature, the dislocation density reduction rate is proportional to the existing stored dislocations, according to the Mecking-Kocks model [91]. f_v is a recovery coefficient depending on the initial deformation condition.

Finally, the dynamic recrystallisation effect on dislocation density is represented by the third term, $H(\rho - \rho_{cr}) f_x S \dot{\epsilon}_p$, where $H(\rho - \rho_{cr})$ is a step function to determine whether the dynamic recrystallisation process occurs. For DDRX in low stacking fault energy materials, if the current dislocation density, ρ , is higher than the critical dislocation density, ρ_{cr} , the dynamic recrystallisation is triggered. Otherwise, there is no dynamic recrystallisation [89], and this dynamic recrystallisation effect on the dislocation term will be set as zero. S is the dynamic recrystallisation volume fraction, which can be determined by an exponential function of strain, and f_x is a coefficient for dynamic recrystallisation.

Regarding the grain size part in Equation 8, the grain size evolves with the dynamic recrystallisation process and it is expressed as [89]:

$$d = S d_{drx} + (1 - S) d_0 \quad (8)$$

where S again is the dynamic recrystallisation volume fraction, d_{drx} and d_0 are the mean grain size of the dynamic recrystallised grains and the mean grain size of the original grains, respectively. d_{drx} is strongly dependent on the temperature, strain rate and initial grain size and can be determined through the Arrhenius equation as [92]:

$$d_{drx} = A_r d_0^{m_r} \left[\dot{\epsilon}_p \exp\left(\frac{Q}{RT}\right) \right]^{n_r} \quad (9)$$

where Q and R are the activation energy for dynamic recrystallisation and the universal gas constant ($8.31 \text{ J K}^{-1} \text{ mol}^{-1}$), respectively. A_r, m_r, n_r are material constants. Note that n_r is negative because high strain rate and high temperature will promote dynamic recrystallisation by suppressing dynamic recovery, which results in more grain boundaries and finer grain size [89].

In materials with high stacking fault energy, CDRX occurs through the transformation from LAGB into HAGB. Such a transformation process is achieved through a gradual increase of the misorientation angle of LAGB. Researchers attempted to incorporate the LAGB and misorientation angle into the dynamic recrystallisation process in physically-based constitutive models [50, 93]. For example, Gourdet et al. [50] proposed the link of the misorientation angle change rate, $\dot{\theta}_{LAGB}$, in LAGB with grain size (d) and strain rate ($\dot{\epsilon}_p$) through Equation 10:

$$\dot{\theta}_{LAGB} = \frac{b}{2N} (1 - \beta) r \rho d \dot{\epsilon}_p \quad (10)$$

where r denotes the dynamic recovery parameter and is a function of strain rate and temperature; $r\rho$ represents the recovery dislocation density, for which a fraction of it (β) forms new LAGBs and the rest ($1 - \beta$) increases the angle of existing LAGBs; N is the number of dislocations stored in the boundary.

The physically-based constitutive models could not only generate the stress-strain curves but also predict the microstructure evolution, such as grain size, dislocation density or misorientation angles under various thermal-mechanical loading conditions. One example of modelling the CDRX on commercial purity aluminium is shown in Fig. 2.14. Both the modelled stress-strain curves and the grain size are in good consistent with experimental data.

It should be mentioned that as more physical mechanisms are considered in the physically-based constitutive model, more equations and materials constants are required. Determination a large number of material constants is a very time-consuming process and solving a large number of non-linear equations is non-trivial. These issues prohibit model development and adoption [71].

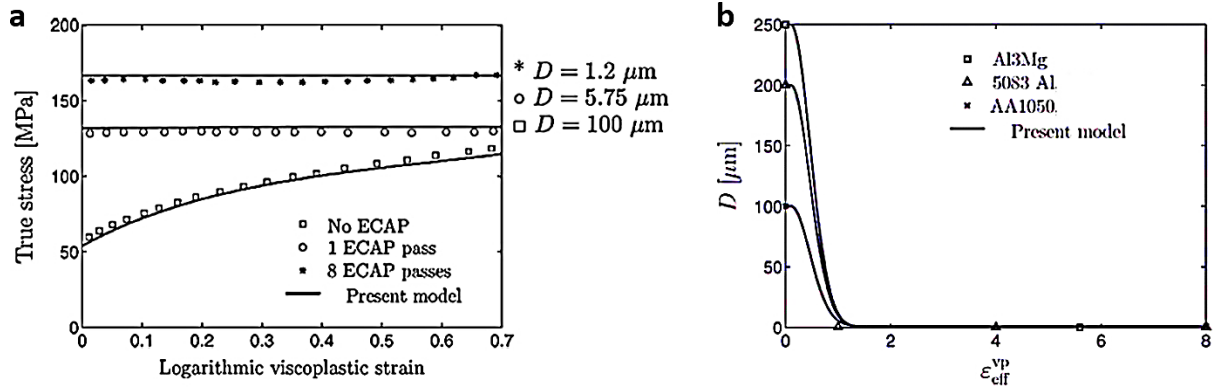


Fig. 2. 14 (a) The stress-strain curves comparing the experimentally obtained and physically-based constitutive modelled ones on AA1050 aluminium after different passes. (b) The grain size evolution at various strains between the experimental one and modelled one [94].

2.6.2.2 Unified physically-based constitutive model (Lin's model)

The development of a unified physically-based constitutive model and their application in metal forming processes is attributed to Lin's pioneering work [37, 85] integrating the classical mechanics developed by Ford [95], Johnson [96], Hill [97] and *etc.*, with physical metallurgy [98]. The main framework was established in 2005, developed and matured in 2015 [37].

Comparing to other physically-based constitutive models, Lin's modelling framework comprises three main novelties. First, the utilisation of state-variables, describing the state of a system and to determine its future behaviour and consisting of first-order differential equations [99], a set of ordinary differential equations is formulated to describe the stress-strain and microstructure evolution [100]. Second, the key driver (dislocation density) was unified for microstructure evolution and flow stress variation [85]. Third, algorithms, *e.g.* genetic evolutionary algorithms, are applied to efficiently determine a large number of material constants for ordinary differential equations [101-102]. These three novelties will be demonstrated through examples in the following.

A typical set of unified constitutive equations used to model the stress-strain response of the hot rolling process [85] is shown in Equation 11-17. This model consists of seven ordinary differential equations, in which the individual physical phenomena are modelled using state variables. For example, the evolution of plastic strain rate ($\dot{\epsilon}_p$) is expressed as functions of stress (σ), isotropic hardening (R), initial yielding stress (k) and grain size (d) in Equation 11. Each physical variable is modelled in the subsequent equations. The isotropic hardening, R , evolves as a function of the normalised dislocation density ($\bar{\rho} = 1 - \rho_i/\rho$, where ρ_i is the

initial dislocation density prior to plastic deformation and ρ is the current dislocation density). At the onset of plastic deformation, $\bar{\rho}$ is zero. The $\bar{\rho}$ is approximately equal to one when dislocation density saturates. This normalised dislocation density has a significant impact on determining the materials constants, which will be discussed later. The evolution of dislocation density is described in Equation 13, in which the strain hardening, dynamic recovery; static recovery and dynamic recrystallisation effect on dislocation density are presented in the first, second and third terms, respectively. The dynamic recrystallisation volume fraction (S) and grain size (d) evolution is represented in Equation 14-16. Both dynamic recrystallisation grain nucleation and existing grain growth are taken account. The $[]_+$ signs in Equation 11 and Equation 15 are used to determine whether the process will take place. If the term in the square bracket is positive, the equations will be solved. Otherwise, it will be set to zero, meaning that the criteria for the occurrence of the processes are not reached. For example, if $(\sigma - R - k)$ is negative, it implies that stress is too low to initiate plastic deformation and the deformation is still in the elastic region. The flow stress (σ) evolution is calculated based on Hook's law in Equation 17 (, the elastic strain is determined by subtracting the calculated plastic strain ε_p from the total strain ε_t).

$$\dot{\varepsilon}_p = A_1 \sinh[A_2(\sigma - R - k)]_+ d^{-\gamma_4} \quad (11)$$

$$\dot{R} = B \bar{\rho} \quad (12)$$

$$\dot{\bar{\rho}} = \left(\frac{d}{d_0}\right)^{\gamma_d} (1 - \bar{\rho})|\dot{\varepsilon}_p| - c_1 \bar{\rho}^{c_2} - c_3 \frac{\bar{\rho}}{1 - S} \dot{S} \quad (13)$$

$$\dot{d} = \alpha_0 d^{-\gamma_0} - \alpha_2 \dot{S}^{\gamma_3} d^{\gamma_2} \quad (14)$$

$$\dot{S} = Q_0 [x\bar{\rho} - \bar{\rho}_c(1 - S)]_+(1 - S)^{N_q} \quad (15)$$

$$\dot{x} = A_0(1 - x)\bar{\rho} \quad (16)$$

$$\dot{\sigma} = E(\dot{\varepsilon}_t - \dot{\varepsilon}_p) \quad (17)$$

where $A_1, A_2, \gamma_4, \gamma_d, Q_0, \rho_c, N_q, c_1, c_2, c_3, A_0, B, A_0, \alpha_0, \gamma_0, \alpha_2, \gamma_2, \gamma_3$ are material constants; R and k represent the isotropic hardening and initial yield stress, respectively; $\bar{\rho}$ is the normalised dislocation density in the material; d is the current average grain size, while d_0 denotes the

initial grain size; S is the volume fraction of dynamic recrystallisation and x represents the recrystallisation incubation time.

In this unified physically-based constitutive model, solving these ordinary differential equations with good accuracy is extremely challenging due to the different units for various variables, which raises considerable difficulties in error assessment as the errors calculated from the individual equation cannot be compared directly. The level of difficulties of this problem increases exponentially with more material constants.

To solve these problems, two effective remedies were given, the employment of the normalisation approach on the variables (*e.g.* dislocation density) with different units [85] and the utilisation of the advanced artificial intelligence, *i.e.* machining learning to solve the more significant number of constants [101-102]. These two approaches tackled the problems in the model and allowed the stress, grain and dislocation density evolution in C-Mn steel during uniaxial tensile deformation; its results and the comparison with experimental data are reported in Fig. 2.15 [85]. An excellent agreement was reached.

In addition, to the author's knowledge, it was the first time that the grain size and dislocation density evolution were outputted from the constitutive equations. This modelling framework has been further developed and used in various applications such as superplastic forming [103], creep age forming [104], hot form quench (HFQ) of aluminium panel components [105], and forging two-phase titanium alloys [106].

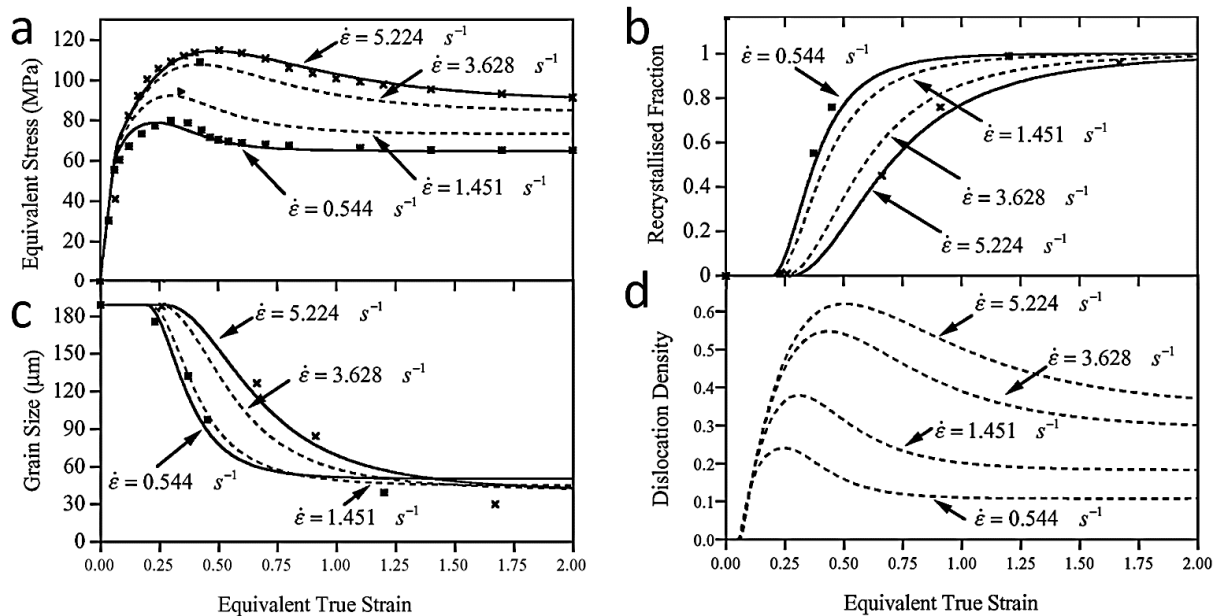


Fig. 2. 15 The comparison of model prediction [85] and experimental results [107] of a C-Mn steel for uniaxial loading cases with strain rates of 0.544, 1.451, 3.628 and 5.224 s^{-1} at 1100 °C on (a) stress, (b) recrystallisation fraction, (c) grain size; and (d) dislocation density (only modelling results).

The current state of the art, research gaps and future perspectives for constitutive models can be summarised in the following. In the past several decades, significant advances have been made in the development of the constitutive models, describing the flow stress behaviour as functions of strain, strain rate and temperature, either through explicit relationships by phenomenological models or implicitly through the physically-based constitutive models.

Compared with the physically-based models, the phenomenological ones have been relatively well developed and widely used in practical FE simulation of metal forming processes, due to their high accuracy, computational robustness and simplicity. While the physically-based constitutive models have also made considerable progress in embedding underlying dislocation, grain and secondary particle evolution in them; applying advanced intelligent optimisation algorithms to determine the material constants and normalise the state variables to reduce the overall errors for searching the optimal constants, physically modelling the viscoplastic deformation process on aluminium alloys is still at its infancy phase.

Prof. Lin established the modelling framework to coupling the microstructure evolution and viscoplastic flow stress behaviour, its accuracy and applications are currently limited to specific cases, mainly owing to three reasons. First, the state variable, dislocation density, probably the most important driver for viscoplastic behaviour, can be hardly calibrated in the model due to few experimental characterisation techniques suitable for acquiring a statistical representative dataset within a reasonable timespan and cost [108]. Second, comprehensive microstructural data for calibrating the physically-based models are unavailable. Most material constants are currently determined through fitting against the stress-strain curves only, and only a few grain size data, which raises the question of whether there is a difference between the phenomenological and physically-based models as they are all determined through fitting the same data [71].

Finally, despite decades of research work on viscoplastic deformation mechanism in aluminium, the in-depth understanding of the strain rate and temperature effect on the kinetics of dislocation accumulation, dynamic recovery and dynamic recrystallisation, is still limited [23]. Hence, these three areas deserve future research attention, with the aim to realise the accurate prediction of both material flow and microstructure in various metal forming processes.

2.7 Summary

The review presented in this chapter has resulted in following main findings: from the basic material and technique to the mechanisms and models. The main points are summarised below:

- i. AA7050 has been widely used in the aviation industry such as wing skins, fuselage frames and bulkheads of aircraft, due to their excellent properties: of high strength to weight ratio and good formability. The basic knowledge of AA7050, from material properties to its thermal-mechanical processing history have been introduced.
- ii. The forging techniques and forging conditions for AA7050 have been presented. The recommended forging temperature range for AA7050 is 400~450 °C and strain rate is 0.01-0.0001 s⁻¹. The minimum recrystallised starting temperature is found to be 340 °C at a strain rate of 0.01 and 0.58 tensile deformation strain.
- iii. The DRX mechanism in aluminium has been found to be mainly CDRX due to its high stacking fault energy and fast recovery rate. CDRX involves the formation of LAGBs from rearranging stored dislocations and transformed into HAGBs. However, quantitative analyses of GND density, LAGB and HAGBs in CDRX are still lacking in the literature. Its relationships with strain rate and temperature have been also reviewed, which are largely unknown since only few research exists.
- iv. Finally, material constitutive models describing the viscoplastic material flow have been reviewed. The phenomenological and physically-based models have been presented and discussed. Unified physically-based constitutive model has the advantages of predicting materials flow as well as DRX mechanism. However, very few models exist for predicting CDRX in aluminium alloys.

Chapter 3

Experiment and Material Characterisations

3.1 Introduction

This chapter provides a detailed description of the test material, equipment and experimental procedures used in this study. Two sets of experimental programmes have been designed:

- i. Thermomechanical tests using the Gleeble 3800 thermal-mechanical simulator to study the viscoplastic behaviour of AA7050. Stress-strain curves were obtained for four compression temperatures and four strain rates.
- ii. Material microstructure characterisation using a scanning electron microscope (SEM) and electron backscatter diffraction (EBSD) to reveal the underlying microstructure evolution, including information regarding the geometrically necessary dislocation (GND) density, low-angle grain boundaries (LAGB) and high-angle grain boundaries (HAGB).

3.2 Materials

The material employed in this work was an aluminium alloy, AA7050, with the nominal chemical composition shown in Table 3.1. This material was supplied by the industrial collaborator, the First Aircraft Institute (FAI), China. The as-received material was in T7451 temper state, meaning that it had been solution heat treated, stress-relieved by stretching and then subjected to a two-stage ageing treatment ($120\text{ }^{\circ}\text{C} \times 6\text{ hours} + 177\text{ }^{\circ}\text{C} \times 7\text{ hours}$) [109] that led to moderate corrosion resistance, good strength and low residual stress.

Table 3.1 Nominal chemical composition of AA7050 (wt. %).

Element	Zn	Mg	Cu	Mn	Cr	Ti	Zr	Fe	Si	Al
wt %	5.7-6.7	1.9-2.6	2.0-2.6	≤ 0.10	≤ 0.04	≤ 0.06	0.08-0.15	≤ 0.15	≤ 0.12	Bal.

Cylindrical specimens with 8 mm in diameter and 12 mm in height were machined from a rolled thick plate, with the long axis of the cylinder perpendicular to the rolling direction, as shown in Fig. 3.1.

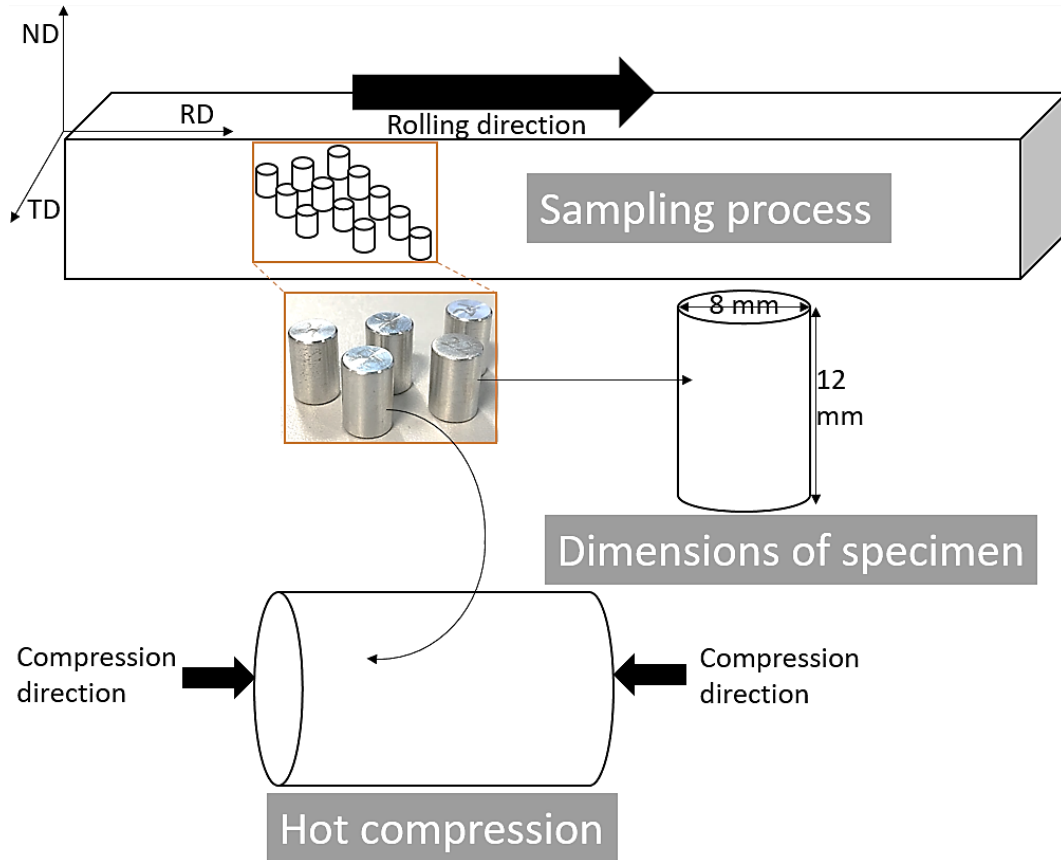


Fig. 3. 1 The sampling process and compression for the test samples.

3.3 Gleeble hot compression test

The fully annealed samples were then prepared for the thermal-mechanical testing using the Gleeble 3800, as displayed in Fig. 3.2(a). The Gleeble 3800 is an appropriate machine for this study because of its precise thermal and mechanical control. Its temperature is raised using a resistant heating method because of its fast-heating rate and relatively uniform thermal gradient. The control of the thermal temperature and thermal profile during testing is programmed in the control system sitting next to the testing chamber as indicated in Fig. 3.2(b). The real-time temperature on the sample is measured and monitored using thermocouples, welded using the Gleeble 3800-associated spot-welding machine. Care was taken to ensure that the welded thermocouples were strongly attached and would not unfasten from the sample surfaces during the test.

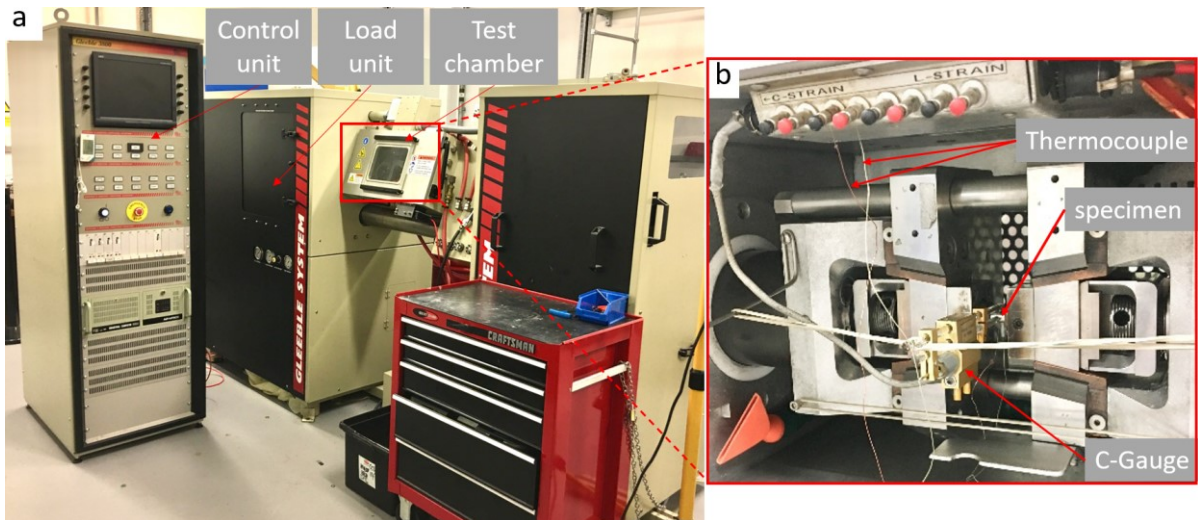


Fig. 3. 2 Gleeble 3800 thermal-mechanical materials simulator: (a) The main units and (b) Set-up for hot compression experiment within test chamber.

The overall thermal-mechanical testing programme is shown in Fig. 3.3, including annealing and hot compression. To simulate the hot forging process, the material was prepared to a soft state prior to the hot forging, such that the lowest forging load is required to achieve the material plastic flow and part's final shapes. This will lower the specifications for the required forging machines and allow larger and more complex-shaped parts to be forged. Thus, these machined samples were fully annealed to reduce the precipitation and dislocation density hardening effect. The annealing was carried out according to the standard industrial hot forging procedure [110], at 413 °C for 2 hours in an electric resistance furnace, followed by furnace cooling.

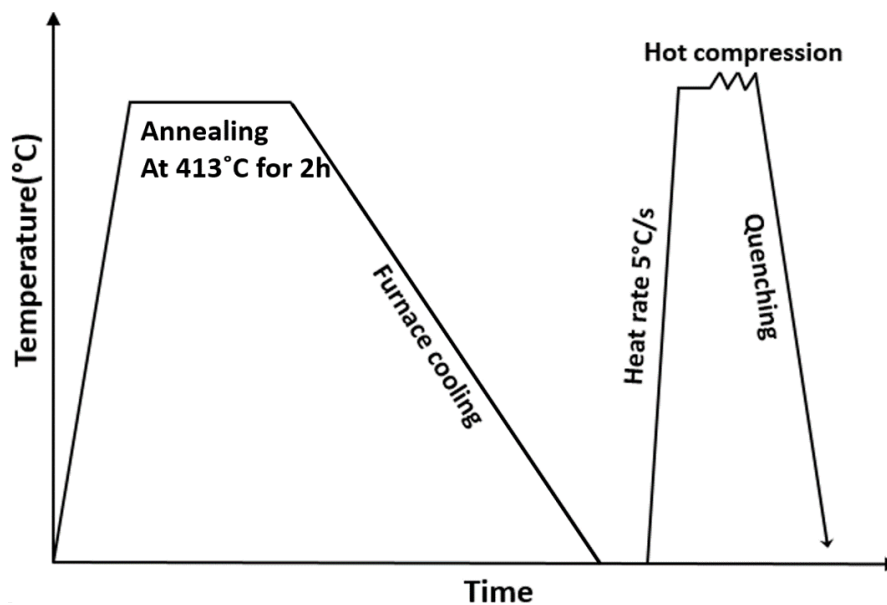


Fig. 3. 3 Schematic temperature-time profile for annealing and hot compression test.

To confirm that the annealed material was softer, a hardness test was carried out for the as-received and fully annealed samples respectively, using a Vickers hardness machine (Zwick/Roell 2HV). The results indicated an expected significant drop (47%) in hardness from 163 to 86 HV. The load for the tests was 1 kgf, and the dwell time was 10 s. The test was performed five times to calculate the average hardness. Each test was carried out at least two indents away from the previous indent to ensure that the plastic deformation wouldn't interfere with the tests.

The thermal profile of the samples during the compression test is also shown schematically in Fig. 3.3. The samples were heated at $5\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$ and then held at the test temperature for three minutes to minimise temperature gradients before compression was started. During the set-up, a nickel-based lubricant and graphite foils were applied between the anvils and the specimen to reduce the friction. The friction coefficient between sample and anvil has been calculated and will be presented in Chapter 3.3.2 in detail. The final true strain (ε) in the samples was approximately 0.58. After the hot compression, the samples were water quenched to room temperature to preserve the microstructure at the end of the deformation. The stress-strain curves were calculated based on the cross-section (C-gauge) changes and the force recorded by the load cell in Gleeble. During the test, the sample diameter (d) changes were monitored using a C-gauge, clipping at the middle section of the sample. The true stress and strain were calculated according to the following equations:

$$\text{True strain } (\varepsilon): \quad \varepsilon = 2 \cdot \ln \left(\frac{d_0}{d_0 + \Delta d} \right) \quad (18)$$

$$\text{True stress } (\sigma): \quad \sigma = \frac{F}{\left[\frac{\pi(d_0 + \Delta d)^2}{4} \right]} \quad (19)$$

where ε is true strain, d_0 is initial specimen diameter, Δd is the diameter increment measured by C-gauge, σ is the true stress, and F is the force line obtained from the Gleeble data library.

To investigate the strain rate sensitivity and temperature effects on the material behaviour, a Gleeble test matrix was designed as shown in Table 3.2. Five test temperatures (380, 400, 420 440 and 460 $^{\circ}\text{C}$) and five strain rates (0.0005, 0.005, 0.05, 0.5 and 5 s^{-1}) were used in this study. In addition, a set of interrupted thermomechanical tests under conditions of 420 $^{\circ}\text{C}$ and 0.05 s^{-1} were performed to different strain levels to obtain insights into the microstructural

evolution mechanisms during the deformation process. The interrupted compression tests were conducted to the strains of 0.05, 0.15, and 0.40.

Table 3.2 Hot compression test matrix.

Strain rate (s ⁻¹) Temperature (°C)	0.0005	0.005	0.05	0.5	5
380			√		
400			√		
420	√	√	√	√	√
440			√		
460			√		

3.3.1 Temperature uniformity

As the thermal gradient across the samples may affect the activation and kinematics of dynamic recrystallisation, the thermal gradient across the cylindrical samples was checked by attaching three pairs of thermocouples along the left-hand side of a sample. The testing sample was heated to 420 °C and the temperatures were monitored and recorded. The obtained results suggested there is only 3 °C thermal variation through the designed sample geometry in this study. Hence, it is reasonable to state that the temperature distribution on the sample is uniform during the hot compression process.

3.3.2 Friction and friction coefficient

To minimise the differences between the nominal and actual plastic strain imposed on the testing samples, and the errors in selecting the regions of interest for microstructure characterisation, the non-uniform plastic strain, *i.e.* the barreling effect, under hot compression was considered and evaluated. The barreling effect is caused by the friction between the testing sample and anvils, which will hinder the material flow along the interface. When friction is increased, less plastic deformation will take place at both ends, compared to the middle area of the sample, such that a barrel-shaped sample is formed. To minimise such effect, nickel-based lubricant and graphite foils were used as lubricants.

The specimen tested at 420 °C and a strain rate of 0.05 s⁻¹ was examined in detail to check the lubricant performance. A vernier caliper which has an accuracy of 0.02 mm was used to measure the diameter and height of the specimen. As shown in Fig. 3.4 and Table 3.3, the

results indicated that small barreling occurred under testing conditions. The coefficient of friction (μ) can be determined according to the following equation [111]:

$$\mu = \frac{m}{\sqrt{27(1 - m^2)}} \quad (20)$$

where m can be calculated based on barreling shape according to Equation 21 [112-113]:

$$m = \frac{\left(\frac{R}{H}\right)b}{\left(\frac{4}{\sqrt{3}}\right) - \left(\frac{2b}{3\sqrt{3}}\right)} \quad (21)$$

As seen in Fig. 3.4, R is average radius of cylinder after compression $R = R_0\sqrt{H/H_0}$, R_0 and H_0 are respectively the initial radius and height of the specimen before compression, H is the height of specimen after compression. $b = 4\frac{\Delta R}{R}\frac{H}{\Delta H}$, in which $\Delta H = H_0 - H$ is the reduction in height, and $\Delta R = R_M - R_T$ is the difference between maximum radius (R_M) and minimum radius (R_T). The measured values are shown in Table 3.3. Thus, the coefficient of friction was determined as 0.087, which indicates a small frictional force in the interface.

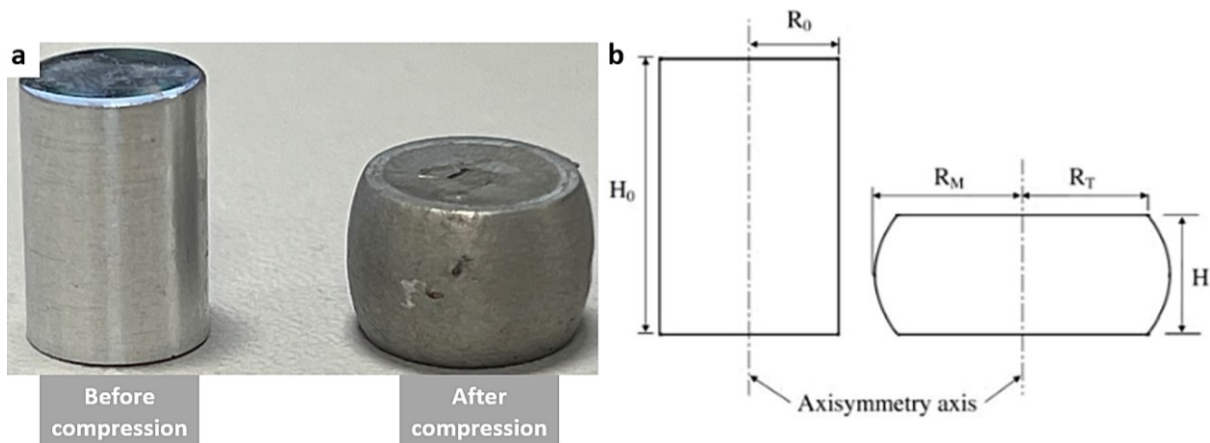


Fig. 3. 4 Specimen geometry. (a) Picture of specimens before and after compression and (b) Schematic diagram showing the geometric parameters used for friction factor (m) calculation.

Table 3.3 The dimension changes in mm before and after hot compression

Variables (mm)	R_0	R	H_0	H	R_M	R_T	ΔR	ΔH	b
Values	3.96	5.17	11.93	6.99	5.46	4.74	0.71	4.94	0.78

3.4 Microstructure characterisation

3.4.1 Sample preparation

To obtain the microstructure features and minimise the post-deformation recrystallisation, the hot compressed samples were immediately water quenched. As the as-received materials were rolled plate with elongated grains present in the rolling direction, the morphology of grains would result in different statistical measurements if different planes had been sectioned. Identifying the rolling direction is vital to ensure a consistent comparison between the results. More reasons for the selected plane will be provided in the grain size measurement section. The identification process is shown in Fig. 3.5. The top surfaces of the samples were ground and polished using a SAPHIR 520 polishing machine (Fig. 3.5(a)). Then the samples were etched using Keller's etchant with a composition of 1.5% HCl + 1% HF + 2.5% HNO₃ + 95% distilled water and cleaned with distilled water as displayed in Fig. 3.5(b). The etched samples were examined under a standard optical microscope to identify the rolling direction and thus the rolling-normal plane, as shown in Fig. 3.5(c).

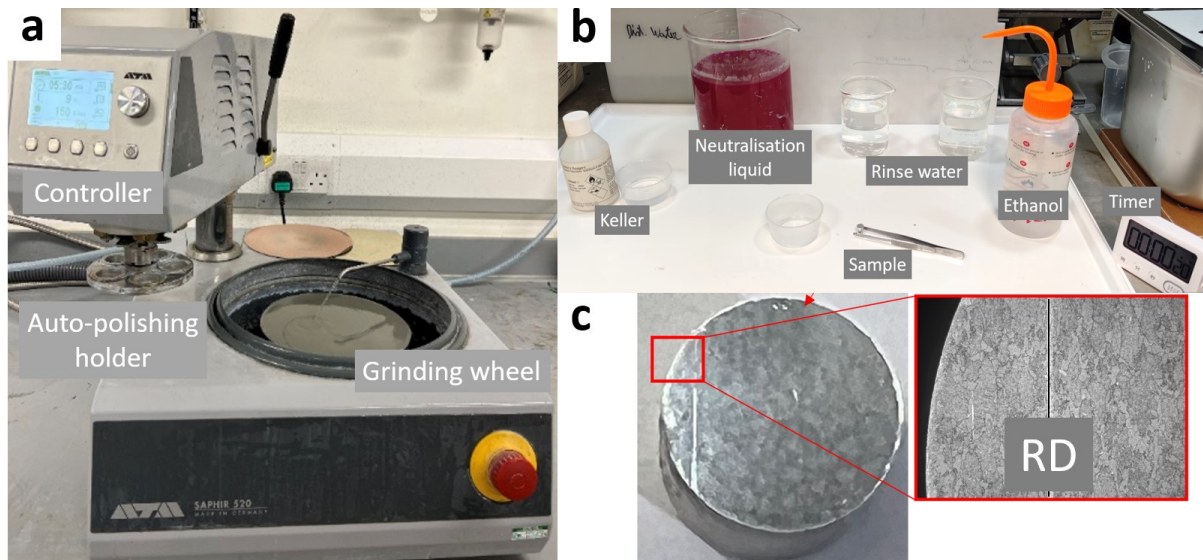


Fig. 3. 5 Rolling direction identification process: (a) Grinding machine (b) Set-up for top surface of sample etching process and (c) Rolling direction identification on the top surface of sample. RD represents the rolling direction.

On the rolling-normal plane, the central region was selected for EBSD orientation mapping. The rolling-normal plane of these samples was sectioned using the BRILLANT 220 cutting machine (Fig. 3.6(a)) and the sectioning strategy is illustrated in the schematic diagram in Fig. 3.6(b). For EBSD characterisation, the sample surfaces should be stretch-free with a mirror finish. The sectioned samples were mounted in sample holders using Bakelite, and

ground progressively using silicon carbide papers from 400, 800, 1200, 2000 grit to 4000 grits, as seen in Fig. 3.5(a). These samples were then polished using a solution of 25% colloidal silica suspension with 75% distilled water for final polishing, under 5 N force for thirty minutes, such that a mirror finish was achieved. This final polishing involves both mechanical and chemical polishing to the extent that the surface deformation induced by the previous cutting process can be completely removed. However, because the oxidation activation energy of aluminium alloys is very low, a layer of thin aluminium oxide film is immediately formed during the polishing [114]. This oxide film is a thermal-mechanically stable insulator, which prohibits the electrons emitted in a scanning electron microscope (SEM) from the sample surface, resulting in poor signal strength for EBSD characterisation. To completely remove the oxide layer, the polished samples were then electropolished using 10% perchloric acid and 90% ethanol solution with a voltage of 20 V and optimised current for 45 s at room temperature, as shown in Fig. 3.6(c) [115]. As the presence of small residual solution on the free surface, no or very thin oxide film will form on the free surface.

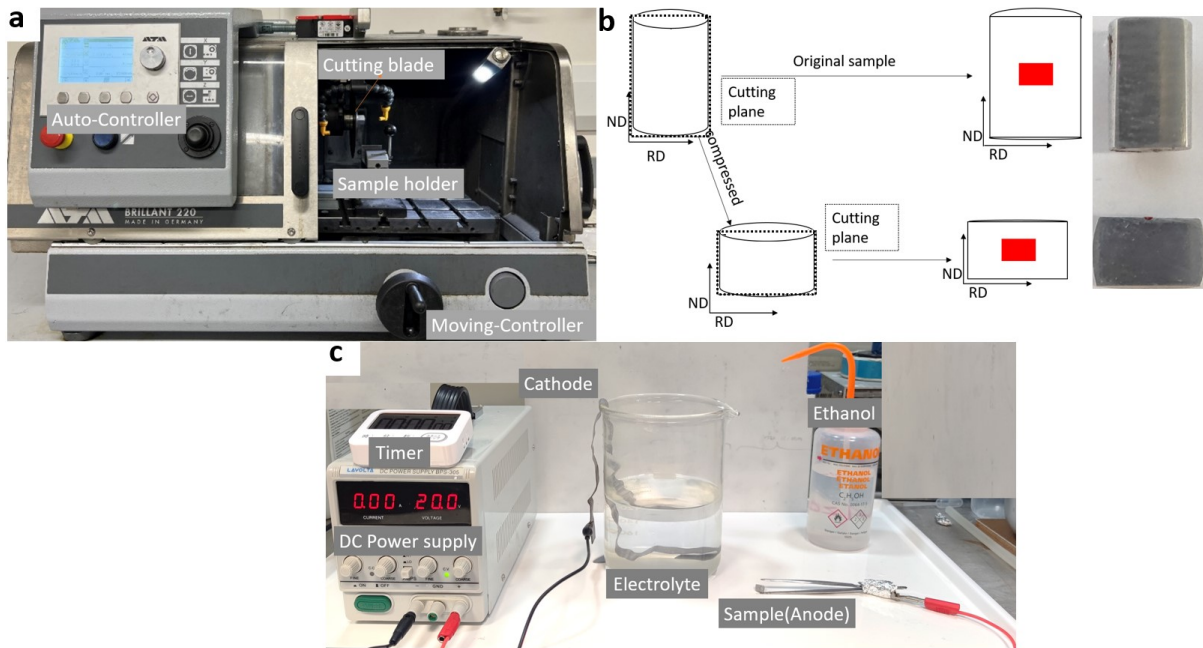


Fig. 3. 6 EBSD specimen preparation: (a) Cutting machine (b) Sample sectioning scheme for EBSD characterisation as highlighted as red square. RD represents the rolling direction and ND is the normal direction (c) Set-up for electropolishing process.

3.4.2 Electron Backscattered Diffraction (EBSD) analysis

The electropolished samples were characterised using a high-resolution SEM equipped with a field emission gun, TESCAN CLARA, and a high-speed EBSD system, Oxford Instrument SYMMETRY 2, as shown in Fig. 3.7. At the central region of the samples, a size of $1000 \times$

1000 μm region was selected for EBSD mapping using a step size of 2 μm . The selection of the map and scanning step size was based on the consideration of three main factors: the EBSD scanning time per map (maximum time is 10 hours due to the equipment access), statistically meaningful by containing >1000 grains, high fidelity of the data revealing details of GND density, LAGB and HAGB distribution and fine morphologies. The SEM was set as acceleration voltage of 30 kV, large current and 15 mm working distance respectively, to maximise the backscatter electron signal strength for EBSD acquisition. Note that the practical EBSD acquisition rate in this study is 200 points per second. The weaker electron signal strength may be attributed to the very thin aluminium oxide film formed on the free surfaces.

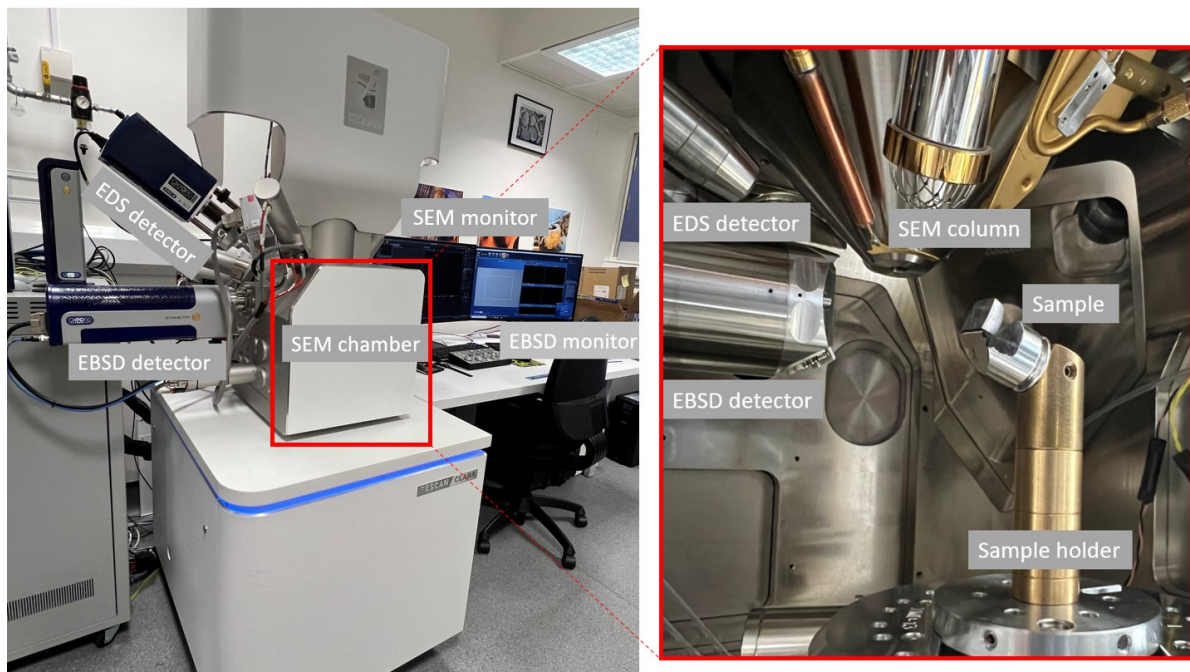


Fig. 3. 7 Tescan SEM equipped with Oxford EBSD detector and the SEM chamber configuration.

3.4.3 Post-processing for Misorientation, LAGB and HAGB

To reveal the GND density, LAGB and HAGB information, the acquired EBSD orientation maps were post-processed using the MTEX EBSD analysis software package [116-117], developed in Matlab, rather than the commercial EBSD analysis software, AZTech, as MTEX provides higher flexibility for high-end users to data-mine the EBSD maps and write their own scripts to extract information according to their requirements.

Misorientation is the crucial term for defining GND density, LAGB and HAGB, hence it is necessary to understand its calculation in EBSD. The mathematical framework for calculating

the misorientation angle has been explained by Randle and Olaf [118]. The crystal orientation of a point in the EBSD map is defined by a transformation of crystal unit cell from a sample reference frame to its measured orientation, through three rotations with respect to its x, y, and z' axis, where x, y are the original reference frame axes, and z' are the new axes of the rotated unit cell. These three rotations are measured and expressed by three Euler angles (Φ , ϕ_1 , ϕ_2). The orientation matrix of an EBSD point (g) can be expressed as:

$$g = \begin{bmatrix} c\phi_1 c\phi_2 - s\phi_1 s\phi_2 c\Phi & s\phi_1 c\phi_2 + c\phi_1 s\phi_2 c\Phi & s\phi_2 s\Phi \\ -c\phi_1 s\phi_2 - s\phi_1 c\phi_2 c\Phi & -s\phi_1 s\phi_2 + c\phi_1 c\phi_2 c\Phi & c\phi_2 s\Phi \\ s\phi_1 s\Phi & -c\phi_1 s\Phi & c\Phi \end{bmatrix} \quad (22)$$

where c and s represent cosine and sine respectively.

If the orientation of point of interest is described by matrix g_a and its four neighbouring points' orientations are g_{b1} g_{b2} g_{b3} g_{b4} , the average misorientation matrix (Δg) of this point is determined by:

$$\Delta g = \text{mean}(g_{b1}g_a^{-1} + g_{b2}g_a^{-1} + g_{b3}g_a^{-1} + g_{b4}g_a^{-1}) \quad (23)$$

Hence the average misorientation angle, θ , is calculated as:

$$\theta = (g_{11} + g_{22} + g_{33} + 1)/2 \quad (24)$$

The GND density, LAGB and HAGB in this study are fundamentally defined by their average misorientation angle (θ). It should be noted that the boundaries between GND density, LAGB and HAGB are unclearly defined in the community, and their values varied in previous literature [6]. In this study, if θ is in a range of $0^\circ < \theta \leq 2^\circ$, the misorientation is caused by the presence of GND density. If θ is in a range of $2^\circ < \theta \leq 15^\circ$, this point is defined as a LAGB point. If θ is larger than 15° , this point is a HAGB point.

3.4.4 GND density

HAGB and LAGB points can be determined directly from EBSD misorientation maps through the misorientation angle, but the GND density (ρ_{GND}) needs to be further processed. The EBSD GND density mathematical framework used in MTEX was based on Pantleon's work [32]. The detailed method will not be presented here, but briefly, EBSD measures the

orientation matrix ($\mathbf{g}$) of each probed point, and the misorientation matrix ($\Delta\mathbf{g}$) can be readily obtained based on Equation 24. Using the $\Delta\mathbf{g}$, and the previously obtained average misorientation angle (θ), the misorientation vector $\theta_i = \theta r_i$ where r_i is the rotation axis, can be determined. This will allow the curvature tensor ($\mathbf{k}$) to be calculated using Equation 25, and the GND density Nye's tensor (α) can be determined using Equation 26, assuming the elastic strain contribution is negligible, which was later validated by Wilkinson et al. [33].

$$k_{ij} = \frac{\partial \theta_i}{\partial x_j} (i, j = 1, 2, 3) \quad (25)$$

$$\alpha_{ij} = k_{ji} - \delta_{ij} k_{mm} \quad (26)$$

Though EBSD can provide the rotation angle vector (θ_i) along three sample frame axes (r_i , $i=1, 2, 3$), the EBSD map is only a 2D map, *i.e.* x_j , $j=1, 2$, as the third axis ($j=3$) is inaccessible in a conventional EBSD system. Thus, only six out of nine k_{ij} curvature components and five out of nine GND density tensor components can be obtained. On the other hand, there are twelve dislocation slip systems in a face-centred cubic (FCC) material. To solve the dislocation density on each slip system with only five known GND density tensor components, this becomes an underdetermined problem. Linear optimisation must be applied to estimate the total GND density, ρ_{GND} . Thus, the sum of GND density from all slip systems is estimated by the least energy configuration existing in the crystal, which means the estimated GND density value is an underestimated one. More details and the complete mathematical framework can be found in Pantleon's paper [32].

The upper bound of GND density ($\max \rho_{GND}$), and lower bound of GND density ($\min \rho_{GND}$) can be determined as $3.05 \times 10^{12} \text{ m}^{-2}$, *i.e.* 12.48 at $\log_{10}$ scale, and $6.10 \times 10^{13} \text{ m}^{-2}$, *i.e.* 13.79 at $\log_{10}$ scale, respectively, according to Equation 27 and Equation 28:

$$\min \rho_{GND} = \frac{\min \theta}{x \times b} \quad (27)$$

$$\max \rho_{GND} = \frac{\max \theta}{x \times b} \quad (28)$$

where the $\min \theta$ is the angular resolution of EBSD technique ($\sim 0.1^\circ$) [119] and $\max \theta$ is defined as 2° in this study; x is the EBSD scanning step size (2 μm) and b is the Burgers vector (0.286 nm for aluminium).

3.4.5 Grain and subgrain sizes

The locations of LAGB and HAGB can be determined and plotted as LAGB and HAGB maps, using EBSD through the measurement misorientation between the probed and its adjacent points. Converting these measured LAGB and HAGB to grain size and subgrain size is a standard practice for isotropic equiaxial-shaped grains [120], so that the effective grain size (d) simply equals to the reciprocal of the average number of intersections between the testing straight lines and all grains, per unit length. Although the grain boundaries manifest themselves as curved lines at the grain-grain interfaces in the 2D map, in 3D, grain boundaries are the free surface of a grain. Hence, grain boundary area per unit volume (S) is another commonly used term to describe the grain size, which can be estimated by the stereological theory [121]. Smith [122] established the general stereological equations for less biased estimation of the total grain boundary area per unit volume (S), based on the measured effective grain size (d) and is expressed in the following equation:

$$d = 2/S \quad (29)$$

where d is the effective grain size in μm and S is the grain boundary area per unit volume in $\mu\text{m}^2/\mu\text{m}^3$.

S can be determined in EBSD map through the following equation:

$$S = \frac{\text{grain boundary area}}{\text{volume}} = \frac{\text{EBSD grain boundary length}}{\text{EBSD map size}} \quad (30)$$

However, the above methodology was established and proved to be valid based on equiaxial-shaped grains. For elongated grains, as the grain morphologies are different in various sectioned planes, three orthogonal planes were recommended by the American Society for Testing and Materials (ASTM) E-112 [120] to measure grain size based on using a set of perpendicular straight test lines. Also, Gokhale and Drury [123] developed an alternative sampling method called “trisector”, using just three vertical metallographic planes, which

demonstrated a more reliable estimation of d and S from an arbitrary and inhomogeneous microstructure.

Nevertheless, sampling several planes for microscopic analysis is very time-consuming and costly. Morris et al. [124] proposed an alternative method with the aim to use only a single plane to estimate the grain size of elongated grains. In their method, the rolling-normal plane was selected to statistically estimate the grain size because most grain boundaries are not parallel to the normal direction. This method was validated by a series of rolling tests, conducted on a low-carbon steel with reduction from 10% to 80%, such that elongated grains were produced. Then, ASTM-E-112, trisector and these single plane methods were all investigated and compared. Their results suggested that the proposed single plane method is sufficient to estimate the d and S of heavily cold-rolled material reliably and efficiently with considerably elongated grain shapes. Such a relationship is valid for elongated grains, only if the rolling-normal plane is sectioned and examined as done in this study. Note that although EBSD can estimate the average grain size straightforward by calculating the areas inside grains, the methodology for reliably estimating the grain size of the elongated grains, the adoption of general stereological relationship is still required.

Chapter 4

Dynamic recrystallisation mechanism during hot forging

4.1 Introduction

This chapter investigates the dynamic recrystallisation mechanism in AA7050 alloys under typical hot forging conditions. Interrupted hot compression was conducted using Gleeble to the strain levels of 0.05, 0.15, 0.40 and 0.58 respectively, at 420 °C and strain rate of 0.05 s⁻¹. Electron backscatter diffraction (EBSD) was used to characterise the grain orientation, grain boundaries and geometrically necessary dislocations (GND) density at various strain levels. Qualitative and quantitative analyses were undertaken to reveal how GND density, low-angle grain boundary (LAGB) and high-angle grain boundary (HAGB) evolve as a function of strain. The correlations of GND density, LAGB and HAGB with strain and stress were examined, and the evolution process of these variables at grain scale was revealed.

The aim of this chapter is to reveal and quantify the detailed dynamic recrystallisation mechanism in AA7050 alloys under typical hot forging conditions. The detailed transformation mechanisms from GND to LAGB and from LAGB to HAGB will be examined. A series of hot compression tests to different strains were carried out in a Gleeble 3800 thermal-mechanical simulator, where the temperature profile and changes can be precisely controlled, and the post-deformation recrystallisation can be minimised. These samples under various plastic strains can reveal the dynamic recrystallisation process and hence the mechanisms. The latest EBSD system (Oxford Instrument, Symmetry 2) was used to scan relatively large orientation maps to enable statistical analysis. The estimated GND density, and characterised LAGB and HAGB were qualitatively and quantitatively analysed as a function of plastic strain. Based on direct observation and the statistical analysis, the detailed dynamic recrystallisation mechanisms will be proposed and discussed.

4.2 Procedures

To investigate the thermomechanical behaviour and microstructure evolution of aluminium alloy 7050, Gleeble 3800 has been employed to simulate the hot forging process for AA7050 and material characterisation techniques (EBSD) would also be used to analyse the microstructure evolution for interrupted tests with various strains (0.03, 0.15, 0.40 and 0.58) at a temperature of 420 °C and a strain rate of 0.05 s⁻¹ as mentioned in Chapter 3. After these compression tests, the samples were immediately water quenched to preserve the deformed microstructure. These thermal-mechanical tests and characterisation are to systematically examine the interactions between viscoplastic behaviour and the corresponding microstructural evolution (GND density, LAGB and HAGB).

4.3 Experimental results

4.3.1 Stress-strain curves

The annealed AA7050 cylindrical samples were hot compressed in Gleeble at 420 °C and 0.05 s⁻¹ to the strain levels of 0.05, 0.15, 0.40 and 0.58, respectively. The obtained true stress-strain curves are shown in Fig. 4.1. The consistency of stress-strain behaviour of all five curves was high, which suggests little variation in microstructure between samples. A clear trend is shown where the flow stress increased rapidly to a peak stress of 53 MPa at about 0.02 strain. As the strain further increased, gradual softening was seen. The flow stress dropped slightly and continually until reaching the final strain of 0.58, and the final flow stress of 48 MPa. This initial hardening followed by a gradual softening trend is characteristic and has been seen in many aluminium alloys [125-128].

The initial hardening process is likely caused by dislocation hardening, in which dislocations are generated for a slip to accommodate the imposed plastic deformation but pinned by various forms such as the tangling of dislocations from different slip systems and the pile-up of dislocation near slip obstacles such as grain boundaries and secondary-phase particles. As the dislocation density increases, the recovery rate will increase and recrystallisation will be activated, resulting in reduced dislocation content and lower flow stress. The low softening rate indicates a smooth transformation of microstructure, but the changes may be subtle as the stress only dropped slightly (14%). These points will be clarified and discussed in the

subsequent microstructure analysis. Note that relatively uniform deformations across the deformed samples had been achieved, as exhibited on the right-hand side of Fig. 4.1.

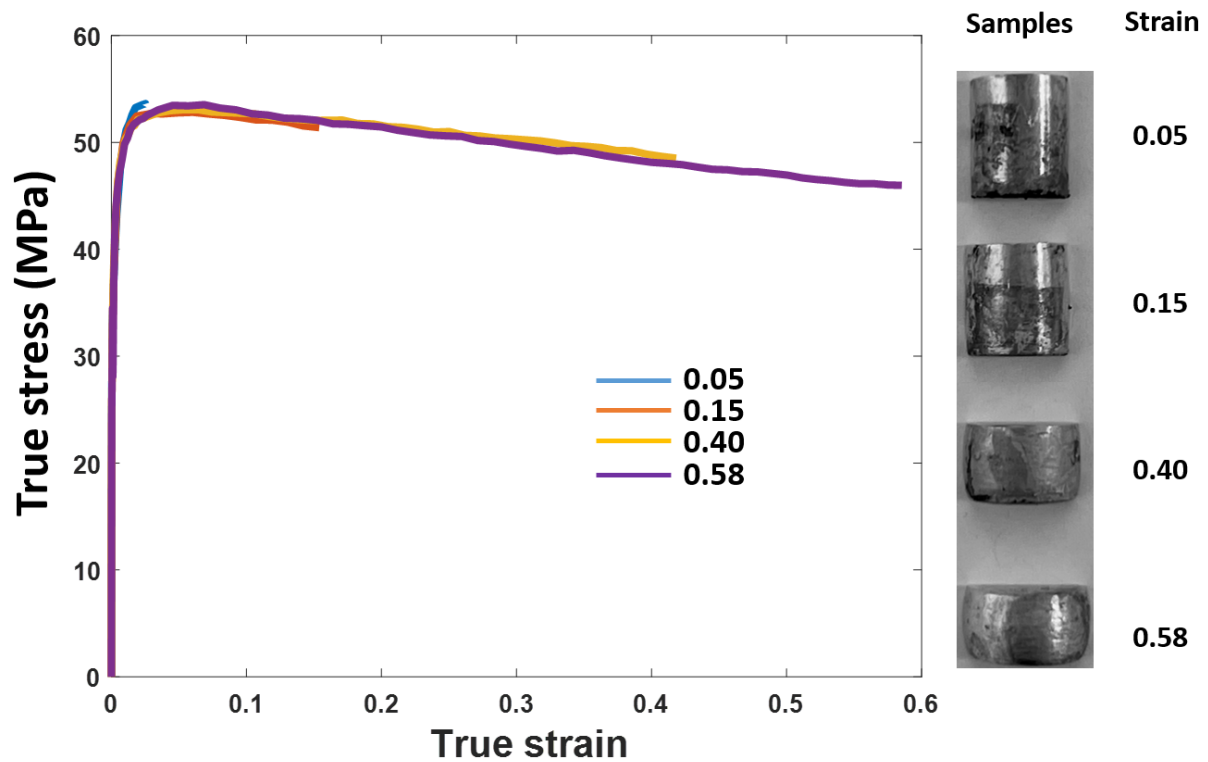


Fig. 4. 1 The true stress-strain plot and deformed samples carried out at 420 °C and 0.05 s⁻¹ at various strain levels.

4.3.2 Grain orientation and dislocation density evolution

An EBSD orientation and GND density maps contain a large amount of data, as each probed point is a data point and there are 250,000 data points per map to process and analyse. To obtain an overall detailed GND density distribution and evolution without missing important information, systematic data mining was undertaken. The qualitative and quantitative GND density evolution analysis approaches, as a function of plastic strain, are presented in Fig 4.2 and Fig 4.3, respectively. In Fig 4.2, the first row shows the general EBSD grain orientation maps are displayed as inverse pole figures (IPF) along the y axis of the map (the loading axis).

The second and third rows illustrate the GND density maps and their histograms. These allow the qualitative grain morphology, orientation, GND morphology and distribution as a function of plastic strain to be inspected directly from the maps. For more statistical quantitatively analysis to reveal the overall distribution and trends are shown in the forms of histograms. For these histograms, to correlate the GND density with stress and strain, the

flow stress as a function of the average of GND density and standard deviation of it (error bars); and the number of probed GND density points within each map (area fraction of GND, the average) and also standard deviation of GND density as a function of strain are computed and plotted in Fig 4.3(a) and (b) respectively.

The general grain distribution and evolution as plastic strain increases are presented in the first row of Fig 4.2. Initially, prior to the hot compression, the fully annealed sample still had the elongated large as-rolled grains, and the orientation of these grains seemed rather random. Within these grains, large misorientation is seen as a faint colour variation. Some small more equiaxial grains could be found adjacent to grain boundaries, and little orientation variation could be observed in these smaller grains. As the plastic deformation started, at the small strain level of 0.05, the small grains grew larger, and the size difference was smaller between the as-rolled large grains and the small but more equiaxial grains.

However, as the deformation proceeded to 0.15, grains were more compressed, flowing horizontally (normal to the loading axis). The orientation variations within grains became increasingly significant. Such trends continued at the strain levels of 0.4 and 0.58, respectively. In addition, the general grain orientations became more uniform. Only the green- and red-coloured grains could be observed, which may be due to either texture evolution or recrystallisation. Severely compressed thin grains together with very fine grains at either grain boundaries or grain interiors are shown in the 0.58 strained sample (Fig. 4.1). More detailed statistical analyses of HAGB and LAGB will be reported in the subsequent sections.

There are interesting findings from the GND density analyses in the second and third rows of Fig. 4.2 and Fig. 4.3(a) & (b). Firstly, based on the GND density maps, it can be seen that in the as-received fully annealed sample, GNDs presented in two distinct forms, either speckle-like or cell-like, with a mix of high and low GND densities in the large, elongated grains, or string-like patterns existing in those small near-equiaxial grains. These grains contain the majority of the lower GND density points, shown here as light green- and yellow-coloured. The white regions in the map are mostly the LAGB (subgrain) points, which are analysed separately in the subsequent section. These LAGB points were reversed to GND density points at 0.05 plastic strain, while the grain size of those near-equiaxial grains became larger and the GND density within them was increased as the dark blue points turned to light green. This indicates that the two types of grains underwent different processes. The dynamic

recovery took place in the large, elongated grains, as more LAGBs were turned back to GND density. Whereas the dislocation accumulation occurred in the near-equiaxial grains and resulted in increasing GND density within them, which implies that these grains accommodated probably most of the imposed 0.05 plastic strain.

As the plastic strain increased to 0.15 or higher levels, more dislocations were generated and stored, which resulted in the disappearance of low GND density points, and the increase in the white regions is seen in these maps. These are confirmed by the histogram plots showing that GND density points tend to move toward the upper bound of the GND value, which corresponds to 2° misorientation, while the standard deviation is reduced as there are less GND density points with low density. Also, there was a general decreasing trend for the number of GND density points as the plastic strain increased, which means more GNDs were transformed into LAGBs. These trends are confirmed in Fig 4.3(b) by displaying the correlations of the number, means and standard deviations of GND density points with the plastic strain.

The in-grain GND density structure evolution as a function of plastic strain is very interesting. It should be acknowledged that given the uniform strain distribution at the macroscopic scale, the local grain-grain strain distribution is significantly inhomogeneous [129] review by DIC and EBSD. Based on the GND density map inspection in the third row in Fig. 4.2, the GND structure evolution trend can be deduced as the following.

Initially, with a small amount of plastic deformation, dislocations formed a string-like network in the moderately deformed grains and the other area of these grains was occupied by low GND density points without specific structure as seen in the near-equiaxial grains in the GND map of 0.05 strain one. The present data are consistent with previous results showing the developed GND network structure in various grains characterised by high-resolution EBSD in deformed copper samples [130].

As the plastic deformation increased, the number of strings increased and eventually evolved into a regular fine cell structure. This structure is probably the most energetically stable form and can accommodate the maximum number of dislocations, *i.e.* the saturated state, as seen in those large, severely deformed grains. With further deformation, as seen in the GND density maps of the 0.40 and 0.58 strains, only this type of cell structure exists within the grains, and an increasing number of GND density points are transformed into LAGBs.

It is interesting to see the negative correlation between the flow stress and GND density in Fig. 4.3(a). Contrary to our expectations, the flow stress was reduced with increasing GND density. In other words, higher GND density requires less flow stress to push dislocations to slip. This finding is less surprising if we consider all aspects of microstructure evolution: GND density, LAGB and HAGB, rather than solely GND density in the hot deformation region, when the deformation temperature is higher than 0.7 of the melting temperature of the materials. In this case, the deformation temperature is already near 0.9 of the melting temperature, and the strain rate sensitivity (m) value is determined as 0.206. Based on the stress-strain curves obtained from Subroto et al. [131], $m = 0.206$ indicates the occurrence of a fast diffusion process [132]. The extensive diffusion occurs at grain boundaries, dislocations and grain interiors. These diffusion processes lower the effects of dislocation and grain boundary hardening and enable the grain boundary sliding process to take place, softening the material and lowering the required flow stress.

A detailed discussion about the relationship between flow stress and grain boundary will be presented in the HAGB section. However, this GND density and flow stress data provide convincing evidence, suggesting that although dislocation density still increases as a function of plastic strain, dislocation hardening effects on pinning oncoming dislocations is no longer the predominating mechanism to the flow stress behaviour.

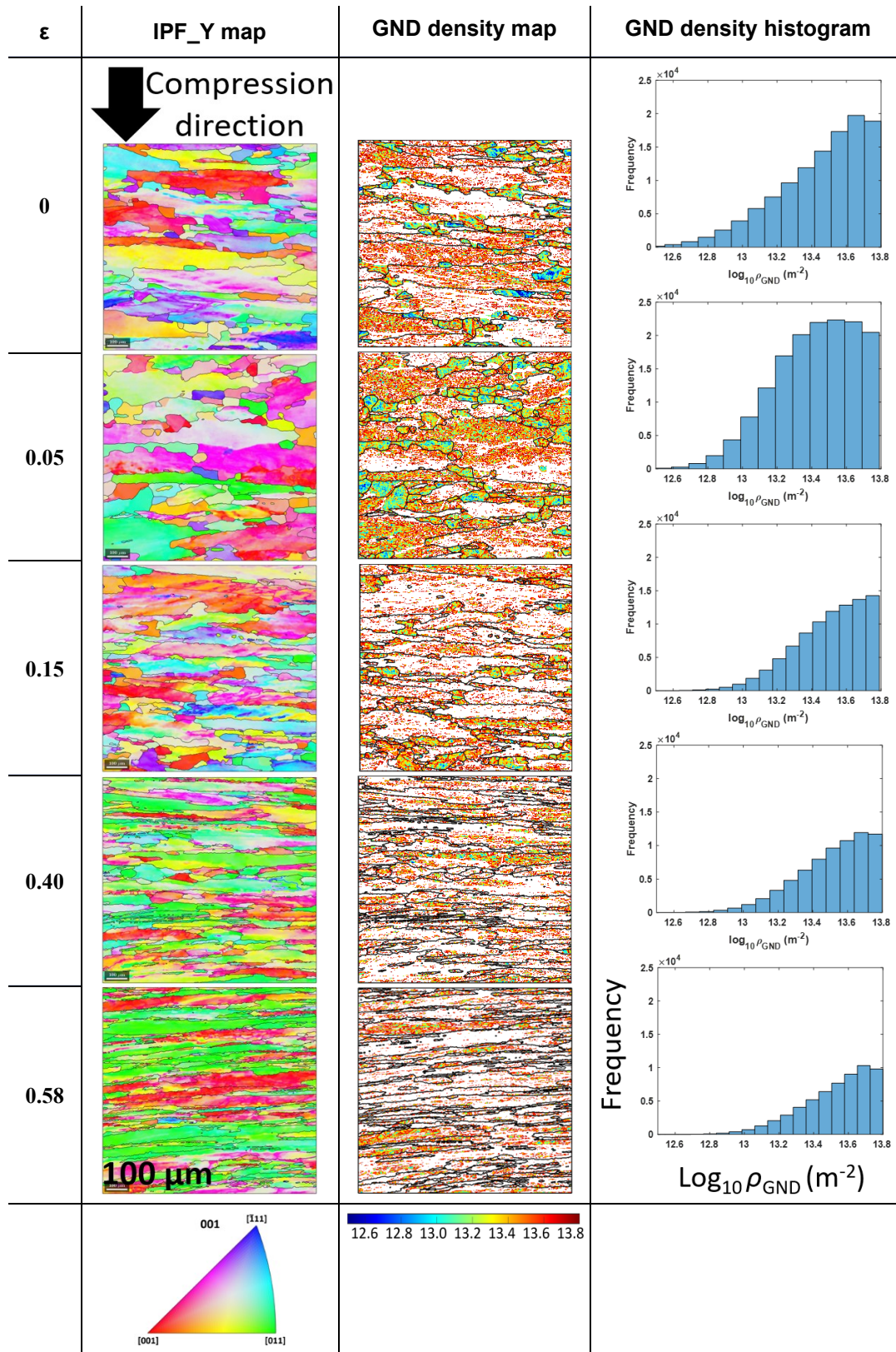


Fig. 4. 2 The evolution of grain orientation, GND density and its statistical distribution as a function of increasing plastic strain. The first row shows the plastic strain level; the second row shows the EBSD inverse pole figure (IPF) with respect to the y (the loading) axis; the third row shows the GND density maps in $\log_{10}$ scale overlaid with the high-angle grain boundaries, showing in black lines; the fourth row shows the histograms of GND density distribution.

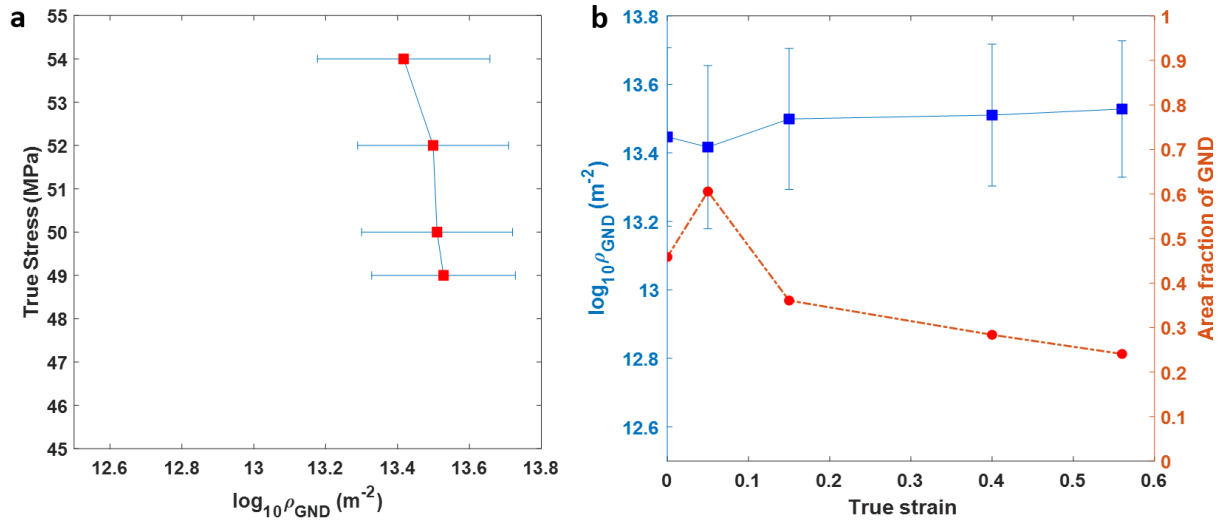


Fig. 4. 3 (a) The flow stress as a function of the average of GND density and standard deviation of it (error bars); (b) Area fraction of GND, the average and standard deviation of GND density as a function of strain.

4.3.3 Low-angle grain boundary evolution

The length of LAGBs indeed increases with increasing plastic strain, as illustrated in Fig. 4.4. The distribution of LAGBs can be observed in the second row in Fig. 4.4, where more LAGBs exist in those large, elongated grains, having high dislocation content with the fine cell-like structure, whereas the near-equiaxial grains are nearly LAGB-free. This corresponds with previous GND density results in transforming LAGBs from the saturated dislocation cell structure. If GNDs are still in their infant stage, *i.e.* a string-like network, no LAGB should be formed.

In the sample deformed to 0.05 strain, most strains are likely accommodated by the near-equiaxial grains to form a more string-like network, and the large grains are under a recovery state such that some of the LAGBs are transformed back to GNDs. However, this trend did not continue as the plastic strain increased. The larger plastic deformation resulted in more LAGBs, transformed from the saturated GND cells. The size of LAGB-formed cell-like subgrains is very small. The highest LAGB intensity emerged near some of the HAGBs as inferred in the LAGB maps of the 0.4 and 0.58 strain. Interestingly, a closer inspection of these two LAGB maps infers that these HAGBs with high LAGB intensity formed arrays of ultra-fine grains with a similar size to subgrains. These fine grains must be the newly recrystallised grains, showing convincing evidence of a strong association between LAGBs and HAGBs. This finding is consistent with the general CDRX theory that HAGBs are formed through LAGB transformation [6].

The quantitative analyses of these LAGB maps as a function of their misorientation angle were undertaken and plotted as histograms, shown in the third row in Fig. 4.4. The general distribution is found to be a half of a normal distribution shape, with the peak population at the lower end (near 2° misorientation) and declining gradually until the 15° misorientation. This distribution is similar in all LAGB maps regardless of their deformation level. Between these maps, some variations can still be found. For example, the number of LAGBs dropped from the strain level of 0 to 0.05, as explained earlier. This is due to the recovery and back-transformation to GNDs in these large grains. From the strain level 0.05 to 0.15, there is a rapid increase in the number of LAGBs. However, with a further increase in deformation level, the increase in LAGBs is very slight between 0.15, 0.40 and 0.58, while the peak value shifted slightly to a higher misorientation from 2° to 3° and the general distribution is slightly more spread, showing more LAGB points with 15° misorientation.

The mean and standard deviations were calculated from these histograms as a function of strain, as shown in Fig 4.5(b). The statistical trends clarify the evolution of LAGB length and misorientation angle, showing a slight increase with increasing plastic strain. It might seem counterintuitive that the means of misorientation angle do not increase toward 15° . Still, this finding is consistent with previous TEM dislocation and subgrain measurement results in dynamic recrystallised aluminium alloys [65]. They found that the misorientation angle in LAGBs saturated at 2° even after a shear strain of 10.08, in which geometrical dynamic recrystallisation (CDRX) is the dominating mechanism, in which, instead of gradual rotation of LAGBs into HAGBs, HAGBs are formed directly by pinching off LAGBs. It seems the observed DRX fit to the CDRX. However, it should be noted that our dataset is limited to the strain level of 0.58 with only a small fraction of dynamically recrystallised grains. It is possible that much larger strain is required to show the CDRX process.

It is challenging to directly compare our LAGB findings with the literature, since either GNDs and LAGBs are treated as one variable or the LAGB distribution is normalised with respect to the total grain boundaries (LAGBs+HAGBs) [51, 67, 133]. Therefore, the number of LAGBs cannot be compared directly from one plot to another plot as the total grain boundaries are increasing.

Even though the majority of grains are occupied with a high intensity of LAGBs and small dynamically recrystallised grains, there are still few virtually LAGB-free grains, as shown in the LAGB maps subjected to 0.4 and 0.58 strain. Linking with the GND density maps in Fig.

4.2, we can see these LAGB-free grains are occupied with high GND density. This implies that deformation or GND/LAGB formation processes are very heterogeneous and orientation and neighbouring-grain dependent in polycrystalline materials. Some preferentially oriented grains with similar neighbours are likely to be deformed and forming GND and LAGB structure first, while those ‘hard’ grains will be subsequently deformed, forming the GND network and later LAGBs. These findings confirmed the correlation between the deformation-GND-LAGB and grain orientations as found in previous studies of aluminium [67] and copper [130], respectively.

In addition, the total numbers of LAGBs in the LAGB maps are converted into the length of LAGBs and plotted as a function of stress in Fig. 4.5(a). Like the GND density, a negative correlation is obtained between the LAGB length and stress, as seen in Fig. 4.5(a). Generally, LAGBs are believed to be low-energy, stable dislocation structures, acting as effective dislocation barriers and responding to the strain hardening [134]. Since the flow stress behaviours are determined by several factors, such as grain size, dislocation density *etc.*, this negative correlation should not be over interpreted. Nevertheless, it does imply that the hardening effect of LAGBs, similar to GND density hardening, has been overtaken by other softening factors. More discussion about the flow stress will be presented in the subsequent HAGB section.

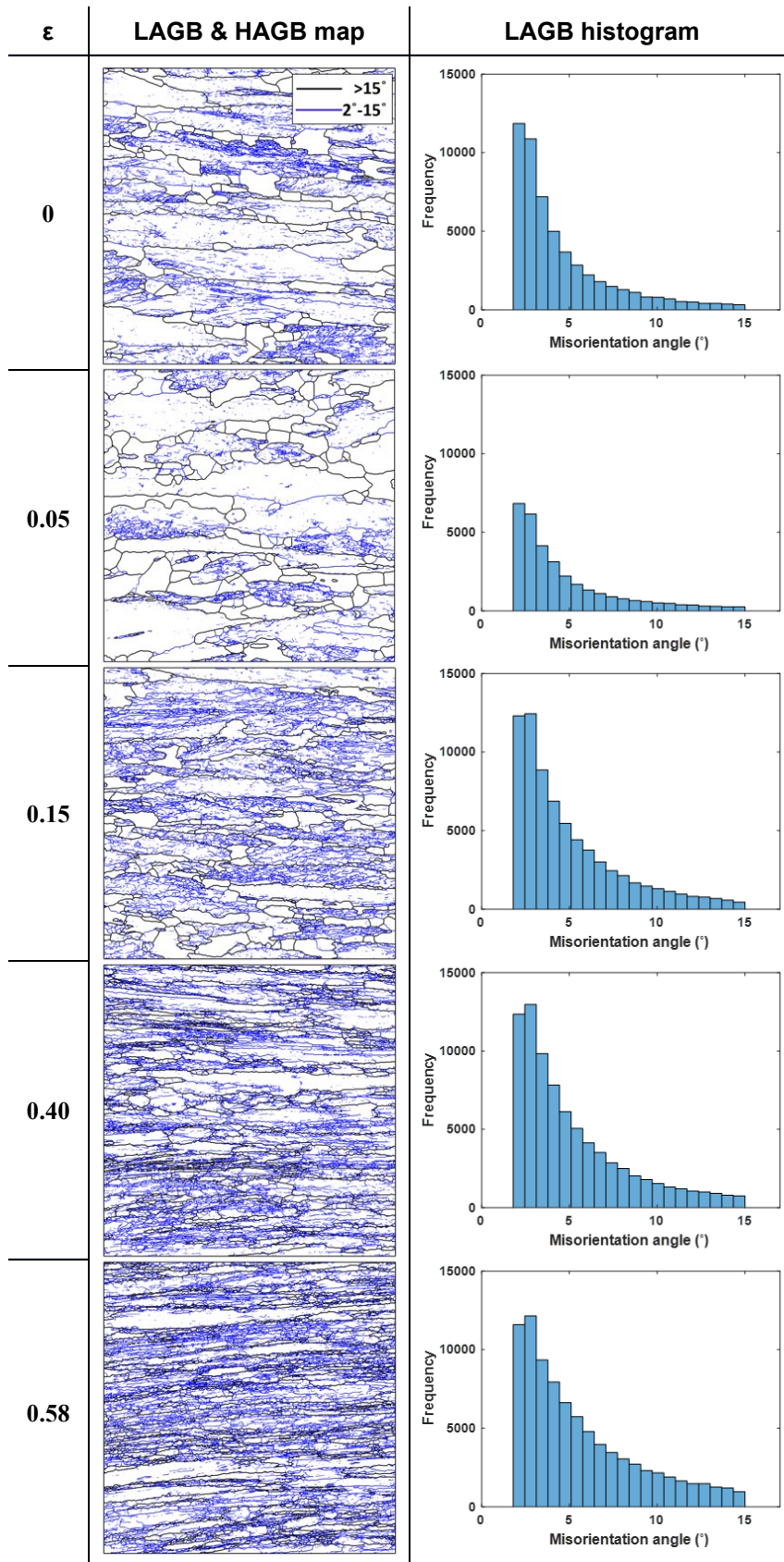


Fig. 4. 4 The evolution of LAGB and its statistical distribution as a function of increasing plastic strain. The first row shows the plastic strain level; the second row shows the LAGB maps overlaid with HAGBs, showing in black lines; the third row shows the histograms of LAGB misorientation distribution.

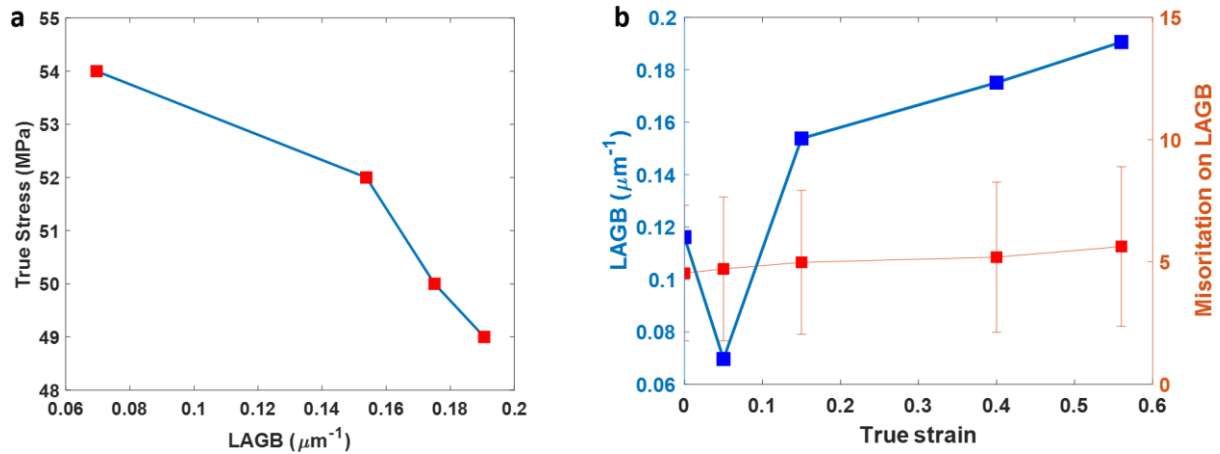


Fig. 4. 5 (a) The flow stress as a function of the length of LAGB; (b) the length of LAGB as a function of imposed strain and the average and standard deviation of misorientation on LAGB as a function of imposed strain.

4.3.4 High-angle grain boundary evolution

The relationships between GND density, LAGB and HAGB have been discussed in the previous sections. This section focuses on the formation of new dynamic recrystallised grains and discusses their effects on the flow stress. At a glance at the HAGB maps in Fig. 4.6, that the length of HAGBs per unit area increased extensively, almost doubled as illustrated in Fig. 4.7(b), with increasing plastic strain. The morphologies of the grains also changed from the initial undeformed state. For example, in the maps subjected to 0.4 and 0.58 strain, compared to the initial grains, the thickness of the elongated as-rolled grains was reduced markedly, and those near-equiaxial grains were squeezed into elliptical shapes. Also, as noticed in the LAGB maps, there are a number of ultra-fine grains formed on some of the HAGBs.

Care should be taken when examining the HAGB relationships with stress and strain. The geometric effects on grain boundary length in elongated large grains under severe plastic deformation cannot simply be ignored. Briefly, as the samples are compressed, the thickness of the elongated grains is reduced and more grains, and grain boundaries, are pushed in the characterisation window. This results in an increase of grain boundary length, even without any recrystallised grains.

These geometric effects are less significant if the shapes of the initial grains are more circular in the 2D map. However, in this study with elongated large grains, the geometric effects on grain size are considerable and must be removed from the final HAGB length. Only rough estimation can be made under some assumptions, as the deformation results in 3D grain shape changes. However, we only have 2D information and accessing the third dimension is

extremely challenging. The method used to estimate the geometrical effects for grain boundary length change is shown in Appendices A. Fig. A1.

The geometric effects were removed from the initially determined total grain boundary length, and the results are reported in Fig. 4.6. The general trend revealed is that the number of HAGBs shows a rapid increase with increasing plastic deformation. This increase in HAGB length is due to dynamic recrystallisation, and can be confirmed by the HAGB maps at 0.4 and 0.58 strain where many ultra-fine grains are formed near original HAGBs. The distribution of HAGB misorientation suggests statistically no correlation with plastic strain, since the dynamically recrystallised grains are very small and only contribute to a tiny fraction of the total HAGBs. Hence, the dynamically recrystallised grains would not affect the general misorientation distribution, even if they were transformed from LAGBs. Further studies with complete dynamic recrystallisation will be needed to clarify this point.

Combining the undertaken GND density, LAGBs and HAGBs analyses, our findings provide convincing evidence that the flow stress seems to be dominated by the grain boundary sliding softening process at the testing condition ($0.9 T_m$) rather than hardening through pinning dislocation slip by the formed dislocation structure, LAGBs or HAGBs. Though all these dislocation-slip obstacles increased with plastic deformation, the flow stress decreased, which implies that the HAGBs had altered their functions in viscoplasticity from strengthening the material by pinning dislocation to softening the material through grain boundary sliding, as the increasing dislocation density or LAGB length cannot exert softening effects on flow stress. Therefore, the longer HAGB length, as a result of dynamic recrystallisation, leads to flow stress softening. The other possible reason proposed for the observed softening was due to texture changes [25, 125-127]. However, this is unlikely the reason for this study, the texture due to deformation would strengthen the material, and due to recrystallisation is not strong.

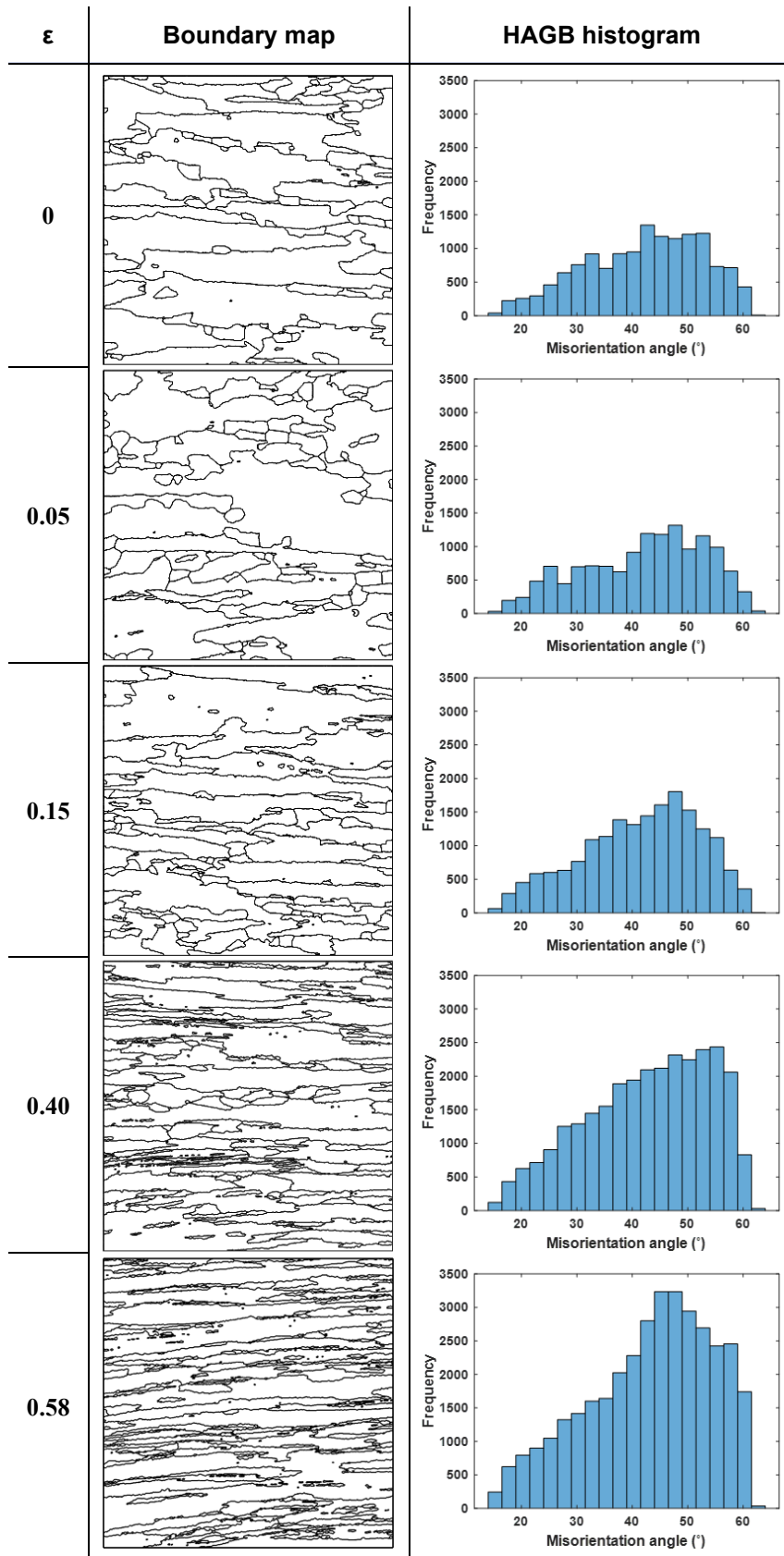


Fig. 4. 6 The evolution of HAGB and its statistical distribution as a function of increasing plastic strain. The first row shows the plastic strain level; the second row shows the HAGB density maps; the third row shows the histograms of HAGB misorientation distribution.

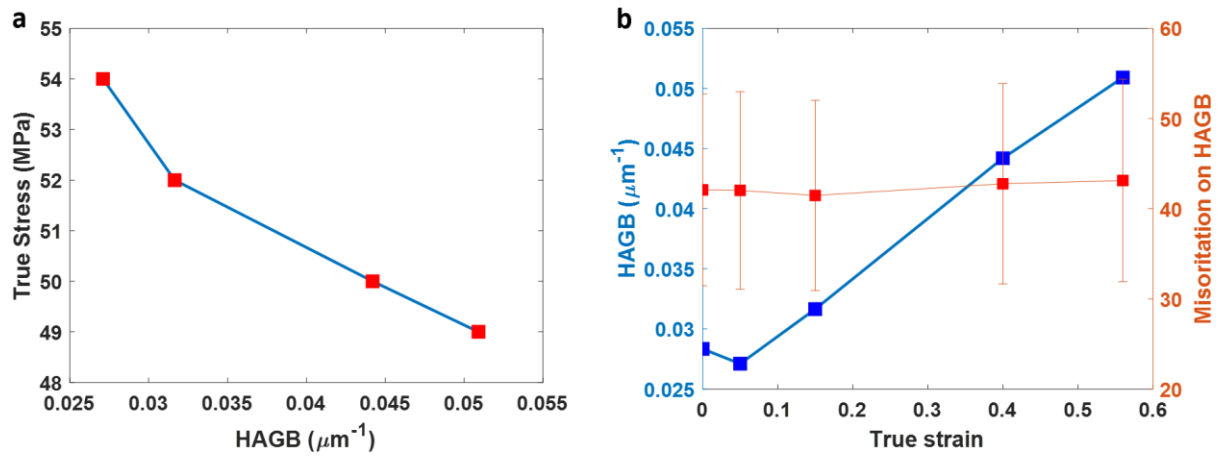


Fig. 4. 7 (a) The flow stress as a function of the length of HAGB; (b) the length of HAGB as a function of imposed strain and the average and standard deviation of misorientation on HAGB as a function of imposed strain.

4.3.5 Dynamic recrystallisation mechanism

Based on the obtained findings of GND density, LAGBs and HAGBs in this study, the general dynamic recrystallisation picture is summarised and schematically illustrated in Fig. 4.8. At the hot deformation temperatures ($> 0.6T_m$), as the plastic deformation starts, dislocations are generated and slip to accommodate the imposed plastic strain. The dislocations from different slip systems interact, jump and cross-slip to form a string-like dislocation network within the grains. As plastic strain increases, the number of strings increases and the dislocation network evolves to form a more regular fine cell-like structure. This structure seems to be the saturated (steady-state) dislocation structure. Further plastic strain and dislocations do not alter the dislocation network morphology or the average dislocation density, rather than transferring them into LAGBs.

As the deformation proceeds, the dislocation density does not vary any further. More dislocation cell structures are transformed into fine near-equiaxial subgrains. The number/length of LAGBs increases significantly, also the LAGBs start to rotate by increasing their misorientation angles, especially for those ones adjacent to grain boundaries. Gradually, some of the LAGBs were transformed into HAGBs and formed ultra-fine grains, similar in size to those subgrains near grain boundaries. This dynamic recrystallisation mechanism appears to support the existing dynamic recrystallisation theory in the literature [23]. However, the dislocation network evolution from string-like to cell-like structure and the transformation from dislocation structure to LAGBs were missing, which is a critical part linking the inhomogeneous plastic deformation at grain scale to dynamically recrystallised grain nucleation [6].

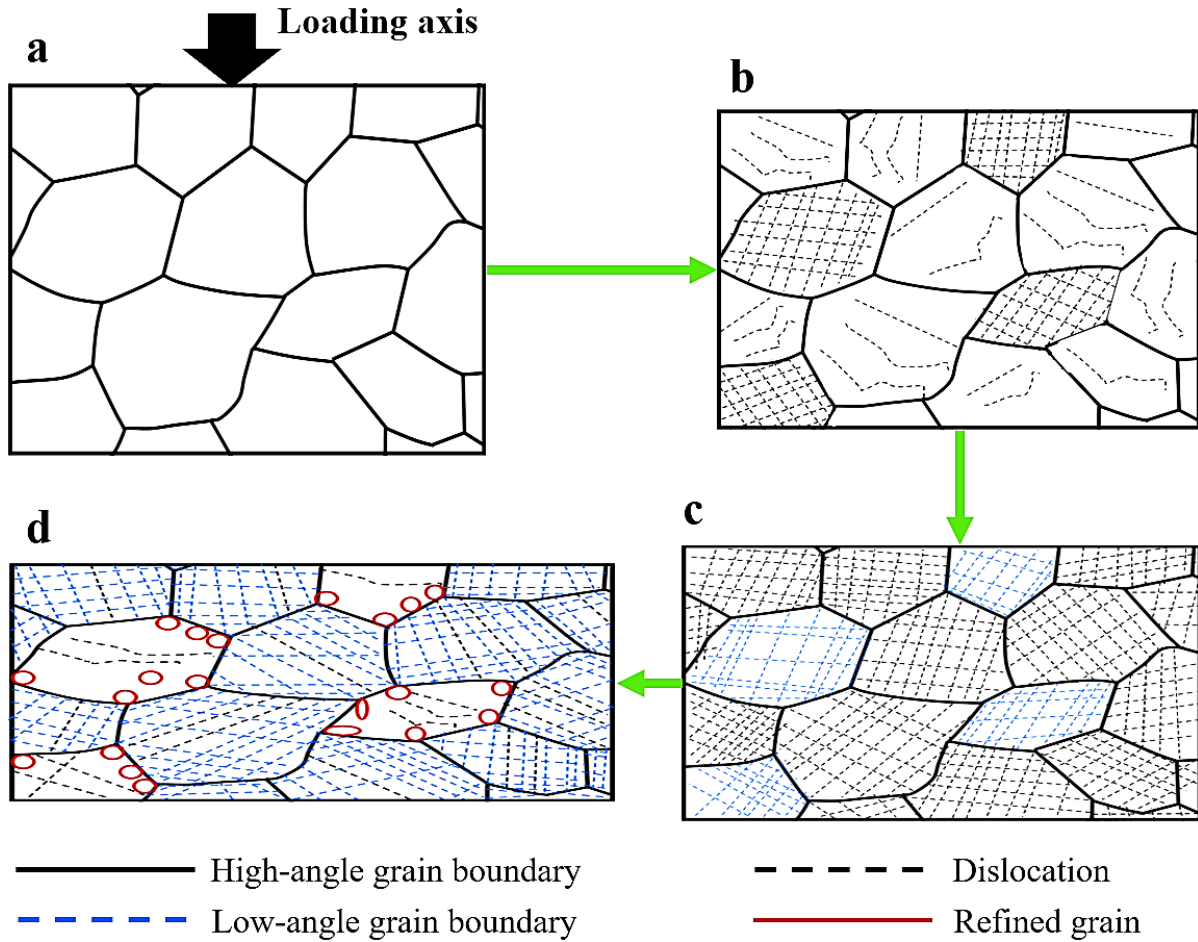


Fig. 4.8 Schematic diagrams illustrating continuous dynamic recrystallisation processes (a) undeformed grains; (b) the initial deformation stage showing the dislocation structure; (c) intermediate stage showing the dislocation structure and subgrain formation; (d) the last stage showing the recrystallised grains with HAGBs.

A few assumptions should be acknowledged and discussed prior to generalising our findings. The grain and dislocation density samplings are assumed to be statistically sufficient. Sampling a statistically sufficient number of grains using EBSD was challenging in this study, due to the existence of large, elongated grains in the as-received material, as seen in Fig. 4.2. This raised a sampling issue for EBSD mapping, because EBSD requires point-by-point scanning, and the scanning speed is limited. Per EBSD session (12 hours), there is a maximum number of scanning points. Hence, the EBSD map size of 1000 mm $\times$ 1000 mm at a step size of 2 μ m was set in the methodology chapter. The statistical representativity was confirmed in this section by analysing three different regions of interest on the annealed sample, as displayed in Appendices B, Fig. B1. The distribution of GND density, LAGB and HAGB misorientation is consistent, which confirms the statistical meaningfulness of the obtained data.

The effects of precipitates are ignored in this study by assuming their distribution does not vary from one strain level to another. It is well known that second-phase particles, *i.e.* precipitates, play an important role in dynamic recrystallisation. Fine precipitates tend to aggregate at or near grain boundaries, hindering boundary motion and slowing down recrystallisation and grain growth through the Zener drag effect [23, 135-136]. The Zener drag pressure equals grain boundary energy times the precipitate volume fraction, divided by the average size of precipitates. Studying either fine or coarse precipitate effects on dynamic recrystallisation is very challenging. The size of fine effectively precipitates for strengthening is typically a few nm [137], which is beyond the resolution of an SEM.

Though coarse precipitates can be characterised under an SEM, their dislocation plasticity behaviour is very localised at a grain-grain level. Micromechanical testing and a characterisation method would be necessary [138] to reveal such mechanisms, but such work is beyond the scope of this study. Nevertheless, as shown in Fig. 3.3, to reduce the hardness of the materials, the as-received the fine-sized precipitates in as T7451 temper state were fully annealed at 413 °C for 2 hours, in which most of the precipitates were expected to grow into a considerable coarse stable size, little further growth is shown in the following hot compression test. The distribution of precipitates prior to and post-hot compression was confirmed to be similar using the backscattered electron (BE) detector on the SEM, as seen in Appendices C, Fig. C1.

Finally, clear dynamically recrystallised grains can be found in the sample at the strain level of 0.58. The number of dynamically recrystallised grains is small as the dynamic recrystallisation process did not occur extensively. Higher plastic strain or temperature is required to further investigate the LAGB and HAGB misorientation rotation.

4.4 Summary

This chapter investigated the dynamic recrystallisation mechanism in AA7050 alloys under typical hot forging conditions, using an interrupted hot compression method on Gleeble to the strain level of 0.58 at 420 °C and strain rate of 0.05 s⁻¹. EBSD has been used to characterise the grain orientation, grain boundaries and GND density at various strain levels. The qualitative and quantitative analyses have been undertaken to reveal how GND density,

LAGBs and HAGBs evolve as a function of strain. The results yielded the following conclusions:

- i. With small plastic deformation, a string-like dislocation structure is firstly formed in some of the grains. As the deformation increases, this string-like dislocation structure evolves into a more homogenous fine cell-like dislocation structure.
- ii. The average dislocation density saturates at 13.5 at $\log_{10}$ scale at the strain level of 0.15 and further straining to 0.58 led to 35% of the dislocation cell structure transforming into LAGBs (subgrains).
- iii. From the strain level of 0.15-0.58, the misorientation angle of LAGB increased slightly from 4° to 5° , but the area fraction of LAGB per unit volume increased markedly from 1.6×10^5 to $1.9 \times 10^5 \text{ m}^{-1}$.
- iv. Some of the LAGBs were transformed into ultra-fine grains formed near grain boundaries, resulting in a rapid increase in the area fraction of HAGB per unit volume from 5×10^3 to $15 \times 10^3 \text{ m}^{-1}$. However, the misorientation angle of HAGBs did not vary markedly.
- v. The strain softening effect is found due to the increasing of HAGBs to facilitate the GB sliding, overtaking the dislocation density, LAGB and HAGB hardening effects by pinning dislocations.

Chapter 5

Effects of processing parameters on dynamic recrystallisation

5.1 Introduction

The aim of this chapter is twofold. Firstly, to undertake a qualitative and quantitative assessment of the GND density, LAGB and HAGB maps, in order to explore the effect of deformation temperature and strain rates on the dynamic recrystallisation mechanism in aluminium alloy 7050. Secondly, to attempt to identify the key underlying mechanism governing the flow stress behaviour.

The results and discussion are organised into six sections. Section 1 provides an overview of the thermal-mechanical behaviour of AA7050 and its variation as a function of deformation temperatures and strain rates. Sections 2, 3 and 4 are dedicated to exploring the effect of strain rate and temperature on GND density, LAGB and HAGB distribution, and their link to flow stress respectively. Section 5 provides in-depth analysis on the overview of relationships of GND density, LAGB and HAGB with the integrated strain rate and temperature effect, *i.e.* the Zenner-Hollomon parameter, to understand how flow stress and underlying microstructure evolution as a function of Zenner-Hollomon parameter, with the aim to identify the dominating mechanism governing the flow stress. The final section discusses the underlying dynamic recrystallisation mechanisms based on the obtained data in Sections 2 - 4. The possible reasons, driving forces and also assumptions and limitations in this study will also be presented and discussed in this section. Again, the secondary particle effect on dynamic recrystallisation lies beyond the scope of this thesis.

5.2 Procedures

The hot compression tests were heated to evaluated temperatures which are 380, 400, 420, 440 and 460 °C and held for 3 minutes within Gleeble. Then, a constant deformation level

was set to 0.58 for the compression process, and the samples were compressed to around 7 mm in height with various strain rates (0.0005, 0.005, 0.05, 0.5 and 5 s⁻¹). EBSD was employed to do the microstructural analysis for compressed samples. More details about the experimentation set-up are shown in Chapter 3.

5.3 Results and discussion

5.3.1 Stress-strain curves

The hot compression flow stress responses under various strain rates at a constant temperature of 420 °C are shown in Fig. 5.1(a). It can be seen that at 420 °C, AA7050 exhibits a significant strain rate hardening behaviour, where the flow stress increased from a mere 20 MPa at 0.0005 s⁻¹ to 110 MPa at 5 s⁻¹, indicating a strong strain rate-dependent viscoplastic behaviour. The general shapes of these stress-strain curves resemble one another, for which all curves rapidly reached the peak stresses followed by slight and gradual softening. For example, the curves for the strain rates of 0.005 s⁻¹ and 0.0005 s⁻¹ seem to show a steady-state after the peak stresses. Further analyses were undertaken to examine the softening rate which is defined by the ratio of the stress difference between the peak stress and the final stress, over the corresponding peak stress. This may imply that AA7050 samples tested under various strain rates are subject to a similar deformation mechanism.

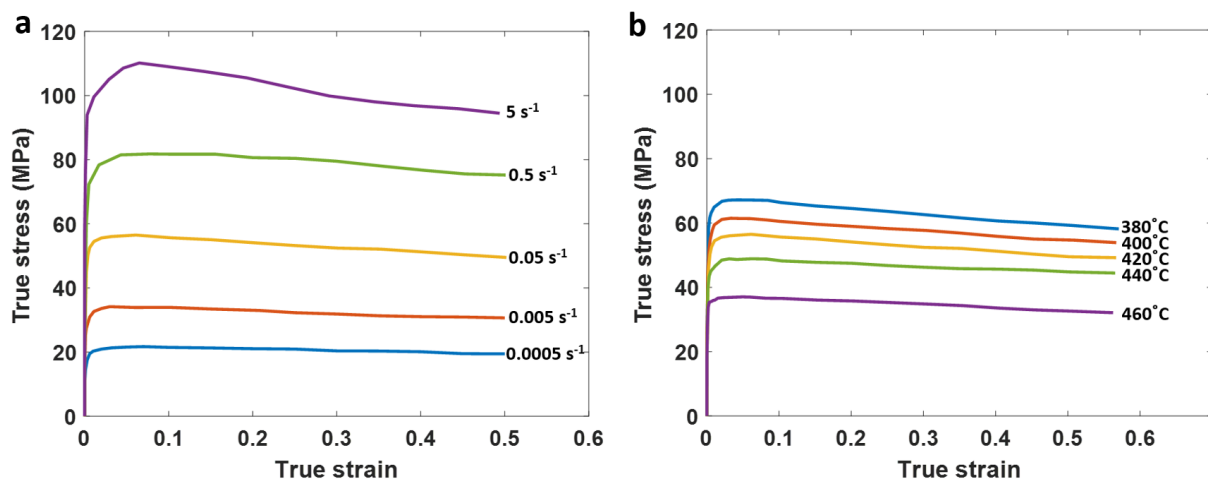


Fig. 5. 1 True stress-strain curves obtained under (a) a range of strain rates (0.0005 – 5 s⁻¹) at constant temperature of 420 °C and (b) a range of temperatures (380 – 460 °C) at a constant strain rate of 0.05 s⁻¹ to the strain level of 0.58.

Under the constant strain rate of 0.05 s⁻¹, the stress-strain data at the temperature range of 380 to 460 °C were obtained and the temperature effects on the true stress-strain curves are

presented in Fig. 5.1(b). The general shapes are similar to those of different strain rates. First, the initial flow stress increased rapidly. Once the flow stress reached peak stress, a gradual smooth decline took place and remained so until the end of the test. Also, the flow stress was found to be strongly influenced by the deformation temperature. As the temperature increased, lower flow stresses were required to deform the material. For example, the peak stress reduced from 68 to 38 MPa from 380 to 460 °C. Compared to the strain rate effects, the variation of stress due to temperature change is not as remarked. The likely reason might be the selected temperature range only varied less than 100 °C, while the strain rate changed by 4 orders of magnitude. With regard to the softening rate, the material displayed a similar softening rate across all testing temperatures, which may indicate that a consistent underlying mechanism exists for all testing temperatures.

5.3.2 Effects on dislocation density

The qualitative and quantitative investigation of the roles of strain rate and temperature on the dislocation density is presented in this section. The analyses on the strain rate effect will be shown first, followed by the analyses of temperature effects. The EBSD orientation maps (inverse pole figures), GND density maps and GND density statistical histograms are presented in Fig. 5.2 as a function of strain rate. The EBSD orientation maps infer that the texture is stronger as the strain rate increases since there are fewer red grains and more and more grains change their colours to green. This implies that the orientation of grains becomes more uniform as the deformation speed increases. Possibly, the green grains may be formed through dislocation slip resulting in grain rotation caused by the imposed plastic deformation, *i.e.* deformation texture, or another possibility is the formation of recrystallised grains, which could have preferential orientation, *i.e.* recrystallisation texture. This finding will be further discussed in the subsequent HAGB section.

It is of interest to see that the GND density increases significantly with increasing strain rate. Combining the GND density maps and histograms, it appears that at the lowest strain rate (0.0005 s^{-1}), the mean GND density is also the lowest, approximately 13.05 m^{-2} at $\log_{10}$ scale, and the majority of the points within grains are GND density points. The distribution of GND density within grains is relatively uniform, filled with a cell-like structure in all grains. As the loading speed increases, there is less time for recovery and the GND density increases rapidly

to the maximum mean value of 13.6 m^{-2} at $\log_{10}$ scale at the strain rate of 0.05 s^{-1} , which corresponds to the maximum dislocation density where the misorientation is less than 2° . Beyond this value, the point is defined as LAGBs. The GND density reaches a saturated state at 0.05 s^{-1} with uniform dislocation cell structure, and the total number of GND density points drops significantly, as shown in the histogram. Further increase in the strain rate reduces the total number of GND density points within the map. At the highest strain rate of 5 s^{-1} , only a few saturated GND density points exist within the GND density map.

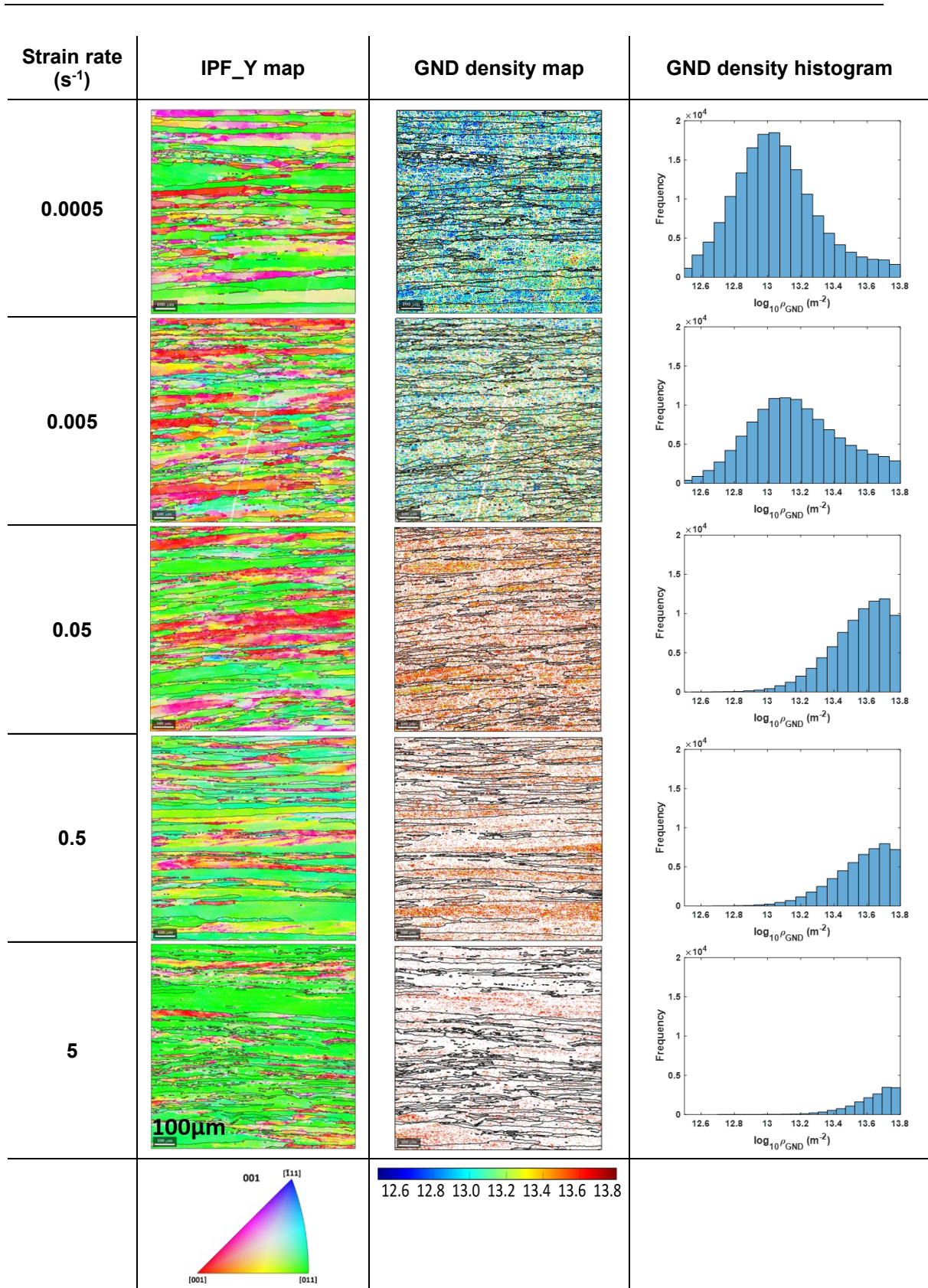


Fig. 5. 2 The evolution of grain orientation, GND density and its statistical distribution as a function of increasing strain rate at 420 °C: The first column shows the strain rate; the second column shows the EBSD inverse pole figure (IPF); the third column shows the GND density maps in log₁₀ scale overlaid with the high-angle grain boundaries, showing in black lines; the fourth column shows the histograms of GND density distribution. The y-axis is the number of points rather than fraction of total data.

More in-depth statistical analyses and correlation with flow stress were conducted, and the results are shown in Fig. 5.3. A strong positive correlation between the flow stress and dislocation density is shown in Fig. 5.3(a), which indicates that higher dislocation density requires higher flow stress and may suggest that dislocation density hardening is considerable. The relationships between dislocation density and strain rate are presented in Fig. 5.3(b).

Clear trends exist showing that dislocation density increased with increasing strain rate, while the number of dislocation density points (area fraction) was reduced almost linearly with rising strain rate. This might imply that as the strain rate increases, there is less time for recovery to take place and significantly more dislocation density would be stored in the material, resulting in the extensive transformation from dislocation boundaries to other boundaries, *e.g.* LAGB and HAGB.

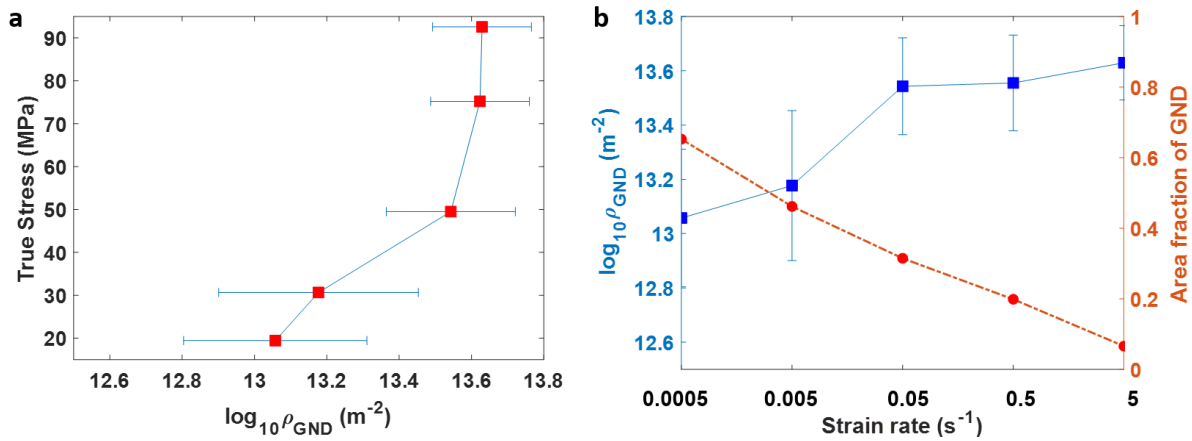


Fig. 5. 3 Statistic values for GND density and its relations with flow stress and strain rate. (a) The relationship between the flow stress and the average GND density and its standard deviation (error bars); (b) Area fraction of GND, the average and standard deviation of GND density as a function of strain rate. The overview of temperature effects on dislocation density is presented in Fig.5.4. Interestingly, the grain orientation data also shows a clear orientation variation trend as a function of the deformation temperature. As the temperature increased, the grain orientations were transformed from ‘red’ [001] grains to ‘green’ [011] ones, which is similar to the previous trend in strain rate analysis. It suggests that the temperature has a similar effect on grain orientation changes, which could be due to dislocation slip resulting in deformation texture, recrystallisation texture, or grain boundary sliding resulting in grain rotation.

The general trend in GND density with decreasing temperature could be found as the GND density increases as the temperature drops, due to the suppressed dynamic recovery at lower temperatures. Hence, the GND density was the highest at the lowest temperature of 380 °C

with the minimum number of GND density points. Further discussion on LAGB and HAGB of these two maps will follow later in Chapters 5.2.3 and 5.2.4, respectively.

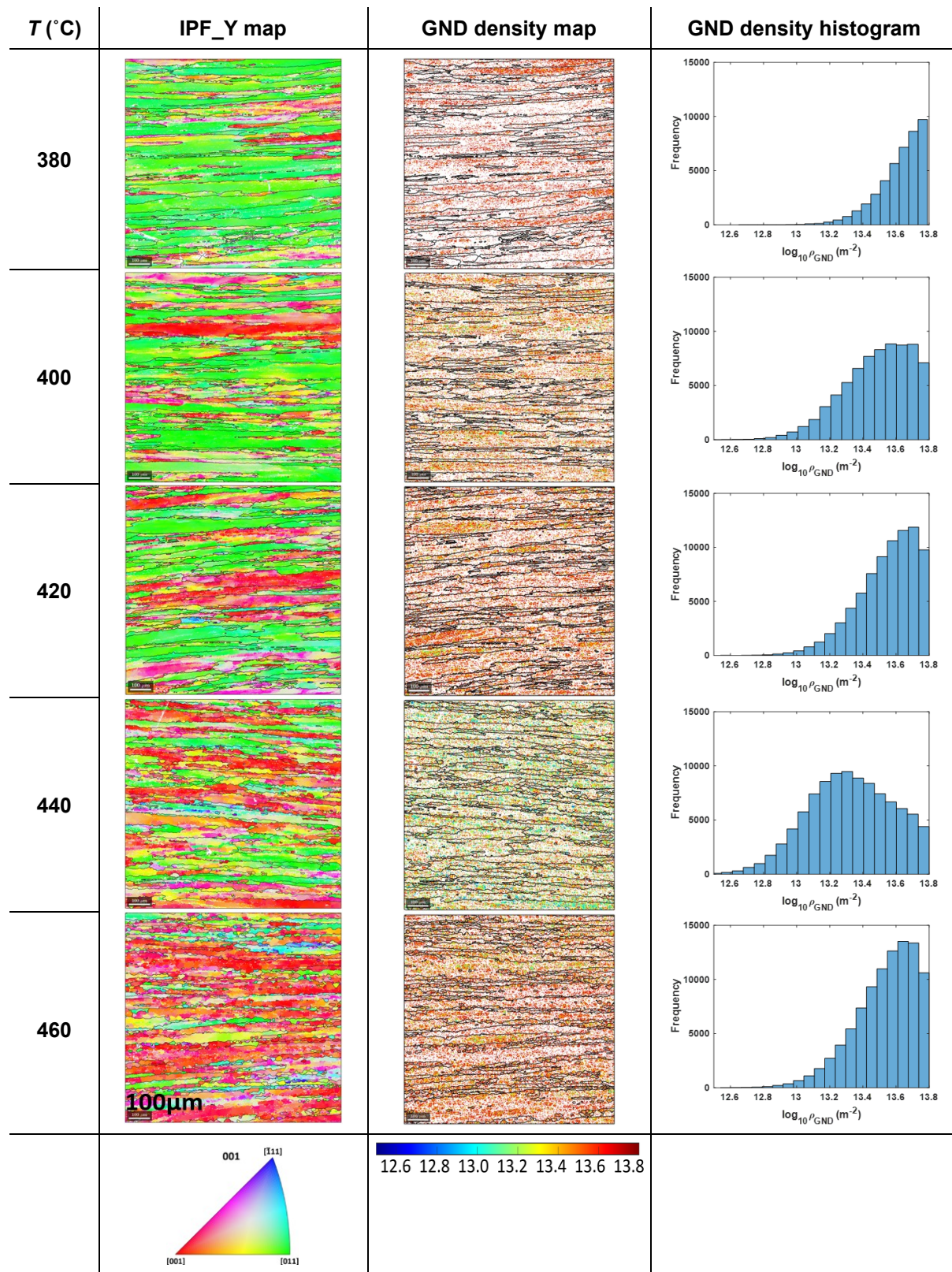


Fig. 5. 4 The evolution of grain orientation, GND density and its statistical distribution as a function of increasing temperature at the strain rate of 0.05 s^{-1} . The first column shows the temperature; the second

column shows the EBSD inverse pole figures (IPF) with respect to the y (the loading) axis; the third column shows the GND density maps in $\log_{10}$ scale overlaid with the high-angle grain boundaries in black lines; the fourth column shows the histograms of GND density distribution. The same scale bars and axes are used for figures in each column.

The summary of the dislocation density relationship with deformation temperature and flow stress is shown in Fig. 5.5. As the temperature decreases from 460 °C to 380 °C, the stored dislocation density rises, due to the reduced thermal energy to facilitate dislocation annihilation. Whereas the number of dislocation density points reduces, due to the increasing transformation to LAGB. Regarding the stress-dislocation density relationship (Fig. 5.5(a)), the general trend seems to suggest a positive correlation in that higher dislocation density requires larger flow stress, which is consistent with the strain rate effect. Nevertheless, as mentioned previously, the dislocation density estimated at deformation temperatures of 420 °C and 460 °C seem to be outliers, which did not follow the general trend.

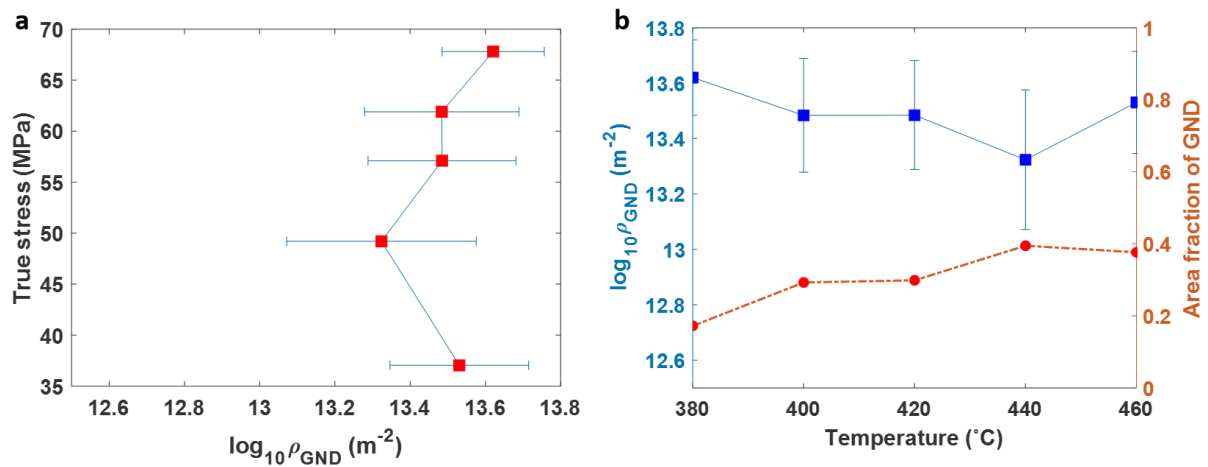


Fig. 5.5 (a) The flow stress as a function of the average of GND density and standard deviation of it (error bar); (b) Area fraction of GND, the average and standard deviation of GND density as a function of temperature.

5.3.3 Effects on LAGB

The strain rate effects on LAGB are presented in Fig. 5.6. The boundary maps show the LAGB intensity and morphology changes from low to high strain rate. It can be seen that the LAGB varies significantly from one strain rate to another. For example, at the lowest strain rate (0.0005 s^{-1}), there is only a small number of LAGBs, and they have large sizes, dividing large grains into smaller ones or similar sizes to those small equiaxial grains. As the strain rate increased to 0.005 s^{-1} , a relatively homogeneous LAGB distribution is found. The morphology of LAGB-formed subgrains is similar to that of the recrystallised grains. It should be mentioned that all grains, regardless of their orientations, are occupied with a large number of LAGBs or subgrains.

However, this trend changed when increasing the strain rate from 0.05 to 5 s⁻¹, where the LAGBs became more intensive and the subgrain sizes became very fine in some grains, while other grains were almost LAGB-free. These findings confirm that at high strain rate, the development of LAGB is grain-orientation-dependent, which is supported by the high-resolution EBSD GND density measurement in deformed copper [130] and the TEM characterisation on hot deformed single crystal aluminium with different orientations [67] in that different grain orientations exhibit a different level of dislocation density and LAGBs. It is unnecessary to have similar contents of dislocation density and LAGBs in all grains, since some of the grains were less likely to be deformed than others; the neighbouring grains could accommodate more strains and ‘shield’ some particular grains.

Also, the current data show that this LAGB orientation dependence trend is more considerably under high strain rate and diminishes under low strain rate. This implies that the deformation coordination between grains is considerably better at a low strain rate, in which sufficient time allows the deformation to be accommodated roughly equally in all grains and leads to homogenous LAGB distribution (*e.g.* 0.005 s⁻¹). These high strain rate LAGB maps raise the reliability question as to the previous TEM subgrain size measurement in hot deformed polycrystalline materials, as the LAGB variation is significant from grain to grain.

Regarding the LAGB misorientation statistical distribution, the histograms show that the general LAGB misorientation distribution remains the same for all strain rates. The highest population of LAGB data has the smallest LAGB misorientation angle (2°). The number declines towards higher misorientation, though the total number of LAGBs in the maps increased with increasing strain rate.

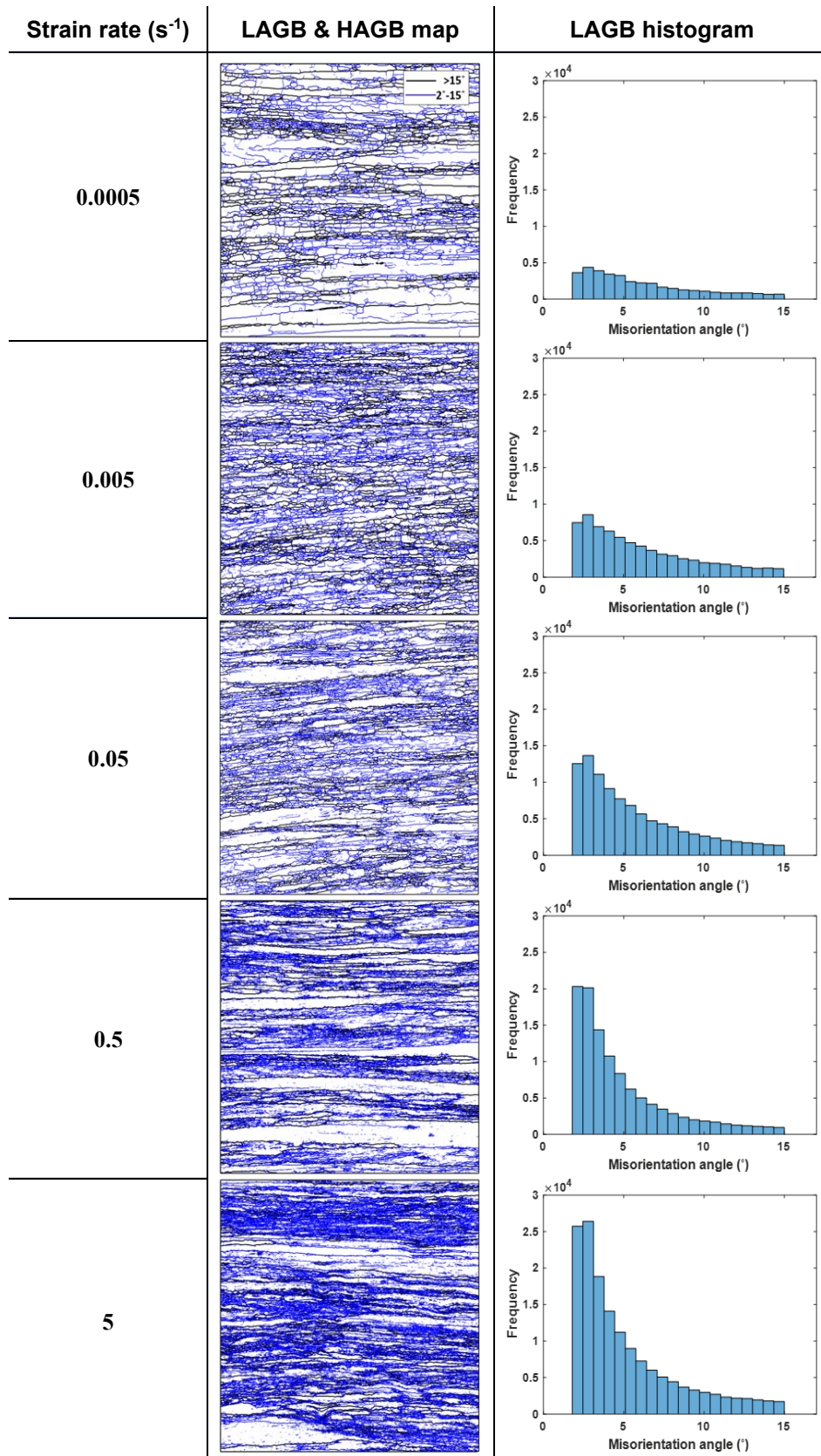


Fig. 5. 6 The evolution of length of LAGB and its statistical distribution as a function of increasing strain rate at the constant testing temperature of 420 °C. The first column shows the strain rate; the second column shows the high- and low-angle grain boundaries, showing in black and blue lines respectively; the third column shows the histograms of LAGB.

The relationships between LAGB number, misorientation angle with strain rate and flow stress are shown in Fig. 5.7. Clear trends are found showing that higher strain rate results in a larger LAGB area per unit volume, and the LAGB area per unit volume displays a positive correlation to the flow stress, meaning that more LAGBs exist at a higher stress state. These findings are consistent with the strain rate effects on dislocation density. Furthermore, the misorientation angle shows a gradual decrease with increasing strain rate, which could be explained by the LAGB misorientation histograms shown in Fig. 5.6, in that there is an increasing number of LAGBs at the peak misorientation (around 2°). This slightly lowers the mean misorientation values, as seen in Fig. 5.7(b).

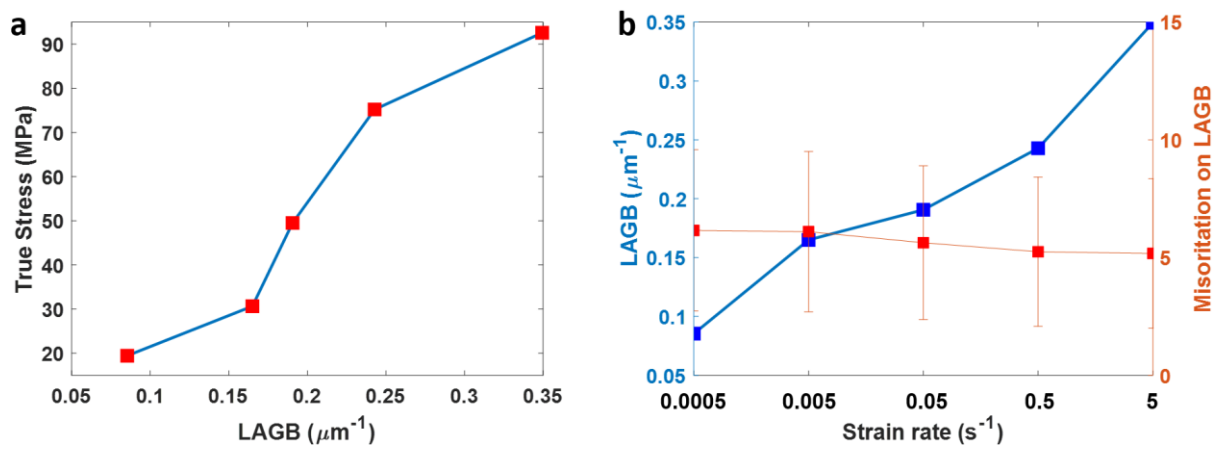


Fig. 5. 7 (a) The flow stress as a function of the length of LAGB; (b) the length of LAGB, the average and standard deviation of misorientation on LAGB as a function of strain rate at the constant temperature of 420°C .

The temperature effects showed opposite trends to the strain rate effects, as displayed in Fig. 5.8. As the temperature decreased from 460°C to 380°C , the LAGB evolved into a more intensive and grain-orientation-dependent inhomogeneous distribution. Under the high deformation temperature of $460\text{--}440^\circ\text{C}$, the LAGBs were relatively homogeneously distributed within all grains. From the LAGB and HAGB maps, we can see that the subgrain sizes are comparable to the recrystallised grains. As the temperature is lowered, the subgrains are finer and only emerge in certain grains. For example, at 380°C , the highest contrast of highly intense LAGB grains and LAGB-free grains was found. Again, like the strain rate effects, the general LAGB misorientation angle shows similar distribution among the samples tested in all temperatures. The number of LAGB points increased with decreasing temperature and accumulates at the minimum LAGB misorientation (2°).

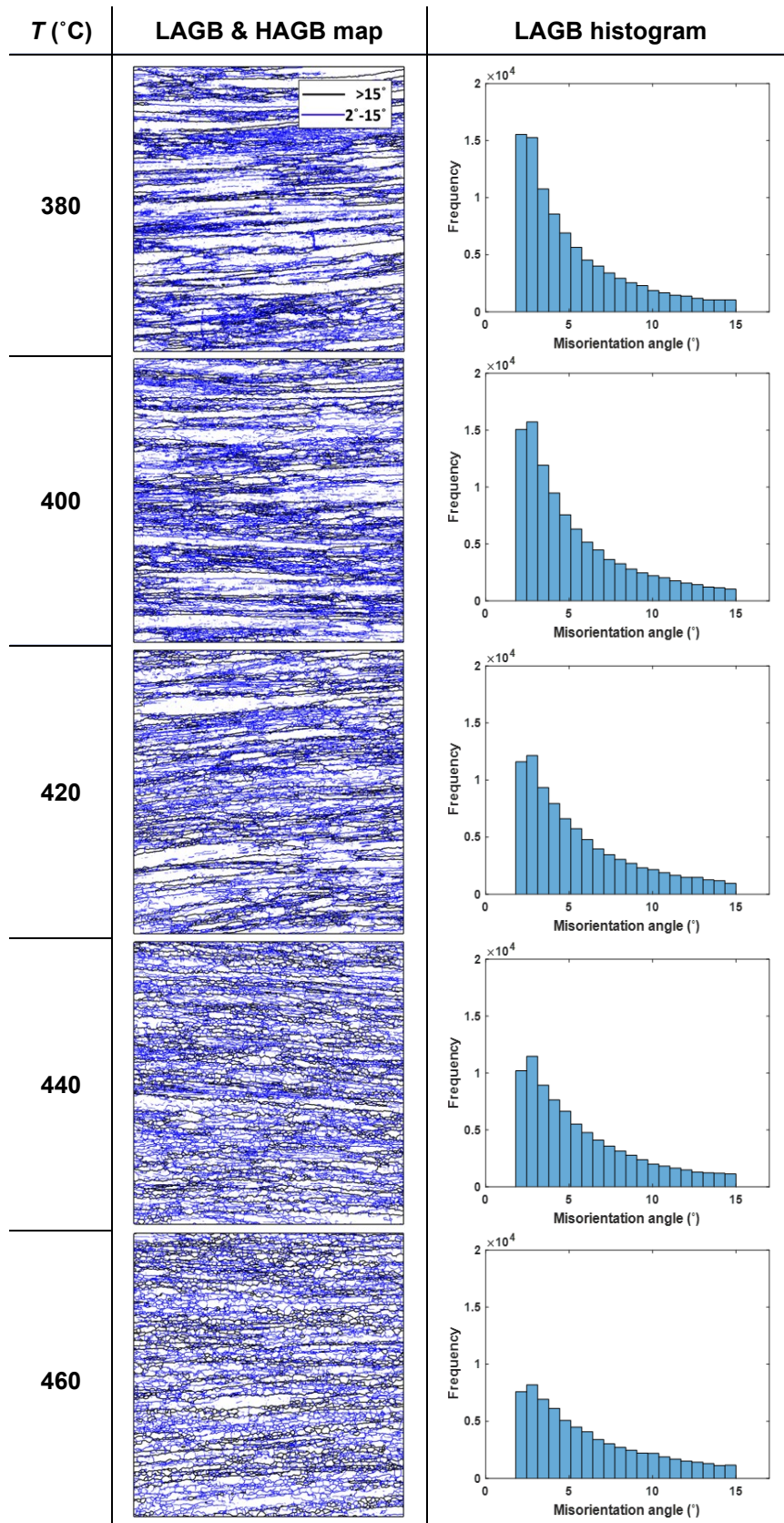


Fig. 5. 8 The evolution of length of LAGB and its statistical distribution as a function of increasing temperature under the constant strain rate of 0.05 s^{-1} : The first column shows the temperature; the second column shows the high- and low-angle grain boundaries, showing in black and blue lines respectively; the third column shows the histograms of LAGB.

The relationships of LAGB area per unit volume and misorientation distribution with testing temperature and flow stress are presented in Fig. 5.9. The temperature effects on LAGB are more consistent than the previous dislocation density, in that lower deformation temperature results in a larger LAGB area and a slight reduction in average misorientation angle. The LAGB area and flow stress, again, show a positive correlation. The findings suggest that as the testing temperature decreases, there is less thermal energy to facilitate dislocation recovery. Hence, more dislocation could be stored within the grains and transformed into LAGBs. The LAGB plays the hardening role to increase the required flow stress for further deformation. It should be noted that although the samples of the 420, 440 and 460 °C exhibit a fluctuation in GND density, the trend in LAGB is very clear in that as the temperature increases, there are fewer LAGBs.

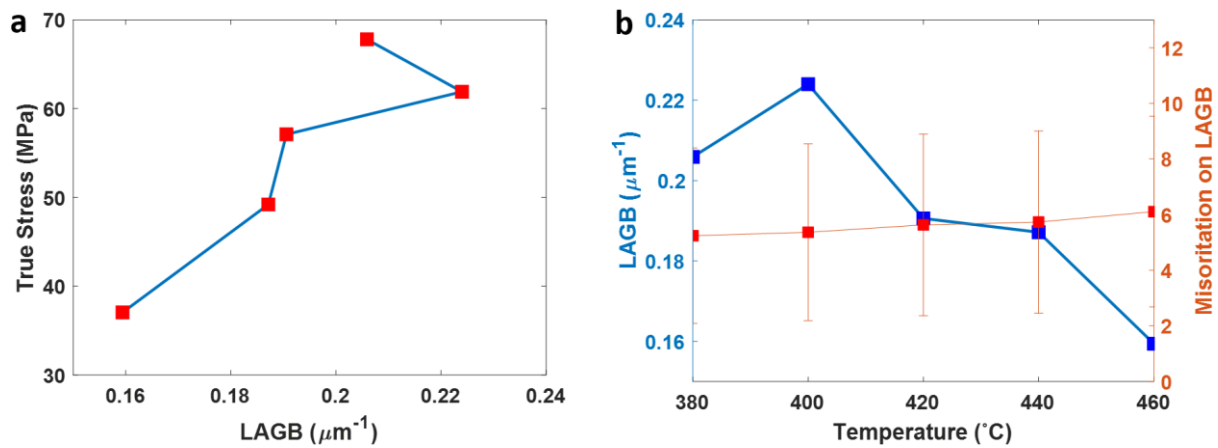


Fig. 5. 9 (a) The flow stress as a function of the length of LAGB; (b) the length of LAGB, the average and standard deviation of misorientation on LAGB as a function of temperature under the constant strain rate of 0.05 s^{-1} .

5.3.4 Effects on HAGB

The effects of strain rates and temperature on HAGBs during hot compression are presented in this section. It should be clarified that since all samples were hot compressed to the same final strain (~ 0.58), unlike the plastic strain effects discussed in the previous chapter, the geometrical effects on grain size variation were identical for all samples in this section. Any variation in HAGB or grain size was due to dynamic recrystallisation. The strain rate effect on HAGB is shown in Fig. 5.10. Unlike the GND density and LAGB, the HAGB does not exhibit a simple trend with an increasing strain rate. At the lowest strain rate of 0.0005 s^{-1} , there were still a few large, elongated grains and smaller grains formed adjacent to these elongated grain boundaries. Some smaller grains were also formed within the grain interior. The HAGB distribution does not vary markedly from the initial undeformed one, which could

be attributed to the attainment of steady-state, reaching the equilibrium between the dislocation generation and annihilation. However, there is a considerable increase in HAGB at the strain rate of 0.005 s^{-1} , the sizes of grain are more homogeneous. All HAGBs are serrate, probably due to the non-uniform pinning affected by the secondary particles. Nevertheless, these serrate HAGBs do imply that the HAGBs are moving or attempting to migrate. Further increase in strain rate to 0.5 s^{-1} does not lead to more HAGBs or more homogenous distribution. On the contrary, there were fewer HAGBs. The general size distribution, as well as its misorientation distribution, is similar to the lowest strain rate, 0.0005 s^{-1} , as shown in the HAGB histograms in Fig. 5.10. However, this trend did not continue to the highest strain rate (5 s^{-1}), which showed the largest HAGB. These HAGBs were very distinct from other strain rates. The recrystallised grains were considerably smaller, and they concentrated in certain grains, while other grains were almost free from recrystallised grain. Also, these HAGBs were smoother than the ones at lower strain rates, which means that these boundaries were unable to migrate due to the limited deformation time. Meanwhile, due to the limited time and suppressed dynamic recovery, the higher stored dislocation density and more LAGBs allowed more HAGBs to be formed. However, the distribution of these recrystallised HAGBs was very heterogeneous, and only concentrated in certain grains, due to the poor deformation coordination among grains at a high strain rate.

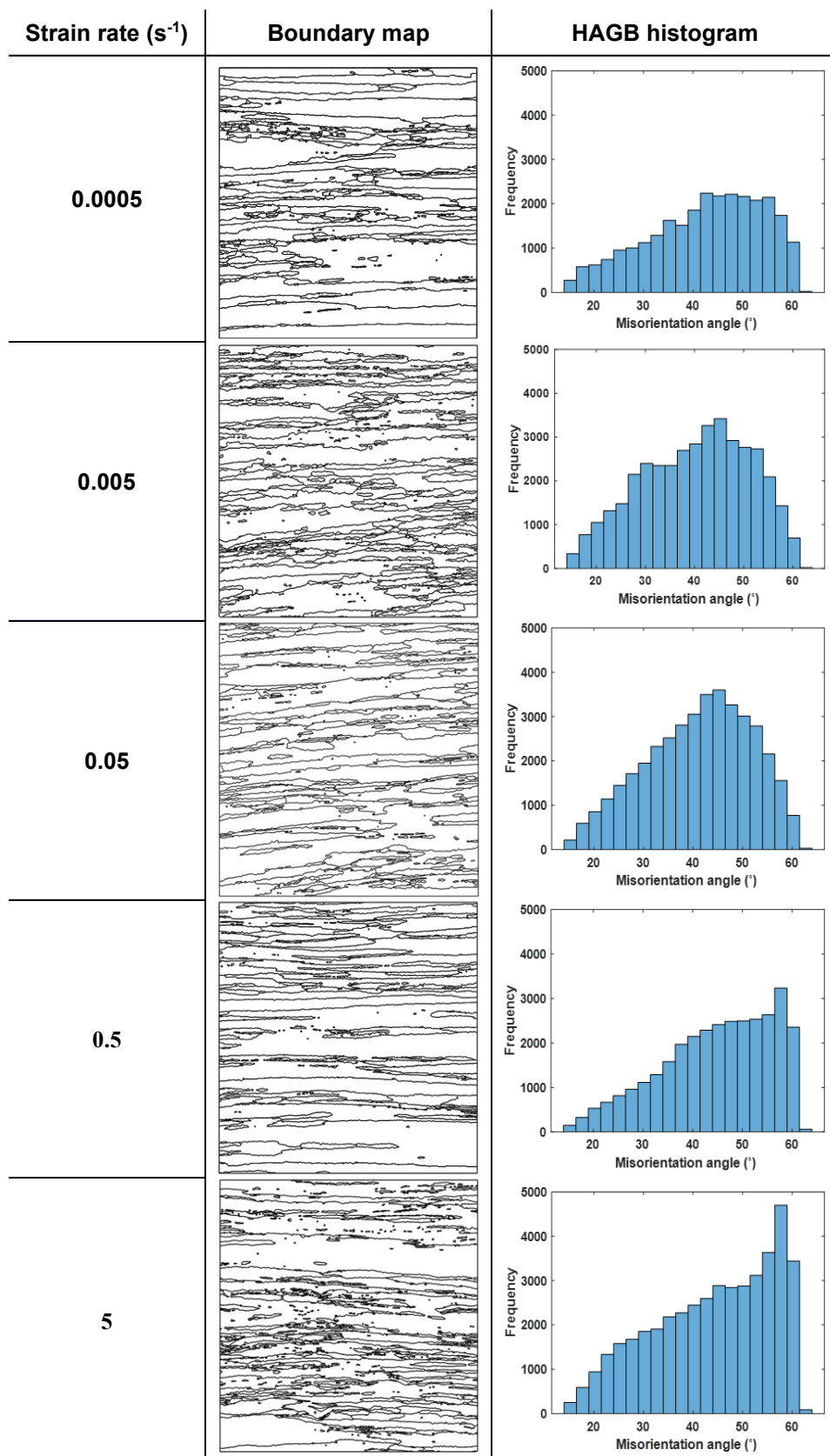


Fig. 5. 10 The evolution of length of HAGB and its statistical distribution as a function of increasing strain rate at the constant temperature of 420 °C. The first column shows the strain rate; the second column shows the high-angle grain boundaries, showing in black lines; the third column shows the histograms of HAGB.

The as-observed general trends in Fig. 5.10 are confirmed by the statistical analysis shown in Fig. 5.11. As shown in Fig. 5.11(a), an S-shaped line plot is found between the flow stress and HAGB areas. Combining with the strain rate and HAGB areas in Fig. 5.11(b), the relationship could be drawn that the largest HAGB area is formed under the highest strain rate condition, while the smallest HAGB area exists under the lowest strain rate. The overall trend means that the HAGB area increases with an increasing strain rate. However, within the strain rate range of $0.005\text{--}0.5\text{ s}^{-1}$, a reversed trend emerged showing that more HAGBs formed with a decreasing strain rate.

These findings may not be entirely unreasonable, if one considers the two essential conditions for new grains to be formed during CDRX. The first one is the stored energy (dislocation density or LAGBs) in the material. Under the same amount of deformation strain, the higher strain rate would have more stored energy, *e.g.* higher dislocation density and more LAGBs, in the material. The second essential condition is the sufficient time for LAGBs to be transformed into HAGBs through gradual rotation.

The lower strain rate would require extra time to allow the LAGBs to be transformed into HAGBs. These two essential conditions are conflicting since a longer deformation time will reduce the stored energy through DRV. Therefore, there must be an optimal point at which both stored energy and time are sufficient to achieve the maximum CDRX and hence the largest HAGB area, *i.e.* the finest grain size. In the present study, the deformation condition 0.005 s^{-1} seems to be the optimal point with the finest and most homogenous dynamic recrystallised grain structure within the range of $0.0005\text{--}0.5\text{ s}^{-1}$. Lower than 0.005 s^{-1} , the low dislocation density and LAGB area prohibit HAGB formation. Once the material has stored sufficient energy, exceeding the critical value, the longer deformation time would result in a more complete CDRX. This explains why the HAGB increases with a reduced strain rate from $0.5\text{--}0.005\text{ s}^{-1}$.

This CDRX could not rationalise the largest HAGB area observed at the highest strain rate (5 s^{-1}). It is likely due to the presence of a different dynamic recrystallisation mechanism: discontinuous dynamic recrystallisation (DDRX) with little diffusion involved. Although the boundary between CDRX and DDRX is ambiguous, it is possible that new grains can be formed instantaneously from points containing high dislocation density without gradually increasing the misorientation angle, as the recrystallised grains formed in low stacking fault energy materials such as steels [139]. This indicates that there could be another critical stored

energy, above which the grains can be formed instantaneously. These agree with the recent review on dynamic recrystallisation in aluminium alloys that different dynamic recrystallisation mechanisms have been observed under various strain rates and temperatures in aluminium alloys [21].

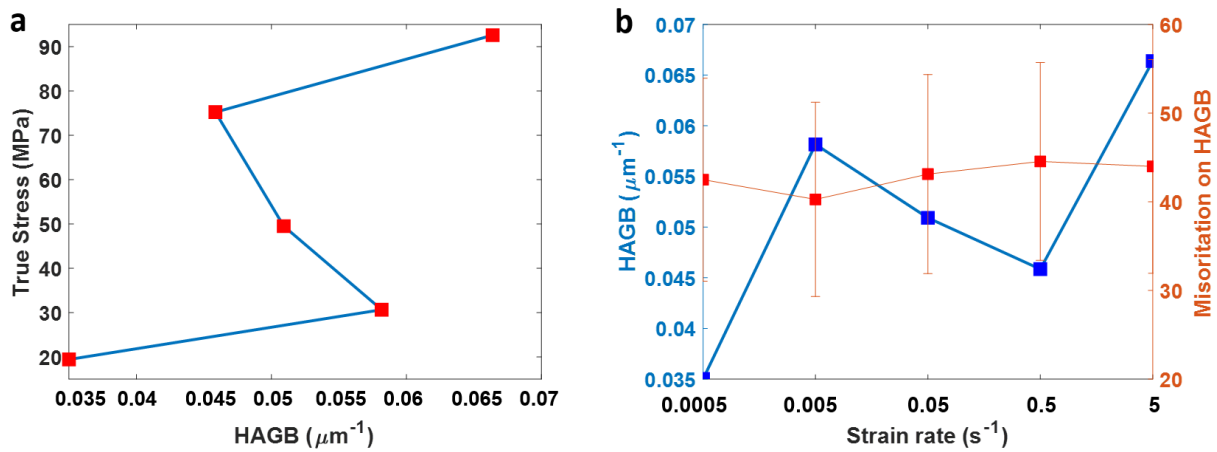


Fig. 5. 11 (a) The flow stress as a function of the length of HAGB; (b) the length of HAGB, the average and standard deviation of misorientation on HAGB as a function of strain rate at the constant temperature of 420 °C.

The qualitative and statistical misorientation analyses of temperature effect on HAGB evolution are presented in Fig. 5.12. When increasing temperature from 380 to 460 °C, two clear trends in HAGB distribution could be observed. The first is that the HAGB area increased. This is mainly due to the enlarged sizes of the newly formed grains with increasing temperature.

The second is that the HAGB morphology was transforming from the smooth boundaries found at 380 °C to wavy and serrate boundaries, as seen at 460 °C. The misorientation angle was shifting from 60° peak to 45°. These findings indicate that the HAGBs are unable to migrate at low temperatures, resulting in smooth morphology. Some very fine recrystallised grains could be found scattered around both grain boundaries and the interior. With increasing temperature, *i.e.* thermal energy, under the same strain rate and strain, the HAGBs have sufficient energy to migrate. However, probably due to the secondary particles, some points of the HAGBs are pinned, resulting in serrate morphology. On the other hand, the fine recrystallised grains are able to grow into larger ones.

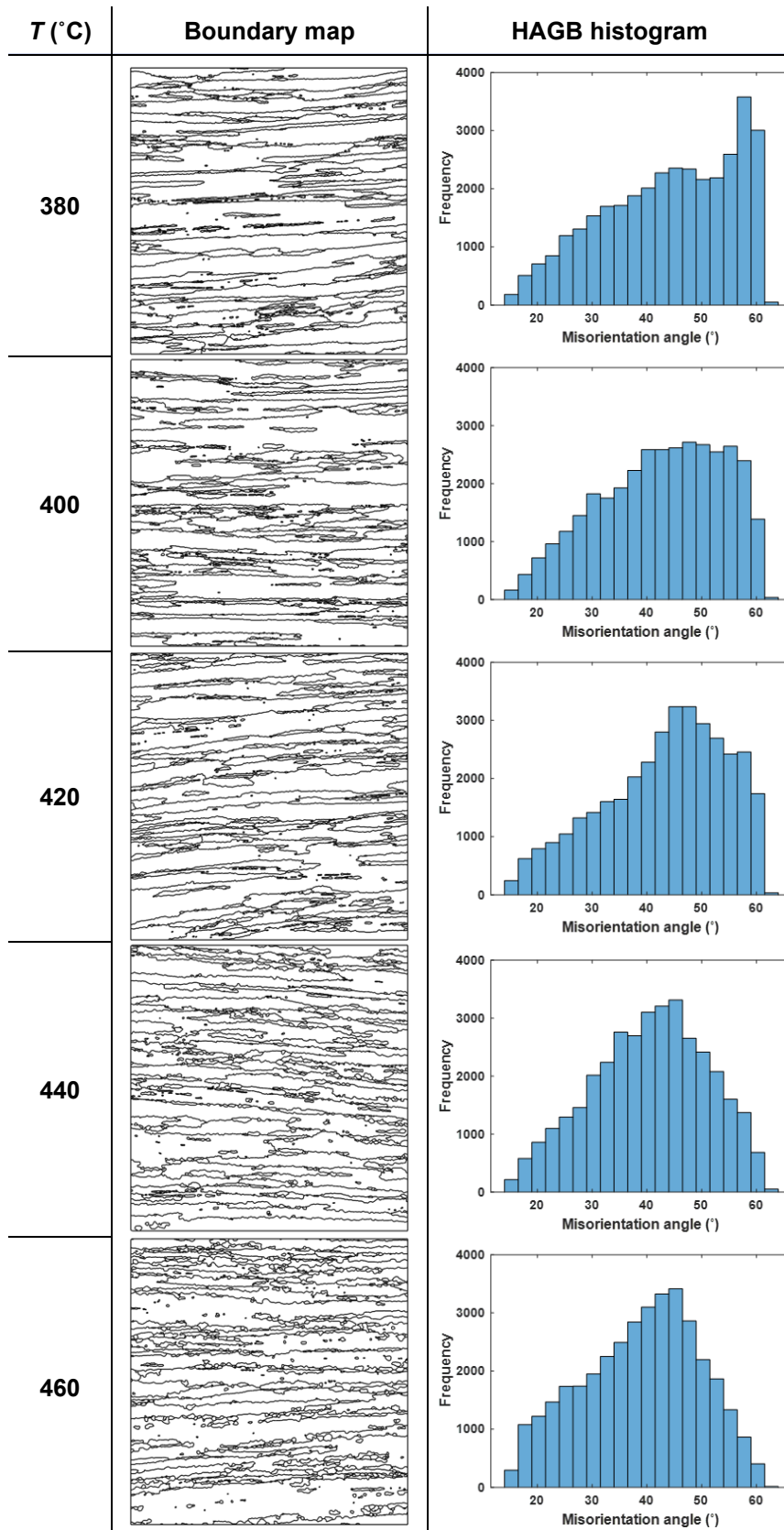


Fig. 5. 12 The evolution of length of HAGB and its statistical distribution as a function of increasing temperature at the constant strain rate of 0.05 s^{-1} . The first column shows the temperature; the second column shows the high-angle grain boundaries, showing in black lines; the third column shows the histograms of HAGB.

The quantitative analyses of the association of stress with HAGB and its relationship with temperature are reported in Fig. 5.13. The trends are very clear, showing that as the temperature increases, there is a larger HAGB area per unit volume. This increasing HAGB area shows a strong negative correlation with flow stress. It means that the flow stress decreases with increasing HAGB area, *i.e.* decreasing grain size. Note this is in contrast with the Hall-Petch relation at room temperature.

Combining the previous dislocation density and LAGB analyses on temperature effect as described in the previous Chapters 4.2 and 4.3 that the dislocation density and LAGB decreased with increasing temperature – the relationship with stress could be interpreted that as the temperature increases, there are less dislocation density and LAGB due to the effective recovery process. However, the HAGB increased due to grain refinement. Therefore, it would be the combining effects of a reduced number of dislocation barriers, *i.e.* dislocation network and LAGBs, the activation of grain boundary migration/sliding and increasing HAGBs, that results in the lowered flow stress.

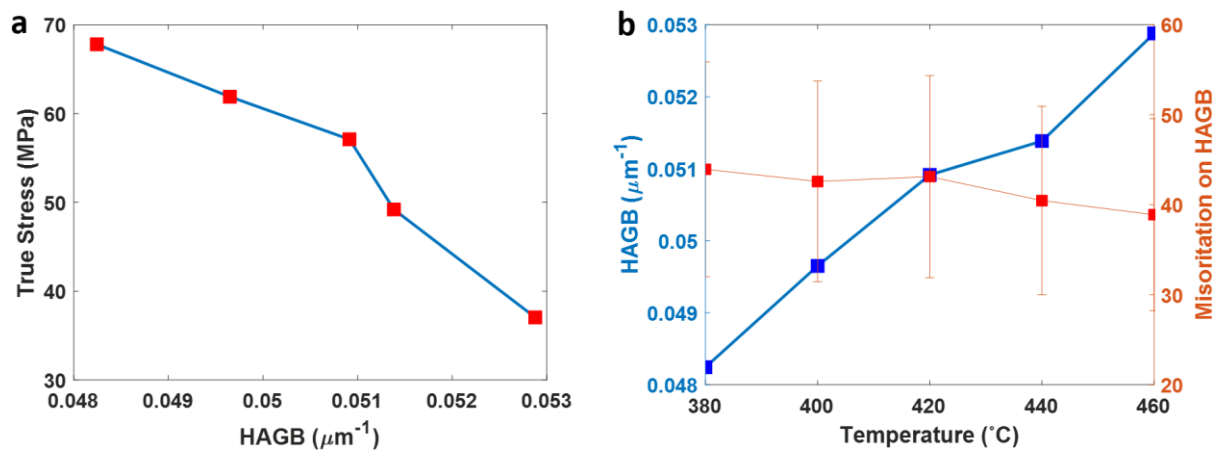


Fig. 5. 13 (a) The flow stress as a function of the length of HAGB; (b) the length of HAGB, the average and standard deviation of misorientation on HAGB as a function of temperature at the constant strain rate of 0.05 s^{-1} .

5.3.5 Z parameter effects on dislocation density, LAGB and HAGB

Based on the data and discussion presented in the previous sections, one can see that the effects of strain rate and temperature on flow stress, dislocation density, LAGB and HAGB are very complex, especially the strain rate effect on HAGB. Since both the strain rate and temperature affect the diffusion kinetics through the duration and thermal energy, and all viscoplastic mechanisms are fundamentally associated with the diffusion process, it is convenient to combine the strain rate and temperature into a single parameter, the Zener-

Hollomon (Z) parameter, to account for the diffusion process. The Zener-Hollomon parameter was described in the literature review chapter in detail. Equation 1 is used to calculate the Z value in this study, where the activation energy Q is taken as 160.3 K/mol (close to the bulk diffusion activate energy in aluminium) [46-47]. Further in-depth analyses are undertaken and shown in Fig. 5.14.

The flow stress and $\ln(Z)$ show an approximately linear relationship, where the flow stress increases with increasing $\ln(Z)$ value (higher strain rate and lower temperature), as seen in Fig. 5.14(a), which is consistent with the previous studies of aluminium alloys [140-141].

As displayed in Fig. 5.14(b-c), it is very interesting to see the strong correlations between dislocation density and LAGB area per unit volume as a function of $\ln(Z)$. As the $\ln(Z)$ value increased, the dislocation density and LAGBs exhibited virtually linear relationships, even though there were some fluctuations in dislocation density. The general relationships clearly show that the dislocation density and LAGB rise with increasing Z . This is less surprising if we consider that as Z increases, the diffusion-driven processes, *e.g.* dislocation annihilation and recovery through cross-slip and jump, or grain boundary migration, are suppressed.

Hence, there should be higher dislocation density and more LAGBs stored in the material. However, the near-linear relationship no longer exists between HAGB and Z , as shown in Fig. 5.14(d). An S-shaped relationship emerges. This is consistent with the HAGB relationship with strain rate in Fig. 5.11; also, it is interesting to see that the temperature effect on HAGB (Fig. 5.13(b)) fits well into the middle linear part of the general S-shaped trend.

Examining the overall stress, dislocation density, LAGB and HAGB relationships with $\ln(Z)$, the governing mechanism for viscoplastic flow stress could be determined as the stored dislocations either in the form of GND or LAGB depending on the resulting misorientation angle. This means that the GND density or LAGB area per unit volume could act as the sole indicator for flow stress. Within the studied $\ln(Z)$ range, the direct HAGB effects, *e.g.* grain boundary hardening or sliding (softening), on flow stress could be neglected. Nevertheless, the HAGB effect on dislocation density, *e.g.* grain boundary growth resulting in dislocation density reduction, should be considered. This finding provides very helpful insight into the development of constitutive equations, determining the contribution of dislocation density and grain boundary on the flow stress, for which it has been very ambiguous or sometimes

arbitrary [142], because of the mixed active underlying mechanisms, and unidentified governing mechanism. The near-linear relationship between LAGB and flow stress is also found in a previous study by Huang and Humphrey [143] on hot deformed pure aluminium. However, the effects of HAGB on flow stress are overlooked, and hence, the governing mechanism of flow stress remains unexplored.

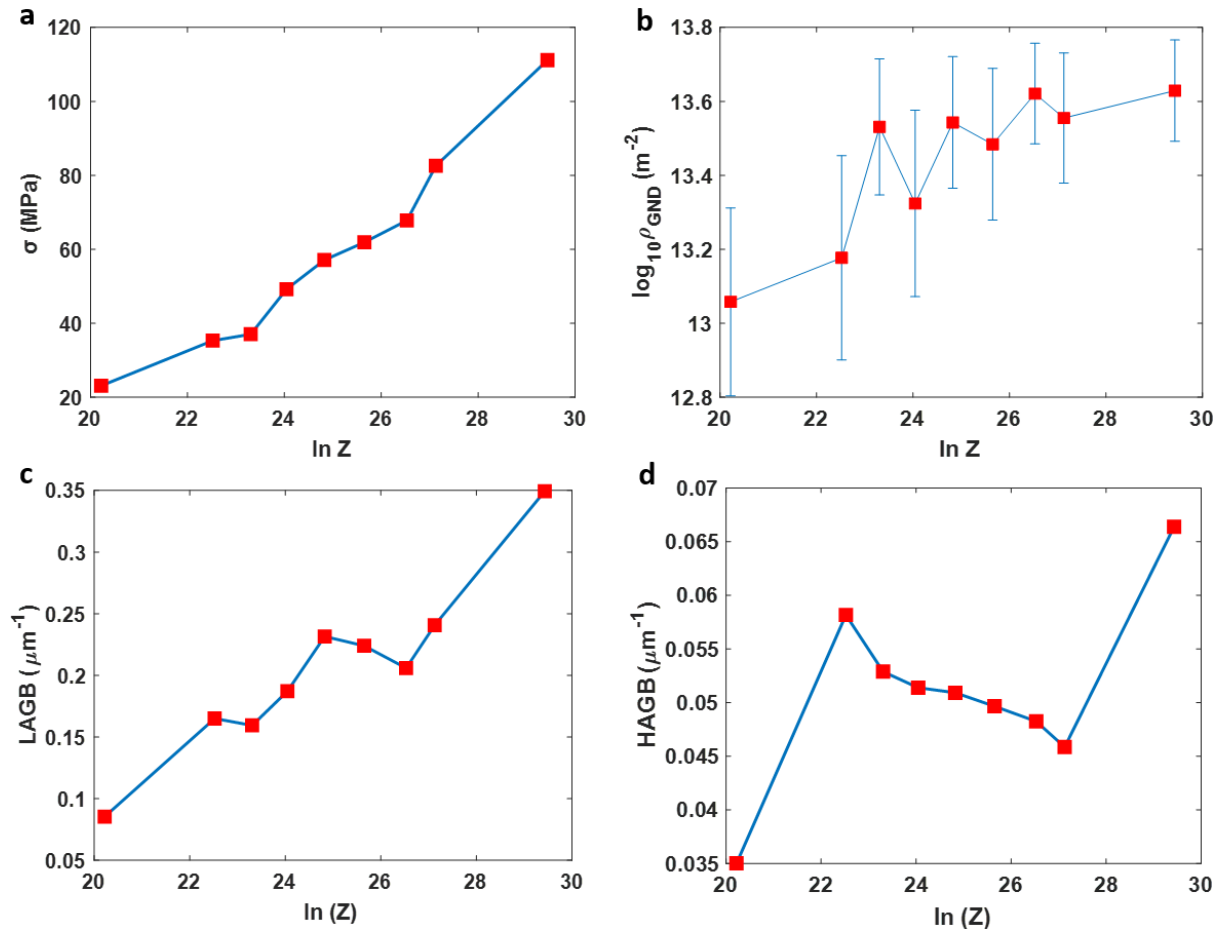


Fig. 5. 14 The relationships of (a) Stress; (b) GND density; (c) LAGB area per unit volume and (d) HAGB area per unit volume with Zener-Hollomon parameter, Z , at $\ln$ scale.

5.3.6 Strain rate and temperature (Z) effect on dynamic recrystallisation

Considering the GND density and LAGB evolution and the strain rate and temperature (as expressed by $\ln(Z)$) effects on HAGB, a general picture of underlying dynamic recrystallisation mechanisms emerges. As schematically illustrated in Fig. 5.15, the relationship between HAGB and $\ln(Z)$ comprises three parts. Firstly, when the $\ln(Z)$ value is low, *i.e.* high temperature and low strain rate, the diffusion process is very active, that the associated mechanisms like dislocation jump, cross-slip, annihilation, *i.e.* DRV, and grain boundary sliding are very active. As a result, the stored energy (dislocation density and

LAGB) in the majority of grains is too low to initiate dynamic recrystallisation, and the HAGB area remains constant as received one.

However, if the $\ln(Z)$ value further decreases, which is beyond the typical practical forging conditions as highlighted in the yellow shaded area in Fig. 5.15, the original HAGB will migrate to form coarser grains. Thus, the HAGB area per unit volume is speculated to be further reduced, as illustrated by the dashed line on the left-hand side of the diagram in Fig. 5.15. On the other hand, when $\ln(Z)$ increases, *i.e.* lower deformation temperature and higher strain rate, the diffusion process driven by DRV will be suppressed, and more dislocations will be stored in the material to form dislocation boundaries and LAGBs. These LAGBs will be gradually transformed into HAGBs, driven by an active diffusion process.

Hence, the HAGB area per unit volume increases and reaches a peak value, in which relatively homogenous grain size could be achieved. Further increasing of $\ln(Z)$ will further suppress the diffusion process. Although more dislocations will be stored in the materials, there will be ample of LAGBs. The limited diffusion process, however, will not allow LAGBs to be transformed into HAGBs and will not allow the HAGBs' growth. The number and size of recrystallised grains will be very small to considerably affect the value of the HAGB area. Hence, within this region, after passing the 'sweat point' of $\ln(Z)$ (~ 21.5 in this study), the HAGB area decreases with increasing $\ln(Z)$. This region is where the typical hot forging conditions are and could explain why, in previous studies of the dynamic recrystallisation in aluminium alloys [46, 67, 144-145], HAGB area per unit volume /grain size positively correlates with $\ln(Z)$ in some cases, while the trend reversed in others.

However, this HAGB decreasing trend with $\ln(Z)$ does not continue with further increase in $\ln(Z)$ value, mainly due to the dynamic recrystallisation mechanism transition from CDRX to DDRX. The HAGB area per unit volume increases with increasing $\ln(Z)$ within this region. While there is little time for DRV to take place, resulting in high dislocation density accumulated in the material, the temperature is still higher than the critical recrystallisation temperature to nucleate recrystallised grains. As evidenced in the present study, there is a large number of recrystallised grains formed in the material at 5 s^{-1} and 420°C and their sizes are very small as there is not sufficient time and thermal energy for them to grow.

Also, the recrystallised grains have high orientation dependence, *i.e.* some grains have a high intensity of recrystallised fine grains, while some grains are free from recrystallised grains. It

is speculated that in this DDRX region, a peak value of HAGB area per unit volume will be reached as the $\ln(Z)$ increases, followed by a HAGB drop with a further increase of $\ln(Z)$, since it will gradually reduce the thermal energy and fewer recrystallised grains will be formed. Eventually, the deformation process will become plastic rather than viscoplastic, since the diffusion process will be neglectable. Therefore, the HAGB area per unit volume will be constant and insensitive to the change of Z values, *i.e.* room temperature plastic deformation.

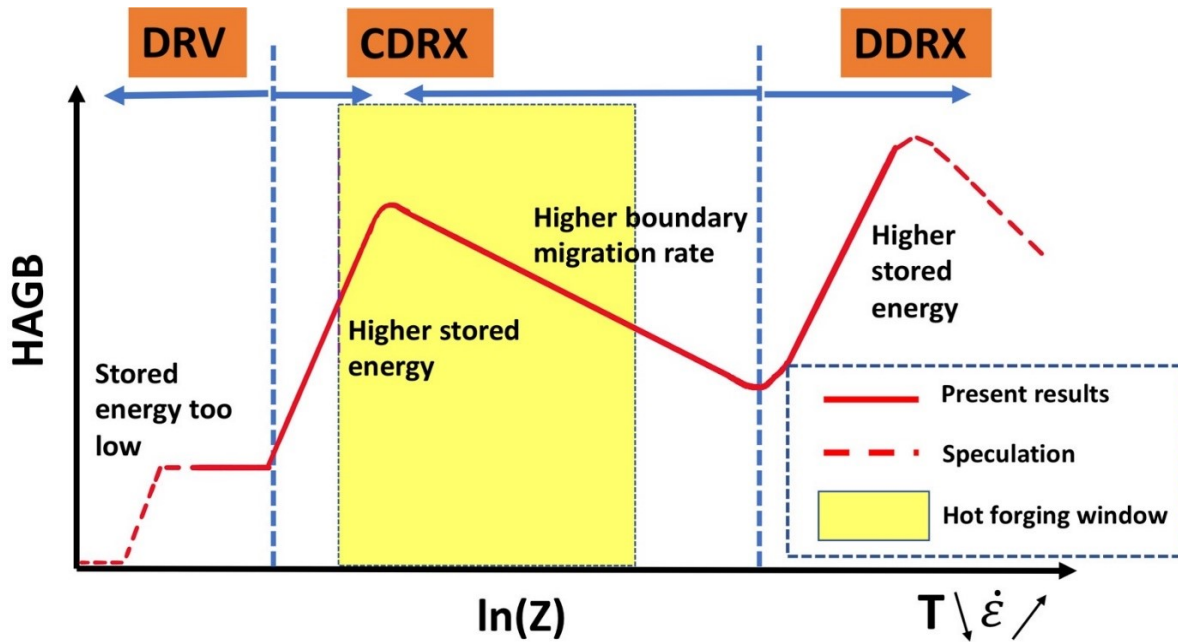


Fig. 5. 15 The schematic diagram showing the dynamic recrystallisation mechanisms as a function of $\ln(Z)$. Z is the Zener-Hollomon parameter.

Three more hot compression tests were conducted to validate the mechanisms proposed above, as shown in Fig. 5. 16. The lower $\ln(Z)$ is 19.441 at 440 °C and 0.0005 s⁻¹, the HAGB value is 0.0350 μm⁻¹ per unit volume is nearly the same as the $\ln(Z)$ value of 20.22 at 420 °C and 0.0005 s⁻¹, which explains the dynamic recovery process dominated with lower $\ln(Z)$ values.

As the recrystallisation mechanics shifted from CDRX to DDRX, with the $\ln(Z)$ values (28.833) increasing at 380 °C and 0.5 s⁻¹, HAGB decreased to 0.0305, which agrees with the concept mentioned before. After this, HAGB increases due to nucleation in DDRX and reach a peak. With further increasing of the $\ln(Z)$ values (31.136) at 380 °C and 5 s⁻¹, the HAGB dropped again to 0.0443, which means that less DDRX occurs at low temperature and high strain rate.

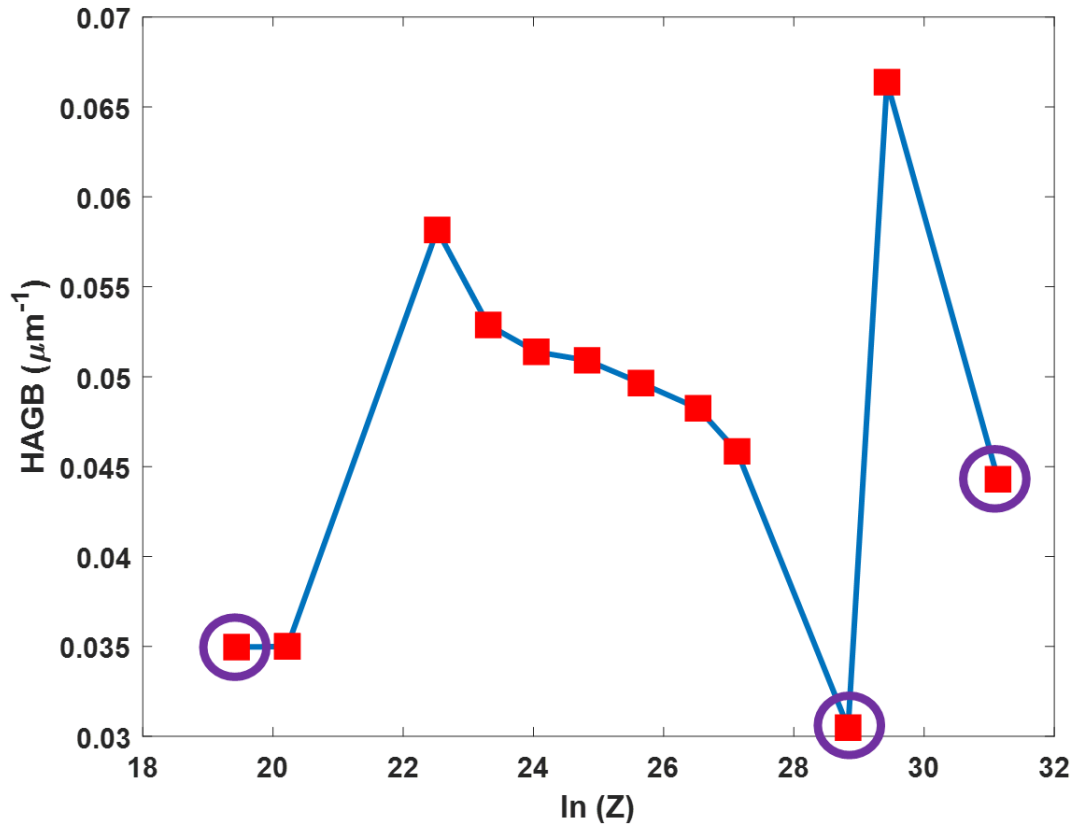


Fig. 5. 16 Three more experiment points for the HAGB area per unit volume with Zener-Hollomon parameter.

It should be mentioned that the proposed overall dynamic recrystallisation mechanism is mainly based on the obtained data in the current study of AA7050, devoting to the hot forging window, which would be adequate to facilitate the model development and hot forging process optimisation. However, generalising the proposed dynamic recrystallisation mechanism to all aluminium alloys under the entire viscoplastic range is beyond the scope of the current study.

Also, comparing the absolute values of the Z and HAGB/grain size from various studies would be unlikely to lead to a strong correlation, because the values of Z and HAGBs also depend on both the initial microstructure state, *e.g.* dislocation density, LAGB and HAGB, and plastic strain levels. The present study is unable to examine the effects of the initial microstructure state and plastic deformation history on the value of Z and HAGB. Further research on these effects would be encouraged. Nevertheless, the general trends of HAGB with $\ln(Z)$ would be expected to be consistent with the current study. In addition, more research on the secondary particle effect on dynamic recrystallisation should be undertaken in future studies.

5.4 Summary

The effect of strain rate and deformation temperature on dynamic recrystallisation has been studied in this chapter. Hot compression tests were conducted using the Gleeble 3800 thermal-mechanical simulator to the strain level of 0.58, at the strain range of 0.0005 to 5 s⁻¹ and deformation temperature range of 380 to 460 °C. In-depth qualitative and quantitative analyses have been undertaken on the GND density, LAGB and HAGB. The relationships between the flow stress behaviour, strain rate and temperature, and underlying microstructure evolution have been determined, and the overall underlying dynamic recrystallisation mechanism has been proposed, based on the relationship between GND density, LAGB and HAGB, using the Zener-Hollomon parameter. The following conclusions can be drawn:

- i. The GND density, LAGB and flow stress exhibit near-linear relationships with the Zener-Hollomon parameter. High dislocation density and more LAGBs are formed at lower temperature and higher strain rate.
- ii. The dislocations in GND density and the LAGB area are found to be the dominating factor, governing the flow stress in the tested ranges of strain rates and temperatures, regardless of the change of HAGBs.
- iii. The relationship between HAGB area per unit volume and the Zener-Hollomon parameter is complex, comprising three regions: namely DRV, CDRX and DDRX. The boundary values of $\ln(Z)$ for AA7050 in this study are less than 20 (0.0005 s⁻¹, 420 °C) for DRV, 20-27.5 for CDRX, and larger than 27.5 (0.5 s⁻¹, 420 °C) for DDRX.
- iv. The largest HAGB area per unit volume within the CDRX region is found at the $\ln(Z)$ value of 22.5 (0.05 s⁻¹, 420 °C), where relatively homogenous grain structure is observed due to the balance of the grain boundary growth and the transformation from LAGBs.
- v. The largest HAGB area per unit volume within the DDRX region is found at the $\ln(Z)$ value of 29.5 (5 s⁻¹, 420 °C), where a large number of fine recrystallised grains are found within grains with certain orientations.

Chapter 6

Development of a physically-based constitutive model

6.1 Introduction

This chapter focuses on developing a set of physically-based, experimentally verifiable constitutive equations to model the viscoplastic behaviour and underlying microstructure evolution in AA7050 under typical hot forging conditions. The insights of dislocation density, low angle grain boundaries (LAGB) and high angle grain boundaries (HAGB) evolution, obtained from the previous experimental chapters, will be embedded in the model development. Nevertheless, the main purpose of this model is more engineering-focused, capturing the macroscopic flow stress-strain and microstructure evolution, which has a pronounced influence on optimising the forming process and the mechanical properties of formed parts.

Many smaller-scale mechanisms, such as the discrete dislocation dynamics, will be generalised in this model. Furthermore, the experimental findings of dislocation density, LAGB and HAGB evolution in the current study form the bases of the model equations. Although similar mechanisms are expected to exist in other aluminium or high stack fault energy metals and the proposed model is likely to work well in them, validating the model in other materials or other processing conditions, *e.g.* severe plastic deformation, is beyond the scope of the current study. Furthermore, based on the early experimental studies in this thesis, the HAGB transformation only involves the frequent pinch-off from the LAGBs. No LAGB rotation, grain boundary migration or grain growth has been observed, and hence these processes will not be accounted for in the model.

This chapter comprises three main sections. The first section presents the constitutive model development with an explanation, premises, assumptions, and their justifications. The second section reports the process of material constants determination. Finally, the model prediction

results, their comparison with experimental data, and the comparison with other existing models will be presented and discussed in the third section.

6.2 Constitutive model development

6.2.1 The underlying physics of viscoplasticity

Although the underlying mechanisms in viscoplasticity are complex, these temperature and strain rate dependent mechanisms are fundamentally governed by a single physical process: diffusion. The relationship between diffusion and viscoplasticity is schematically illustrated in Fig. 6.1. At the atomic scale, the change in strain rate and temperature influences the diffusion process among atoms. For example, if the strain rate is low and the temperature is high, the atoms have a longer time and more thermal energy (or kinetic energy) to migrate to other places through various paths such as lattice vacancies in lattice, dislocation cores, or grain boundaries. Consequently, the diffusion process drives the microstructure rearrangement, namely, dislocation annihilations through bulk or dislocation diffusion [146], LAGB to HAGB transformation through dislocation diffusion [147], and HAGB sliding by grain boundary diffusion [148]. These three mechanisms together influence the flow stress, leading to plastic deformation. Therefore, the development of the constitutive model will be primarily based on the proposed diffusion-driven or dislocation movement framework in Fig. 6.1.

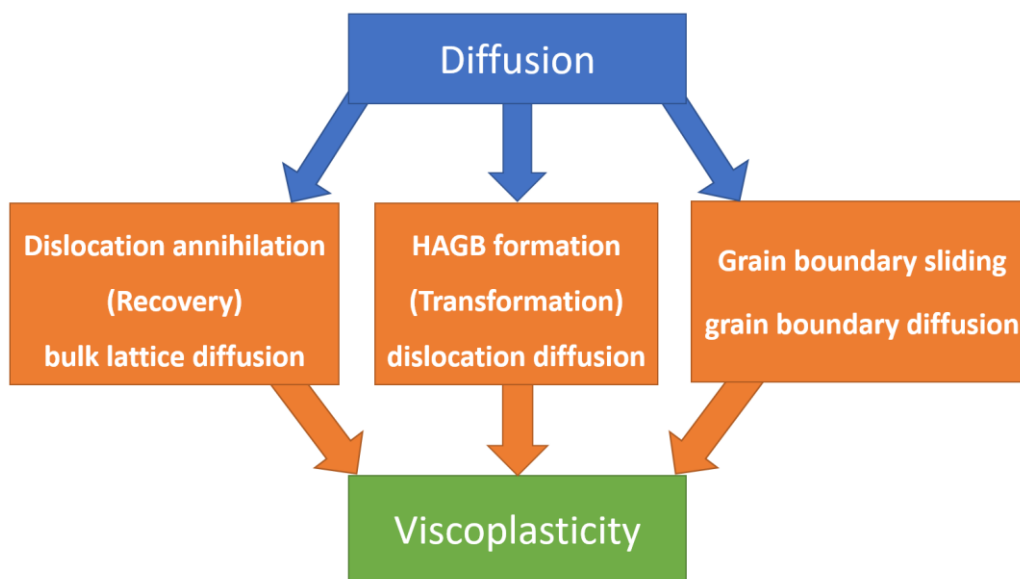


Fig. 6. 1 Schematic illustration of relationships among various mechanisms in viscoplastic deformation.

Within each microstructure evolution mechanism, microscopically, there are complex physical processes. For example, the HAGB formation process involves LAGB-HAGB transformation concerning the HAGB transformation rate. For modelling, these detailed processes are scientifically important, which could be very useful to embed the model into the Phase Field or cellular automaton modelling techniques to model some important microstructure movement. However, modelling these processes at such a level of detail will significantly increase the complexity of the model by introducing a considerably large number of material constants [149], which will prohibit the model from modelling the general various forming processes. Since the aim of this study on constitutive modelling is to capture the macroscopic stress-strain and microscopic GND, LAGB and HAGB evolution in a statistical way, and the driving force for these microstructure changes in the diffusion process, the proposed model will directly link the microstructure evolution with their driving diffusion process.

In the model, the diffusion kinetics (rate) will be determined through the widely used Arrhenius equation in the following form [150]:

$$D = A \dot{\epsilon} e^{-\frac{Q_a}{RT}} \quad (31)$$

where D is the diffusion rate constant (frequency of diffusion); A is a temperature-independent constant for diffusion; Q_a is the activation energy for diffusion with the unit of J mol⁻¹.

6.2.2 Viscoplastic strain evolution

To model the viscoplastic strain evolution, a widely used sinh-law equation [142] is adopted here due to its suitability to model a wide range of strain rates and stress levels [151]:

$$\dot{\epsilon}_p = A_1 \sinh[A_2(\sigma - R - L - H - k)] \quad (32)$$

where A_1 and A_2 are athermal constants, σ is the viscoplastic flow stress. For viscoplastic flow to occur, σ must be larger than the sum of hardening terms to resist dislocation glide or grain boundary sliding, namely, dislocation density hardening (R), LAGB hardening (L) and HAGB hardening (H). Note that compared with previously proposed viscoplastic strain evolution equations [37], the temperature-dependent material constant is not explicitly shown in the equation, since the temperature influence has been incorporated in the hardening terms

to describe the dislocation annihilation, grain boundary sliding and HAGB transformation, as shown in the following.

6.2.3 Yield stress

The sum of the resisting stress for dislocation glide and grain boundary sliding equals to the yield stress (σ_y). The resistance stresses comprise short-range thermal-dependent stress (k) and long-range athermal stresses (R , L and H), that are formulated in Equation 33 [152].

$$\sigma_y = k + R + L + H \quad (33)$$

where k is attributed to the short-range internal resistant stress that generates from solvent and solute atoms or precipitates within lattice, prohibiting dislocation glide [153]; R , L and H are the long-range resistant stresses for dislocation glide, generated from the obstacles such as dislocation network, LAGBs or HAGBs, respectively.

Regarding the value of k , as the deformation temperature (T) increases, there is sufficient thermal energy facilitating the dislocations to overcome this short-range stress. It is confirmed that the internal stress is equal to zero when the temperature is higher than 0.25 times the melting temperature of the material (T_m) of the material in steels (BCC) [153]. Even though this thermal effect should be less pronounced in aluminium (FCC), the typical hot forging temperature range of AA7050 is between 653-733K (0.85-0.95 T_m). Thus, it is reasonable to assume that k is zero.

In aluminium alloys, the total stored dislocation density (ρ_t) is divided into two parts by their misorientation angles (θ), and they are treated as separated hardening terms. The first part is the GND density (ρ_g), in which the underlying dislocations resulted in lattice misorientation with the θ range between 0-2°. Due to the high stacking fault of aluminium, the majority of dislocation dipole multiples, *i.e.* Statistically stored dislocations, SSDs, (ρ_s), are annihilated through the active dislocation cross-slip and jumps facilitated by thermal energy. Thus, the majority of dislocations in the hot deformation aluminium exist in the form of GNDs, such that it is assumed that $\rho_g = \beta_1 \rho_t$ within the range of $\theta \in [0, 2^\circ]$, where β_1 is 0.9. It means that within a given θ range, ρ_g represents ρ_t behaviour; ρ_g and ρ_s show the same trends; however, ρ_s only accounts for a fraction of ρ_t (10%). This assumption is supported by increasing evidence on GND density study in uniaxial tensile deformation, from various

techniques, such as the X-ray in-situ synchrotron measurement shows that the ρ_g and ρ_s exhibit very similar strain hardening trends [36, 154], fitting well to the Taylor hardening theory; high-resolution EBSD demonstrated that ρ_g could be used in the Taylor hardening theory to represent the ρ_t [34]. Thus, the strain hardening term R here is mainly attributed to ρ_g , which has $\theta \in [0, 2^\circ]$.

As the θ of the dislocation boundary further increases ($\theta \in (2^\circ, 15^\circ)$) by accumulating more dislocations of the same sign, these boundaries are defined as LAGBs or subgrain boundaries. The LAGB is conventionally treated as a separate term from a general dislocation formed boundary LAGBs play a critical role in forming HAGB. It is commonly accepted that in high stacking fault energy materials, the formation of HAGBs is through the transformation of LAGBs [6]. Differing from the recrystallisation in low stacking fault energy materials, there is no clear recrystallisation grain nucleation and growth stage.

Nevertheless, fundamentally, LAGBs are also dislocation formed boundaries but have higher dislocation density than GNDs. The magnitude of LAGBs are expressed in the form of area per unit volume (S_l), which is suggested to be proportional to ρ_t , as $S_l = c_2 \rho_t$, for $\theta \in (2^\circ, 15^\circ)$ where c_2 is a material constant depending on the saturation state of LAGBs [50]. Therefore, in modelling the viscoplastic deformation of aluminium, the total dislocation density is separated as GND density and LAGBs depending on their misorientation angles.

6.2.3.1 Dislocation hardening

The term R corresponds to dislocation hardening (*i.e.* isotropic strain hardening), which is proportional to the square root of dislocation density (ρ_h) as typically modelled by the classic Taylor's hardening theory [155]:

$$R = B_1 \sqrt{\rho_g / \beta_1} \quad (34)$$

where B_1 is the dislocation hardening coefficient. β_1 is the fraction of GNDs in total dislocations and is assumed to be 0.9 in this study. This Taylor hardening relationship has been recently validated in uniaxial tensile deformed AA7020, which the ρ_g was measured using in-situ X-ray synchrotron [156], which probably is the most accurate dislocation density measurement technique since it can separate the line broadening effects from micro-strain and dislocation density. Note that the initial ρ_g may not be near the fully annealed or

fully recrystallised state as the as-received materials are often cold deformed and only partially annealed. There is often a moderate amount of dislocation density stored in the material.

As discussed earlier, owing to the high stack fault energy of aluminium and the testing temperatures ($> 0.7 T_m$), dislocation cross-slip and jump can easily take place to annihilate SSDs and form more geometrically necessary boundaries. It leads to a significant fraction of ρ_g in ρ_t , and it is reasonable to assume 90% of the stored dislocation density are GNDs, *i.e.* $\rho_g = \beta_1 \rho_t$ and $\beta_1 = 0.9$.

6.2.3.2 LAGB hardening

LAGB area per unit volume (S_l) is used to represent the LAGB content within the material and is found to be proportional to the hardening effect (L) [83, 143, 157-158]. Thus, S_l is related to the LAGB hardening (L) as:

$$L = B_2 S_l \quad (35)$$

where B_2 is the LAGB hardening coefficient, and S_l is the LAGB area per unit volume and is inversely proportional to the subgrain size (d_{sub}) by $S_l = 2/d_{sub}$ [122].

6.2.3.3 HAGB hardening

The HAGBs that form grains are described by the area per unit volume (S_h), which has a relationship with the grain size (d) as $S_h = 2/d$ [122]. The HAGB hardening effect (H) is described by the classic Hall-Patch relationship [159]:

$$H = B_3 \sqrt{S_h} \quad (36)$$

where B_3 is a diffusion-dependent grain boundary hardening coefficient, can be determined by $B_3 = (\beta_2 - D_1)$, where β_2 is a temperature-independent material constant; $D_1 = A_1 \dot{\epsilon} e^{-\frac{Q_1}{RT}}$ where A_1 is a temperature-independent material constant Q_1 is the activation energy for grain boundary sliding. It means when the influence of diffusion is significant, B_3 value will decrease from positive to negative. Physically, it means that grain boundary starts to slide and acts as softening mechanism when the diffusion process is significant. Whereas grain boundary acts as dislocation glide obstacles to harden the material when the diffusion process is slow.

Based on the early discussion, the evolution of hardening processes by dislocation, LAGB and HAGB relies on the development of dislocation density (ρ_t), LAGB (S_l) and HAGB (S_h). These microstructure variables are defined as state variables in the model. Their evolution is mathematically described and presented in the following sections.

6.2.4 Dislocation density evolution

The classic Kocks and Mecking's dislocation evolution equation [160-161], as presented in (, is adopted here to model the dislocation density evolution rate $\dot{\rho}_t$, due to its simplicity (only two constants), suitability for CDRX in aluminium, for which the dynamic recovery is considered. The widely used Lin's dislocation density evolution equation [85] is not selected here since the effect of grain boundary sliding on the kinetics of dislocation density accumulation is accounted for in the Hall-Patch effect in the current model. Furthermore, the recrystallisation effect on dislocation density in CDRX is negligible since the grain size and HAGB have primarily formed through LAGB transformation rather than grain boundary migration, as typically seen in DDRX.

The total dislocation evolution rate $\dot{\rho}_t$, as shown in Equation 37, is governed by the competition between dislocation accumulation and dislocation recovery. Note that the recovery part only accounts for dislocation annihilation. The dislocation transformation into LAGBs and HAGBs will be accounted for as the transformation part, which will not influence the total dislocation density here. This will keep the modelling and experimental results consistent.

In the current study, ρ_t is defined as the average dislocation density and calculated from the points (n) within grains excluding the points at LAGBs or HAGBs ($\rho = (\sum_1^n \rho_i)/n, i = 1 \dots n$), the dislocation transformed LAGB point will be simply excluded from the dislocation density calculation ($n-1$). In Equation 37, the first term represents the dislocation accumulation process, which is suggested to be an athermal process [162]. During such a process, the mobile dislocations become immobile and stored in the material, after gliding a certain distance, being inversely proportional to the average spacing between dislocations which is expressed by $\sqrt{\varepsilon_p}$.

Thus, c_1 is an athermal constant and can be calculated from the hardening rate of the stress-strain curve of the material tested at room temperature. Whereas the second term is suggested to represent the thermally activated dislocation annihilation rate, driven by a diffusion

process. which can be expressed as a function of temperature and the dislocation density (ρ_t) since the presence of dislocations accelerates the diffusion rate by providing more diffusion channels for atoms to migrate [163-164].

$$\dot{\rho}_t = c_1(1 - \frac{\rho_t}{\rho_t^*})\dot{\epsilon}_p^{1.07} - D_2\rho_t \quad (37)$$

where c_1 is an athermal material constant; D_2 is a diffusion-dependent constant and can be determined by an Arrhenius equation: $D_2 = A_2\dot{\epsilon}_p e^{-\frac{Q_2}{RT}}$, where Q_2 is the activation energy for dislocation annihilation. It is apparent that when the recovery rate, D_2 , is small, *e.g.* at low temperature and high strain rate, the rate of dislocation density $\dot{\rho}_t$ is high, indicating fast dislocation multiplication. As D_2 increases, the dislocation annihilation process becomes more considerable. The accumulation and recovery process will gradually reach an equilibrium state in which the total dislocation density will be constant.

6.2.5 GND density and LAGB evolution

Both the GND density rate ($\dot{\rho}_g$) and LAGB area per unit volume rate ($\dot{S}_l$) are proportional to the total dislocation density rate ($\dot{\rho}_t$). As discussed earlier, GNDs and LAGBs are just two forms of dislocation boundaries, for which GND is defined as the point with a misorientation angle (θ) lower than 2° , while LAGBs are the points with higher misorientation angles in the range of $[2^\circ, 15^\circ]$. The misorientation angle between two neighbouring points (distance of x) is generated by the presence of dislocations [165], which can be simply estimated through $\theta = \rho_g b x$ [20, 166], where b is the Burgers vector of the material. In the current study, ρ_g is assumed to account for 90% of ρ_t in hot deformed aluminium. Hence, the misorientation angle (θ) is directly proportional to the total dislocation density ρ_t .

When the plastic deformation is small, virtually all dislocation density will contribute to the increase of GND density. Thus, the change in GND density rate can be assumed to be the same as the total dislocation density changing rate, *i.e.* $\dot{\rho}_g = \dot{\rho}_t$ as shown in Equation 38. As the plastic deformation proceeds, the total dislocation density reaches a level corresponding to the misorientation angle of 2° , the dislocation formed boundaries are defined as LAGBs. Further increase of plastic deformation or total dislocation density will fully contribute to LAGB. Since the LAGB area per unit volume (S_l) is proportional to the total dislocation

density (ρ_t) as suggested by Gourdet et. al. [50] that the evolution of $\dot{S}_l$ is also proportional to $\dot{\rho}_t$, as seen in Equation 39.

$$\dot{\rho}_g = \dot{\rho}_t \quad \rho_t \in [0, \rho_t(2^\circ)] \quad (38)$$

$$\dot{S}_l = c_2 \dot{\rho}_t \quad \rho_t \in [\rho_t(2^\circ), \rho_t(15^\circ)] \quad (39)$$

where the form of $\rho_t(X^\circ)$ represents the dislocation density corresponding to the misorientation angle $\theta = X^\circ$, and c_2 is an athermal material constant dependent on the saturating dislocation density within LAGBs.

6.2.6 HAGB evolution

The HAGB evolution rate ($\dot{S}_h$) is determined by two premises, namely, the available LAGBs (S_l) and the LAGB to HAGB transformation frequency (f). Based on the previous chapter on the strain effect on dynamic recrystallisation, the HAGB evolution mechanism in AA7050 under typical hot forming ranges is that as S_l increases, there is more recrystallisation occurred. It agrees with the general understanding of dynamic recrystallisation in aluminium [6] that a large S_l is necessary for recrystallisation to take place.

The observed dynamic recrystallisation mechanism appears to fit well with the description of GDRX also proposed by McQueen [167], in which the misorientation angle distribution does not change any further once the peak value reaches 2° . HAGBs seemed to be transformed through suddenly pinch-off from the existing LAGBs. The gradual rotation from LAGB to HAGB, as typically suggested for CDRX, was not observed.

As discussed in the previous Chapter 6.2.5, S_l is proportional to the total dislocation density, which is governed by the dislocation accumulation and annihilation processes. The diffusion promotes dislocation annihilation. It means that under high temperature and low strain rate, high diffusion rate D will accelerate the dislocation annihilation, such that the total dislocation density and S_l may increase slowly or decrease, which will be undesirable for dynamic recrystallisation. On the other hand, if the temperature is low and the strain rate is high, there will be more dislocations and larger S_l to promote dynamic recrystallisation.

The second premise for dynamic recrystallisation is found to be f , which seems to be proportional to D , as shown in the Chapter 5 on strain rate and temperature effects on dynamic recrystallisation. When the temperature is high and the strain rate is low, the high D

stimulates the LAGB to HAGB transformation and results in an increase in dynamic recrystallisation, yet the low D will suppress f and dynamic recrystallisation. Thus, based on the earlier experimental findings, the S_l and f can be drawn as the two essential conditions for dynamic recrystallisation and HAGB evolution.

The diffusion rate D has opposite effects on S_l and f , resulting in a complex trend in HAGB evolution ($\dot{S}_h$). The relationships of S_h , f , $\dot{S}_h$ and D are schematically shown in Fig. 6.2. Fig. 6.2(a) that S_l will decrease considerably with increasing diffusion rate as described by the Arrhenius relationship for the dislocation annihilation process. On the contrary, in Fig. 6.2(b), the LAGB-HAGB transformation frequency (f) increases with increasing diffusion rate. Coupling these two effects on $\dot{S}_h$ leads to Fig. 6.2(c), which shows an initial increase of $\dot{S}_h$, *i.e.* dynamic recrystallisation rate, as the diffusion rate increases. This increasing trend continues until it reaches a peak. Then, a further increase in the diffusion rate results in a reduction in the dynamic recrystallisation rate. In a physical sense, it means that at the low diffusion rate, the dynamic recrystallisation rate is very slow mainly constrained by the low HAGB transformation frequency, although there are adequate LAGBs.

Increasing the diffusion rate accelerates the dynamic recrystallisation rate due to the higher HAGB transformation frequency. This trend leads to the optimal dynamic recrystallisation condition with a fast rate, where sufficient LAGBs are transformed into HAGB at high frequency. However, further increasing the diffusion rate reduces the available LAGBs. The insufficient LAGB leads to an overall reduction in dynamic recrystallisation rate, although the transformation frequency is higher. This trend agrees with the observed HAGB evolution in the strain rate and temperature effect study in Chapter 5. Therefore, the HAGB evolution is determined by the product of LAGBs and the transformation frequency as presented in Equation 40.

$$\dot{S}_h = f S_l = D_3 S_l \quad (40)$$

where D_3 is a diffusion rate constant, corresponding to HAGB transformation frequency, which is determined by $D_3 = A_3 \dot{\epsilon}_p e^{-\frac{Q_3}{RT}}$, where A_3 is an athermal constant and Q_3 is the activation energy for LAGB to HAGB transformation.

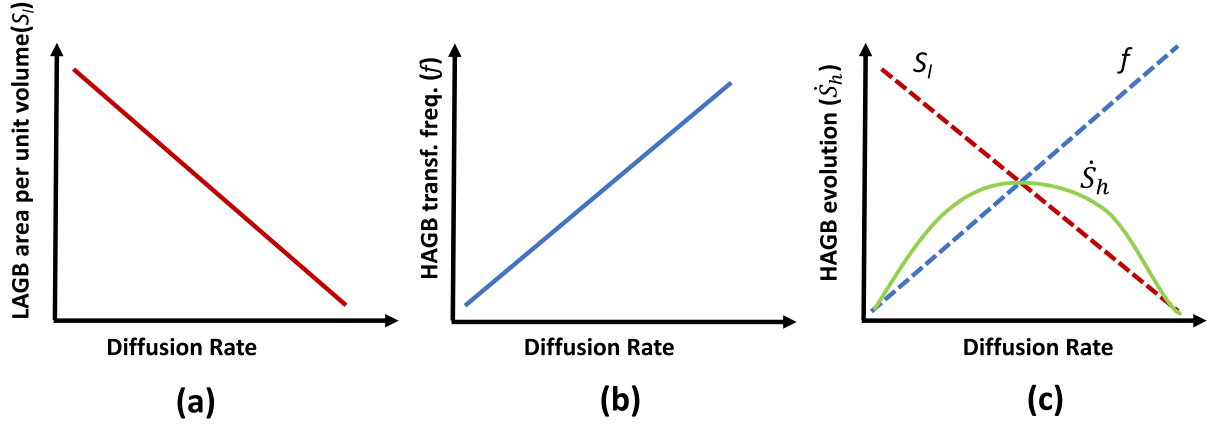


Fig. 6. 2 The schematic plots of the LAGB area per unit volume, LAGB to HAGB transformation frequency and HAGB evolution as a function of diffusion rate.

As the HAGB is transformed from LAGB, the LAGB area will reduce. Hence, the LAGB evolution equation in(, should add a reduction term due to the HAGB transformation. The final form of the LAGB evolution is expressed in Equation 41.

$$\dot{S}_l = c_2 \dot{\rho}_t \dot{\epsilon}_p^{0.1} - \dot{S}_h \quad \rho_t \in [\rho_t(2^\circ), \rho_t(15^\circ)] \quad (41)$$

6.2.7 Flow stress evolution

Finally, the flow stress evolution can be easily computed using Hook's law, as shown in Equation 42.

$$\dot{\sigma} = E(\dot{\epsilon}_t - \dot{\epsilon}_p) \quad (42)$$

where $E = E_0 e^{(-KG \times T)}$ is the Young's modulus of the material (71.7 GPa) at room temperature, $\dot{\epsilon}_t$ is the applied total strain rate.

6.2.8 Formulating new constitutive equations

Based on the previous sub-sections, the complete physical-based constitutive model proposed is shown in Equation 43. The flow stress is governed by three hardening terms, namely, the dislocation (R), LAGB (L) and HAGB (H) hardening, in which the three microstructure variables, namely, the dislocation density (ρ_g), LAGB ($\dot{S}_l$), and HAGB transformation ($\dot{S}_h$), are modelled as state variables. The grain boundary sliding, dislocation annihilations and HAGB transformation due to diffusion are explicitly considered in the model.

$$\begin{aligned}
\dot{\varepsilon}_p &= A_1 \sinh[A_2(\sigma - R - L - H - k)] \\
\dot{R} &= B_1(\rho_g/\beta_1)^{-0.5} \dot{\rho}_g \\
\dot{L} &= B_2 \dot{S}_l \\
\dot{H} &= B_3 S_h^{-0.5} \dot{S}_h \\
\dot{\rho}_t &= c_1(1 - \frac{\rho_t}{\rho_t^*}) \dot{\varepsilon}_p^{1.07} - D_2 \rho_t \\
\dot{\rho}_g &= \dot{\rho}_t; \quad \rho_t \in [0, 13.83] \\
\dot{S}_l &= c_2 \dot{\rho}_t \dot{\varepsilon}_p^{0.1} - \dot{S}_h; \quad \rho_t \in [\rho_t(2^\circ), \rho_t(15^\circ)] \\
\dot{S}_h &= D_3 S_l \\
\dot{\sigma} &= E(\dot{\varepsilon}_t - \dot{\varepsilon}_p)
\end{aligned} \tag{43}$$

6.3 Material constants determination

Even though the number of constants in this model has been considerably reduced from more than 30 to around 10, determining these constants is still nontrivial due to the highly nonlinear nature of those ordinary differential equations. A Matlab script (as attached in Appendices) was created to calculate the material constants by embedding these constitutive equations, experimental stress-strain curves, GND density, LAGB and HAGB data. To run the script, the initial values of all variables must be set. The initial values are shown in Table 6.1. Most of the initial microstructure variables such as the GND density, LAGB and HAGB can be read directly from experimental measurement without making any assumptions, which ensures the initial values are truly physical.

Table 6.1. Initial values (at $t = 0$) for state variables.

Variable Name	Unit	Value
Flow stress (σ)	MPa	0
Plastic strain (ϵ^p)	-	0
dislocation density (ρ_t)	$\text{Log}_{10} m^{-2}$	13.47
GND density (ρ_g)	$\text{Log}_{10} m^{-2}$	13.44
Work hardening (R)	MPa	1.4
Low-angle boundary hardening (L)	MPa	0.4
Grain boundary hardening (H)	MPa	0.5
Surface of High-angle grain boundary per unit volume (S_{HAB})	μm^{-1}	0.0271
Surface of High-angle grain boundary per unit volume (S_{lab})	μm^{-1}	0.0696

These initial values are used to determine the constants in the Matlab script, in which Equation 43 are embedded. The initial values of the constants are estimated based on previous constitutive modelling studies of AA7050 [149]. Applying these initial constants into Equation 43, the rates of all variables are computed in the first step. Then, the current variable values for the first step can be determined by adding the initial values with the increments, which is the product of the rate of variables multiplied by the time step. For the second step, the variable rate and values can be calculated in the same way but updating the initial values with the first step determined values. This process will continue until the specified number of time steps (5000) are reached. The 5000 steps are selected to balance the fitting accuracy and computational cost, as a larger number of steps tends to improve its accuracy but require increasing computational cost.

The calculated variables are then compared with experimentally determined curves. The constants are tuned to minimise the difference between the experimental and computational curves until a reasonable agreement is reached, in other words, until the errors between the experimental and computational curves are acceptable. Several optimisation schemes are available in Matlab, such as the evolutionary programming method [168] and the least square method [169], which were used previously to facilitate the constant determination process to some extent. However, the mathematical relationships of the proposed constitutive equations

are considerably different from previous models by directly considering diffusion processes; setting the ranges of these constants for these optimisation methods is problematic. Hence a time-consuming, manual tuning was applied to determine the material constants. The determined constants are shown in Table 6.2 and 6.3.

Table 6.2. List of the determined athermal constant values.

Athermal Material Constants	A_1 (s^{-1})	A_2 (MPa^{-1})	β_1 ($-$)	β_2 ($-$)	B_1 ($-$)	B_2 (MPa)	c_1 ($-$)	c_2 ($-$)
	7.2×10^{-4}	0.114	0.80	0.92	91.00	9.30	475.00	0.99
	d_1 ($MPa \cdot s$)	d_2 ($-$)	d_3 ($-$)	ρ_t^* (μm^{-2})	Q_b ($J \text{ mol}^{-1}$)	E_0 (MPa)	KG (MPa)	R ($J \text{ mol}^{-1}$)
	97.00	0.19	0.24	14.00	134.00	71.7×10^3	5.4×10^{-4}	8.314

Table 6.3 List of the determined diffusion-dependent constant values.

Diffusion Dependent Constants	$B_3(MPa)$	$D_2 (s^{-1})$	$D_3 (s^{-1})$
	$\beta_2 - d_1 \dot{\epsilon}_t^{0.01} e^{-\frac{Q_b}{RT}}$	$d_2 \dot{\epsilon}_t e^{\frac{Q_b * 60}{RT}}$	$d_3 \dot{\epsilon}_t e^{-\frac{Q_b}{RT}}$
	$k (MPa)$	$E (MPa)$	
	$209.98 - 0.2905 \times T$	$71.7 \times 10^3 e^{(-KG \times T)}$	

6.4 Results and discussion

The constitutive stress-strain curves at various temperatures and strain rates are presented in Fig. 6.3. As seen in Fig. 6.3, the new constitutive model can capture reasonably well the viscoplastic flow behaviour, that the maximum flow stress increases with the decrease in temperature or the increase in strain rate.

Furthermore, the new model is able to simulate the common softening behaviour mainly due to grain boundary sliding occurring in all stress-strain curves. Admittedly, small dispersions can still be seen in Fig. 6.3(a) in the curves at either the highest or lowest strain rates. These small dispersions could be caused by either systematic experimental errors at very high and low strain rates, at which the strain and stress measurement could be less accurate, or further improvement on the material constants optimisation can be made. Despite the small dispersions, overall, the proposed constitutive equations have captured the characteristics of

the viscoplastic behaviour of AA7050; and reasonable accuracy can be achieved in the selected hot forging window.

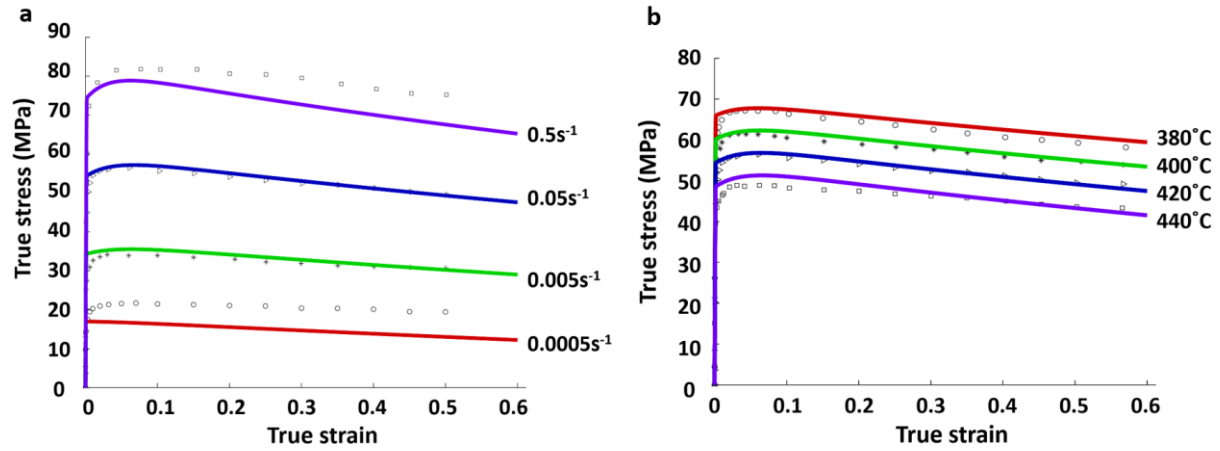


Fig. 6.3 True stress-strain curves for AA7050 from experiments (symbols) and modelling (lines), (a) $T = 420\text{ }^{\circ}\text{C}$; (b) $\dot{\epsilon} = 0.05\text{ s}^{-1}$.

Since these macroscopic stress-strain curves are determined through the underlying microstructure evolution based on Equation 43, the modelled microstructure evolution relationships are shown in Fig. 6.4. Fig. 6.4(a)-(c) report the GND, LAGB and HAGB evolution as a function of plastic strain at one of the selected hot deformation conditions: $420\text{ }^{\circ}\text{C}$ 0.05 s^{-1} . The modelled GND density, LAGB and HAGB results, shown as the blue lines, fit well with the red experimental trends, that at the beginning, the GND density and LAGB increase rapidly with increasing plastic strain.

However, the rate of increase decreases considerably at the later stage, which indicates the equilibrium state between dislocation generation and recovery. Nevertheless, HAGB exhibits an almost linear relationship with plastic strain due to the occurrence of CDRX. This linear relationship is well captured by the proposed model by balancing the LAGB population and diffusion-driven LAGB-HAGB transformation rate. These suggest that the proposed GND density, LAGB, and HAGB evolution processes seem reasonable and are able to accurately describe the underlying mechanisms.

Under the same plastic strain (~ 0.58), the GND density, LAGB and HAGB evolution as a function of the diffusion process, *i.e.* Zener-Hollomon parameter, is presented in Fig. 6.4(d)-(f). Although there are some fluctuations in the experimental data, the general trends are clear that GND density and LAGBs increase with increasing Z value. Physically, it is due to the suppression of dislocation annihilation with the less diffusion process. However, the GND density and LAGBs trends are different, since the LAGBs also depend on the LAGB-HAGB

transformation. These observed trends for GND density and LAGBs have been correctly modelled by the proposed dislocation density and LAGB evolution equations in Equation 43, especially for the LAGBs, as shown in Fig. 6.4(e) that there is a very small difference between the experimentally measured and modelled LAGB area fractions under a wide range of thermal, mechanical testing conditions.

Although this trend has been correctly captured in Fig. 6.4(d), there are some dispersions at lower Z values, *i.e.* at higher temperature and lower strain rates. A closer inspection reveals that the experimental GND density in the first two points ($\sim 13.1 \text{ m}^{-2}$ at $\log_{10}$) is even considerably lower than the as-annealed materials ($\sim 13.4 \text{ m}^{-2}$ at $\log_{10}$, as seen at the first point in Fig. 6.4(a)). This GND density reduction suggests the occurrence of GND density drops from grain growth. This phenomenon was not considered since it is extremely rare to occur under typical hot forge processes due to the required very high temperature or very low strain rate. This phenomenon could be accounted for in more generalised aluminium hot deformation models in future.

Regarding the dynamic recrystallisation and HAGBs, it is of great interest to notice that the proposed three phases of dynamic recrystallisation in the previous chapter are correctly modelled. In the previous chapter, it was proposed that dynamic recrystallisation consists of three phases: In the first phase, HAGB will increase with Z value until it reaches the first peak due to the increasing LAGB; further increasing in Z will result in a lower HAGB value due to the lower LAGB and HAGB transformation rate; further increase in Z at very high strain rate will reach another peak due to the direct transformation of LAGB to HAGB with little diffusion process involved. These three phases are correctly modelled in Fig. 6.4(f), suggesting the competing mechanisms between the LAGB area and LAGB-HAGB transformation rate as discussed in Chapter 4.2.

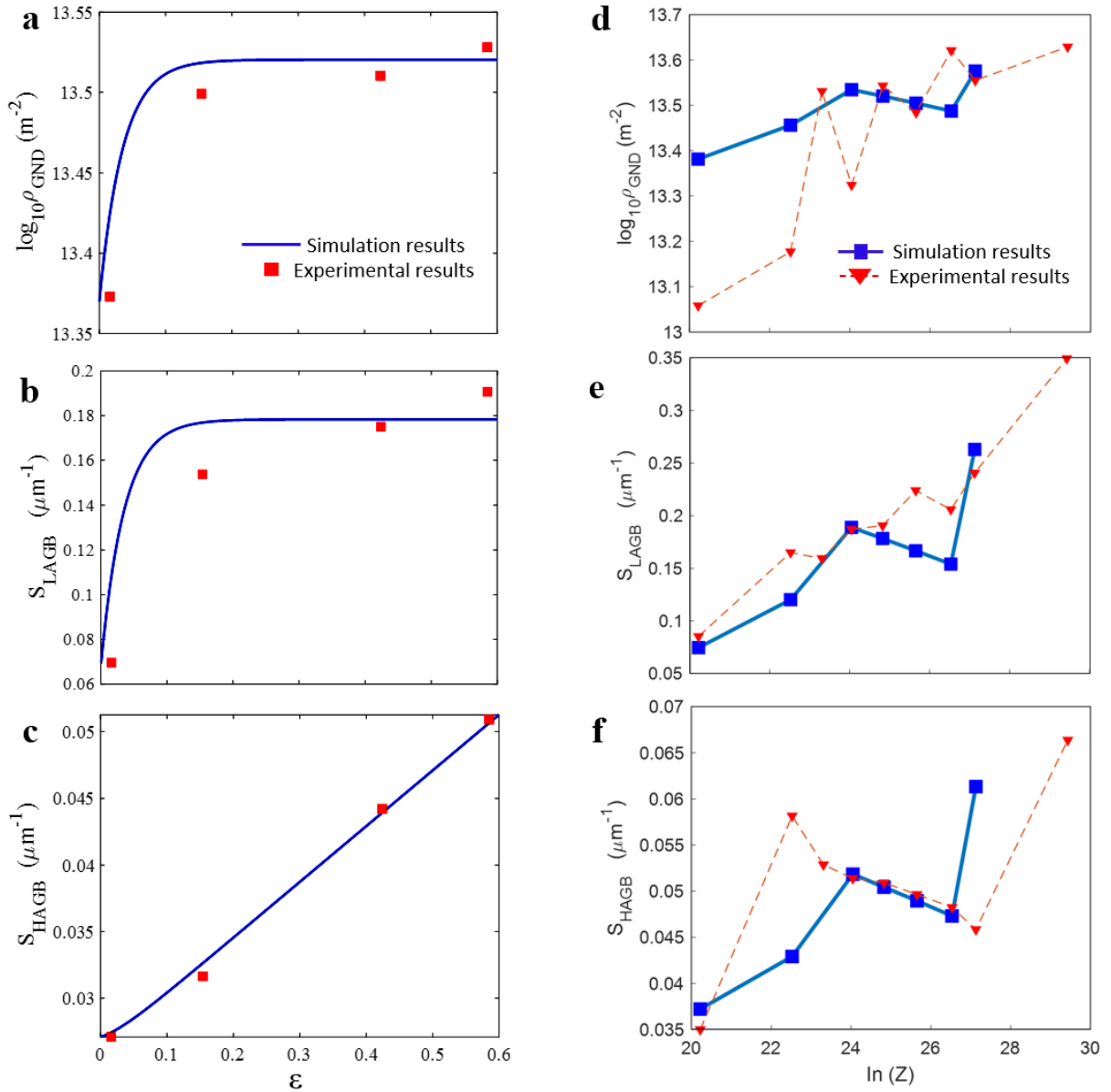


Fig. 6. 4 The evolution of $\log_{10}\text{GND}$ density, area fractions of LAGB and HAGB per unit volume as functions of plastic strain: (a), (b) & (c) at 420°C , 0.05 s^{-1} ; and as functions of Zener-Hollomon parameter (Z): (d), (e) & (f). Red lines and squares represent the experimental data, while the blue curves and downward-pointing triangle correspond to the modelled results.

Compared with existing dynamic recrystallisation viscoplastic constitutive models for aluminium alloys [21, 23, 149, 170], a number of novelties and advantages can be demonstrated in this new model. First, almost all existing models model the continuous rotation of LAGB into HAGB in hot deformation of aluminium alloys, which, however, was not observed for this study. Instead, CDRX seems to be the primary recrystallisation process.

To the author's knowledge, this is the first diffusion-driven (dislocation movement) constitutive model attempt to model CDRX by considering the two competing processes,

namely, the LAGB population and the LAGB-HAGB transformation possibility. Both processes are determined by the diffusion process. Second, the GND density is introduced in the constitutive equations as a state variable. This variable allows dislocation density to be partially experimentally verifiable, which is a significant advance compared to previous physically-based constitutive models [71, 142], in which dislocation density is an unverifiable term and often casts concerns about the proposed physics behind these models.

Although, in this study, the GND density and total dislocation density are assumed to have a simple linear relationship because of the high stacking fault energy of aluminium alloys, this GND and total dislocation density relationship can be adjusted according to the materials and their underlying mechanisms. Consequently, all three key microstructure variables in the model, namely, GND density, LAGB and HAGB, can now be directly verified against experimental measurement. Third, the proposed model is a diffusion-based model. All diffusion activated viscoplastic phenomena, namely, the grain boundary sliding, dislocation annihilation and LAGB and HAGB transformation, are explicitly modelled with a simple addition or subtraction relationship. This approach simplifies the model and reduces the complexities caused by the observed phenomenological processes. As a result, this simple but experimentally verifiable model considerably reduces the required number of material constants and hence significantly saves time and effort on material constants determination and curves fitting.

This developed constitutive model aims to model the viscoplastic behaviour and microstructure evolution during the hot forging of AA7075 alloy. As demonstrated in this section, the current model shows a relatively high accuracy in predicting the key variable evolution process, including dislocation accumulation, recovery, LAGB and HAGB transformation. However, it should be noted that although the current model has incorporated the primary CDRX mechanism, which should be applicable to other aluminium alloys and a wide range of high stacking fault energy alloys, depending on the strain rate and temperature range, other DRX mechanisms including DDRX and GDRX could be the dominating mechanism. In that case, the current model would have limited accuracy. In addition to the governing mechanism, the accuracy of the model largely relies on the available experimental data. Large range of testing temperatures, strain rates and EBSD characterisation would be essential to ensure the accuracy of the model.

6.5 Summary

A physically-based, diffusion-driven (dislocation movement) constitutive model has been proposed to model the viscoplastic behaviour of AA7050 under typical hot forging conditions, based on the experimental observations in the previous chapters. The following conclusions can be drawn:

- i. The GND density, LAGB and HAGB behaviour driven by athermal and diffusion processes have been modelled explicitly. In the model, dislocation density accumulation, recovery, and transformation into GND density and LAGBs have been considered. Meanwhile, the LAGB formation from dislocation density and the LAGB-HAGB transformation process have been taken into account.
- ii. The GND density has been introduced into the constitutive model as a state variable, such that the GND density can be verified through EBSD measurement.
- iii. The CDRX process has been modelled based on the LAGB population and LAGB-HAGB transformation rate. The S-shaped HAGB curve as a function of diffusion level has been correctly modelled.
- iv. The proposed model can accurately capture the general trends of stress-strain, GND density, LAGB and HAGB behaviour as a function of strain and diffusion level, expressed as Zener-Hollomon parameter.
- v. The grain growth has not been considered in this model.

Chapter 7

Modelling of isothermal open die forging

7.1 Introduction

This chapter aims to verify the proposed constitutive model developed in the previous chapter on a hot forging process. From the engineering aspect, this study is to simulate the hot forging process using the developed physically-based constitutive model, such that the macroscopic materials flow, as well as the microstructure evolution and distribution, can be accurately predicted in hot forged AA7050 parts, and the hot forging process can be optimised according to the designer's specifications.

Isothermal open die hot forging (upsetting) is conducted by hot compressing cylindrical samples using Gleeble 3800. Although the hot forging procedure is the same as the previous uniaxial compression test, the hot forging test focuses on validating the developed model by comparing the inhomogeneous strain and strain rate distribution across the samples, due to the friction at the interfaces between the forged samples and the anvils, which is closer to the industrial practical hot forging process, while the purpose of those uniaxial hot compression tests is to establish the essential AA7050 viscoplastic behaviour database, by obtaining the stress-strain, GND, LAGB and HAGB evolution at the central points of the samples, under various temperature and strain rate conditions.

Therefore, in this chapter, the developed unified physically-based constitutive model is embedded into a subroutine of Finite Element Analysis (FEA) software, *i.e.* ABAQUS, to simulate an isothermal open-die forging process. Meanwhile, lab-scale isothermal hot forging is conducted to validate the accuracy of the proposed model by examining the macroscopic geometrical changes, GND density, LAGB and HAGB distribution at various positions of the hot-forged part. Note that more complex industrial forging verification with non-uniform temperature, friction, strain, and strain rate distribution was planned, yet it was unable to be undertaken due to the unexpected pandemic outbreak in the past two years. Hence a relatively simple isothermal lab open die forging process is conducted to validate the model to some extent.

This chapter consists of three main parts: 7.2 describes the FE model set-up, including the selection of element type and size, model development and its boundary conditions. Also, how the subroutine is developed and embedded into ABAQUS will be explained; 7.3 presents the model verification by comparing the experimental and FE modelled the stress-strain data at the central point of the hot-forged part; 7.4 validates the model microstructure prediction by evaluating and comparing the experimental and modelled GND density, LAGB and HAGB at various positions of the hot forged parts.

7.2 FE model set up

7.2.1 Isothermal open die hot forging and boundary conditions

The isothermal hot compression test in Gleeble 3800 is schematically shown in Fig. 7.1. Similar to the previous uniaxial hot compression test in which a cylindrical sample was placed between two anvils acting as forging die, which was connected to the loading machine through clamps and steel jaws. The forging load is applied along the cylindrical axis by moving one of the anvil-clamp-jaw toward the other. The heating is achieved through electrical resistance, monitored, and controlled by a pair of attached thermal thermocouple wires at the centre of the sample.

To simulate this hot forging process, a FE model needed to be established with sample geometry, loading and boundary conditions is shown on the right-hand side of Fig. 7.1. To account for the friction between the die and workpiece, the interfaces between the anvils (forging die) and the workpiece were modelled. The forging die was assumed to be rigid due to its high stiffness, and the friction coefficient between the die and the workpiece was determined as 0.087. The detailed friction coefficient determination will be presented in Chapter 7.2.3.

The workpiece dimensions are reported in Fig. 7.1. Since the workpiece has a cylindrical shape, it is axially, horizontally (x-axis) and vertically (y-axis) symmetrical. Due to these symmetries, a 2D quarter area is sufficient to model and represent the entire cylindrical workpiece, as shown as the shaded area in the inset of Fig. 7.1. This 2D modelling approach would significantly reduce the computational cost without compromising the model accuracy. To account for these symmetries in the model boundary conditions, the movement edge along the y-axis on the cylindrical axis (*i.e.* the left-hand side of the 2D model) is constrained along

the y-axis. In other words, this edge can move along the y axis but cannot move along the x or z-axis. Whereas the edge along the x-axis at the centre of the workpiece (*i.e.* the bottom side of the 2D model) is free to move along the x-axis but unable to move along the y and z-axis. Finally, the loading is applied along the y axis by controlling the displacement at the top edge of the model. Note that as the experiment is at a near isothermal hot forging condition, constant temperature distribution across the workpiece is assumed in this simulation.

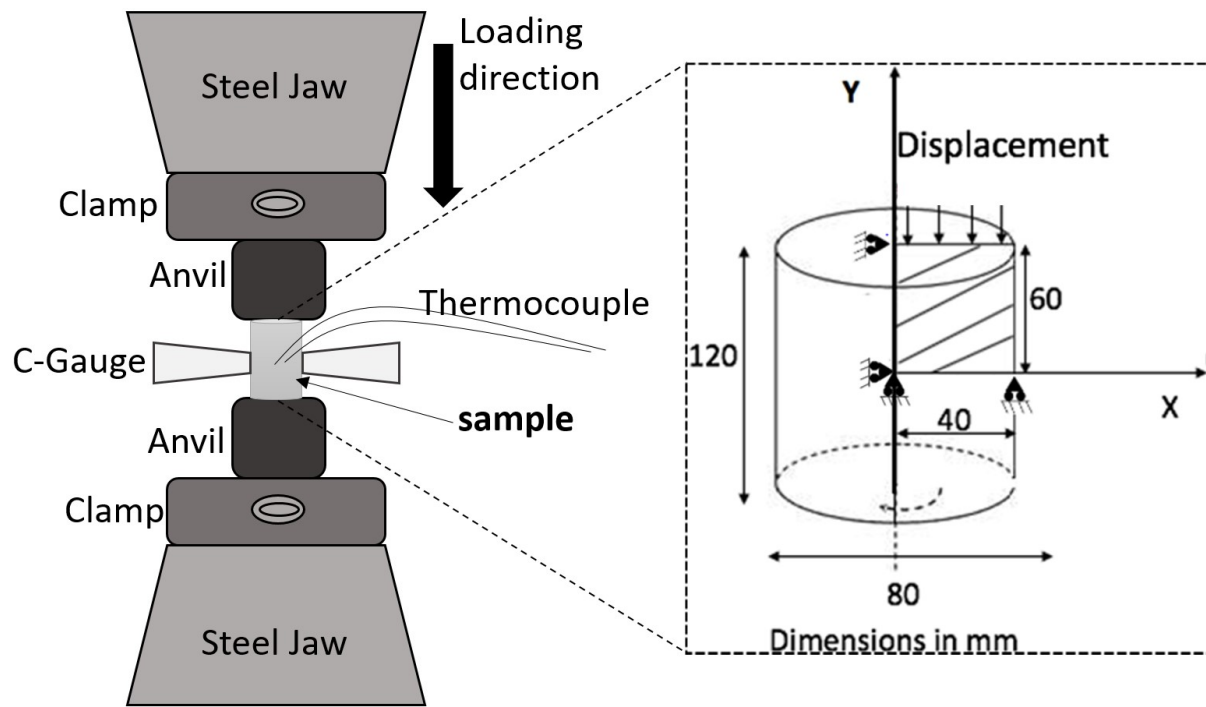


Fig. 7. 1 Schematic illustration of the hot forging process within Gleeble, the workpiece geometry and boundary conditions

7.2.2 FE model establishment, element type and size selection

Based on the model discussed in Chapter 7.2.1, a FE model was built in ABAQUS and shown in Fig. 7.2. Due to the highly non-linear characteristic of the constitutive model and the dynamic deformation process, ABAQUS/EXPLICIT was chosen as the solver to model the forging process. In ABAQUS, the explicit algorithm can calculate the new state of variables directly from the current state. At the same time, the implicit method has to solve a coupled system of non-linear equations, typically using the Newton-Raphson method. Generally, the implicit approach offers better accuracy but requires considerably larger computational power. In contrast, the explicit method with an appropriate step size can achieve a good balance between computational cost and accuracy. Therefore, ABAQUS/EXPLICIT is superior in solving extremely non-linear, dynamic problems than

ABAQUS/STANDARD (IMPLICIT), which is better at solving static, linear or near-linear problems. Hence, the explicit solver was selected.

For each element, the material properties are assigned as AA7050 with isotropic properties. As ABAQUS does not automatically apply the axial symmetrical condition to the model, the radial symmetry without twist boundary condition must be specified by the element type. Hence, CAX4R, the axisymmetric solid element with 4-node bilinear reduced integration with hourglass control, was selected as the element type used in this model. Note that the CAX4R is the most effective element of the axisymmetric solid element available in ABAQUS/EXPLICIT with four nodes. Utilising the CAX4R element optimises the model's efficiency and accuracy. The bilinear quadrilateral could significantly save computational time without compromising accuracy. Also, the reduced integration could avoid shear-lock and meshed fine elements. Meanwhile, the hourglass control could avoid the hourglass effect, which causes inaccuracy by reduced integration with a limited number of elements.

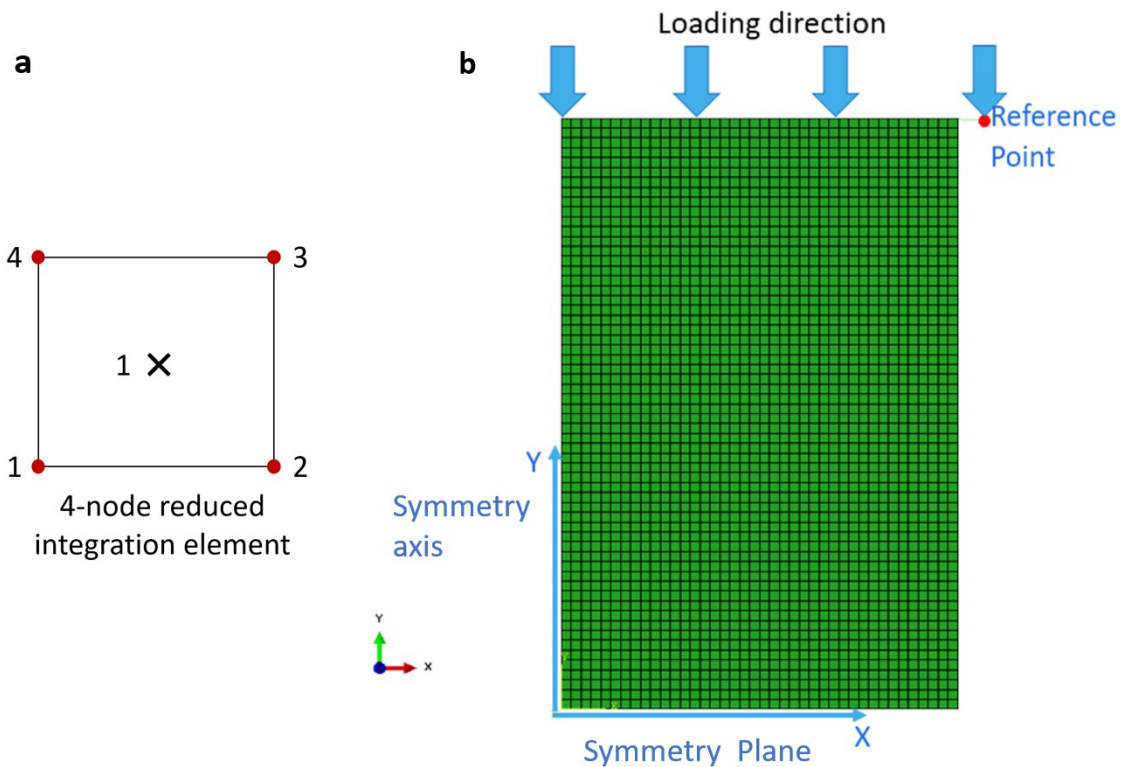


Fig. 7. 2 (a) 4-node reduced integration element in 2D quadrilateral elements. The red dot and black cross represent the node and integration points. (b) The illustration of the finite element mesh, symmetry planes and coordinate system of the axisymmetric model.

7.2.3 Implementing constitutive equations using user-defined subroutine

The proposed unified physically-based constitutive equations for AA7050 are embedded into ABAQUS/EXPLICIT by a user-defined subroutine, VUMAT, using FORTRAN. It should be

noted that the established constitutive equations were calibrated using uniaxial stress–strain test data. It is necessary to generalise these uniaxial constitutive equations for multiaxial ones in the VUMAT subroutine to be used in the FE analyses. The uniaxial to multiaxial transformation is presented in Fig. 7.3, where the overall working principle is outlined.

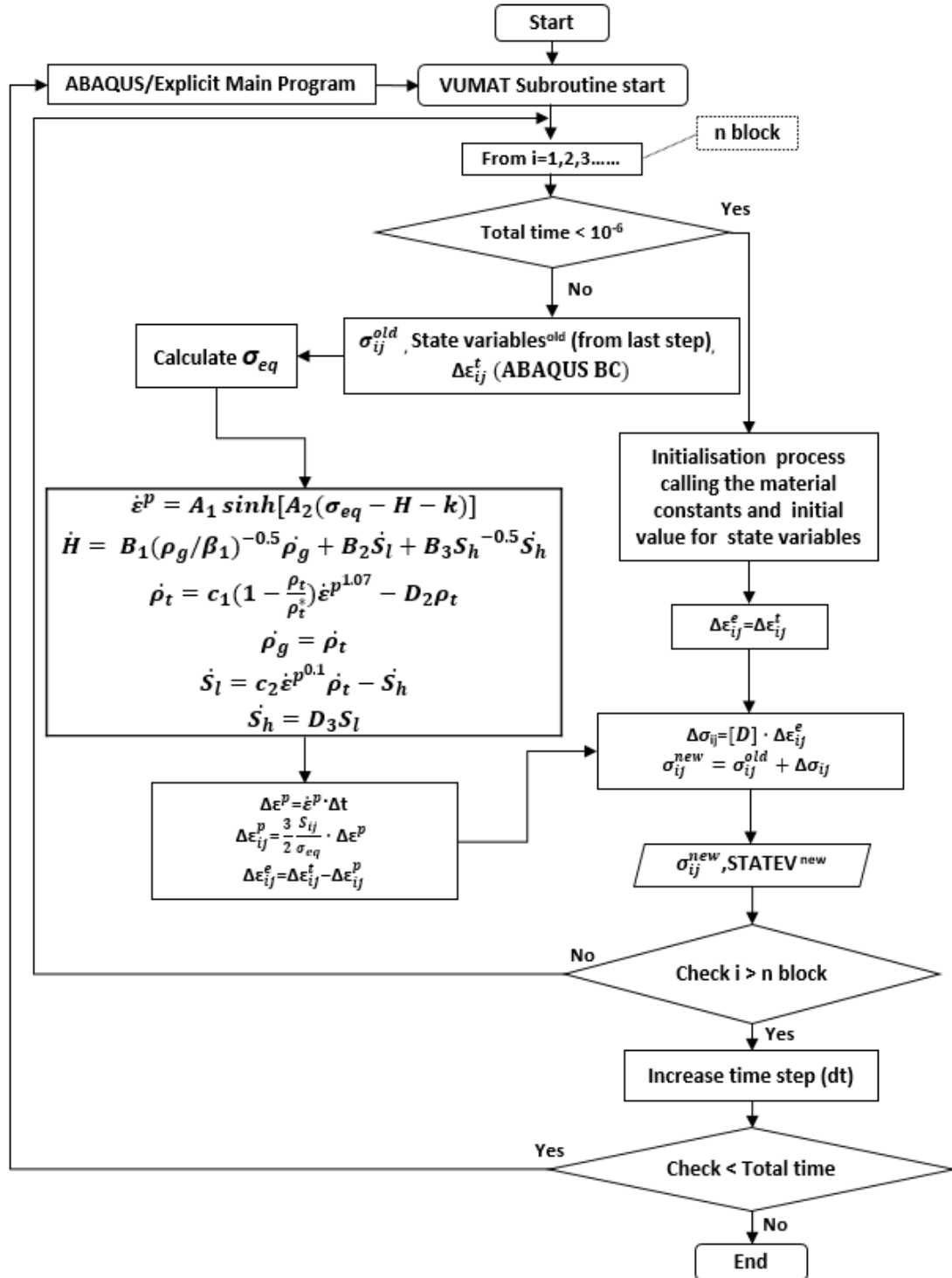


Fig. 7.3 The flowchart describes the user defined VUMAT subroutine.

The model is loaded by a displacement (strain rate) control. The total strain tensor (ε_{ij}^t) can be specified according to the loading condition and constantly increases with time. At the initial loading stage, the deformation is elastic. Since this elastic region is very short in viscoplasticity, it can be defined as the elastic loading region ($\varepsilon_{ij}^e = \varepsilon_{ij}^t$) when the loading time is shorter than 10^{-6} s. The corresponding stress tensor (σ_{ij}) can be calculated straightforward using Hooke's law ($\Delta\sigma_{ij} = D\Delta\varepsilon_{ij}^e$, where D is the elastic modulus). This process will continue incrementally by the predefined time step (dt), and the stress and strain will be accumulated and updated step by step until the loading time exceeds 10^{-6} s. Then the material enters subroutine uses equations in the plastic deformation region.

At the first step of the plastic deformation, the 3D stress tensor σ_{ij} from the previous time step is converted into a 1D scalar by computing the equivalent von-Mise stress (σ_{eq}) [171]. σ_{eq} will be passed to the constitutive equations. All state variables, *e.g.* $\dot{\sigma}$, $\dot{\varepsilon}^p$, $\dot{H}$, $\dot{L}$ and $\dot{\rho}$ will be calculated. The plastic strain increment ($\Delta\varepsilon^p$) is thus determined by the product of $\dot{\varepsilon}^p \Delta t$. This 1D equivalent plastic strain $\Delta\varepsilon^p$ is decomposed into a 3D plastic strain tensor ($\Delta\varepsilon_{ij}^p$) by considering plastic strain energy in von-Mise behaviour ($\Delta\varepsilon_{ij}^p = \frac{3}{2} \frac{S_{ij}}{\sigma_{eq}} \Delta\varepsilon^p$, where S_{ij} is the deviatoric stress obtained through $\sigma_{ij} - \delta_{ij}\sigma_{ij}/3$) [37]. The determined $\Delta\varepsilon_{ij}^p$ is subtracted from the total strain tensor $\Delta\varepsilon_{ij}^t$ to determine the elastic strain tensor, $\Delta\varepsilon_{ij}^e$. Then, Hook's law ($\Delta\sigma_{ij} = D\Delta\varepsilon_{ij}^e$) can be applied to determine the stress increment, and hence the flow stress. All other variables, *e.g.* ρ , L , H , will be updated at the end of a time step.

This calculation process will be undertaken on all integration points (known as 'n block') for all elements in the developed FE model. Then, the calculation processes to the next time step and repeats until the predefined number of the time step is reached.

7.3 FE hot forging simulation results and discussion

The overview of FE isothermal open die hot forging simulation results, which incorporate the developed dynamic recrystallisation constitutive model for AA7050 and undertaken at 420°C and $0.05s^{-1}$, is presented in Fig. 7.4, including variable distribution and evolution examples, such as the equivalent plastic strain, total dislocation density, GND density, LAGB area per unit volume and HAGB area per unit volume.

The equivalent plastic strain 3D distribution and evolution are displayed in Fig. 7.4 (a). As the hot forging starts, the workpiece is deforming with inhomogeneous equivalent plastic strain distribution, for which the highest plastic strain (~ 0.85), the “hot spot”, can be found in the central region. In contrast, the lowest plastic strain (~ 0) occurs at the interface centre area between the workpiece and die due to friction. At the workpiece-die interface, due to the “friction hill” [172], which suggests the highest friction occurs at the centre of the interface and the minimum friction exists at the edge, the plastic strain distribution appears to be a normal distribution, with minimum strain close to zero at the centre and the maximum strain of 0.8 at the rim. The variation of plastic strain in other regions of the workpiece is less, within the range of 0.36-0.57. This friction-caused inhomogeneous plastic deformation results in the characteristic open die forging feature: barrelling, which will be validated with experimental results in a later section.

Total dislocation density distribution and evolution exhibit different behaviour to equivalent plastic strain, as shown in Fig. 7.4 (b) that the dislocation density rises rapidly to reach a near saturation state with only moderate plastic deformation (0.29). Further increase in plastic deformation to 0.58 does not increase the dislocation density considerably. Nevertheless, the distribution of high dislocation density within the workpiece evolves slightly from a ‘round’ shaped pattern to a ‘butterfly’ shaped one. This agrees with the underlying dislocation density evolution mechanism during the dynamic recrystallisation process in aluminium alloys [67, 173], that the dislocation density increases rapidly at the early stage of plastic deformation and is followed by a steady-state due to the counterbalance of dislocation accumulation and fast recovery. It is of interest to observe the dislocation density distribution pattern evolution in the later stage of deformation in a forging workpiece.

GND density and LAGB area fraction per unit volume exhibit similar trends to total dislocation density, seen in Fig. 7.4 (c-d). This similar behaviour is expected, since GND density account for 80% of total dislocation density in the proposed model, and LAGB has a linear relationship to total dislocation density. Nevertheless, the LAGB shows a more uniform distribution pattern in the central regions than the GND density. This uniform distribution could attribute to the LAGB to HAGB transformation process, in which a high LAGB area per unit volume region is more likely to be transformed into HAGBs and hence reduce LAGBs, as discussed in the preceding chapters.

Finally, considering HAGB area per unit volume behaviour, as seen in Fig. 7.4 (e), its trends are exciting. As the workpiece is deformed to 0.29, unlike dislocation density or LAGBs, there is a moderate, relatively uniform increase of HAGB across the entire workpiece. Further increase of plastic deformation results in inhomogeneous, ‘butterfly’ shaped distribution, where the highest HAGB is at the centre of the workpiece and spreads to the rims of the interfaces between the workpiece and die. These results demonstrate that the critical stored LAGBs are necessary to initiate the dynamic recrystallisation process. The LAGB-HAGB transformation is a diffusion process driven for which duration is an essential factor to be considered. Hence, higher plastic strain, *i.e.* longer duration, will allow more LAGBs to be transformed into HAGBs.

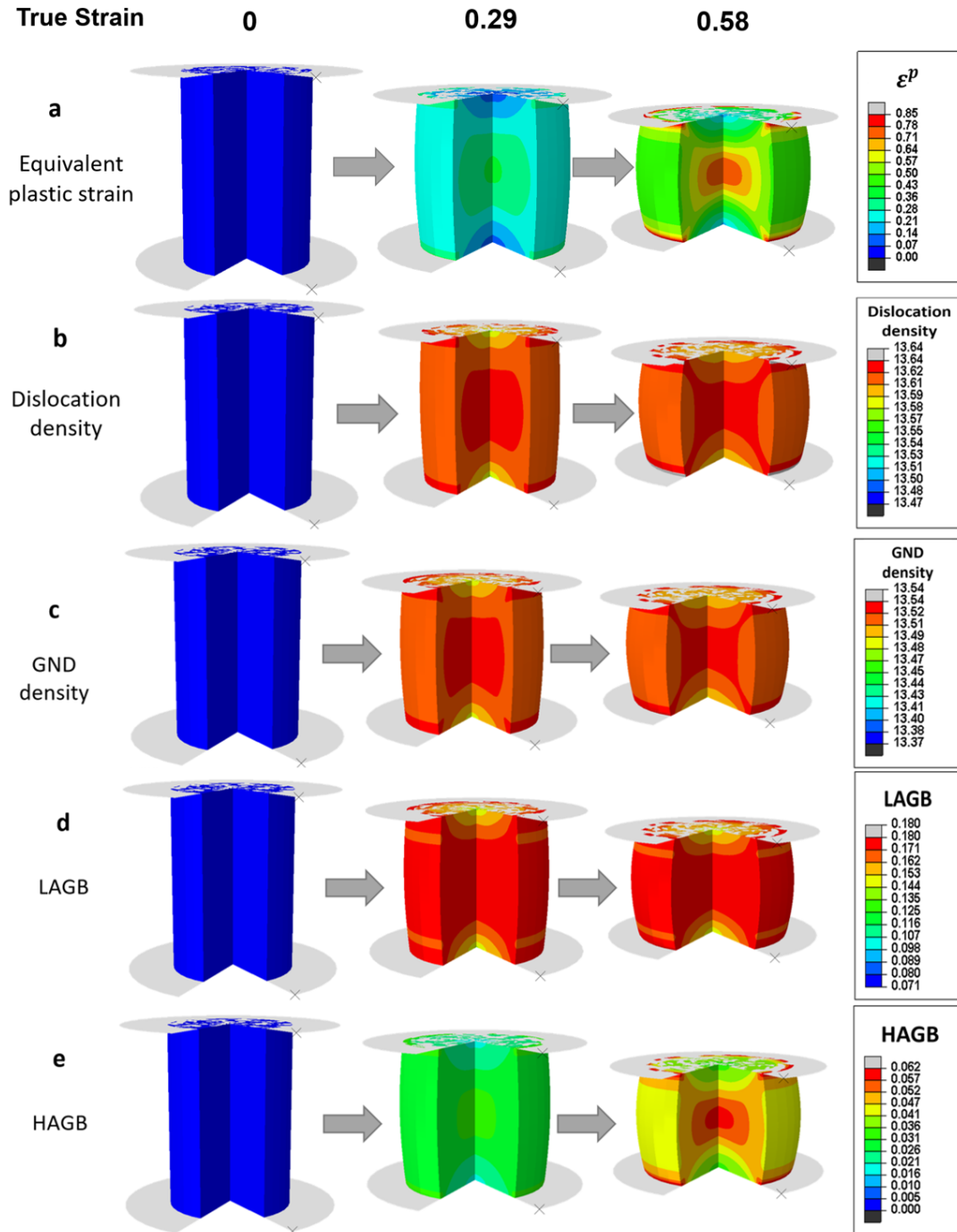


Fig. 7. 4 ABAQUS 3D hot forging model at 420 °C and 0.05 s⁻¹: (a) The equivalent plastic strain; (b) total dislocation density at log₁₀ scale m⁻²; (c) GND density at log₁₀ scale m⁻² (c); the length of low angle grain boundary per unit area m⁻¹; (d) the length of high angle grain boundary per unit area m⁻¹.

7.4 FE hot forging simulation validation

To validate and test the accuracy of the developed FE forging simulation, two isothermal open die hot forging at 380 °C, 0.05 s⁻¹ and 420 °C, 0.05 s⁻¹ were conducted experimentally and simulated numerically, respectively. The extensions at the central regions of the

workpieces were monitored by C-gauge (True strain for the specimen) and their underlying microstructure were revealed by EBSD. These experimental results are used to compare with the numerical results and determine the accuracy of the simulation.

7.4.1 Macroscopic behaviour and validation

Local stress-strain curves and overall geometrical changes are used to compare the material's macroscopic behaviour. Due to the presence of friction at the interface between the workpiece and forging die, their stress-strain distribution is inhomogeneous. The local stress-strain relationship at the middle section of the workpiece was experimentally measured using C-gauge, as illustrated in Fig. 7.5(a). The expansion of the middle section was monitored during the loading. Hence, the instantaneous stress and cross-sectional area change were determined. In the simulation, stress-strain data at the middle section of the workpiece were calculated by averaging the stress-strain values of the points at the edge, middle and centre of the middle sections, as illustrated in Fig. 7.5(b). The comparison of FE simulation and experimental results is presented in Fig. 7.5(c) and (d). For both forging cases, FE simulated stress-strain curves show good consistency with the experimentally measured ones. A reasonable agreement has been reached at a macroscopic scale.

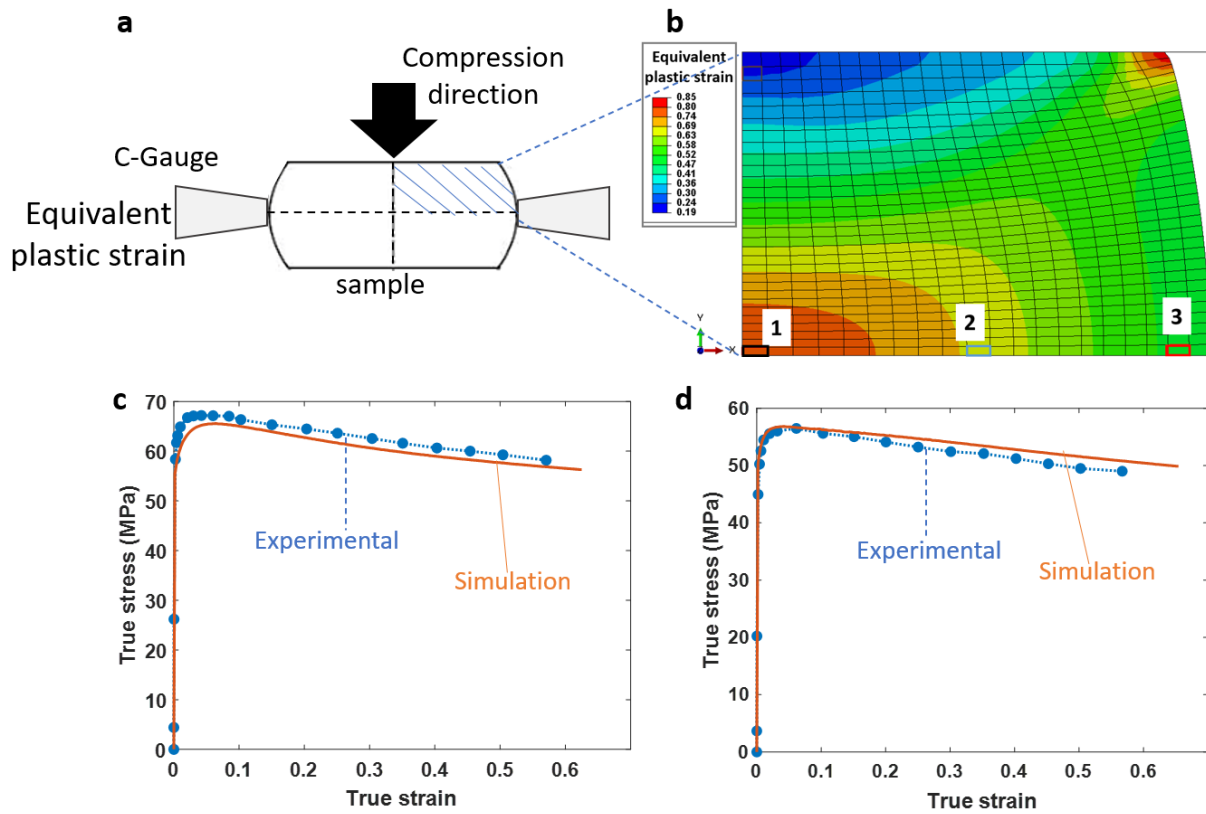


Fig. 7.5 (a) Schematic illustration of the middle section stress-strain measurement by C-gauge, FE simulation area is labelled with blue lines; **(b)** the FE modelled equivalent plastic strain distribution at a strain level of 0.58 under 420 °C, 0.05 s⁻¹, selected three points in the middle section for stress-strain calculation; and FE simulated and experimentally measured stress-strain curves at **(c)** 380 °C, 0.05 s⁻¹ and **(d)** 420 °C, 0.05 s⁻¹, respectively.

The macroscopic response of the developed FE model is further validated by comparing the barrelling contour between FE and experimental measurement for the two forging cases. The comparison data is presented in Fig. 7.6. Fig 7.6(a) shows a workpiece hot forged at 420 °C, 0.05 s⁻¹. Its middle section and the general barrelling phenomenon are exhibited at the side edges. The FE simulated forged half workpiece is shown in Fig. 7.6(b). Quantitative analyses were undertaken by comparing the radius along the cylindrical axis of the workpiece between the ones experimentally measured by vernier calliper and FE modelled displacement results, as shown in Fig. 7.6(c) and (d). In Fig. 7.6(c), the experimental and modelled radius profiles are virtually identical. For the 420 °C, 0.05 s⁻¹ case, as shown in Fig. 7.6(d), even though there is a slight deviation near the top surface of the workpiece, in general, the FE results are consistent with the experimental data. Therefore, the material's macroscopic behaviour has been accurately captured in the developed FE model, which indicates the applied loading condition, boundary conditions, as well as the material model, mesh type and friction condition are appropriate.

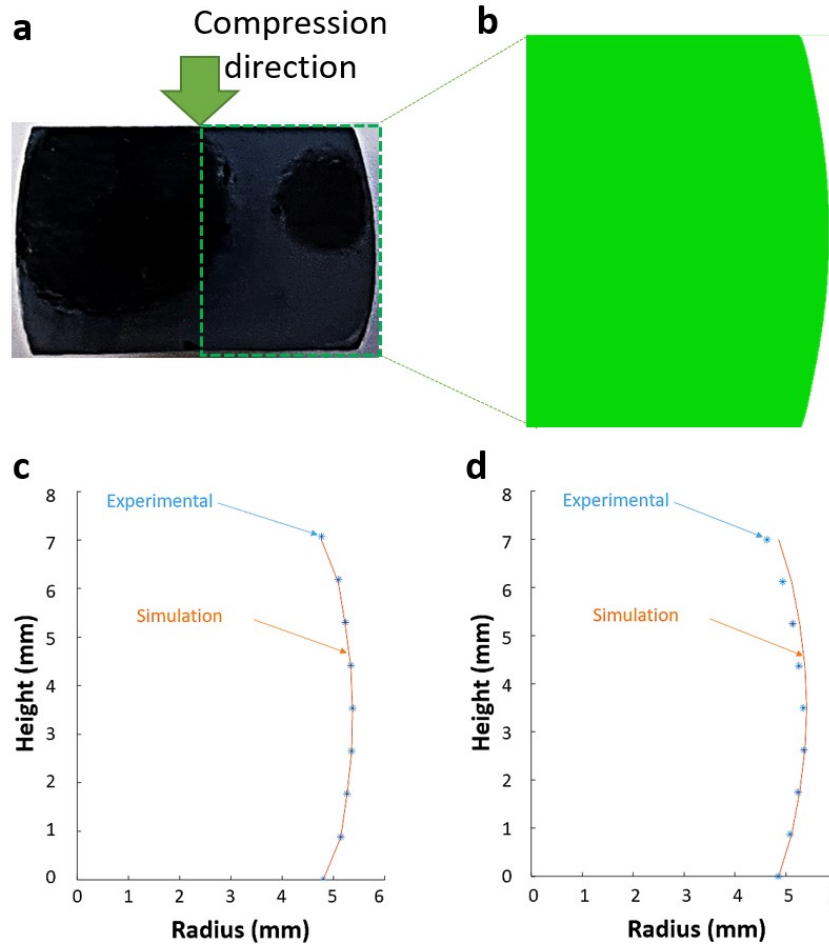


Fig. 7. 6 Comparison of the barrelling contour of (a) experimentally forged, and (b) FE half model at 420 °C, 0.05 s⁻¹, and comparison of the final forged geometric profile under (c) 380 °C, 0.05 s⁻¹, and (d) 420 °C, 0.05 s⁻¹.

7.4.2 Microscopic behaviour and validation

To validate the accuracy of microstructure prediction of the FE simulation, three regions at the top centre, the middle centre and the middle edge of hot forged workpieces are selected, as schematically illustrated in Fig. 7.7, to compare the experimental and numerical results. These three regions represent the largest, lowest, and medium plastic deformation occurring in the hot-forged workpieces. The experimentally EBSD scanned three regions (1 mm x 1 mm) on the hot-forged workpieces are indicated as black squares in Fig. 7.7(a). Map averaged values are used for subsequent quantitative analyses. At the same time, the three corresponding simulated regions are highlighted with white rectangles in Fig. 7.7(b). The following subsections will present the comparison of the experimental and numerical results in these three regions in terms of GND density, LAGBs and HAGBs.

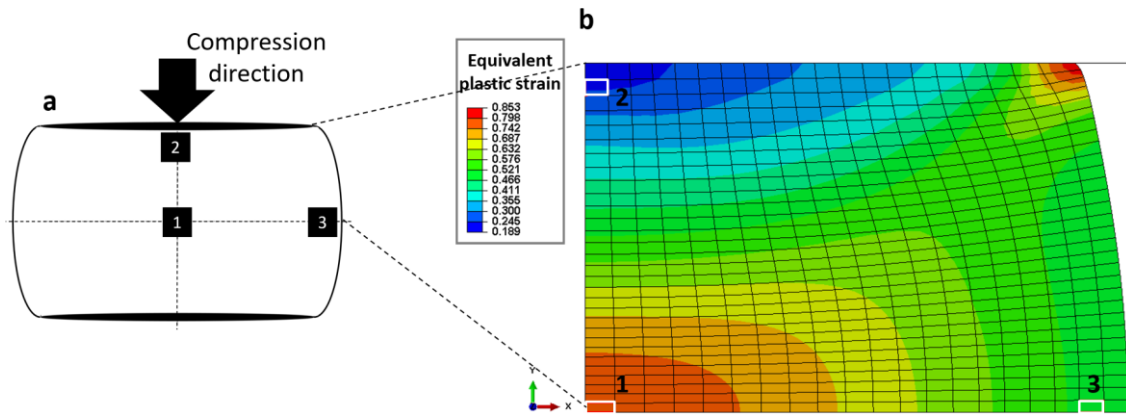


Fig. 7. 7 (a) Sample cutting scheme of the three selective positions for EBSD scanning is in dark colour and (b) the three correspond positions in ABAQUS model in white colour.

7.4.2.1 GND density

The FE predicted GND density contour maps of the forged workpieces under 0.05 s^{-1} at 380°C and 420°C , are shown in Fig. 7.8(a) and (b), respectively. The general distribution of GND density is similar for both forging cases. However, the overall magnitude of GND density drops as the deformation temperature increases from 380°C and 420°C , which is reasonable, due to the rise of the recovery process with temperature.

A quantitative comparison is undertaken between the numerical and experimental results at the three selected regions for both forging cases, see Fig. 7.8(c) and (d). As GND density rapidly reaches its saturation state at the very early stage of deformation, the variation in GND density distribution at the end of plastic deformation is expected to be relatively small, which is confirmed in Fig. 7.8(c) and (d). Nevertheless, the rank of GND density values should still be: region 1, region 3 and region 2, according to their plastic deformation levels. In both cases, the simulated results predict the expected trends.

Experimentally, the highest GND density values are always found at the edge of the middle section of the workpieces (region 3, medium plastic deformation). However, its trends in regions 1 and 2 are reasonable that GND density in region 1 (highest plastic deformation) is higher than in region 2 (lowest plastic deformation). One of the possible explanations for the observed highest GND density measured at region 3 is due to the highest cooling rate at the free surface of the workpiece after the hot forging, which gives a longer recovery time in regions 1 and 2 near the cylindrical axis and results in lower dislocation density. The workpiece cooling process was not considered in the current simulation. The other possible reason could be due to the operation of different dislocation accumulation mechanisms under uniaxial deformation and multiaxial deformation modes. The proposed dynamic

recrystallisation constitutive model is primarily based on uniaxial hot compression conditions, where underlying dislocation slip systems are activated and operated by compression stress, *i.e.* region 1, while the loading stress in region 3 has both compression and shear stress generated by friction strain. Recent research [42] suggests the multiplication of dislocations under the mix of compression and shear stress is complex and its underlying mechanism currently is still elusive.

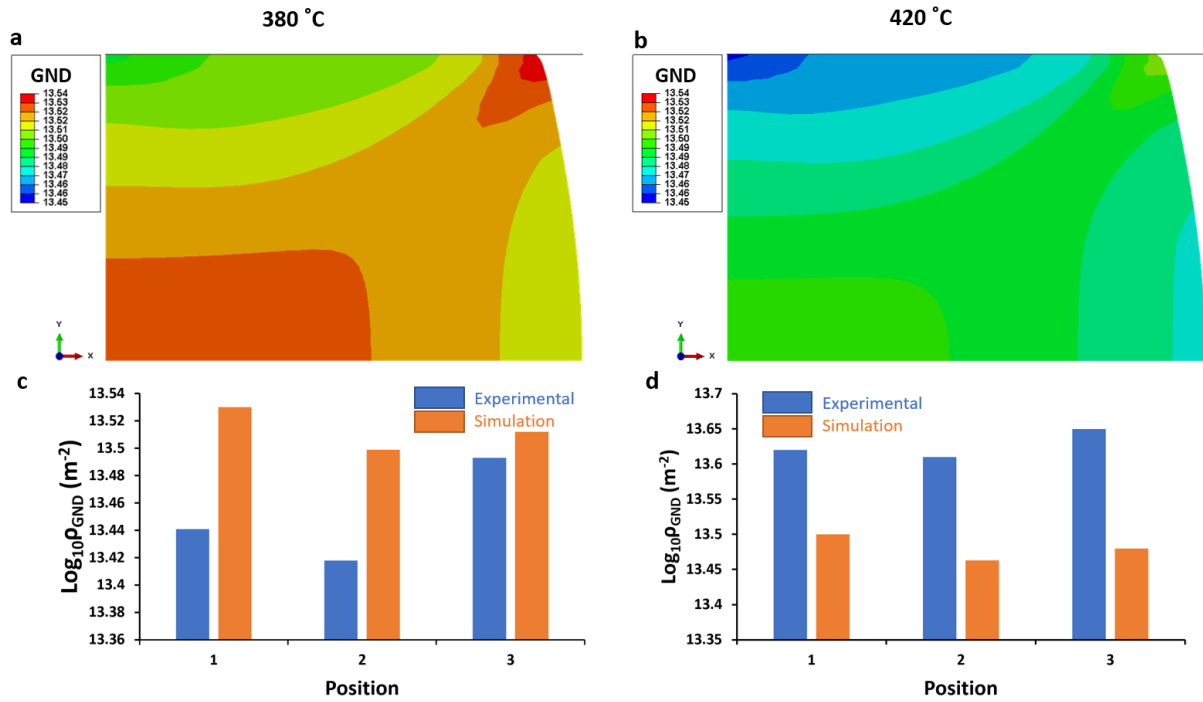


Fig. 7. 8 (a-b) Simulated GND density contour maps at 380 °C and 420 °C 0.05 s⁻¹, respectively and (c-d) the GND density of three selected regions at 380 °C and 420 °C 0.05 s⁻¹. Experimental results are in blue, while simulation results are coloured in orange.

7.4.2.2 LAGB area fraction validation

The LAGBs exhibit slightly different contour patterns to GND density mainly due to the LAGB-HAGB transformation process. FE simulation predicts lower LAGBs at higher deformation temperature (420 °C), as displayed in Fig. 7.9(a) and (b), for which the lower LAGBs stem from the higher diffusion rate and hence the recovery rate. The rank of LAGBs among the three selected regions remains the same as GND density and plastic strain in region 1, region 3 and region 2. The quantitative evolution of these three regions is reported in Fig. 7.9(c) and (d). The general trends agree well between the experimental and simulation data, which suggests the high accuracy of the FE model for predicting LAGB distribution in hot forged parts.

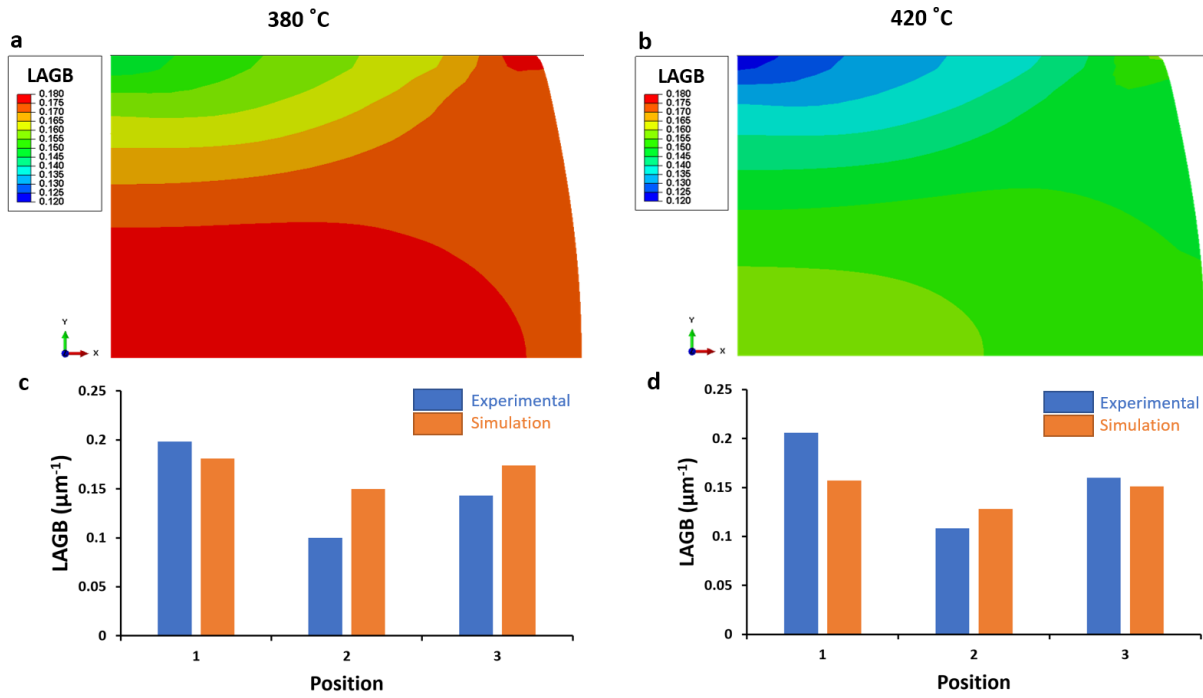


Fig. 7.9 (a-b) LAGB area per unit volume contour maps at 380 °C and 420 °C 0.05 s⁻¹, respectively and (c-d) the experimental and simulation data comparison in three selected regions in the forged workpieces; experimental results are denoted by blue colour and simulation results are highlighted in orange.

7.4.2.3 HAGB area fraction

Finally, the HAGB (grain size) FE prediction is compared with experimental measurement, as shown in Fig. 7.10. The general trends as well as the magnitudes between the experimental and simulated results in both forged cases agree well. The highest HAGB area per unit volume is found and predicted in region 1, while the lowest HAGBs exist and are accurately predicted in region 2. Moreover, comparing the lower temperature forged case with the higher temperature one, simulation accurately predicts that higher HAGBs are present in the higher temperature forged case. This is due to the higher diffusion rate and hence the LAGB-HAGB transformation rate at higher temperature. Therefore, the developed FE model can accurately predict the distribution of HAGBs as well as the diffusion effects in hot forging.

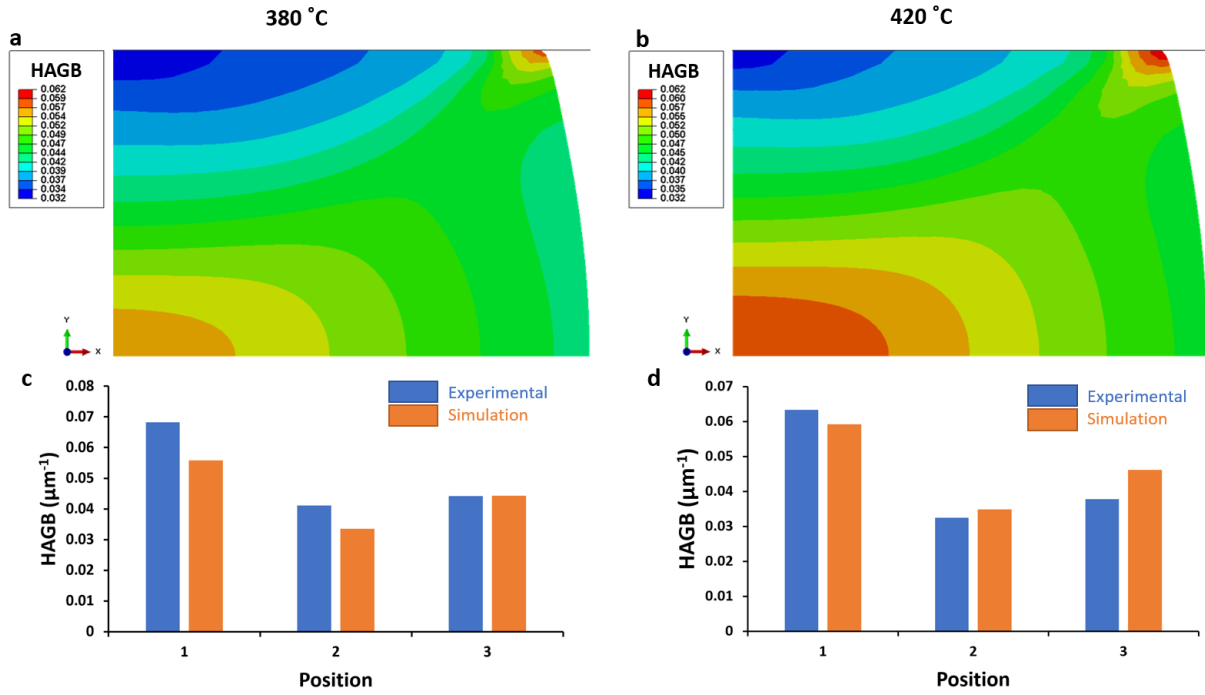


Fig. 7.10 (a-b) HAGB area per unit volume contour maps at 380 °C and 420 °C 0.05 s⁻¹, respectively and (c-d) the comparison of experimental and simulated results at the three selected regions; the experimental data are coloured in blue colour, while orange colour is used to colour FE predicted results.

Based on the above validation, the developed FE simulation with the proposed material model seems to be able to predict accurately the strain accurately, LAGBs and HAGBs in isothermally open die hot forged AA7050 parts, in addition to the overall forged shape. The successful validation of the FE forging model at both macro and microscopic scales suggests an accurate adequate description of the relationships between LAGBs and HAGBs with strain and diffusion processes in the material model. GND density can be accurately predicted to some extent. Some further study is needed to identify the reasons for some dispersed results.

Although hot forging simulation has been developed and proved to be reasonably accurate, which forms the basis of the forging model framework, it should be noted that isothermal open die forging is probably one of the simplest forging processes. In practice, most of the aerospace AA7050 components are produced through multistage close die forging [174], where the material flow within dies is considerably more complex; temperature distribution and heat transfer between workpieces and dies are complicated [175]; also, friction coefficient varies significantly as the lubricant changes at various temperature and stress. These factors need to be individually studied and carefully validated to apply the developed model to a wider range of practical forging applications.

The established processing model has demonstrated to be able to accurately simulate both material flow and microstructure evolution in the hot forging process of AA7050. Consequently, the optimal forging conditions, such as forging temperature, speed and die design for material flow can be efficiently determined through FEA simulation. This will result in a significant saving in lead time, high material wastage and manufacturing costs to the aircraft companies, especially for large or extra-large components.

7.5 Summary

An isothermal open die hot forging FE simulation, incorporating the proposed physically-based, unified viscoplastic constitutive model through user defined subroutine VUMAT, has been successfully developed. To validate the developed model, hot forging processes at 380 °C and 420 °C, 0.05 s⁻¹ have been simulated and experimentally conducted. The following conclusions have been reached.

- i. The developed FE hot forging simulation can successfully show the distribution and evolution of stress-strain, total dislocation density, GND density, LAGBs and HAGBs in the forged workpieces.
- ii. The macroscopic deformation of the forged workpieces has been accurately predicted by the model by comparing with experimental stress-strain and radius variation along the loading axis.
- iii. The FE prediction of LAGBs and HAGBs distribution within the hot forged parts has been proved to be very accurate, consistent well with experimentally EBSD measured values.
- iv. Experimentally EBSD measured GND density has the highest value at the edge of the middle section, which is different from FE simulation and requires further investigation.

Chapter 8

Conclusions and future work

8.1 Conclusions

The overall aim of accurately predicting the stress-strain relation and microstructural evolution of AA7050 in hot forging has been successfully achieved in this study. Experimental evidence and new understandings of microstructural evolution under a typical hot forging window range (strain level: 0-0.58, strain rates: 0.0005 to 5 s⁻¹ and temperatures: 380-460 °C) have been obtained through Gleeble uniaxial compression testing and EBSD GND density, LAGB and HAGB mapping. These understandings have allowed a set of new dynamic recrystallisation constitutive equations to be proposed to model AA7050's viscoplastic stress-strain behaviour as well as microstructure evolution. This model has been implemented in FE software, ABAQUS, using a VUMAT subroutine to simulate open die hot forging processes, which have been subsequently compared and validated with experimental data. The specific conclusions from this study can be drawn as follows.

8.1.1 Experimental

- i. A strong viscoplastic behaviour and temperature-dependent behaviour of AA7050 has been observed under the hot forging conditions. If the strain rate dropped from 0.5 to 0.0005 s⁻¹, the flow stress was reduced by more than 70% at a constant temperature of 420 °C. When the temperature was increased from 380 to 440 °C, the flow stress was reduced by around 27% at a constant strain rate of 0.05 s⁻¹. Dynamic recovery and recrystallisation of AA7050 leads to the softening of the material during deformation at elevated temperature.
- ii. When the plastic deformation is small, a string-like dislocation structure is firstly formed in some of the grains. As the deformation increases, this string-like dislocation structure evolves into a more homogenous fine cell-like dislocation structure at 420 °C and a strain rate of 0.05 s⁻¹.

- iii. From the strain level of 0.15-0.58, the misorientation angle of LAGB increased slightly from 4° to 5°, but the area fraction of LAGB per unit volume increased markedly from 1.6×10^5 to $1.9 \times 10^5 \text{ m}^{-1}$ at 420 °C and a strain rate of 0.05 s^{-1}
- iv. Some of the LAGBs were transformed into ultra-fine grains formed near grain boundaries, resulting in a rapid increase in the area fraction of HAGB per unit volume from 5×10^3 to $15 \times 10^3 \text{ m}^{-1}$. However, the average misorientation angle of HAGBs (around 42°) did not vary markedly at 420 °C and a strain rate of 0.05 s^{-1}
- v. The strain softening effect is found due to the increasing of HAGBs to facilitate the GB sliding, overtaking the hardening effects of dislocation density, LAGB and HAGB.
- vi. The GND density, LAGB and flow stress exhibit near-linear relationships with the Zener-Hollomon parameter. High dislocation density and more LAGBs are formed at lower temperature and higher strain rate.
- vii. The dislocations in GND density and the LAGB area are found to be the dominating factor governing the flow stress in the tested ranges of strain rates and temperatures, regardless of the change of HAGBs.
- viii. The relationship between HAGB area per unit volume and the Zener-Hollomon parameter is complex, comprising three regions: namely DRV, CDRX and DDRX. The boundary values of $\ln(Z)$ for AA7050 in this study are less than 20 (0.0005 s^{-1} , 420 °C) for DRV, 20-27.5 for CDRX, and larger than 27.5 (0.5 s^{-1} , 420 °C) for DDRX.
- ix. The largest HAGB area per unit volume within the CDRX region is found at the $\ln(Z)$ value of 22.5 (0.05 s^{-1} , 420 °C), where a relatively homogenous grain structure is observed due to the balance of the grain boundary growth and the transformation from LAGBs.
- x. The largest HAGB area per unit volume within the DDRX region is found at the $\ln(Z)$ value of 29.5 (5 s^{-1} , 420 °C), where a large number of fine recrystallised grains are found within grains with certain orientations.

8.1.2 Modelling and simulation

- i. A diffusion-driven (dislocation movement) constitutive model has been proposed to model the viscoplastic behaviour of AA7050 under typical hot forging conditions, based on the experimental observations in the previous chapters.
- ii. The GND density, LAGB and HAGB behaviour driven by athermal and diffusion processes have been modelled explicitly. In the model, dislocation density accumulation, recovery, and transformation into GND density and LAGBs have been considered. Meanwhile, the LAGB formation from dislocation density and the LAGB-HAGB transformation process have been taken into account.
- iii. The GND density has been introduced into the constitutive model as a state variable, such that the GND density can be verified through EBSD measurement.
- iv. The CDRX process has been modelled based on the LAGB population and LAGB-HAGB transformation rate. The S-shaped HAGB curve as a function of diffusion level has been correctly modelled.
- v. The proposed model can accurately capture the general trends of stress-strain, GND density, LAGB and HAGB behaviour as a function of strain and diffusion level, expressed as the Zener-Hollomon parameter.
- vi. An isothermal open die hot forging FE simulation, incorporating the proposed physically-based, unified viscoplastic constitutive model through user-defined subroutine VUMAT, has been successfully developed.
- vii. The developed FE hot forging simulation can successfully show the distribution and evolution of stress-strain, total dislocation density, GND density, LAGBs and HAGBs in the forged workpieces.
- viii. The macroscopic deformation of the forged workpieces has been accurately predicted by the model by comparing the stress-strain and radius variation along the loading axis with experimental results.
- ix. The FE prediction of LAGBs and HAGBs distribution within the hot forged parts has been proved to be very accurate, agreeing well with experimentally EBSD measured values.
- x. Experimentally EBSD measured GND density has the highest value at the edge of the middle section, which is different from FE simulation and requires further investigation.

8.2 Research limits and considerations for future work

Some interesting findings have been obtained in this study. To further explore these, new investigations will be required in future studies. Also, due to the defined research scopes of the current study, to generalise the obtained findings and the developed model to generate a larger impact, the following suggestions are provided for future work:

- i. As the LAGB to HAGB transformation occurs continually, using an interrupted compression test method at various strain levels, to deduce the process based on quantitative analysis, may not be the best route. *In-situ* hot compression/tensile test within Tescan EBSD can be conducted to provide direct evidence by observing such transformation process in a selected region of interest.
- ii. The current dynamic recrystallisation study only focuses on the uniaxial compression test. However, as discussed, compression and shear deformation may have different effects on dynamic recrystallisation kinetic. To separate the influence of compression, tensile and shear stress on dynamic recrystallisation, hot compression, torsion and compression-torsion tests can be carried out under the same thermal-mechanical conditions.
- iii. Although 2D simulation has the advantages of saving computational time and cost with acceptable predicting accuracy, it would be of interest to further explore the 3D process modelling, especially for temperature and friction that are no longer uniform along the third direction, which is closer to practical hot forging processes.
- iv. The current model is implemented in the FE software, ABAQUS, via the VUMAT subroutine, which can yield the results with good accuracy. However, ABAQUS is not designed to handle large plastic deformation due to mesh crash or non-convergence issues. It would be recommended to implement the proposed material model into FE forming software such as Qform, which handles large plastic deformation effectively and efficiently through automatic element remeshing algorithm. This allows more complex forging with large deformation to be simulated with high efficiency and accuracy.
- v. The current FE hot forging model is validated against open die hot forging samples. To generalise the current model for practical hot forging uses, it is necessary to

validate against large or extra-large AA7050 components where the variation of strain, dislocation density, LAGB and HAGB would be more significant than smaller ones.

- vi. Assuming uniform temperature distribution and friction coefficient in the current study is not unreasonable as conditions are well controlled under in-lab conditions. However, to generalise the proposed model for practical uses, it is necessary to further study the actual friction factor under practical operating conditions and temperature variation during the whole forging process.
- vii. Once the proposed model is validated under more practical conditions, it would be very interesting to carry out a hot forging optimisation process through FE modelling, *i.e.* ‘digital twin’, where forging operational parameters, dies and lubrication selection can be optimised.
- viii. The optimal forging parameters, such as forging temperature and forging speed, die-design for optimal material flows, etc., could be determined in a more effective and more efficient manner by implementing the recommendations given above for the further improvement of the FE models of the forging process.

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Appendices

A. Geometric effect on grain size reduction

The geometric effect on grain size reduction is considered here due to the presence of large, elongated grains and relatively large deformation to further elongate these grains. The simplified process is shown in Fig. A1, when the grains are large and elongated, only partial of the large grains can be captured within the EBSD maps. After the plastic deformation to 50% of the initial height, more grains are squeezed into the EBSD maps without the need for recrystallisation. The geometric effect is taken into account by assuming that the large, elongated grains have rectangular shapes, with thickness t_0 , and the perimeters of these rectangular grains are conserved in 2D. The EBSD map size has the length, L , the height, H and the boundary length of small grains, X_0 . The initial grain boundary length within the map area equals to $L \times (H / t_0) + H + X_0$. After the deformation of ϵ , the length of the rectangle, L , does not change, the width/thickness, t , is reduced to $t_0 \exp(-\text{abs}(\epsilon))$. As a result, the original grains are further elongated along the horizontal axis, as seen in Fig. A1, and more grains are compressed in the region of interest. The total grain boundary length is $L \times [H/(t_0 \exp(-\text{abs}(\epsilon)))] + H + X_0 \times \exp(\text{abs}(\epsilon))$.

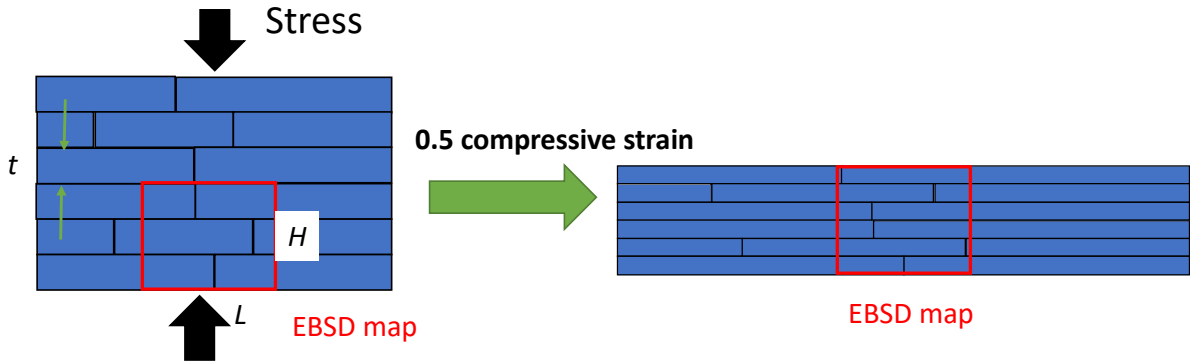


Fig. A1. The schematic illustration of geometrical effect on grain size reduction

In this study, the EBSD map, L equals to $H = 1$ mm and $X_0 = 10.336$ mm. The $t_0 = 0.0625$ mm estimated from EBSD initial map.

B. Statistical sampling

The statistical representativity of EBSD sampling is examined in this section by acquiring three 1 mm × 1 mm maps at three locations in the fully annealed sample, as displayed in Fig. B1. The GND density, misorientation angles of HAGB and LAGB analyses were undertaken, and their histograms and the mean and standard deviations are reported in Fig. B2 and Table B1, respectively. They confirm the reasonable consistency between the statistical results of the maps, which indicates that the sampling size and data is statistically meaningful.

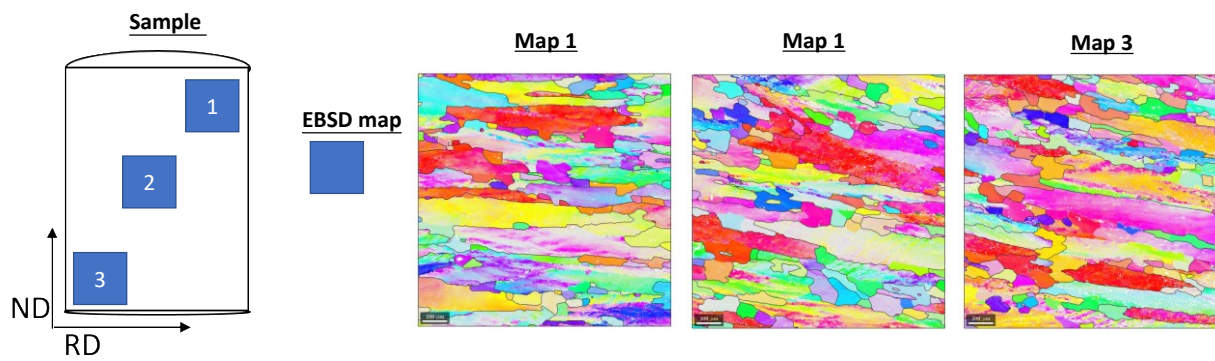
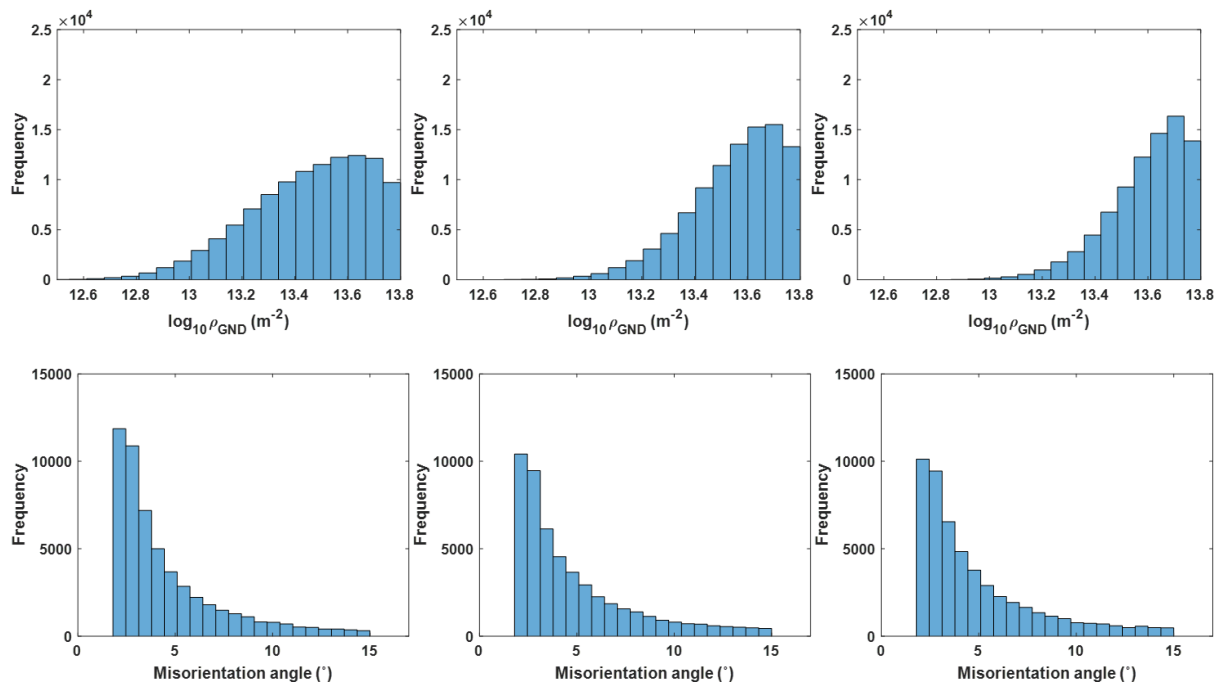


Fig. B1. The three EBSD map locations on the sample and their orientation maps



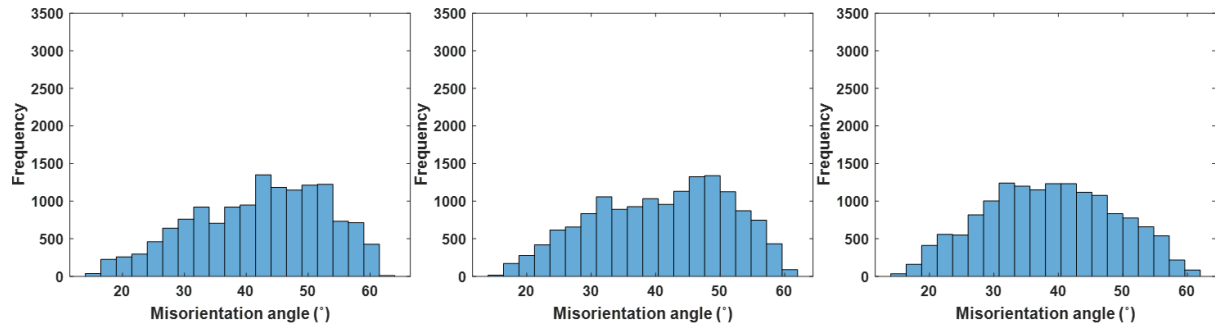


Fig. B2. Histograms of dislocations density, the misorientation angles of LAGB and HAGBs calculated from the three EBSD maps in Fig. A2.

Table B1. Mean and standard deviations of microstructure variables based on EBSD map 1-3

EBSD Map	1	2	3
GND density ($\log_{10} \text{ m}^{-2}$)	13.5 ± 0.2	13.6 ± 0.2	13.6 ± 0.2
LAGB points	116122	111662	112672
Misorientation on LAGB	$4.5^\circ \pm 2.8^\circ$	$4.8^\circ \pm 3.0^\circ$	$4.9^\circ \pm 3.0^\circ$
HAGB points	28336	29824	29766
Misorientation on HAGB	$42.1^\circ \pm 10.6^\circ$	$40.8^\circ \pm 10.4^\circ$	$38.6^\circ \pm 10.0^\circ$

C. Precipitates effects

The precipitates prior to and post the hot deformation were characterised using backscatter diffraction imaging. The images at various magnifications were acquired and are shown in Fig. C1. The data confirms that the general distribution of precipitates did not vary extensively during the hot compression.

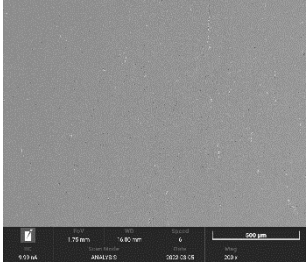
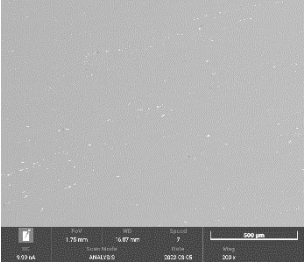
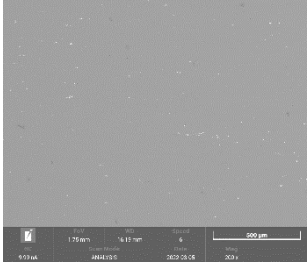
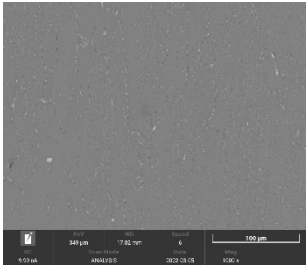
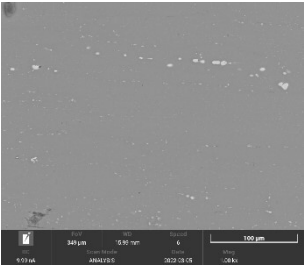
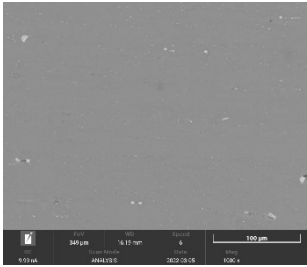
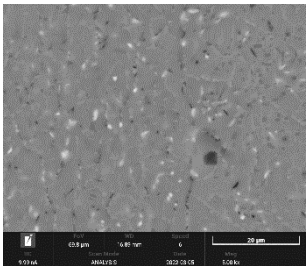
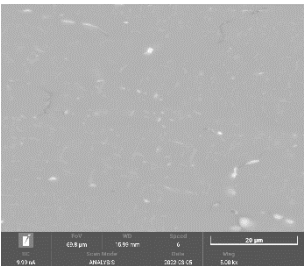
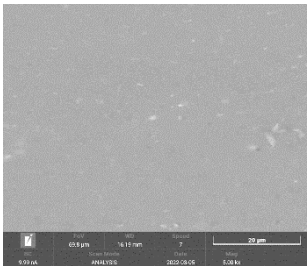
Magn.	Sample condition		
	Annealed	0.4 strain	0.58 strain
x200			
x1k			
x5k			

Fig. C1 the backscatter diffraction images of the undeformed and deformed samples at various magnifications. The white regions are precipitates.

D. Matlab script

```
clear;
close all
%% ***** Data input *****

testdata=readmatrix('Jiangshuai7050-0dot05');
seq = 4;

% seq1: T=380,ep=0.05;
% seq2: T=400,ep=0.05;
% seq3: T=420,ep=0.05;
% seq4: T=440,ep=0.05;

exstrain(:,1)=testdata(:,1);
exstress(:,1)=testdata(:,2);
exstrain(:,2)=testdata(:,3);
exstress(:,2)=testdata(:,4);
exstrain(:,3)=testdata(:,5);
exstress(:,3)=testdata(:,6);
exstrain(:,4)=testdata(:,7);
exstress(:,4)=testdata(:,8);
exstrain_420=testdata(:,9);
rho_g_420=testdata(:,10);
exstrain_420_gz=testdata(:,11);
SLAB_420=testdata(:,12);
exstrain_420_g=testdata(:,11);
SHAB_420=testdata(:,13);
%% ***** Material Parameters *****

max_strain=0.6; % Experiemtal engineering strain
E = 71.7e3;      % Elastic modulus, Mpa
Qb = 134.0;      % Active energy for grain boundary diffusion, KJ/mol
KG= 5.4e-4;      % for the Shear modulus, G=G0*exp(-KG*T)
R = 8.314;       % Gas constant, J/mol
%% ***** Initial Values for calculation*****

ep0=0.0;         % initial plastic strain rate
rho_t0=13.47;    % initial dislocation density
rho_g0=13.37;    % initial GND density
S_hab0=0.0271;   % initial aera of the HABs
S_lab0=0.0696;   % initial aera of the LABs

Wh_rho0=3.4;     % initial work hardening stress, wh-->Work Hardening dislocation
Wh_lab0=2.4;     % initial work hardening stress, wh-->Work Hardening LAGB
Wh_hab0=3.5;     % initial work hardening stress, wh-->Work Hardening HAGB

stress0=0.0;     % initial equivalent stress
%% ***** Model Parameters for Tuning *****

A1=7.2e-4;
A2=0.114;
B1=91;
B2=9.3;
beta1=0.8;
beta2=0.92;
c1=475;
c2=0.99;
```

```

d1=97;
d2=0.19;
d3=0.24;
rho_t1=14;

%% ***** Test Conditions *****
strain_rate_1=0.05;      % strain rate
dt_1=0.002;             % time step (Total_time=max_srain/strain_rate_1)
t1=380;

strain_rate_2=0.05;      % strain rate
dt_2=0.002;             % time step (Total_time=max_srain/strain_rate_1)
t2=400;

strain_rate_3=0.05;      % strain rate
dt_3=0.002;             % time step (Total_time=max_srain/strain_rate_1)
t3=420;

strain_rate_4=0.05;      % strain rate
dt_4=0.002;             % time step (Total_time=max_srain/strain_rate_1)
t4=440;
%% ***** Matrix Initiation *****

strain_rate=[strain_rate_1,strain_rate_2,strain_rate_3,strain_rate_4];
t=[t1,t2,t3,t4];
T=[t1+273,t2+273,t3+273,t4+273];
dt=[dt_1,dt_2,dt_3,dt_4];

matrixSize=int64(max_strain/(strain_rate_1*dt_1)+1); % Be sure strain_rate*dt
keep the same!
% total steps: max_strain/(strain_rate_1*dt_1)
total_strain=zeros(matrixSize,seq);
total_time=zeros(matrixSize,seq);
total_strain(:,1)=0:(dt_1*strain_rate_1):(max_strain);
total_time(:,1)=0:dt_1:(max_strain/(strain_rate_1));
total_strain(:,2)=0:(dt_2*strain_rate_2):(max_strain);
total_time(:,2)=0:dt_2:(max_strain/(strain_rate_2));
total_strain(:,3)=0:(dt_3*strain_rate_3):(max_strain);
total_time(:,3)=0:dt_3:(max_strain/(strain_rate_3));
total_strain(:,4)=0:(dt_4*strain_rate_4):(max_strain);
total_time(:,4)=0:dt_4:(max_strain/(strain_rate_4));
D1=zeros(1,seq);
D2=zeros(1,seq);
D3=zeros(1,seq);
B3=zeros(1,seq);
k=zeros(1,seq);
EE=zeros(1,seq);

ep_dot=zeros(matrixSize,seq); % 1:Plastic strain rate
ep=zeros(matrixSize,seq);
rho_t_dot=zeros(matrixSize,seq); % 2:Dislocation density rate
rho_t=zeros(matrixSize,seq);
rho_g_dot=zeros(matrixSize,seq); % 3:GND density rate
rho_g=zeros(matrixSize,seq);
Wh_rho_dot=zeros(matrixSize,seq); % 4:Work hardening rate from dislocation
Wh_rho=zeros(matrixSize,seq);
Wh_lab_dot=zeros(matrixSize,seq); % 5:Work hardening rate from LAGBs
Wh_lab=zeros(matrixSize,seq);
Wh_hab_dot=zeros(matrixSize,seq); % 6:Work hardening rate from HAGBs

```

```

Wh_hab=zeros(matrixSize,seq);
S_lab_dot=zeros(matrixSize,seq);           % 7: LAGBs area evolution rate
S_lab=zeros(matrixSize,seq);
S_hab_dot=zeros(matrixSize,seq);           % 8: HAGBs area evolution rate
S_hab=zeros(matrixSize,seq);
stress_rate=zeros(matrixSize,seq);         % 9: Equivalent Stress rate
stress=zeros(matrixSize,seq);

for i=1:seq
    ep(1,i)=ep0;
    rho_t(1,i)=rho_t0;
    rho_g(1,i)=rho_g0;
    S_lab(1,i)=S_lab0;
    S_hab(1,i)=S_hab0;
    Wh_rho(1,i)=Wh_rho0;
    Wh_lab(1,i)=Wh_lab0;
    Wh_hab (1,i)=Wh_hab0;
    Wh(1,i)=Wh0;
    stress(1,i)=stress0;

    D2(i)=16.29552*d2*strain_rate(i)*exp(-Qb*60/(R*T(i)));
    D3(i)=d3*strain_rate(i)*exp(-Qb/(R*T(i)));
    B3(i)=beta2-d1*((strain_rate(i)^(0.01))*exp(-Qb/(R*T(i))));
    EE(i)=E*exp(-KG*T(i));
    k(i)=202.98-0.2905*T(i);
end
%%
for i=1:seq
    for m=1:(matrixSize-1)

        if (stress(m,i)-Wh_rho(m,i)-Wh_lab(m,i)-Wh_hab(m,i)-k(i))<0
            % 1. Plastic Strain rate
            ep_dot(m,i)=0;

            else
                ep_dot(m,i)=A1*(sinh(A2*(stress(m,i)-Wh_rho(m,i)-Wh_lab(m,i)-Wh_hab(m,i)-k(i))));
            end

            rho_t_dot(m,i)=c1*(1-rho_t(m,i)/14)*(0.05^(1.07))-D2(i)*rho_t(m,i);
            % 2. dislocation Evolution

            rho_g_dot(m,i)=rho_t_dot(m,i);
            % 3. GND Evolution

            S_lab_dot(m,i)=c2*rho_g_dot(m,i)*(ep_dot(m,i)^0.1)-S_hab_dot(m,i);
            % 4. LAGB Aera Evolution

            S_hab_dot(m,i)=D3(i)*S_lab(m,i);
            % 5. HAGB Aera Evolution

            Wh_rho_dot(m,i)=B1*((rho_g(m,i)/beta1)^(-0.5))*rho_g_dot(m,i);
            % 6. Work-hardening rate from GND

            Wh_lab_dot(m,i)=B2*S_lab_dot(m,i);
            % 7. Work-hardening rate from LAGB

            Wh_hab_dot(m,i)=B3(i)*(S_hab(m,i)^(-0.5))*S_hab_dot(m,i);
            % 8. Work-hardening rate from LAGB

```

```

stress_rate(m,i)=EE(i)*(strain_rate(i)-ep_dot(m,i));
% 9. Stress rate
% *****Update the variables values*****
stress(m+1,i)=stress(m,i)+stress_rate(m,i)*dt(i);
% Equivalent Stress
ep(m+1,i)=ep(m,i)+ep_dot(m,i)*dt(i);
    % Plastic strain

rho_t(m+1,i)=rho_t(m,i)+rho_t_dot(m,i)*dt(i);
    % Dislocation density

rho_g(m+1,i)=rho_g(m,i)+rho_g_dot(m,i)*dt(i);
    % GND density

Wh_rho(m+1,i)=Wh_rho(m,i)+Wh_rho_dot(m,i)*dt(i);
    % Work hardening dislocation

Wh_lab(m+1,i)=Wh_lab(m,i)+Wh_lab_dot(m,i)*dt(i);
    % Work hardening LAGB

Wh_hab(m+1,i)=Wh_hab(m,i)+Wh_hab_dot(m,i)*dt(i);
    % Work hardening HAGB

Wh(m+1,i)=Wh_rho(m+1,i)+Wh_lab(m+1,i)+Wh_hab(m+1,i);
    % Work hardening stresss

S_hab(m+1,i)=S_hab(m,i)+S_hab_dot(m,i)*dt(i);
    % Area of the HAGBs

S_lab(m+1,i)=S_lab(m,i)+S_lab_dot(m,i)*dt(i);
    % Area of the LAGBs
end
end

%% *****Plot Settings*****
str=strings(1,seq);
str_text=strings(1,seq);
sca_marker=['o','*','>','s','0','x','p','+'];
sca_marker_size=15;
col={[0.85 0.2 0.2],[0 0.85 0.1],[0 0.1 0.8],[0.63 0.13 0.94],[1 0.49804
0.14118],[1 0.5 0],[0 0.5 1]};
stress_point_size=45;
scatter_sz=75;
curvewidth=2;

title_font='Times New Roman';
titlefont_size=10;

xlabel_size=18;
xlabel_font='Times New Roman';

axisfont_size=14;
axis_font='Times New Roman';

legendfont_size=12;
legend_font='Times New Roman';

textfont_size=12;

```

```

text_font='Times New Roman';

unit='^oC';
blank='  ';

str_Measure='Measurement';
line_width_scale=0.4;

Draw_max = 1.02*max_strain;

%% *****Plot*****
figure(1);
for i=1:seq
    str(i)=[num2str(t(i)) unit];
    str_text(i)=[blank num2str(t(i)) unit];
    scatter(exstrain(:,i),exstress(:,i),sca_marker_size,sca_marker(i),...
        'MarkerEdgeColor',[0.4117 0.4117 0.4117],'LineWidth',0.5);
    hold on;
    splot(i)=plot(total_strain(:,i),stress(:,i),'-','Color',col{i});
    hold on;
end
xlabel('\epsilon');
ylabel('\sigma (MPa)');
axis([0 Draw_max 0 80]);
for i=1:seq
    x0=size(total_strain(:,i));
    x1=x0(1);
    text_set(i)=text(total_strain(x1,i),stress(x1,i),str_text(i));
end
set(splot,'LineWidth',1.5*curvewidth);
set(text_set,'Fontname',text_font,'FontSize',textfont_size);
hold off;

set(gca,'Fontname',axis_font,'FontSize',axisfont_size);
set(get(gca,'xlabel'),'Fontname',xlabel_font,'FontSize',xlabel_size);
set(get(gca,'ylabel'),'Fontname',ylabel_font,'FontSize',ylabel_size);
set(gcf,'color','w')

```

E. Pre heat treatment methods

To acquire optimal parameters in good agreement with the practical hot forging process, comparisons with various pre-heat treatments were by softening the samples before hot compression test. One sample was solution heat treated (475 °C for 1 hour and then water quenched to room temperature [176]. Another sample annealed (413 °C for 2 hours and followed by furnace cooling) which leads to a soft one. The last one was compressed straightaway, using the as-received material without any further heat treatment. They were heated up to 400 °C, held for 3 minutes and then compressed with a constant strain rate of 0.005 s⁻¹. As shown in Table E1 and Fig. E1, after annealed, the flow stress is around 8% less than the other two which means less forge force is needed to achieve similar shape for AA7050 components. The main reason is due to the coarsening of the precipitates which weakens the precipitation strengthening effect in the material. Meanwhile, to have consistent initial microstructure for all test samples and replicate the practical industrial process, pre-heat treatment annealing was applied before the hot compression tests.

Table E1. Peak flow stress from various pre-heat treatments.

Pre-heat treatment	Solution heat	Annealed	T7451
Peak flow stress	44.45	41.27	43.58

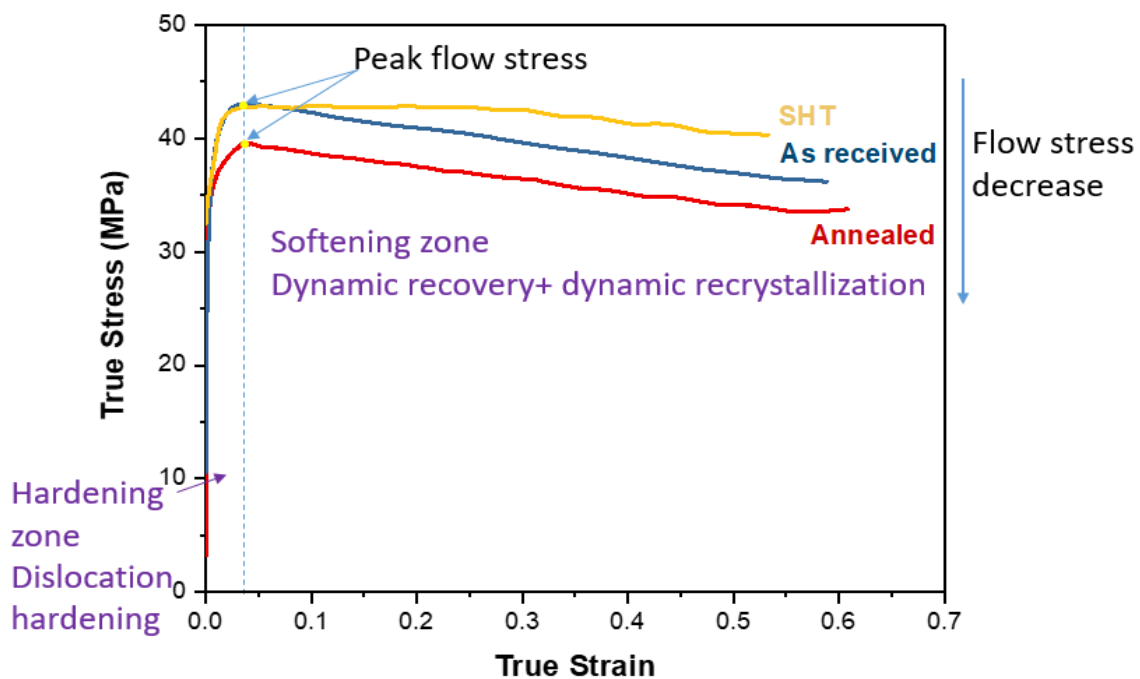


Fig. E1. Variation of flow stress of AA7050 for different pre-heat treatment under the same strain rate and temperature (400 °C and 0.005 s⁻¹).

F. Experimental data

Table F1. Data values from stress-strain curves at different temperatures.

Temperature (°C)	Softening ratio (MPa/100% strain)	Peak flow stress (MPa)	Normalised softening ratio
380	17.5	67.75	0.26
400	14.77	61.89	0.24
420	14.83	57.10	0.26
440	11.60	49.20	0.24

Table F2. Data values from stress-strain curves at different strain rates.

Strain rate (s ⁻¹)	Softening ratio (MPa/100% strain)	Peak flow stress (MPa)	Normalised softening ratio
0.5	18.14	82.60	0.22
0.05	14.77	57.10	0.26
0.005	9.09	35.27	0.26
0.0005	7.04	23.08	0.31

Table F3. Data values for $\ln(Z)$, peak flow stress, GND density, length of low-angle and high-angle grain boundary per unit area under different conditions.

Strain rate (s ⁻¹)	T (°C)	$\ln(Z)$	σ_p (MPa)	GND (m ⁻²)	LAGBs (μm ⁻¹)	HAGBs (μm ⁻¹)
0.0005	440	19.441	19.154	13.051	0.083	0.035
0.0005	420	20.220	23.100	13.058	0.085	0.035
0.005	420	22.522	35.300	13.177	0.165	0.058
0.05	460	23.307	37.038	13.531	0.159	0.053
0.05	440	24.044	49.200	13.324	0.187	0.051
0.05	420	24.825	57.100	13.543	0.232	0.051
0.05	400	25.651	61.900	13.484	0.224	0.050
0.05	380	26.529	67.800	13.621	0.206	0.048
0.5	420	27.127	82.600	13.555	0.241	0.046
0.5	380	28.833	96.882	13.645	0.313	0.030
5	420	29.430	111.150	13.629	0.349	0.066
5	380	31.136	123.192	13.661	0.486	0.044