

**Evaluation of superplastic deformation mechanism
for near- α TA15 Titanium Alloy: Microstructure
Evolution and Constitutive Modelling**

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A thesis submitted for the degree of Doctor of Philosophy of Imperial
College London and the Diploma of Imperial College London

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July 2020

DECLARATIONS

Declaration of originality

I hereby certify that the work presented in this thesis is the result of my own investigations, which were carried out at Imperial College London from March 2016 to July 2020, except where otherwise stated. Collaborations and contributions by other researchers have been truly and properly acknowledged.

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Abstract

TA15 (Ti–6Al–2Zr–1Mo–1V) is a type of near- α titanium alloy. It is famous for its application in the aerospace industry because of its high strength to mass ratio, high weldability, superior creep, and excellent creep resistance at high temperatures up to 550 °C. However, TA15 is challenging to be deformed and processed due to its low plasticity and narrow processing window. Hence, to exploit the superplastic deformation behaviour of TA15 in an effective manner, more fundamental research is necessary. There are limited comprehensive studies on the microstructure evolution in superplastic deformation of titanium alloys, and most studies are devoted to α/β type alloys. Until now, investigations focusing on the superplastic behaviour of a near- α alloy have not been reported in detail, especially regarding its hot deformation mechanism at a temperature above 900 °C with a low strain rate range of 0.0005-0.01s⁻¹. This has necessitated a more detailed microstructural study to understand the superplastic deformation mechanism of TA15 and provides important details for the constitutive modelling of the superplastic blow forming of the alloy. An attempt has been made in this investigation to achieve this employing microstructural characterization of specimens deformed in tension with temperature and strain rate conditions of 880°C/0.01s⁻¹, 900°C/0.01s⁻¹, 880°C/0.001s⁻¹, and 920°C/0.0005s⁻¹. Results show that TA15 alloy exhibits excellent superplastic behaviour for all the temperature and the corresponding strain rates. The microstructure remained equiaxed at all deformation conditions, which is a typical attribute of superplastic deformation. The maximum tensile elongation of 1450% is achieved for 880 °C and a strain rate of 0.001s⁻¹. Flow softening is observed the low elongations at temperature 880 °C and 900 °C with a strain rate of 0.01s⁻¹ while strain hardening is observed for the high elongations at deformation conditions 880 °C/0.001s⁻¹ and 920 °C/0.0005s⁻¹. The probable deformation mechanisms such as grain boundary sliding (GBS) under different deformation conditions are discussed in terms of grain changes, geometrically necessary dislocations (GNDs) and texture evolution. It is observed that the fraction of β -phase is accelerated during the hot deformation and contributed to the enhancement of superplasticity. The results show that the dominant mode of deformation changes at initial, intermediate, and final stages of deformation. Based on the obtained deformation behaviours, a physically based constitutive model combining a series of parameters is established, including grain size

evolution, dislocation density, void fraction, and dynamic recrystallization (DRX). The material constants for the proposed constitutive equations are determined using a Gradient-based optimization technique. Finally, to validate the accuracy of the established constitutive model, the finite element simulation of the superplastic gas-blow forming of TA15 sheet of the industrially relevant problem is performed.

Acknowledgment

First of all I am very grateful to my God for giving strength to complete this work. I would like to thank a number of individuals with whom I have worked and who have provided support, guidance, and friendship during my PhD journey. Special thanks to my supervisory team, Dr. Jun Jiang and Prof. Jianguo Lin, for their time, generous advice, suggestions, and feedback given in conducting this research work and preparing this thesis. Their combined expertise in the field of materials and engineering has immensely influenced the work in this thesis. Both have offered me much encouragement during the course of my study, and I have enjoyed our meetings and discussions. I am thankful to all the companies who allowed access for me to undertake my research, as well as the many individuals within them who freely gave their time, knowledge and expertise, which resulted not only in some fascinating insights, but also some valuable contributions to the ideas included in this thesis. Special thanks to Dr. Christopher Hopper of Intelligent Metal Forming Group at Imperial College London for his contribution to the proofread of this thesis and whose comments have strengthened the arguments and improved the clarity of the theory presented in this thesis. In last but certainly not least, I am greatly indebted to Schlumberger Foundation for awarding me Faculty for the Future Fellowship scholarship to sponsor my PhD.

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Nomenclature

ASTM	American Society for Testing and Materials
BCC	Body-centered cubic
BSE	Back scattered electron
CDRX	Continuous dynamic recrystallization
CEEQ	Equivalent creep strain
DB	Diffusion bonding
DDRX	Discontinuous dynamic recrystallization
DRX	Dynamic recrystallization
DRV	Dynamic recovery
EBSD	Electron backscatter diffraction
FEA	Finite element analysis
GBS	Grain boundary sliding
GND	Geometrically necessary dislocation
HAGBs	High angle grain boundaries
HCP	Hexagonal closest packed
IPF	Inverse pole figure
ISV	Internal state variable
LAGBs	Low angle grain boundaries
MDRX	Metadynamics recrystallization

MRD	Multiples of random distribution
MRU	Multiples of random unit
PDRX	Post-dynamic recrystallization
PF	Pole figure
RD	Rolling direction
SEM	Scanning electron microscope
SFE	Stacking-fault energy
SPF	Superplastic forming
SRX	Static recrystallization
TD	Transverse direction
TEM	Transmission electron microscope
TMP	Thermo-mechanical processing
WH	Work hardening

Chapter 1

Introduction

1.1. Superplasticity and Superplastic Forming (SPF)

Superplasticity is the ability of polycrystalline materials to exhibit a very high value of strain (can be even more than 2000%) without necking in a hot tensile test under specific conditions (Sieniawski and Motyka, 2007). Superplasticity can be exploited for the production of engineering components of complex structure (Langdon, 2010). Examples exist in aerospace, defence, biomedical, sport, and automotive sectors (Barnes, 2007). For example, as shown [Figure 1.1](#) under optimal superplastic conditions, elongations over 2000% have been achieved in what Pearson (Pearson, 1934) first described as metals deforming in a viscous manner, comparable to the behaviour of glass.

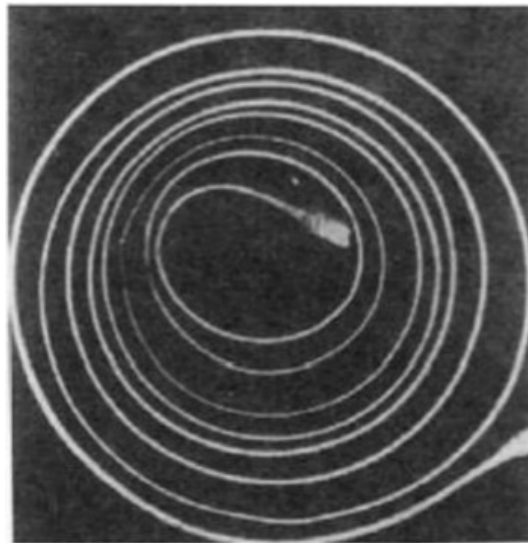


Figure 1. 1. Pearson 1934, Bi-Sn alloy (1950%) (Pearson, 1934).

1.2. Superplastic forming: technological application

Although the superplastic phenomena was discovered in the early 1920s, it was not until much later that superplasticity began to be exploited in the manufacturing of industrial components. Known as superplastic forming (SPF), traditionally, the SPF phenomenon has been employed in the production of deep, complex structures made from metal sheets – structures that cannot be manufactured using conventional press or stamp techniques. The most important and widely used SPF method of recent years is called the blow forming process. This superplastic forming method is illustrated in [Figure 1.2](#), which shows a cross-section of the dies and forming diaphragm. During the blow forming process, the dies and sheet material are normally maintained at the forming temperature, and the gas pressure is imposed over the sheet, causing the sheet to form into the lower die; the gas within the lower die chamber is simply vented to the atmosphere. The rate of pressurization in blow forming usually is established such that the induced strain rates in the forming sheet are maintained within the superplastic range. The rate of pressurization is determined either by trial and error or by the application of analytical modelling methods. This pressure is generally applied slowly rather than abruptly to prevent too rapid a strain rate and consequent rupturing of the material. To retain maximum ductility during SPF process, it is desirable to adjust the forming pressure so as to keep the strain rate within an optimum range throughout the whole forming process. Friction between the die and the sheet has been found to significantly affect the thinning of the component during the blow forming process (Chen et al., 2001). Severe thinning will lead to failure of the component. Therefore, to obtain the successful manufacture of SPF products, it is essential to have a better understanding of all the elements involved in the superplastic forming process.

1.3. Industrial Application of Superplastic forming

Superplastic forming (SPF) is being used increasingly by a range of industries across the world, *e.g.*, aerospace, high-performance automotive, architecture, medical, and defence. A typical example is shown in [Figure 1.3](#). This part has to be made from titanium for reasons of strength and corrosion. It would be impossible to form this part ‘in one shot’ using conventional cold forming processes, and hence an expensive and heavy riveted or welded structure would result.

Rolls-Royce uses SPF process to produce hollow titanium fan blades for their Trent series engine, which results in a weight saving of almost 50%. [Figure 1.4](#) shows the application for SPF process for fabricating the engine air intake lip skin of Boeing 777X.

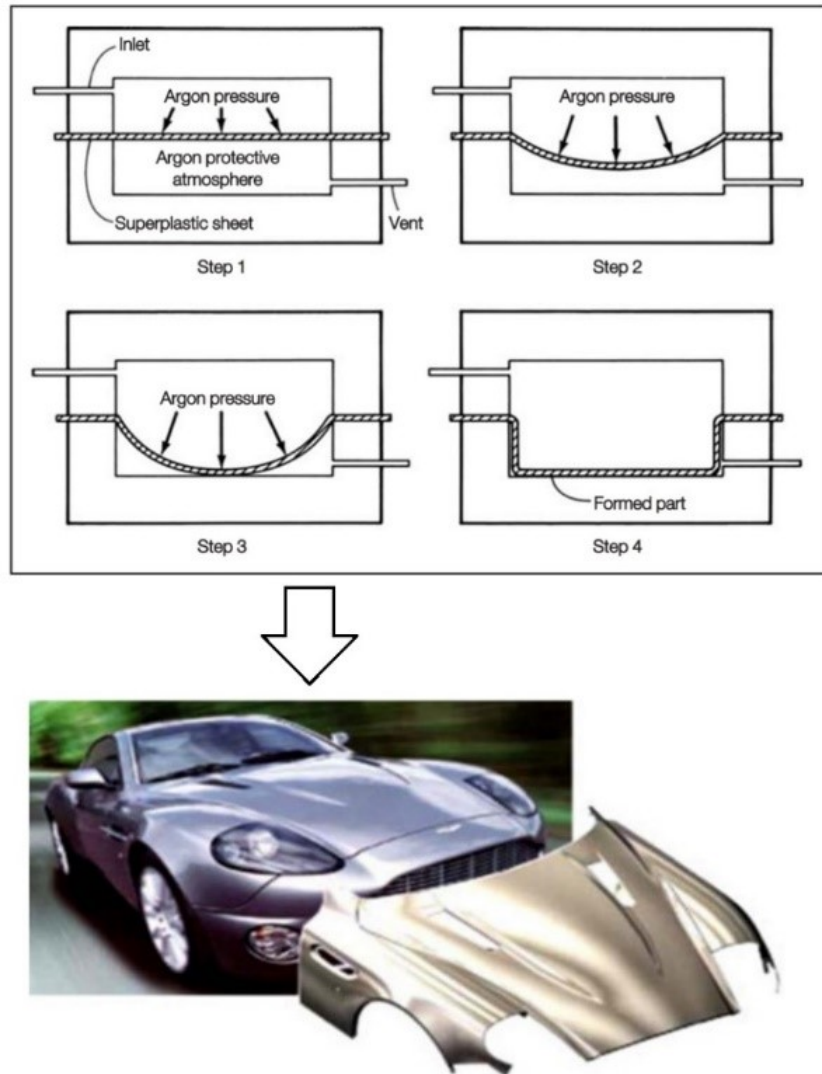


Figure 1. 2. Flow diagram of Superplastic Forming Process and Product (Semiatin et., al, 2006).

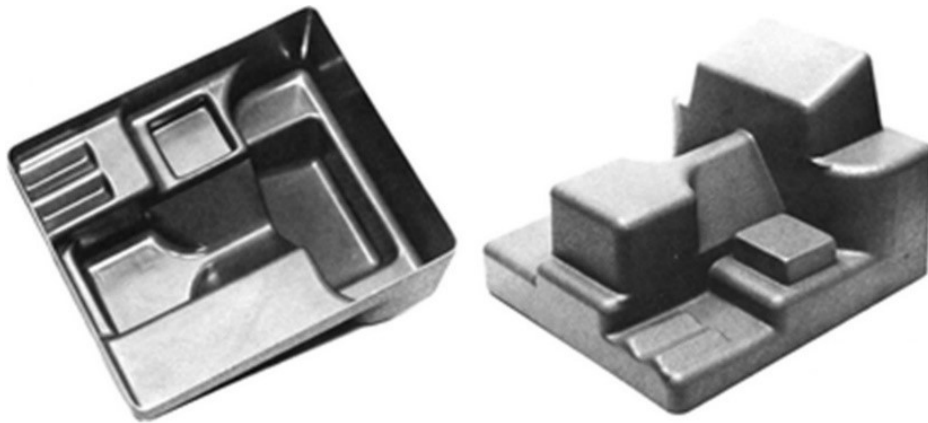


Figure 1. 3. Complex geometry made through SPF produced (Barnes, 2007).

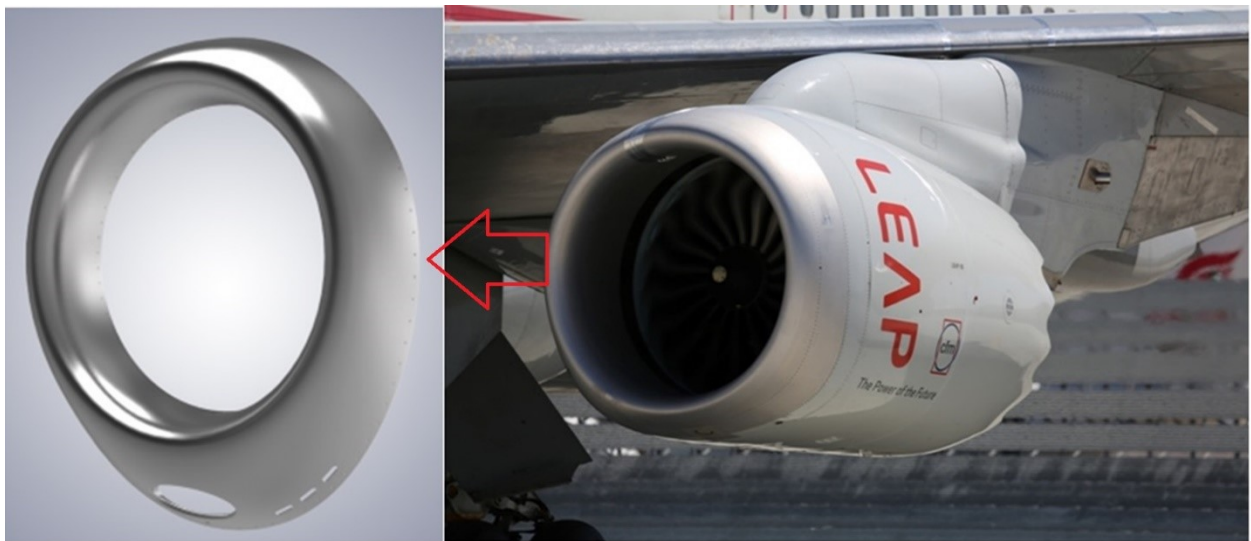


Figure 1. 4. Engine air intake lip skin of Boeing 777X produced by SPF

1.4. Titanium and Superplasticity

Superplastic Titanium alloys provide a combination of excellent mechanical properties of Titanium – high strength and corrosion resistance – with formability advantages of superplasticity. Additionally, the materials exhibit diffusion bondability in the same range

of temperature at which superplasticity occurs, enabling, manufacturing of even more unique architectures. Since its adoption, it has been shown capable of both reducing the cost and facilitating the fabrication of Titanium structures that were otherwise too expensive or too difficult to manufacture using traditional forming methodologies (Sieniawski and Motyka, 2007). Several industries embraced the technology, amongst the most notable example being the aerospace industry where superplasticity in Titanium alloy is used to manufacture hollow fan blades, unity body nacelles, keels, diffusion-bonded multi-sheet parts and structural panels amongst others (Beal et., al, 2006; Barnes, 2007).

1.4.1. Significance of Titanium alloys in the SPF industry

Titanium-based alloys are widely used for superplastic forming (SPF) of complex-shaped aerospace components and have proved to be a technically superior and cost-effective material in a wide variety of aerospace applications. Titanium alloy is one of the critical structural elements in aircraft fuselages and engines, and about 80% of titanium production is consumed by the aerospace industry (Cui, 2011). The future of titanium in the global aerospace market looks promising with opportunities in commercial aircraft, military aircraft, helicopter, general aviation, and regional aircraft (Lucintel, 2018). Recently, the increased use of composite materials in aerospace industry has further stimulated the demand for titanium alloys. It is because of the high compatibility of titanium alloys with composites due to its relatively low galvanic corrosion as compared to other rival aerospace alloys *e.g.* aluminum. For instance, an increase in composite usage from 11% in Boeing 777 (launched in 1994) to 50% in Boeing 787 (launched in 2009) caused an increase in titanium from 7% to 15%, respectively, in respective models. Similarly, the use of titanium alloy on military aircraft has reached 30%~40%. For example, titanium alloys occupy 42% of F-22 Raptor fighter body structure material, mainly for the manufacture of the wings, fuselage, skins, and load-bearing parts (Black and Kohser, 2019).

For example, in the United States, titanium alloys occupy 34% of F-15 fighter body structure material, mainly for the manufacture of the fuselage, wings, skins, and load-bearing components (Liu, 2008).

Similarly, the use of titanium alloy on civil aircraft can also account for about 10% to 15% of the airframe. In the Boeing 787 body, titanium alloy usage is 15%. The more significant part of the literature on superplastic deformation of titanium alloys pay particular attention to $\alpha+\beta$ type alloys, especially Ti-6Al-4V (Mikhaylovskaya et al., 2017). At the same time, near- α titanium alloys are also very attractive for aerospace applications, mainly due to their superior fatigue and creep properties at elevated temperatures. The α/β alloys such as Ti-6Al-4V are limited to in-service temperatures of about 350°C, but the standard near- α alloys are creep resistant up to about 500°C (Verlinden et al., 2007) – hence their superior thermal stability at elevated service temperature may satisfy the increasing efficiency of the turbojet engine (Li et al., 2019a). In recent times, studies on the superplasticity of near- α alloys have considerably increased (Mikhaylovskaya et al., 2017) due to rapidly growing applications of SPF of Ti alloys in aerospace engineering.

TA15 (Ti-6Al-2Zr-1Mo-1V) is a type of near- α titanium alloy and is famous for its application in the aerospace industry because of their high strength to mass ratio, high weldability, superior creep, and their ability to retain its mechanical properties at elevated temperatures up to 550 °C (Wang et al. 2005). As a potential candidate of materials for the aerofoil blade and disc, TA15 alloy has attracted strong interest recently. It is widely used to manufacture structural components in the aerospace industry (Wang et. al, 2010; Fan et. al, 2013; Yang et. al, 2015; Sun et. al, 2016; Fan et. al, 2017). For example, the large-scale integral components of TA15 titanium alloy with thin web and high rib complex shape (e.g. bulkhead) which can reduce the weight of equipment and improve the structural performance, has increasingly extensive application in aerospace field (Yang et. al, 2011; Zhao et. al, 2011). Compared with Ti-6V-4Al alloy, an additional small amount Zr and Mo elements are added to TA15 alloy (Ti-6.5Al-1Mo-1V-2Zr) to control the $\alpha\beta$ phase fraction and also do improve its corrosion and creep resistant. The large-scale integral components of TA15 titanium alloy with thin web and high rib complex shape (e.g. bulkhead) are

produced to reduce the weight of equipment and improve the structural performance (Yang et. al, 2011; Zhao et. al, 2011).

Most research on the superplasticity of titanium alloys has been devoted to the two-phase titanium alloys such as Ti–6Al–4V, however, only few studies have reported on the microstructure evolution, textural changes and superplastic deformation behaviors of near- α titanium alloys having equiaxed microstructure. As a classic near α titanium alloy, TA15 is difficult to be deformed and processed due to its low plasticity and narrow processing window, which leads to large inhomogeneous deformation in the workpiece, undesired thickness variation and poor structural integrity. Moreover, until now, little research on the superplastic behaviour of the TA15 alloy has been reported, especially regarding its hot deformation mechanism at a temperature above 900 °C with a low strain rate range of 0.0005-0.01s⁻¹. Therefore, for widening its industrial applications of TA15 alloy, it is necessary to perform an in-depth study on microstructural evolution, textural changes, deformation mechanisms, and constitutive behaviour of TA15 alloy during the superplastic deformation.

Finite element modelling (FEM) is used commercially in SPF applications for the process-optimization purpose. The main objective is usually to control the process parameters (e.g., gas pressure) to avoid excessive thinning of the deformed parts. However, FE simulation of an SPF process remains very complex because many parameters have to be determined. The reliability of FE simulation results is critical since the pressure-time history used in SPF is dependent on the accuracy of constitutive equations coupled with the ever-changing geometry of the sheet as it fills the die. Therefore, the accuracy of the simulation code to forecast the deformation mechanism of the sheet material is a major factor in optimizing gas pressure cycles that can produce the workpiece with a uniform thickness in the shortest possible time. The penalty of an inaccurate pressure/time cycle can range from sub-optimum forming to excessive thinning or rupture of the sheet. At present, there are many reports available in the literature using FEA to the study of the industrial production of parts by SPF. But most of these FEA models are based on the traditional constitutive models (Fan et al., 2018) available in the built-in materials libraries of commonly employed

commercial software packages (ABAQUS, ANSYS, MARC, and DEFORM-3D). These do not reliably facilitate the users to simulate complex industrial parts. In other words, these built-in models are unable to describe the effects of the thermal and mechanical history and the softening phenomena on the shape and magnitude of the flow curve (Bariani et al, 2000). These effects are predominant after the initial stage of straining where strain hardening prevails. This inaccuracy has been proved to lead to large approximations in calculating the flow stress (Bariani and Negro, 1999). These do not reliably facilitate users to simulate complex industrial parts. Moreover, the observation from the literature survey shows that most of the pressure-time profile schemes for superplastic blow forming titanium alloys have been developed for laboratory-scale simple geometries such as the single stage of a hemispherical dome, semi-circular dome, single-stage rectangular die, simple conical die, and specific applications of simple shape rectangular pan and pockets.

Although there is a series of studies in the literature regarding the study of the industrial production of superplastic forming processes using finite element methods. But most of these studies (Chen et al, 2001; Hambli and Potiron, 2001; Lee and Huh, 2001; El-Morsy et al, 2002; Carrino et al, 2003a; Carrino et al, 2003b; Li et al, 2004; Zhang et al, 2004; Kumar et al, 2006; O'Brien et al, 2007; Luo et al, 2008; Luckey et al, 2009; Bing et al, 2010; Filice et al, 2010; Taleff et al, 2010; Ng et al, 2011; Inanov et al, 2012; Zhou et al, 2015; Sathish et al, 2017; Giuliano, 2019; Aleyari et al, 2020) use simple 'power-law' material expressions which do not contain any information about the microstructure and its deformation path. Hence, developing a reliable constitutive relationship to present the complex strain hardening and dynamic softening behaviour of metals or alloys is fairly paramount. The potential improvements are strongly based on better scientific understanding of dynamic recrystallization, strain hardening, and phase transformation in superplasticity of TA15 titanium alloy.

1.5. Aim and Objectives

The overall aim of this study is to improve the understanding of the superplastic effect in TA15 titanium alloy across a wide range of length scales at which superplasticity is

relevant: from the grain-grain deformation accommodation to the optimization of the manufacturing process of industrial parts.

To accomplish the above aim, the following objectives were formulated:

- i. To improve the understanding of the superplastic deformation mechanism of TA15 alloy based on the microstructural evolution at temperatures ranging from 880 °C to 940 °C and stain rates ranging from $5 \times 10^{-4} \text{ s}^{-1}$ to $1 \times 10^{-2} \text{ s}^{-1}$.
- ii. To characterize the microstructural evolution during superplastic deformation, with reference to the starting microstructure, by using Back Scatter Image (BSI)-Scanning Electron Microscope (SEM) and electron back scatter diffraction (EBSD).
- iii. To correlate the concurrent microstructure evolution to the optimum conditions for superplasticity in TA15 alloy.
- iv. To clarify the contribution of dislocation activity to superplastic deformation through texture measurements
- v. To establish a set of unified physical mechanism-based, visco-plastic constitutive equations with microstructural considerations.
- vi. Fine element modelling to calculate the optimized pressure cycle for a complex shape SPF part of TA15 alloy and to investigate thickness variation in the deformed part.

Chapter 2

Literature Review

Superplasticity is the capacity of certain polycrystalline materials to exhibit exceptionally large elongations under load without necking before failure.

2.1. Conditions for Observing Superplasticity

Generally, the following primary conditions are considered necessary for transforming the material into a superplastic state:

- fine grain size, usually 10 -15 μm or less. The optimum strain rate for superplasticity increases with decreasing grain size when grain boundary sliding (GBS) is the dominant process ([Ma and Mishra, 2014](#)).
- a deformation temperature in the order of $\sim 0.5T_m$, where T_m is the absolute melting temperature of the material. This condition is necessary to allow the activation of the accommodating diffusion processes that go hand in hand with grain boundary sliding ([Verlinden et al, 2007](#))
- a specific range of strain rates (usually from 0.0001 to 0.01 s^{-1}), which is specific to a given alloy. Deformation at strain rates higher than this range involves grain growth and grain elongation parallel to the flow direction, an increase of dislocation density in the grains, and work hardening.
- a thermally stable microstructure, single-phase materials generally do not exhibit superplasticity because grain growth occurs rapidly at elevated temperatures. Hence, the presence of second phases at grain boundaries is required to resist excessive grain growth. A considerable amount of fine, uniformly distributed, and thermally stable second-phase particles is necessary to keep the microstructure stable during superplastic deformation.

- equiaxed grain shape, with equiaxed grains, grain boundaries can experience shear stress, thus easily promoting GBS.
- high-angle grain boundaries (HAGBs, angle $\geq 15^\circ$), in particular, random HAGBs promote GBS, whereas low-angle grain boundaries are generally believed to be not suitable for GBS.

2.2. Superplastic Deformation Mechanism

The exact mechanism of superplastic deformation is a source of continuing debate, and many models supporting different accommodation mechanisms have been proposed. These models make correlations between grain boundary sliding behaviour and diffusional creep, the movement of lattice dislocations along the grain boundary, and intragranular dislocation slip.

2.2.1. Superplasticity as a regime of creep

Creep is the phenomenon of slow deformation of a material under persistent mechanical loading, while superplasticity is the ability of a material to undergo large amounts of deformation without necking at relatively low strain rates. Therefore, it is well-agreed that superplasticity and creep are linked and thus probably accompanied by the same kind of microstructural phenomena. With this in mind, it seems appropriate to introduce the concept of thermally-activated mechanisms of deformation for which diffusive processes are rate-controlling *i.e.* creep. When materials are deformed under constant stress at temperatures approximately greater than $0.4T_m$ they undergo a transient response where the strain-rate becomes constant. This is known as the steady-state creep regime, and its dependence on stress can provide important information about the mechanisms involved.

2.2.2. Grain boundary sliding as a deformation mechanism

Grain-boundary sliding (GBS), the relative motion of grains to one another along their boundaries, is generally considered to be the dominant mechanism of superplasticity, as it represents most of the strain in superplastic flow occurring at high temperatures. There are

two main theories regarding GBS as the deformation mechanism, *i.e.* Rachinger sliding (Rachinger, 1952) and Lifshitz sliding (Lifshitz, 1963). The Rachinger sliding refers to the process where-by adjacent grains move to each other while retaining essentially their original shapes (Langdon, 2006). A distinguishing characteristic of Rachinger sliding is a net increase in the number of grains lying along the direction of the tensile stress within the gauge length. This is achieved with a grain neighbor switching geometry, as shown in Figure 2.1. However, the retention of the grain aspect ratio alone is not sufficient to identify this type of sliding mechanism. This is because Choksi found that Lifshitz sliding along with grain growth could maintain the equiaxed shape of grain after the diffusional creep.

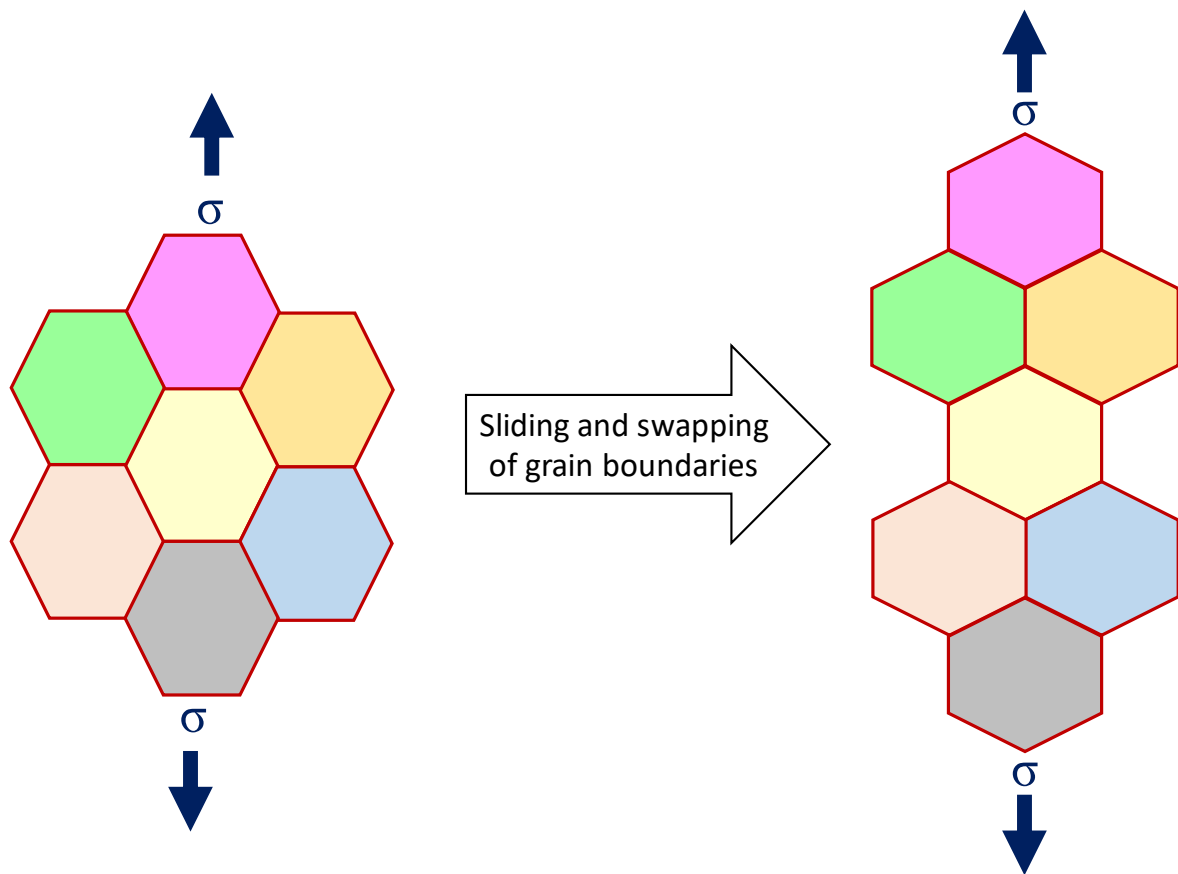


Figure 2. 1. Schematic of Rachinger grain boundary sliding

According to Lifshitz sliding (Lifshitz, 1963), GBS occurs such that the grains elongate along the tensile axis and maintain their adjacent neighbors. The schematic of Lifshitz sliding is depicted in Figure 2.2. It can be observed that grains become elongated without an increase in number along the tensile axis. In summary, Lifshitz sliding considers the relative motion of grains along a common boundary when the accommodating process is a diffusional creep, while when the process of accommodation occurs by glide and climb of dislocations, the GBS process is termed as Rachinger sliding. In the Lifshitz mechanism, the number of surface grains remains constant, whereas the number of surface grains increases under the Rachinger mechanism (Langdon, 1994). However, Rachinger, sliding is widely accepted as the main type of grain boundary sliding (Alabort et al, 2016)

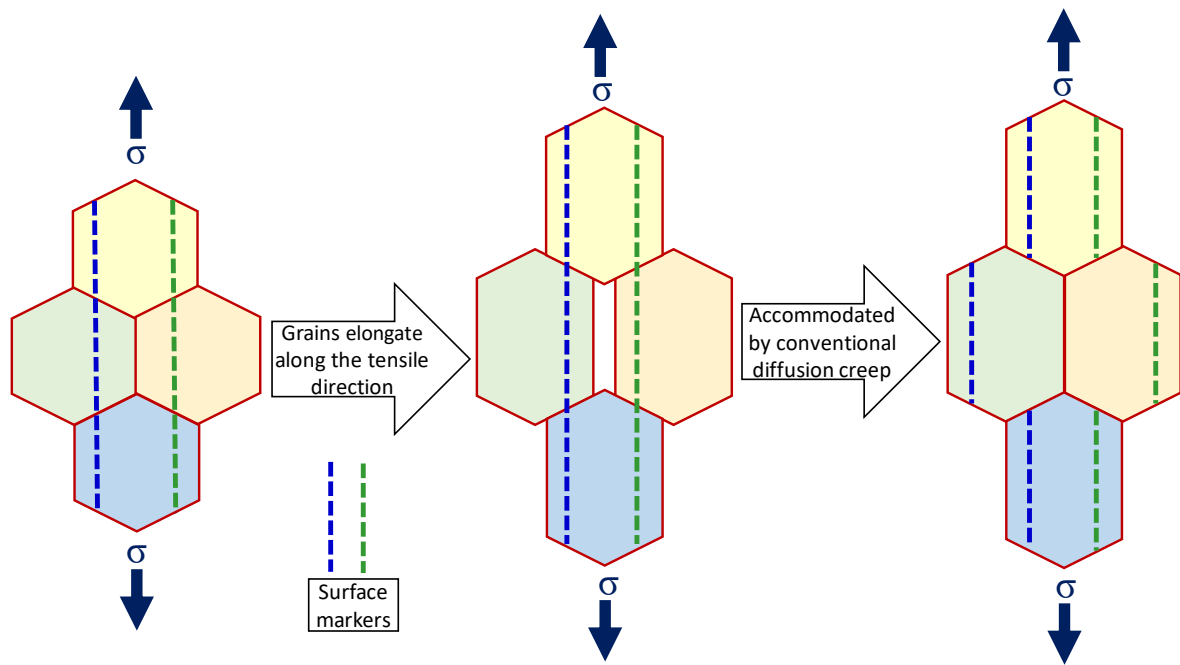


Figure 2. 2. Schematic representation of Lifshitz grain boundary sliding, which accompanies diffusional creep.

2.2.3. The accommodation of grain boundary sliding

As discussed above, grain boundary sliding accounts for the plastic deformation in superplastic flow. However, the grain boundary sliding cannot occur without some accommodation mechanism at the periphery of the grains. Therefore, it appears that sliding is accommodated by some limited intragranular dislocation movement, which serves to facilitate both the sliding and the rearrangement of the individual grains (Ridley, 1995). This accommodation, during which grains undergo deformation to relieve the GBS-induced stress concentration, is regarded as the rate-controlling process of superplasticity (Masuda et. al, 2016). However, in the particular case of the superplastic deformation of titanium alloys, the actual existing accommodation mechanism involved is still a matter of debate. The accommodation mechanisms considered can be divided into three groups:

- i. Diffusional accommodation (Ashby and Verrall, 1973; Spingarn and Nix, 1978; Geckinli, 1983)
- ii. Accommodation by dislocation motion (Ball and Hutchinson, 1969; Mukherjee, 1971; Arieli and Rosen, 1977; Valiev and Langdon, 1993; Bate and Blackwell, 1998).
- iii. Combined models having elements of both dislocation and diffusional accommodation (Ghosh and Raj, 1981; Mayo and Nix, 1989) and
- iv. Cooperative grain boundary sliding (Zelin and Mukherjee, 1995; Kaibyshev et. al, 1998; Emaletdinov, 1999; Mara et. al, 2007)

Diffusional accommodation occurs by the transport of material either along the grain boundaries or through the lattices of grains. These models suggest that the necessary movement of mater at triple grain junctions can be redistributed by diffusional flow with the absence of dislocation activity. Among the diffusional accommodation models, the model of Ashby-Verrall Model (Ashby and Verrall, 1973) holds a special place (Figure 2.3). Ashby and Verrall (1973) proposed a model for sliding accommodated by diffusion in which involving grain switching. Their model explains superplasticity as a transition region between the diffusion-accommodated flow, operative at low strain rates, and dislocation creep at high strain rates. However, the Ashby–Verrall model has (correctly) been criticized over points of greater or lesser importance *e.g.* Spingarn and Nix (1978)

argued that Ashby–Verrall model does not satisfy certain conditions of equilibrium. They suggested that the distributions of normal tractions are symmetric about the mid-points of each grain boundary segment before diffusion flow, which therefore restricts the allowable transient diffusional fluxes. So, there is no means for Ashby–Verrall atom fluxes to occur. The schematic of modification made by Spingarn & Nix (1978) to the Ashby–Verrall model is shown in Figure 2.4. Furthermore, Nix (1985) has suggested that the shape change suggested earlier by Lee (1970) will produce a more realistic transient configuration during the shape change, in which grain neighbour switching occurs by diffusion and grain boundary migration. The process allows each grain in a four-grain cluster to undergo the same (and symmetrical) change in shape and yield the same final configuration. Geckinli (1983) later expanded the concept to three-dimensional space and limited the occurrence of diffusion activity to the grain corners. However, these kinds of models are inconsistent with many of the experimental data, particularly regarding the stress dependence of strain-rate.

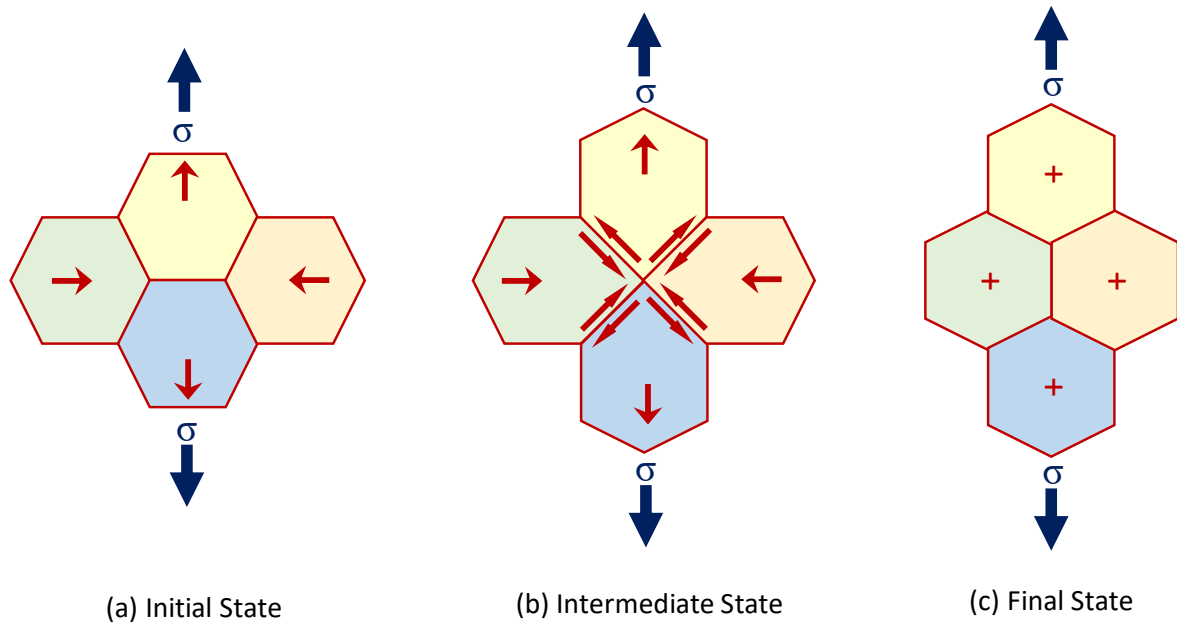


Figure 2. 3. *Ashby-Verrall model of GBS accommodated by diffusional flow*

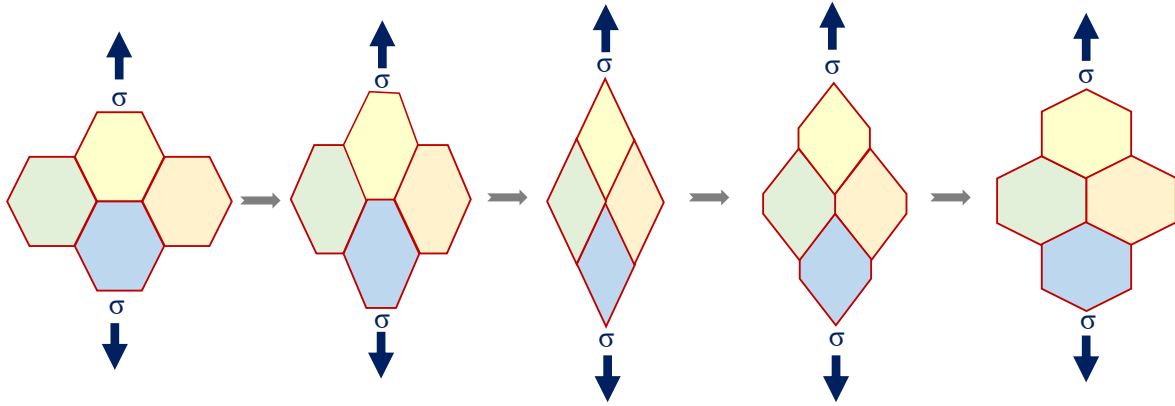


Figure 2. 4. Grain switching through diffusional mass transport as postulated by Ashby and Verrall (1973) and modified by Spingarn and Nix (1978).

The accommodation mechanism of dislocation activity was first observed and proposed by Ishida and Brown (1967). The dislocation-accommodation mechanisms can be grouped into a process due to dislocation pile-ups in the grain interior, and that a process due to dislocation pile-ups in the grain boundary. The accommodation models categorized as pile-ups within the grains (Ball and Hutchinson, 1969; Mukherjee, 1971) are based on the common assumption that dislocations, which are emitted at the grain boundary due to stress concentration, then traverse the grain by a gliding process and are then annihilated at the opposite grain boundary. Ball and Hutchinson (Figure 2.5) suggested that grains slide in bigger groups, and the rate of accommodation is governed by the pile-up and consequent climb of dislocations, which accumulate against opposite grain boundaries of any given grain which impedes the sliding. Mukherjee (1971) has proposed a modification of the above model in which grains slide individually rather than in groups (Figure 2.6). According to Mukherjee (1971), most of the resistance is generated by ledges and protrusions of the boundaries themselves: these act as sources of dislocation activity within the grain lattice. Large ledges or obstacles on the grain boundary surface would cause dislocations to be generated at these sites. The generated dislocations would travel through the blocked grain and pile-up at the opposite grain boundary. The rate of dislocation generation and movement in the pile-ups is controlled by the rate of continuous removal of

the leading dislocation in the pile-up by a climb along the grain boundaries to the annihilation sites at a distance of the order of grain diameter.

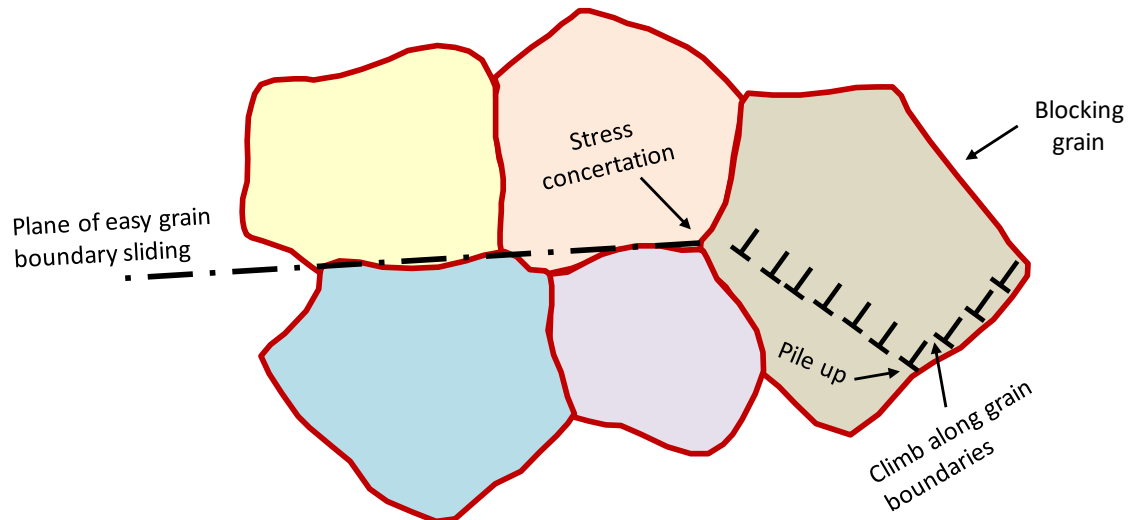


Figure 2. 5. Ball Hutchinson model of GBS accommodated by dislocation movement

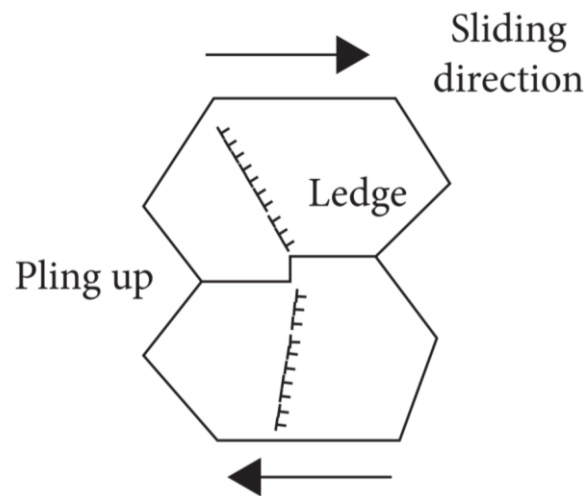


Figure 2. 6. Mukherjee model elaborates dislocation accommodated sliding mechanism of single grain (Adopted from Wang et. al, 2018)

In the models associated with dislocation pile-up in the grain boundary plane (Gifkins Core-Mantle model, 1976), sliding is considered to take place by the motion of grain-boundary dislocations that pile up at triple points. The resulting stress concentration is relaxed by dissociation of the leading grain-boundary dislocation by moving either in the boundaries making up the triple point or by moving into lattice dislocations accommodating sliding. These new dislocations then climb or glide in or near these two boundaries until they meet each other. Then they may annihilate or combine with them to form different grain-boundary dislocations. As shown in Figure 2.7, in the Gifkins' Core-Mantle model, sliding is considered to take place by the motion of grain boundary dislocations, which pile up at triple point. The model suggests that dislocation activity is confined to a narrow mantle region close to the grain boundaries and the dislocations then gliding and climbing. This flow in the mantle of width $\approx 0.07d$ allows grains to rotate. At the same time, grains move in the third dimension so that a grain may appear from above or below at the centre of a four-grain unit (Figure 2.8). However, several studies (Ghosh and Raj, 1981) see superplasticity as involving both diffusion and dislocation mechanisms simultaneously.

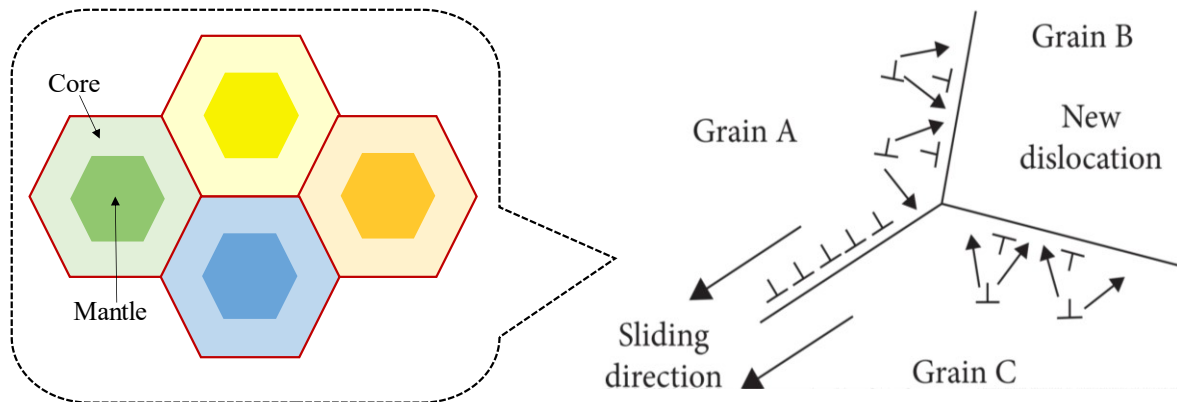


Figure 2. 7. Gifkins core-mantle model

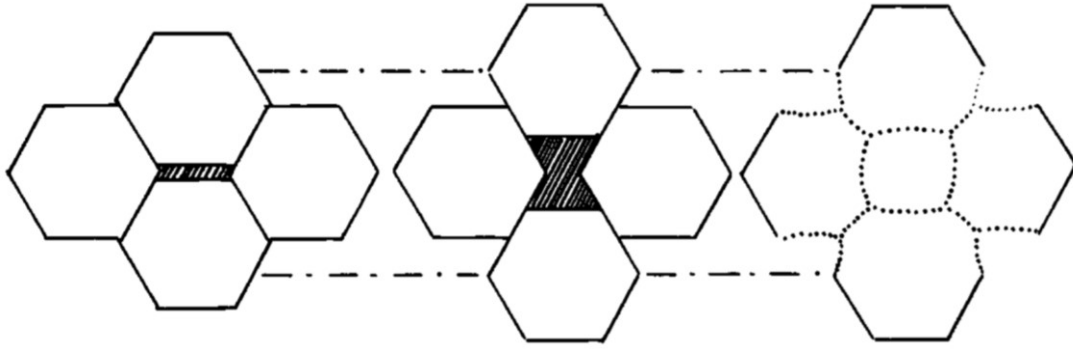


Figure 2. 8. Elongation by grain-boundary sliding and accommodation by the emergence of grains from one layer to the next. A gap forms between four grains and is filled by an emerging grain (from [Poirier, 1985](#))

The composite schematic of the above dislocation based accommodation mechanisms of grain boundary sliding models are shown in Figure 2.9.

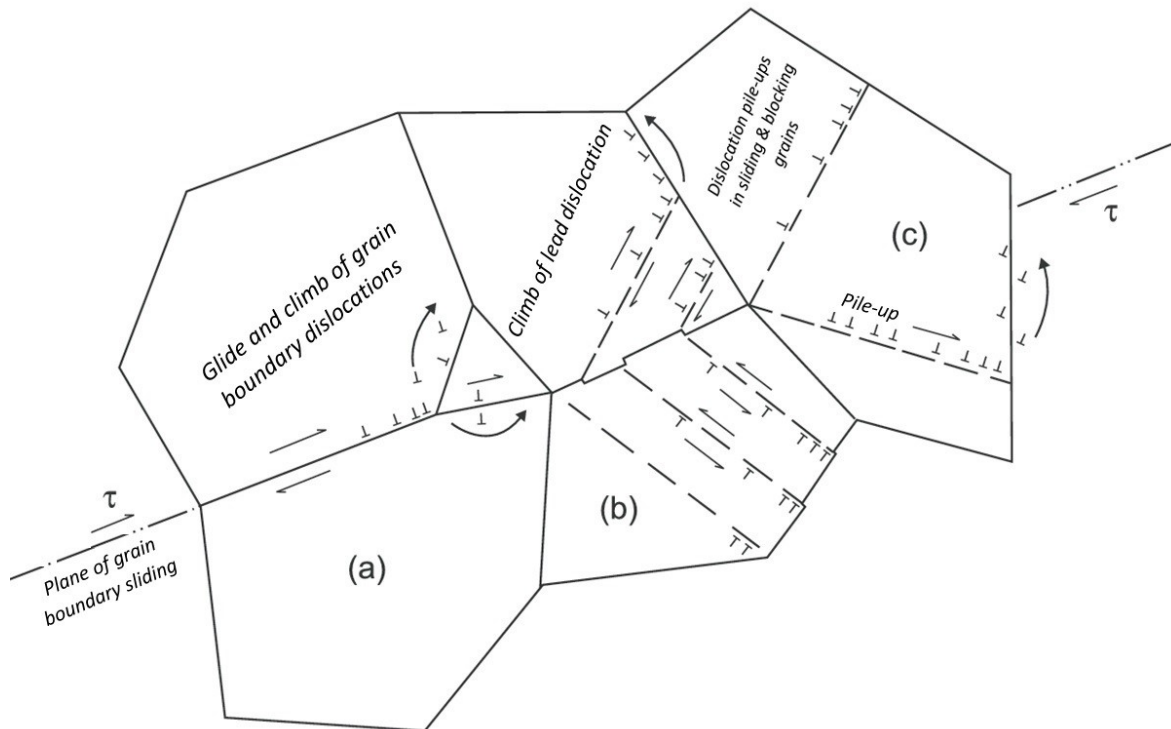


Figure 2. 9. Composite schematic of dislocation based accommodation mechanisms of grain boundary sliding proposed by: (a) Gifkins model (b) Mukherjee model and (c) Ball and Hutchison model (Modified from [Ridley, 2011](#))

Keeping in view the above discussion on different types of GBS accommodation mechanisms during superplastic deformation, the matter still remains controversial and so far, no single theory has been able to explain universally all of the experimental observations (Alabort et. al, 2016). For example, in the aluminium alloys, Rust and Todd (2011); Sotoudeh and Bate (2010); Mikhaylovskaya et. al (2018); for magnesium alloys, Panicker et. al (2009); and for Al–Mg based alloys, Mikhaylovskaya et. al (2015), have all confirmed that diffusion creep – and not dislocation activity – is the dominating mechanism in superplasticity. In contrast, for a Al-Mg based alloy, Tokunaga et. al (2019) ;and for an Al-Li based alloy, Jia et. al (2019); and for a titanium alloy Zhang et. al, (2019) claimed dislocation creep as the accommodating mechanism. Similarly, for aluminium alloys, Kotov et. al (2016); Yakovtseva et. al (2016) and Pozdniakov et. al (2019) observed that GBS takes place through the combined mechanism of dislocation creep and diffusion creep. Investigating the superplastic deformation mechanism of a ferritic steel, Kamikawa et. al, (2016) observed that at a specific temperature (1000 °C), decreasing the strain rate from $1.0 \times 10^{-4} \text{s}^{-1}$ to $1.0 \times 10^{-5} \text{s}^{-1}$, changes the accommodation mechanism from the conventional dislocation creep to diffusional creep. Since many distinct conclusions have been reported, it seems reasonable to believe that the accommodation mechanism of GBS in superplastic deformation may depend upon important alloy characteristics (e.g. phase architecture and grain morphology) and deformation condition (e.g. strain rate and temperature).

2.3. Microstructure Evolution in Superplasticity

Superplasticity depends on the changes in the microstructure that take place during deformation. The main mechanisms that controlling the microstructure evolution during superplastic deformation are work hardening, recovery, recrystallization, and grain growth (Figure 2.10). During the superplastic deformation, the strain energy is stored in the material mainly in the form of dislocations, and this energy is released primarily in the form of three processes, including: recovery, recrystallization, and grain growth (subsequent to recrystallization) (Kassner, 2015). Dynamic grain growth and cavitation affect the extent of achievable superplastic flow in a material (Chokshi and Mukherjee,

1988; Chung et al., 2004; Hirai et al., 2007; Jiang and Bate, 1996; Padmanabhan and Raviathul Basariya, 2009; Rabinovich and Trifonov, 1996). However, dynamic recrystallization, when present in a continual manner, could favor superplastic deformation (Maehara and Ohmori, 1987; Qing et al., 1992), and can enhance the ductility (Pancholi and Kashyap, 2007).

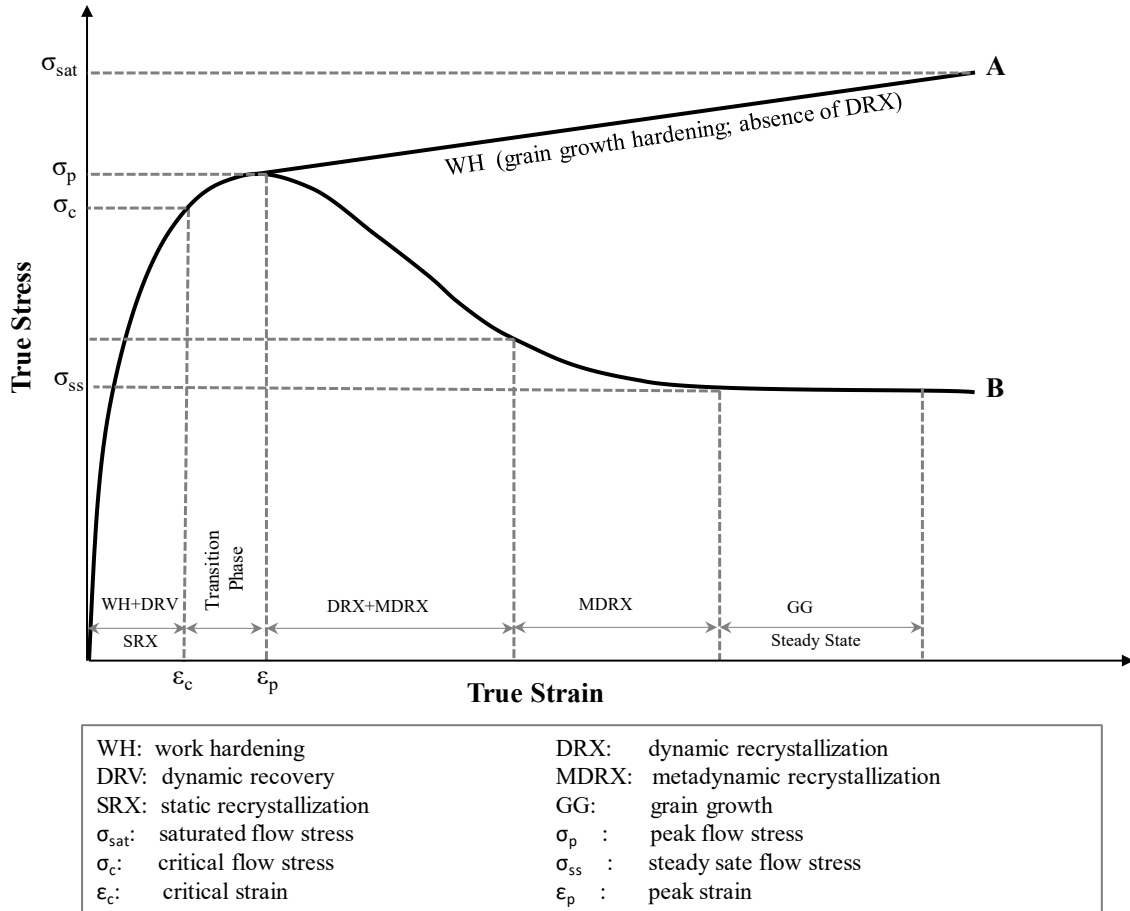


Figure 2. 10. Schematic illustration of a flow stress curve representing the classification of mechanisms associated with the microstructure evolution during superplastic deformation.

2.3.1. Work Hardening

Work hardening (also known as strain hardening) is a measure of how the resistance to plastic flow increases as the metal is deformed. Once the plastic deformation is initiated, the moving dislocations interact with each other and with the grain boundaries to make continued yielding more difficult (Dieter et. al, 2003). In superplastic deformation, the

decrease in work hardening with strain is diminished by a result of the dislocation annihilation and rearrangement process.

2.3.2. Recovery

The definition of recovery includes all processes releasing the stored energy that does not require the movement of high-angle boundaries ([Kassner, 2015](#)). In the context of superplastic deformation, dynamic recovery (DRV) plays a vital role in lowering the flow stress. DRV results from the annihilation of dislocations due to ease of dislocation cross slip, climb, and unpinning, as well as the formation of cells and subgrains, which act as sinks for moving (mobile) dislocations ([Dieter et. al, 2003](#); [Semiatin, 2003](#)). Dynamic recovery occurs principally in pure FCC and BCC metals/alloys where the medium to high stacking fault energy limits the extension of the dislocations, and facilitates thermal activation of cross-glide and climb ([Cohen and von Turkovich, 1989](#)).

Though recovery is an important process, it is often difficult to quantify. It can occur immediately after deformation (known as static recovery), and also dynamically during deformation (known as dynamic recovery). Furthermore, changes in microstructure due to DRV cannot be detected under microscopy ([Verlinden et. al, 2007](#)) but can be observed through TEM (transmission electron microscopy) when the dislocation density is substantially reduced, and the considerable rearrangement of the remaining dislocations has occurred ([Mankins, 2004](#)). Likewise, the texture of the deformed metal necessarily does not change during DRV. DRV affects properties such as dislocation density, subgrain size, and misorientation – but the resolution of such property changes is often poor, and statistically quantifying the changes in the affected structural features is difficult ([Verlinden et. al, 2007](#)).

2.3.3. Recrystallization

In the superplastic deformation of metallic materials, recrystallization often takes place. This, which can be defined as the process of the “formation of a new grain structure in a deformed material by the formation and migration of high angle grain boundaries ($>10^\circ$ –

15°) driven by the stored energy of deformation” (Doherty et. al., 1997). Recrystallization can occur under two broad conditions; static and dynamic (Kassner, 2015). Static recrystallization (SRX) can take place upon subsequent annealing of a deformed sample in the absence of external plasticity. In contrast, dynamic recrystallization (DRX) refers to the occurrence of recrystallization during deformation. DRX is different from static recrystallization in the sense that DRX characteristics are decided by the rate of grain nucleation versus the rate of growth under the given imposed conditions of temperature and strain rate. In SRX, on the other hand, a fixed amount of stored energy (dependent on cold work) is released by thermally activated dislocation recovery and grain boundary migration and may be termed as a kinetic process.

Besides DDRX and CDRX, another concept of Metadynamics recrystallization (MDRX) is also observed during hot deformation, originally proposed by Jonas and co-workers (Djaic and Jonas, 1973; Petkovic et. al, 1979; Sakai et. al, 1988 and Roucoules et. al, 1994). It is also known as post-dynamic recrystallization (PDRX). MDRX occurs after deformation because the strains required to complete the DRX are not continuously achieved. During MDRX, no new nuclei are formed but the dislocation density reduces, and the grain structure transforms into a fully recrystallized structure. During high-temperature deformation, even though straining is stopped (means locally no strain in the grains) annealing continues and the just nucleated DDRX grains will grow with no incubation period into the heterogeneous, partly dynamically recrystallized matrix. Metadynamics Recrystallization (MDRX) can be distinguished from static Recrystallization (SRX) in several ways. The kinetics of MDRX is highly strain-rate dependent but depends only weakly on the amount of deformation and the temperature (López and Rodriguez-Ibabe, 2012). Moreover, MDRX is relatively sensitive to the prior strain-rate but insensitive to pre-strain and temperature, while SRX strongly depends on pre-strain and temperature, but only slight on strain-rate (Kassner, 2015).

In general, DDRX takes place in alloys with low to medium stacking-fault energy (SFE) (e.g. Mg, Zn, α -Ti alloys) at high temperature ($>0.5T_m$) in which dynamic recovery is sluggish (because slow mobility of dislocations results in the accumulation of high dislocation densities), so that the driving force for recrystallization is maintained (Cahn,

1996; Kassner and Barrabes, 2005 and Raabe, 2014). On the other hand, in alloys with high SFE (e.g. aluminium alloys, β titanium alloys), there is the ease of dislocation movement during plastic deformation, and so they are prone to dynamic recovery followed by CDRX rather than DDRX during high-temperature deformation (Guo-Zheng, 2013; Raabe, 2014; Sakai et al., 2014 and Huang and Logé; 2016). Hence, as the α phase of a Titanium alloy has its atoms arranged in an HCP structure, while the β phase has a BCC crystal structure (Campbell, 2008), the HCP structure has a low SFE which gives less opportunity for cross slip (Harrison and Winstone, 1996), while the BCC structure is associated with high SFE. Therefore, the SFE for the hot deformation of titanium alloys depends on the α/β ratio of the alloy.

2.3.4. Grain Growth

Following recrystallization and the elimination of dislocation substructure, the principal driving force for microstructural changes is the reduction of grain-boundary energy (Semiatin, 2003). In superplastic deformation of titanium alloys, grain growth induced by plastic strain and variation in dislocation density will occur subject to availability of sufficient time and the driving force required for grain growth. The grain growth can be broadly classified into two categories: static grain growth and dynamic grain growth. The static and dynamic grain growth works independently and plays a significant role in viscoplastic deformation at a low strain rate and high temperature, such as superplastic deformation. On the other hand, during recrystallization, new grains are nucleated, and the total number of grains increases. Consequently, the average grain diameter decreases.

2.4. Mechanics of Superplasticity

The single most important property for the mechanical description of superplastic materials is the strain rate sensitivity parameter m (Ridley, 2011), which is used to differentiate superplastic and non-superplastic materials (Enikeev, 1997). This is defined as the inverse of stress exponent n ($m = 1/n$). The relation between strain rate sensitivity parameter m , stress, and plastic strain rate can be expressed as a single power-law form as:

$$\sigma = k\dot{\epsilon}^m \quad (2.1)$$

Where σ is the stress, $\dot{\epsilon}$ the plastic strain rate and k is a material constant. Thus one can determine the strain rate sensitivity m as the logarithmic relationship between stress and strain rate as

$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right|_{\epsilon, T} \quad (2.2)$$

The strain rate sensitivity index m can be used to depict the deformation stability, critical to optimizing the amount of deformation that a material can take place before failure. Higher values of m indicate an increased resistance to necking, and thus they allow large elongation to failure (Campbell, 2012). When m is equal to 1, the flow stress is directly proportional to strain rate, and the material behaves as a Newtonian viscous fluid, such as hot glass (Smallman and Ngan, 2014). Superplastic materials are therefore characterized by high m values. In practice, the value of m for superplasticity is usually higher than 0.5 with the majority of superplastic materials having an m value in the range of 0.4 – 0.8 (Campbell, 2011; Ridley, 2011; Humphreys et. al, 2017) whereas in the major mechanisms associated with high temperature creep, the values of m are either ‘0.2’ for control by dislocation climb or ‘0.3’ for control by dislocation glide (Mohamed and Langdon, 1974) and the elongations are no more than ~300% (Shahmir, 2018; Langdon, 2018). This means that relatively high elongations may also be achieved in metals deforming by dislocation glide, and it is, therefore, necessary to differentiate between elongations associated with glide and those of true superplasticity (Langdon, 2018). Accordingly, it is accepted as reasonable to adopt a tensile elongation of at least 400% as evidence for the occurrence of superplastic flow (Langdon, 2009).

The value of ‘ m ’ depends on a number of factors: strain, strain rate, microstructure evolution, deformation mode, type of loading, and others. Therefore, this parameter cannot be considered as a material constant (Padmanabhan, 2018). There are several experimental

methods to determine the value of m (Padmanabhan et. al, 2001; Totten and Mackenzie, 2003; Comley, 2008). The most convenient way is a uniaxial tensile test at a particular constant temperature and at different strain rates, ‘jump test’ (Beer and Barnett, 2006; Smolej et. al, 2009; Khan and Pandey, 2011; Zhou et. al, 2016). In this method, a relationship between the flow stress (σ) and the strain rate ($\dot{\epsilon}$) is obtained first and the ‘ m ’ value is then calculated using Eq. 2.2.

2.4.1. Mechanistic of Superplasticity

The applied stress (σ) for superplastic deformation is found to be strongly dependent on the strain rate ($\dot{\epsilon}$). In general, for the most superplastic materials the logarithmic curve ($\text{Log}\sigma$ vs $\text{log}\dot{\epsilon}$) has a sigmoidal shape, as shown in Figure 2.11, and can be divided into three main regions, labelled I, II, and III, to which a Region 0 may also be added. In some cases, it has been observed that m can initially decrease but further reductions result in a transition to a slope of 1, which is termed as Region 0 where true diffusion creep dominates with an activation energy of grain boundary diffusion (Vale et al, 1979). However, it should be noted that grain growth during slow strain rate deformation can affect the flow stress and complicate the interpretation of this region, as can the different testing techniques used to obtain stress-versus-strain rate data (Ridley, 2009).

The slope of the aforementioned logarithmic curve is denoted as strain rate sensitivity, m . This sigmoidal curve shows that m passes through a maximum in Region II, delineated by a value of $m > 0.3$. This region is associated with optimum superplasticity, and also with a maximum extent of grain boundary sliding. The experimental results in region II are generally consistent with several theoretical models for superplasticity. However, at present, no single model can account for all of the microstructural and mechanical observations. It is even possible that no such universal theoretical model applicable to all polycrystalline solids is attainable (Mukherjee, 1993). Regions I and III, representing the lower and higher strain rate ranges, have m values in the range 0.1 to 0.3. Kashyap and Mukherjee (1985) noted that, during tensile deformation in Regions I and III, the grains elongate in the direction of tension, and the change in the shape of the specimen is

consistent with the change in grain shape. The grain elongation occurs such that the position of adjacent grains remains unaltered, and the number of grains across the cross-section is unchanged. These conditions correspond to deformation under non-SP conditions.

At higher strain rates, region III is usually associated with recovery controlled dislocation creep mechanism (power-law creep). It is generally believed that dislocation climb is the rate-controlling process.

The origin of the low strain rate region I is not exactly clear. The experimental evidence in this region is often limited and contradictory. However, it has been proposed that the existence of regions arising from the segregation of impurity atoms at grain boundaries. Here they interact with the moving boundary dislocations that contribute to grain boundary sliding. A number of publications by Mohamed and his Co-workers ([Chaudhury and Mohammed, 1988](#)) show that if the impurities were reduced to very low levels (i.e. 6 ppm), then there was no evidence for the existence of Region I, although Region II and its transition to Region III were unaffected by purity levels.

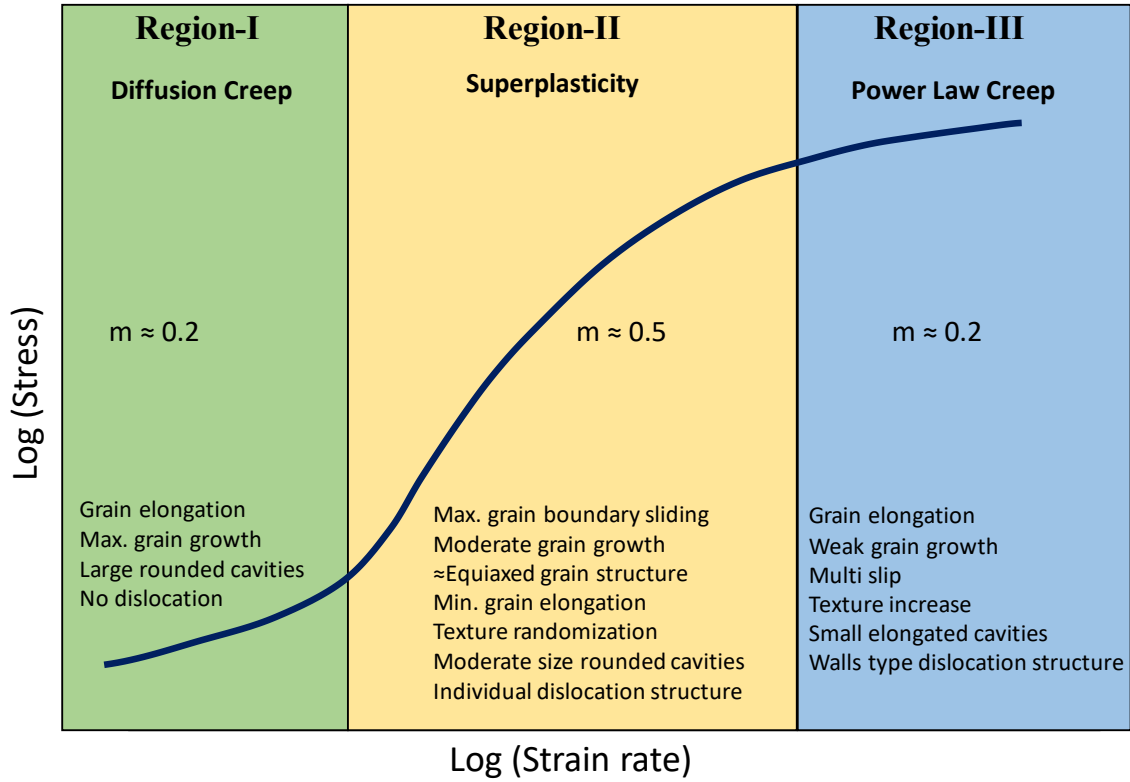


Figure 2. 11. Schematic illustration of the mechanical behaviour of superplastic materials

2.4.1.1. Microstructural effects on Superplastic Region-II

For materials exhibiting superplasticity, grain size is the dominant microstructural factor influencing flow stress and strain rate sensitivity. Typically, in most superplastic materials, increasing the grain size increases the flow stress in the superplastic regime, and tends to reduce the strain rate at which the maximum value of m is observed (Jackson, 2011). The general correlation for diffusion-assisted hot deformation process can be expressed as (Mukherjee et al., 1969):

$$\dot{\epsilon} = \frac{AD_0Gb}{kT} \left(\frac{\sigma}{G}\right)^n \left(\frac{b}{d}\right)^p \exp\left(-\frac{Q}{RT}\right) \quad (2.3)$$

where, $\dot{\epsilon}$ is the strain rate, A is a microstructure and mechanism dependent dimensionless constant, D_0 the diffusion coefficient, G the shear modulus, b the Burgers vector, k the Boltzman constant, T the absolute temperature, n the stress exponent for deformation, σ the

applied stress, p the exponent of inverse grain size, d the grain size, Q the activation energy of diffusion and R is the gas constant. Considering the GBS as the dominant deformation mechanism of superplasticity, the Eq. (2.3) can be written as:

$$\dot{\epsilon} = \frac{AD_{gb}Gb}{kT} \left(\frac{\sigma}{G}\right)^2 \left(\frac{b}{d}\right)^2 \exp\left(-\frac{Q}{RT}\right) \quad (2.4)$$

It can be seen from Eq. (2.4) that reducing the grain size by half can lead to an increase in the optimal superplastic strain rate by four. Every superplastic material has a limited regime in which grain boundary sliding is dominant. The upper boundary of this regime is the maximum superplastic strain rate $\dot{\epsilon}_{SPmax}$: if the strain rate was further increased, then the deformation would occur in region III (Figure 2.11), and different mechanisms would be operating. For region-III as shown in Figure 2.11, hot deformation occurs by an intragranular dislocation process, independent of grain size. In contrast, for the superplastic regime corresponding to region-II, the behaviour depends on grain size, such that the smallest grain size results in the highest strain rate at any selected stress. This is because, in superplasticity, the strain rate varies inversely with grain size raised to a power of 2 (Wazzan, 1965). Therefore, the best-known way to increase the superplastic regime of a material is by refining the grain size. Conventionally, multi-step thermo-mechanical processing (TMP) (*e.g.* a combination of multipass-rolling and appropriate heat-treatments) is used to produce the fine-grained equiaxed microstructure needed for optimum superplasticity. A typical thermomechanical processing route for attaining fine grained microstructure in titanium alloys is shown schematically in Figure 2.12. Generally, primary hot working and recrystallization annealing in the β -phase region is first conducted to eliminate the casting inhomogeneities; this produces a lamellar microstructure (Gao et al, 2020). Subsequently, the lamellar microstructure is commonly broken down (referred to as globularisation) to get the desired equiaxed microstructure by a subtransus processing (Yang et al, 2016).

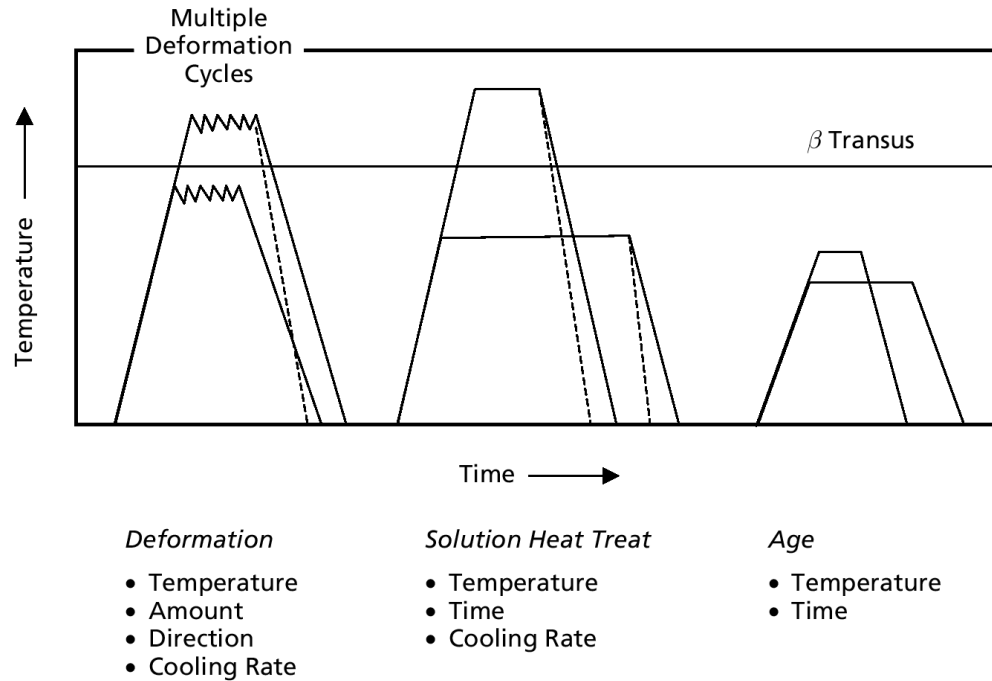


Figure 2. 12. Thermomechanical Treatment of Titanium Alloys ([Campbell, 2011](#))

2.4.1.2. Temperature effects on Superplastic Region-II

As well as grain size, superplastic deformation depends significantly on deformation temperature. In general terms, for a given system, increasing deformation temperature displaces the SP region-II (Figure 2.11) to higher strain rates and m values. However, such an effect is observed only in the temperature range corresponding to a stable microcrystalline structure ([Kaibyshev, 2012](#)). If significant grain growth occurs with increasing temperature, or if the stability of the microstructure is lost as a result of the transformation of the material into a single phase condition, then the superplastic behaviour decreases abruptly or disappears completely ([Kaibyshev, 2012](#)).

2.5. A numerical description of superplasticity

The superplastic deformation behaviour of metallic materials is always accompanied with various interconnecting metallurgical phenomena such as work hardening, dynamic recovery (DRV), dynamic recrystallization (DRX) and flow instability ([Lin et al, 2008](#); [Mandal et al, 2009](#); [Samantaray et al, 2010](#); [Li et al, 2011](#); [Xiao et al, 2012](#)). In addition,

the relationships between the flow stress and these phenomena are non-linear. Consequently, the modelling and prediction of the superplastic deformation behaviours of metallic materials are complex in nature (Quan et. al, 2016). In this regard, a considerable amount of research has been conducted employing various constitutive models or equations to predict the flow behaviour of materials at elevated temperatures. Constitutive equations are equations that describe the mechanical state of materials under differing loading and thermal conditions. The development of mathematical models for the technological processes of superplastic forming, as well as evolving algorithms and methods to solve corresponding boundary value problems, are the final goals. The applicability and reliability of simulation results is strongly dependent on the accuracy of constitutive equations (Zhou and Clode, 1998). Constitutive equations can be broadly categorized into two groups: physical models and phenomenological (engineering) models (Ashby, 1992; Liang and Khan, 1999; Chaboche, 2008; Lin and Chen, 2011). However, the aim of these different models in achieving the accurate prediction of the material behaviour for specific materials remains the same (He et. al, 2018).

2.5.1. Phenomenological constitutive models

Phenomenological constitutive models do not consider the micro-mechanisms of material deformation and only consider the influences of macroscopic deformation parameters (deformation temperature, strain rate, and strain) on flow stress.

These models link the flow stress to the strain, the strain rate, and the temperature and are optimized to best represent the main features of the flow curves. All material constants are determined from macro-experiments only. In principle, they should describe 'best fit' equations, using a specific number of coefficients best to fit the stress-strain curves of the experimental data. These models are the most widely used, because they can be easily be understood and are based on empirical observations (Lin et al, 2016). The flow stress is usually expressed as functions of deformation temperature, strain rate, and strain. The notable feature of these models is that as they use a reduced number of material constants, they can be easily calibrated. However, due to their empirical characteristics and lack of a

physical basis, the phenomenological constitutive models cannot be extrapolated to cover wide ranges of practical forming conditions. The general form of Phenomenological constitutive models can be written as:

$$\sigma = f(\varepsilon, \dot{\varepsilon}) \quad (2.5)$$

Up to now, there have been various phenomenological models proposed, such as those listed below:

$$\ln \sigma = F(\ln \varepsilon, \ln \dot{\varepsilon}) \quad (2.6)$$

Applying Taylor's expansion and disregarding higher-order terms, Eq. 2.6 can be written as:

$$\sigma = K \varepsilon^n \dot{\varepsilon}^m \quad (2.7)$$

Where K is a material constant, m is the strain-rate sensitivity, and n is the strain hardening exponent. The value of m and n can be determined by following relations:

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}} \quad (2.8)$$

$$n = \frac{\partial \ln \sigma}{\partial \ln \varepsilon} \quad (2.9)$$

Expression 2.7. is known as the double power-law model. If the strain hardening component ' n ' in Eq. 2.7 is removed ($n = 0$), one obtains the most fundamental relationship between stress and strain-rate, which is known as a single power-law model, and can be expressed as:

$$\sigma = K \dot{\varepsilon}^m \quad (2.10)$$

This kind of phenomenological model is widely implemented and used in most of the commercial finite element packages, which allow the numerical simulation of superplastic forming. Currently, there are various alternative phenomenological models, such as those listed below:

2.5.1.1. Arrhenius Equation

One of the most commonly used phenomenological models for hot forming is the Arrhenius hyperbolic-sine equation (Lin et al, 2014) proposed by Garofalo (1963) and later popularised by Sellars and Tegart (1972). A number of researchers (Lou et al, 2011; Sun and Zhang, 2011; Samantaray et al, 2011; Wu et al, 2012; Su et al, 2016; Yu et al, 2017) used the Arrhenius Equation to predict the deformation of metals and alloys under hot conditions. It is well accepted that the relationship between the flow stress (σ), strain rate ($\dot{\epsilon}$) and temperature (T) during hot deformation can generally be expressed in the form of an Arrhenius type equation in the form given by Sellars and McTegart (1966).

$$\dot{\epsilon} = A[\sinh(\alpha\sigma)]^n \exp\left(-\frac{Q}{RT}\right) \quad (2.11)$$

As a form of modification, the temperature compensated strain rate is known as Zener-Hollomon parameter, Z (Zener and Hollomon, 1944) can be incorporated into the Arrhenius equation and is expressed as

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \quad (2.12)$$

Then, the flow stress σ , written as a function of the Z parameter, can be given as:

$$\sigma = \frac{1}{\alpha} \ln \left\{ \left(\frac{Z}{A} \right)^{1/n} + \left[\left(\frac{Z}{A} \right)^{2/n} + 1 \right]^{1/2} \right\} \quad (2.13)$$

Where

$\dot{\epsilon}$ = strain rate, (s^{-1})

σ = flow stress (MPa)

A, α = materials constant,

T = absolute temperature (K)

n = stress exponent ($n = 1/m$, m is the strain rate sensitivity),

Q = deformation activation energy for hot working ($kJ/mole$)

R = universal gas constant ($8.314 J/mol/K$)

After partial differentiation, the Eq. 2.13, becomes

$$Q = R \left\{ \frac{\partial \ln \dot{\epsilon}}{\partial \ln [\sin h (\alpha \sigma)]} \right\}_T \left\{ \frac{\partial \ln [\sin h (\alpha \sigma)]}{\partial (1/T)} \right\}_{\dot{\epsilon}} \quad (2.14)$$

According to the above equation, the activation energy Q can be calculated at different temperatures and strain rates.

The major shortcoming of the original Arrhenius equation is that it neglects the effect of strain on the flow stress. Accordingly, efforts have been made to update the Arrhenius equation by considering the strain-compensated effect (Lin et. al, 2008; Lin et. al, 2009; Haghdadi et. al, 2012; Chen et. al, 2019).

The effect of strain can be compensated in the standard Arrhenius equation by considering the parameter Q , α , n , and $\ln A$ in Eq. 2.13 as a polynomial function of strains. Therefore, Eq. 2.13 can be represented by the following expression in which the strain is considered:

$$\sigma = \frac{1}{c(\epsilon)} \ln \left[\left\{ \frac{\dot{\epsilon} \exp(\frac{e(\epsilon)}{RT})}{f(\epsilon)} \right\}^{1/d(\epsilon)} + \left\{ \frac{\dot{\epsilon} \exp(\frac{e(\epsilon)}{RT})}{f(\epsilon)} \right\}^{2/d(\epsilon)} + 1 \right]^{0.5} \quad (2.15)$$

Where $c(\epsilon)$, $e(\epsilon)$ and $f(\epsilon)$ are the polynomial functions of strain, which are used to represent the material constants α , n & A , respectively.

2.5.1.2. Johnson–Cook (JC)

The Johnson–Cook (JC) constitutive model, based on the conventional phenomenological theory, is widely used because of its simplicity and the low number of material constants (Kotkunde et al, 2017) involved. Determining these material constants also requires less experimental work. The JC model was proposed by Johnson and Cook in 1983 for metals subjected to large strains, high strain rates, and high pressure. The JC model has been successfully embedded into many commercial finite element programs such as ABAQUS and DEFORM to describe the deformation behaviour of sheet metal forming (Mabrouki et al, 2008; Chen et al, 2011). In the original JC model, the Von Mises stress is expressed as:

$$\sigma = [A + B(\varepsilon)^n] \left[1 + C \ln \left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \right) \right] \left[1 - \left(\frac{T - T_r}{T_m - T_r} \right)^m \right] \quad (2.15)$$

Strain hardening effect
Strain rate (viscosity) effect
Thermal softening effect

Where σ is the equivalent flow stress, ε and $\dot{\varepsilon}$ are the equivalent plastic strain and strain rate, respectively. $\dot{\varepsilon}_0$ is the reference strain rate (defined as 0.01s^{-1}), A , B , n , C , and m are materials constants.

JC model has some serious limitations. First, in the original JC model, effects of strain hardening, thermal softening and strain rate hardening are independent of each other, and it does not consider the coupling effects of temperature and strain rate. Second, the term for strain-rate hardening describes that the flow stress only increases linearly with the logarithmic strain rate. However, it is often the case that the flow stress initially increases linearly with the logarithmic strain rate; however, at the later stages, the flow stress rises steeply. Third, the JC model describes the thermal softening behaviour as being approximately linearly, but in reality, the thermal softening is somewhat saturated at high temperatures. To characterize the flow behaviour accurately, some improvements are made to the original JC model by introducing the updated strain term (Lin et al., 2018; He et. al, 2018), temperature term (Hou et. al, 2010) and the term reflecting the coupled effects of temperature and strain rate (Lin et. al, 2010).

2.5.2. Physical-based constitutive model

During hot and superplastic deformation processes, the internal microstructure of material changes extensively. However, the phenomenological models are not able to give an accurate interpretation of the microstructure evolution. Physical constitutive models are intended to consider the physical deformation process mechanisms to establish constitutive models that consider the thermal activation energy, dislocation density evolution, kinetics of slips, crystal plastic deformation, etc. Physical-based constitutive models account for physical aspects of the material behaviour. Compared to the phenomenological descriptions, they allow for an accurate definition of material behaviour under wide ranges of loading conditions by making some physical assumptions and considering a larger number of material constants. Although the physically based model can represent the deformation behaviour with greater accuracy over a wide range of temperatures and strain rates, they are not always preferred as they involve larger numbers of material constants than those phenomenological models, and these constants may not be readily available (Khan and Liang, 1999). The most prevailing physical models are the Zerilli–Armstrong model and Kocks–Mecking model.

2.5.2.1. Zerilli–Armstrong (ZA) model

The Zerilli–Armstrong (ZA) (Zerilli and Armstrong, 1987) model is one of the common physically-based constitutive models (He et al, 2014). It is derived based on dislocation mechanisms that play an essential role in the plastic deformation of metallic materials under various loading conditions. It divides the flow stress into two components, thermal and athermal

$$\sigma = \sigma_a + \sigma_{th} \quad (2.16)$$

where σ_a and σ_{th} are the athermal activation and the thermal activation flow stresses, respectively.

The Zerilli–Armstrong model invokes a relatively simple expression as compared to other dislocation-based constitutive models. The main characteristics of the model are that each material structure type will have a different expression due to the different strain rate

controlling mechanism for that particular structure. The thermal activation flow stress for face-centred cubic (FCC) and body-centred cubic (BCC) types of metals are given as:

$$\sigma_{th} = C_1 \exp(-C_3 T + C_4 T \ln \dot{\epsilon}) \quad (\text{for BCC metals}) \quad (2.17)$$

$$\sigma_{th} = C_2 \dot{\epsilon}^{\frac{1}{2}} (-C_3 T + C_4 T \ln \dot{\epsilon}) \quad (\text{for FCC metals}) \quad (2.18)$$

By combining the athermal activation stress and the effect of yield stress on grain size into one component C_0 , two different types of $Z-A$ models can be expressed as:

$$\sigma = C_0 + C_1 \exp(-C_3 T + C_4 T \ln \dot{\epsilon}) + C_5 \dot{\epsilon}^N \quad (\text{for BCC metals}) \quad (2.19)$$

$$\sigma = C_0 + C_2 \dot{\epsilon}^{\frac{1}{2}} (-C_3 T + C_4 T \ln \dot{\epsilon}) \quad (\text{for FCC metals}) \quad (2.20)$$

Where

C_0 ; C_1 ; C_2 ; C_3 ; C_4 ; C_5 ; n are material constants.

Besides the original ZA model discussed above, there are some other important modified ZA models mentioned in the references ([Abed and Voyiadjis, 2005](#); [Noble and Harding, 2006](#); [Lee and Liu, 2006](#); [Özel and Karpat, 2007](#); [Zhang et al, 2009](#); [Samantaray et al, 2009](#); [Gao and Zhang, 2010](#)).

Referring to the existing literature on the accuracy of the two above mentioned groups of models, there is variation about the claims made by the researchers. In some comparative studies on the phenomenological and physical-based constitutive models it is concluded that physical models can better predict the hot deformation of the material than phenomenological models ([He et. al, 2014](#); [Kotkunde et. al, 2014a](#); [Kotkunde et. al, 2014b](#); [Mirzadeh, 2015](#); [Farabi et. al, 2015](#); [Xia et. al, 2017](#); [Li et. al, 2019b](#)) while some studies reported superior accuracy for phenomenological models ([Samantaray et. al, 2009](#); [Li et. al, 2013](#); [Shamsolhodaei et. al, 2014](#); [Shang et. al, 2015](#))

2.5.2.2. Kocks and Mecking (KM) model

The Kocks–Mecking (KM) model (Mecking and Kocks, 1981; Estrin and Mecking, 1984; Follansbee and Kocks, 1988) is an important physical model, that has set the scene for future modelling efforts. This KM model dominates the constitutive modelling philosophy to the present day, (Estrin and Vinogradov, 2013). The model is based on the assumption that the kinetics of plastic flow is determined by a single structural parameter (dislocation density ρ), which represents the entire current structure (Ding and Guo, 2002). Incorporation of dislocation density is important as it is the dislocation evolution and movement that causes both the deformation and strain hardening (Peeters et al, 2001). The classical Kocks–Mecking model relates the dislocation density to the applied stress as follows:

$$\sigma = M\alpha Gb\sqrt{\rho} \quad (2.21)$$

Where M is the Taylor factor reflecting the texture of the material, G is the shear modulus, b is Burger vector and α is a temperature dependent constant. The evolution of dislocation density ρ with respect to the imposed plastic strain can be presented as

$$\frac{d\rho}{d\varepsilon} = k_1\sqrt{\rho} - k_2\rho \quad (2.22)$$

Where

ρ = average dislocation density,

ε = plastic strain,

k_1 = material constant and describes the storage of dislocations at the forest dislocations (*i.e.* the dislocations crossing the slip plane)

k_2 = describes the dynamic recovery of the dislocation density, which is a thermally activated process of dynamic recovery by dislocation cross-slip and recombination or dislocation climb, which is a function of temperature and strain rate, and can be described as:

$$k_2 = k_{20} \frac{\dot{\varepsilon}_0^*}{\dot{\varepsilon}} \exp \frac{Q_{dm}}{RT} \quad (2.23)$$

where $\dot{\epsilon}$ is the strain rate, Q_{dm} is the activation energy for cross-slip and recombination (kJ/mol), R is the gas constant (8.314 J/mol K), T is the absolute deformation temperature (K), and k_{20} is a proportional constant.

Examples of constitutive modelling work based on the KM model can be found in studies conducted by Luo et al, (2010); Roy et al, (2012); Huang et al, (2017); Souza et al, (2019); and Hallberg et al, (2020).

2.5.2.3. Physical models for microstructure evolution

It is well known that the microstructure evolution strongly affects the deformation behaviour and mechanical properties of the material. Therefore, an appropriate representation of mechanical behaviour has to be based on microstructural state variables, which, in turn, are affected by the process history of that material (Li and Li, 2006). Recently, a number of researchers (Fedelich, 1999; Goerdeler and Gottstein; 2001; Karhausen and Roters, 2002; Ganapathysubramanian and Zabaras, 2004; Lin and Dean, 2005; Beyerlein and Tomé, 2008; Luo et al, 2010; Alabort et al, 2018; Hallberg et al, 2019) chose microstructural state variables, such as dislocation density and grain size as internal state variables in the constitutive equations. These works are very helpful in the understanding of the physical mechanisms during the plastic deformation, and hence in the designing of processing parameters. The important microstructure related parameters which are generally taken into account when establishing the theory of microstructure evolution model for plastic deformation, include dislocation density, static and dynamic grain growth, void and cavity nucleation damage, dynamic recrystallization.

2.5.3. Unified constitutive models

Unified viscoelastic constitutive equations refer to the nonseparation of the plastic (rate-independent) and creep (rate-dependent) parts of the inelastic strain. It enables the metal flow and microstructure evolution, such as strain hardening, recovery, recrystallization, grain size, and dislocation density to be coupled and described. Although the approach of the unified model is phenomenological in nature, it is based strongly on the underlying

physics observed during experimentation. These constitutive models usually write the functional dependence of strain rate as:

$$\dot{\varepsilon} = f\{\sigma, T, S_i, P_j\} \quad (2.34)$$

Where P_j is the set of j physical constants and S_i are the state variables that model microstructure related properties: *e.g.* dislocation density, grain size, damage etc. These state variables are generally dynamic: this means that they change as deformation progresses. Thus, another set of equations is introduced:

$$\dot{S}_i = g_i\{\sigma, T, S_i, P_j\} \quad (2.35)$$

Where $\dot{S}_i$ is the rate of change over time of the i th state variable.

Many researchers have developed these formulations to represent superplastic behaviour.

Most of the constitutive works available in the literature regarding superplastic deformation of titanium alloys are either experimental in nature (Gao et al, 2017; Jiang et al. 2018; Maa et al, 2019; Mosleh et al. 2019; Wanget et al. 2019) or takes into account physical models based on tensile deformation at temperature up to 900 °C (Yang et al, 2015; Alabort et al, 2016; Velayet et al. 2016; Yang et al, 2016). Moreover the most of the constitutive studies are devoted to α/β type alloys. Until now, constitutive investigations focusing on the establishment of a mechanism-based constitutive model considering the microstructure evolution of a near- α alloy have not been reported in detail, especially regarding its hot deformation mechanism at a temperature above 900 °C with a low strain rate range of 0.0005-0.01s⁻¹.

Chapter 3

Flow behaviour and governing mechanisms of a titanium alloy during superplastic forming

3.1. Introduction

The superplasticity of material is intrinsically linked to its microstructures ([Sun et al, 2012](#); [Xu et al, 2012](#) and [Wang et al, 2013](#)), where the evolved microstructures concurrently affect the deformation mechanisms and hence govern the superplasticity of the material during SPF. Additionally, the final distributions of the microstructures on the post-formed parts also determine the in-service mechanical performances ([Sinha et al, 2012](#); [Liu et al, 2017a](#)) and the structural integrity of the parts. For instance, the grain size $< 10\mu\text{m}$ of the SPF component is considered to benefit the component fatigue life. Therefore, examining the microstructure evolutions during the deformations are essential for developing optimized processing windows to achieve better formability and the desired final microstructures and in-service performances.

Comprehensive studies on the microstructure evolutions have been performed on α/β type alloys, such as Ti-6Al-4V ([Mikhaylovskaya et al, 2017](#)), in superplastic deformation. However, rare studies were performed on the TA15, which is a promising alloy that has been increasingly used in the high-speed lightweight aircraft.

The deformation temperatures of the near- α titanium alloys are usually 30–50 °C below the β -transus temperature (*i.e.* ~ 985 °C for TA15) to achieve high ductility and thermal stability during SPF and the equiaxed microstructures after SPF ([Ning et al, 2015](#)). These suggested deformation conditions drive the needs for detailed investigations of both the superplastic behaviours and the associated underlying deformation mechanisms and microstructures of the near- α alloys, which have not been focused on, but are greatly significant for the material flow behaviour and the in-service performances.

This chapter provides a better understanding of the superplasticity phenomena required to perform SPF via a detailed study of the tensile deformation behaviour and the microstructure evolutions of TA15 alloy. Moreover, the investigation above 900°C is particularly relevant for the industrial application of superplasticity due to the possibility of combining superplastic forming with diffusion bonding for producing a hollow multilayer sandwich structure such as rocket exhaust nozzle, jet engine casings or turbine blades. Such understanding can be potentially translated into validated materials models, which can accurately reflect the underlying mechanisms of superplasticity with reasonable accuracy. Consequently, this work could contribute to widening the industrial applications of this promising alloy.

With the above in mind, the main objectives of this chapter are as follows:

- i. To evaluate the superplastic flow behaviour of TA15 titanium alloy at temperatures ranging from 880 °C to 920 °C and strain rates ranging from 0.0005s^{-1} to 0.01s^{-1} .
- ii. To investigate the grain size, texture, phase, and GND evolutions.

With the aforementioned objectives, this chapter addresses the following main gaps in the literature.

- i. Other than being the first comprehensive microstructure evolution investigation during superplastic deformation, the novelty of this work lies in the use of interrupted tensile data for grain size, texture, and GND evolution.
- ii. There is no comprehensive study to date, which uses a consistent framework across multiple microstructural attributes (*i.e.* phase change, grain size, grain orientation, texture, and GND) in superplastic deformation.

3.2. Materials and Methods

3.2.1. Material, specimen design and thermal mechanical testing

The current study used a rolled TA15 alloy sheet with a thickness of 2 mm. This thickness corresponds to the practical SPF in industries. The β -transus temperature of this material

is 985°C. The energy dispersive X-ray technique has been used to determine the elemental composition of the alloy, and results are shown in [Table 3.1](#).

Table 3. 1. Chemical compositions of TA15 alloy.

Element	Al	Zr	Mo	V	Ti
Mass%	6.78	2.0	1.24	1.32	Bal.
Impurity	Fe	Si	C	N	
Mass%	0.050	0.01	0.0072	0.0038	

The superplastic tensile tests were performed using LETRY-5kN electro-hydraulic servo mechanical testing machine produced by Lichuang Machine Company, Xi'an, China ([Figure 3.1a](#)). The pull rods of the machine was equipped with cylindrical bearings at both bar ends: upper and lower. This allows the specimen to sit parallel to the applied tension load. The specimen grip boxes were joined to the pull rods using short, double-threaded link rods. The sample dimensions were provided in [Figure 3.1b](#). Superplastic tensile specimens with a gauge length of 15 mm, a width of 6 mm, and a thickness of 2 mm were cut in parallel to the rolling direction of the sheet. The dimensions of the tensile samples were determined according to the tensile test setup. Before the tensile testing, the gauge range of each sample was mechanically polished using a series of grinding silicon-carbide papers and finished with the 600 grade.

In preparation for the superplastic tensile tests, the surfaces of the tensile specimens were coated with oxidation-resistant glass before the tests to avoid the formation of the alpha case layer, which is a surface hardened layer that may be formed at high solubility of oxygen environments ([Leyens and Peters, 2003](#)). The alpha case layer will affect the mechanical properties and lower the tensile ductility during the superplastic tests, and hence reduce the reliability of the tests. On the other hand, to ensure the accuracy of the controlled temperatures, a conventional three-zone furnace was used to guarantee the uniformity of the temperatures. The temperature was monitored with the attached

thermocouples, which were spot-welded on different positions of tested samples inside the upper and lower grip boxes. Additionally, the temperature was recorded and feedback-controlled to ensure that the testing temperature is within $\pm 3^\circ\text{C}$ of the target temperature. This enables higher accuracy of the tests, where the ASTM standard for superplastic testing allows a tolerance of $\pm 6^\circ\text{C}$. The load cell was installed on the bottom fixed crosshead to avoid any heat input due to the radiation from the furnace and enable more accurate load records.

The detailed procedures of the superplastic tensile testing and heat treatment are presented in [Figure 3.1c](#). The heating rate is determined based on the average heating rate of a conventional furnace heating, whilst avoiding any possible temperature overshooting issues. The specimens were firstly heated using $10^\circ\text{C}/\text{min}$ to a temperature that was 100°C lower than the target temperature. Then a slow heating rate, $3^\circ\text{C}/\text{min}$, was applied until the specimens achieving the target deformation temperature, T_{target} . Subsequently, the specimens were held at T_{target} for 15 minutes to eliminate the thermal gradient of the sample and enable the furnace to attain an equilibrium temperature distribution. Finally, the specimens were superplastically deformed at the designed temperatures and strain rates to various strain levels, followed by water quenching to freeze the deformed microstructures. To ensure the superplasticity, the deformation rate must be constant. Thus, the machine must increase the cross-head speed as the sample is subjected to elongation. Assuming constant volume, the machine cross-head velocity V was determined as

$$V = \dot{\epsilon}_t [l_0(1 + e)] \quad (3.1)$$

Where l_0 is the initial gauge length, e is the engineering strain defined as $e = (l - l_0)/l_0$ and $\dot{\epsilon}_t$ is the target or total strain-rate and can be decomposed in different terms as

$$\begin{aligned}\dot{\varepsilon}_t \\ = \dot{\varepsilon}_p + \dot{\varepsilon}_e + \dot{\varepsilon}_m\end{aligned}\tag{3.2}$$

where $\dot{\varepsilon}_p$ is the sample plastic strain rate, $\dot{\varepsilon}_e$ is the sample elastic strain rate and $\dot{\varepsilon}_m$ is the elastic strain rate of the different parts of the loading machine. Since the applied load and strain-rates were relatively low, and the load frame has high rigidity, the elastic strain rate term for the machine was excluded. The sample elastic strain-rate was also omitted since this was estimated a couple of orders of magnitude lower than the plastic counterpart. The target strain rate ($\dot{\varepsilon}_t$) was achieved by measuring and recording the instantaneous length (l) of the specimen in real time and then adjusting and controlling the instantaneous cross-head velocity V of the specimen through the PID control system.

The deformation temperatures are 880°C and 920°C and strain rates are 0.01s⁻¹, 0.001s⁻¹ and 0.005s⁻¹. The full matrix of the testing conditions (*i.e.* deformation temperatures, strain rates, and strain levels) are given in detail in [Figure 3.2](#). For clarity, the tests under each condition were firstly stretched to fracture to obtain the total elongation and the viscoplastic stress-strain curves. Then tests under identical conditions were interrupted at selected strain levels, as given in [Figure 3.2](#), aiming to examine the microstructure evolutions and distributions. Compared to conventional hot tensile testing, superplastic tensile tests take too long and are difficult to perform, and usually only one test per test condition is performed. So only one specimen was tested for each testing condition.

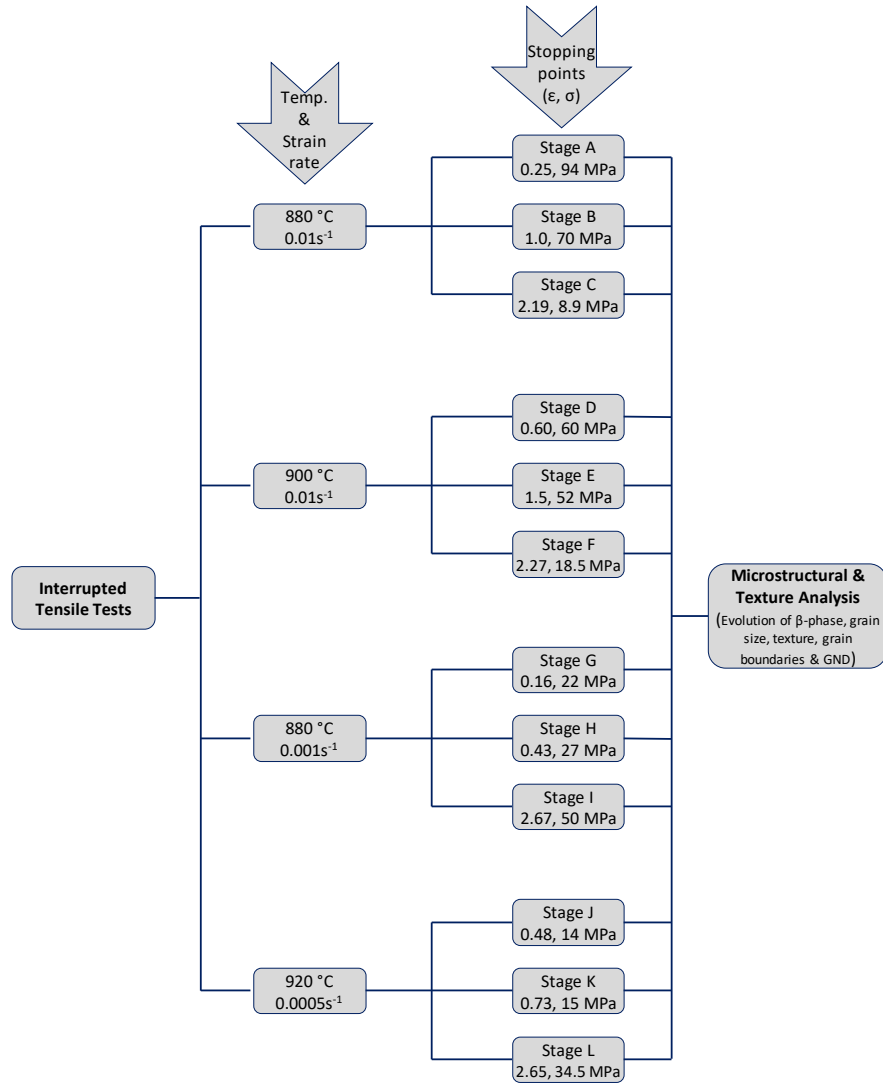


Figure 3. 1. The interrupted thermomechanical Testing conditions for microstructure examinations

3.2.2. Microstructure Observation

The deformed and as-received samples were subjected to a scanning electron microscope (SEM), and electron backscatter diffraction (EBSD) characterization to reveal the superplastic deformation mechanism. The microstructure characterization samples were

cut along the transverse direction, as shown in [Figure 3.1b](#). These samples were mechanically grinded using 2500 and 4000 grit papers followed by polishing with 70:30 colloidal silica (40 nm particle) and hydrogen peroxide solution. The samples for SEM backscatter characterization were subsequently etching by Krolls reagent (1-3mL HF, 2-6mL HNO₃, and 100mL water) for 10 seconds.

EBSD can measure the grain orientation, grain size, and texture variation by point-to-point interrogating and indexing the formed electron diffraction patterns. Also, regarding the stored dislocation density measurement, the EBSD technique can reveal the geometrically necessary ones, which cause geometrical lattice rotation in the crystal lattice. In contrast, the statistically stored dislocations are not accessible by EBSD. Even EBSD cannot reveal the total dislocation density; it is probably the most appropriate technique to quantitatively evaluate the stored dislocation density (GND) in a statistically representative volume as it allows both the observation of the dislocation structures and in the meantime gives quantitative average dislocation density value over a relatively large mapping area ([Jiang et al, 2013](#)). A reasonable correlation was achieved between the EBSD recovered GND density and total dislocation density by comparing various dislocation characterization techniques, such as TKD-TEM ([Liang et al, 2019](#)) and Pitting techniques ([Wu et al, 2019](#)). This confirms the representability of the EBSD estimated GND density for the total stored dislocation density. Thus, EBSD is chosen as the characterization tool in this study.

The EBSD analysis was performed with a step size of 0.4 μm and a scan area of $248.4 \times 186.9\mu\text{m}$. The alpha grain size, orientation maps, pole figures (PFs), inverse pole figures (IPFs), and geometrically necessary dislocations (GND) density were calculated. The EBSD scan surface is on the rolling free surface (RD-TD). [Table 3.2](#) shows the value of parameters specified by the standard for grain measurements using EBSD ([Mingard et al, 2009](#)).

The GND densities were calculated from the EBSD measurements via the MTEX toolbox using a methodology outlined below. Due to the existence of hard and soft orientations in the predominant HCP α phase ([Littlewood and Wilkinson, 2012](#)) at a deforming temperature of 880-920°C, the local deforming mechanism is complex. The local deformation mechanism at the microstructural level can be investigated by calculating

GND density and the distribution in individual grains (He et al, 2016). The state of dislocations represented by a second-order dislocation tensor proposed by Nye (1953) was resolved by studying the rotation gradient tensor from EBSD measurements. As the conventional 2D EBSD can only collect information on the specimen surface, only lattice rotation gradients along x_1 and x_2 directions are measurable. The GND calculation process is based on the method described by Pantleon (2008) using six available curvature tensors (five dislocation density tensors and one difference between two dislocation tensors).

The GND density was calculated using MATLAB™ toolbox MTEX with self-developed routines. A total of 33 potential dislocations are likely to present in α phase: 3 types of $\langle a \rangle$ edge on basal planes; 3 types of $\langle a \rangle$ edge on prismatic planes; 6 types of $\langle a \rangle$ edge on pyramidal planes; 3 types of $\langle a \rangle$ screw; 12 types of $\langle a+c \rangle$ edge on first-order pyramidal slip planes; 6 types of $\langle a+c \rangle$ screw. Different dislocation types have different line energies and contributions to the total energy of the dislocations in the material. The line energy of each kind of dislocations was used as weights; the objective function was to find the minimal weighted sum of all dislocation densities. According to Frank's rule, $E_{el} = \alpha G b^2$ (where α is a constant, G is the shear modulus, b is the magnitude of the Burgers vector), the energy of a $\langle a+c \rangle$ edge dislocation is bigger than a $\langle a \rangle$ edge dislocation by a factor of 3.51. The energy of a $\langle a \rangle$ edge dislocation shall be bigger than $\langle a \rangle$ screw dislocation by a factor of $1/(1-\nu)$ (Pantleon, 2008), where ν is the Poisson's ratio.

Table 3. 2. EBSD operating parameters (Mingard et al, 2009)

Description	Value
Grain misorientation	5 degrees
Points per grain	>100

Cleanup (dilation)	<10% of points modified
Scan area	>50 whole grains
Total no. of grains	>500

The EBSD measurements were performed by Hitachi S-3400N at an operating voltage of 25kV, an aperture size of 120 μm , a working distance of 20 mm and a step size of 0.4 μm . These images were used to quantify the α and β -volume fraction as the amount of β phase retained at room temperature in near α alloy is very low, and it is hard to index this phase reliably using EBSD (Davies, 2009). Note that the α -phase can be either α_p (primary alpha), which is present at high temperature or α_s (secondary alpha) which forms when the β phase transforms during the subsequent cooling. This α_s was used to quantify the β volume fraction at the hot superplastic deformation stage. The ImageJ software was then used to measure the relative percentages of α and β grains by the large contrast between these phases.

3.3. Results

3.3.1. Microstructure of the as-received material

According to the BSE micrograph (Figure 3.3a), the initial as-received microstructure typically consists of a two-phase structure of α (dark region), and β (bright region). The volume fraction of α and β phase are estimated to be 94% and 6% respectively, and β -phase is randomly distributed in α -phase matrix. The histogram map in Figure 3.3b and EBSD map in Figure 3.3c displays the grain size distributions, where the initial material mainly consists of fine equiaxed grains with an average grain size of less than 4 μm . The grain size distribution of the as-received materials, varying from 0.1 μm to 11.5 μm .

Figure 3.3 (c, d & e) depicts the EBSD Inverse Pole Figure (IPF) Z results, showing grain orientation and texture distribution of the as-received material. It can be seen that the initial microstructure consists of fine equiaxed grains but with a preferred orientation, which may be attributed to the pre-rolling and subsequent insufficient annealing treatment. It can be observed that the microstructure of the as-received sheet exhibited strong rolling textures,

which is dominated by basal texture direction [0001] with the maximum texture intensity of 4.9 multiples of random distribution (MRD). The GND density distribution map of this cold-rolled titanium is shown in Figure 3.3f, which demonstrates that the initial material has a high stored dislocations density as the average GND density is calculated as $3.47 \times 10^{14} \text{ m}^{-2}$.

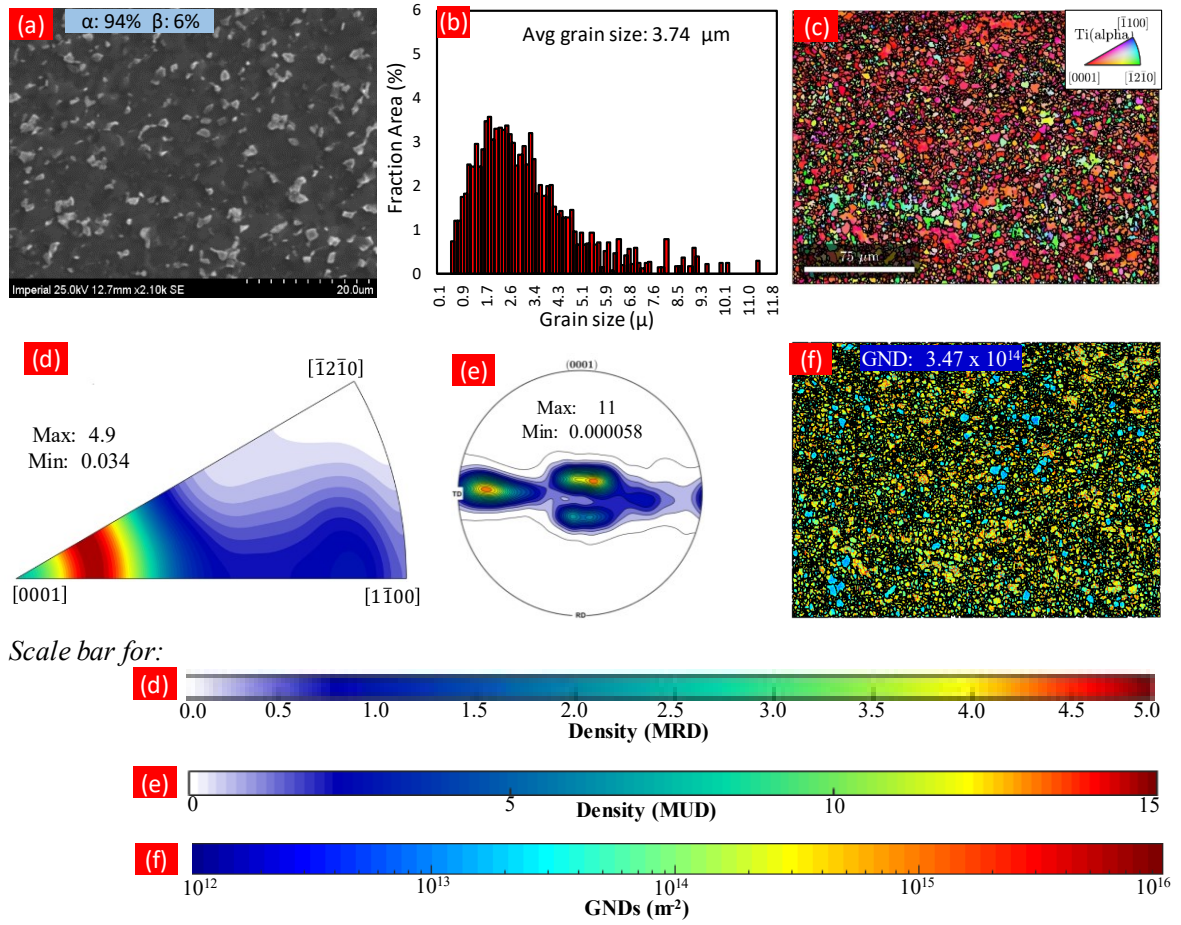


Figure 3. 2. SEM and EBSD analyses of the microstructures in the initial material: (a) BSE image (b) grain size distribution (c) grain orientation map (d) inverse pole figure (e) pole figure (f) GND density map

3.3.2. Viscoplastic behaviors under superplastic conditions

- i. The true stress-strain curves for the hot tensile tests conducted at $880^\circ\text{C} / 0.01\text{s}^{-1}$, $900^\circ\text{C} / 0.01\text{s}^{-1}$, $880^\circ\text{C} / 0.001\text{s}^{-1}$ and $920^\circ\text{C} / 0.0005\text{s}^{-1}$ are presented in Figure 3.4. The effects of temperature on viscoplastic behaviour under the superplastic condition can be evaluated by comparing the flow stress curves of $880^\circ\text{C} /$

0.01s^{-1} and $900\text{ }^{\circ}\text{C} / 0.01\text{s}^{-1}$. Whereas the effects of strain rates can be investigated by comparing $880\text{ }^{\circ}\text{C} / 0.01\text{s}^{-1}$ and $880\text{ }^{\circ}\text{C} / 0.001\text{s}^{-1}$. $920\text{ }^{\circ}\text{C} / 0.0005\text{s}^{-1}$ is an extreme condition with the highest temperature and lowest strain rate as a reference. All flow stress curves exhibit excellent superplastic behaviour, and the achieved elongations are tabulated in [Table 3.3](#). A graphical analysis of the curves confirms the following:

- ii. No steady flow stress was observed under all testing conditions, where significant strain-hardening and -softening behaviors present, depending on the strain rates and temperatures.
- iii. Strain-softening dominants at higher strain rates (i.e. $0.01/\text{s}^{-1}$), while strain-hardening dominants at lower strain rates (i.e. $0.001/\text{s}^{-1}$ and $0.0005/\text{s}^{-1}$).
- iv. The morphologies of the strain-softening flow curves (*i.e.* under $880\text{ }^{\circ}\text{C} / 0.01\text{s}^{-1}$ and $900\text{ }^{\circ}\text{C} / 0.01\text{s}^{-1}$) could be classified into (a) elastic phase, (b) transition phase (c) flow softening phase, and (d) localized necking phase.
- v. The morphologies of the strain-hardening flow curves (*i.e.* under $880\text{ }^{\circ}\text{C} / 0.001\text{s}^{-1}$ and $920\text{ }^{\circ}\text{C} / 0.0005\text{s}^{-1}$) could be classified into (a) elastic phase, (b) transition phase (c) work hardening phase, and (d) localized necking phase.
- vi. At the lowest temperature (880°C) and the highest strain rate (0.01s^{-1}) the flow curve exhibits continuous flow softening;
- vii. At medium temperature (900°C) and the highest strain rate (0.01s^{-1}), the work hardening overcome the flow softening between the true strain of 0.5 (the stage D) and 1.5 (stage E).

Further rationalizations of the stress-strain relationships are discussed in section 3.4.

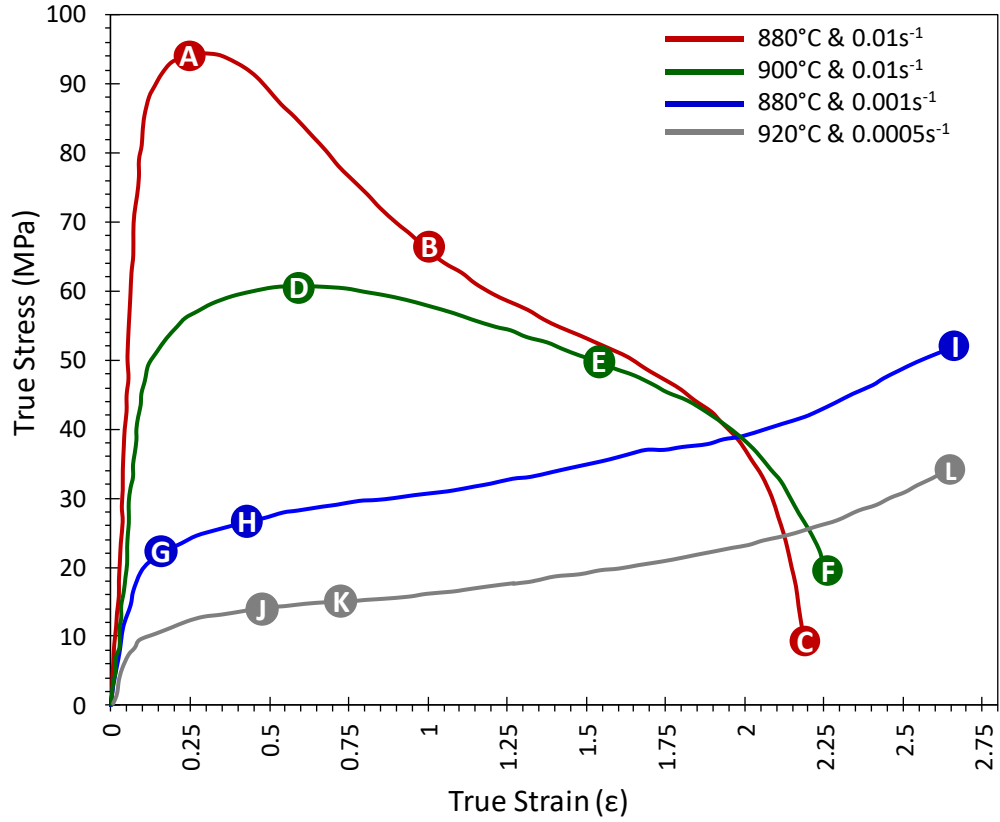


Figure 3. *Viscoplastic behaviours of TA15 under the deformation conditions: 880 °C/0.01s⁻¹, 900 °C/0.01s⁻¹, 880 °C/0.001s⁻¹ and 880 °C/0.0005s⁻¹. Note that labels A to L denote the interrupted stages of the tensile tests, corresponding to Figure 3.2.*

Table 3. 3. Percentage elongation during superplastic deformation of TA15

Temperature (°C)	Strain rate (s ⁻¹)	Elongation δ
880	0.01	749%
900	0.01	800%
880	0.001	1470%
920	0.0005	1400%

To calculate the strain rate sensitivity and activation energy, the relationship between the flow stress (σ), strain rate ($\dot{\epsilon}$) and temperature (T) during hot deformation can be expressed using the below Arrhenius equation:

$$\dot{\epsilon} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (3.3)$$

where $\dot{\epsilon}$ is the strain rate, σ the flow stress, A is a material constant, R is the universal gas constant, T is the absolute temperature in kelvin, Q is the activation energy and n is the stress exponent, which is the inverse of the strain rate sensitivity m :

$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right|_{\epsilon, T} \quad (3.4)$$

Taking natural logarithm on both sides of Eq (3.3), it can be expressed as follows:

$$\ln \dot{\epsilon} = \ln A + n \ln \sigma - \frac{Q}{RT} \quad (3.5)$$

For a constant strain rate $\dot{\epsilon}$ and applying partial derivative of Eq. 3.5, the activation energy, Q , can be expressed as:

$$Q = \left. \frac{R \partial \ln \sigma}{m \partial (1/T)} \right|_{\dot{\epsilon}} \quad (3.6)$$

The schematic representation of the results derived from Eq. 3.4 and Eq. 3.6 are shown in Figure 3.5.

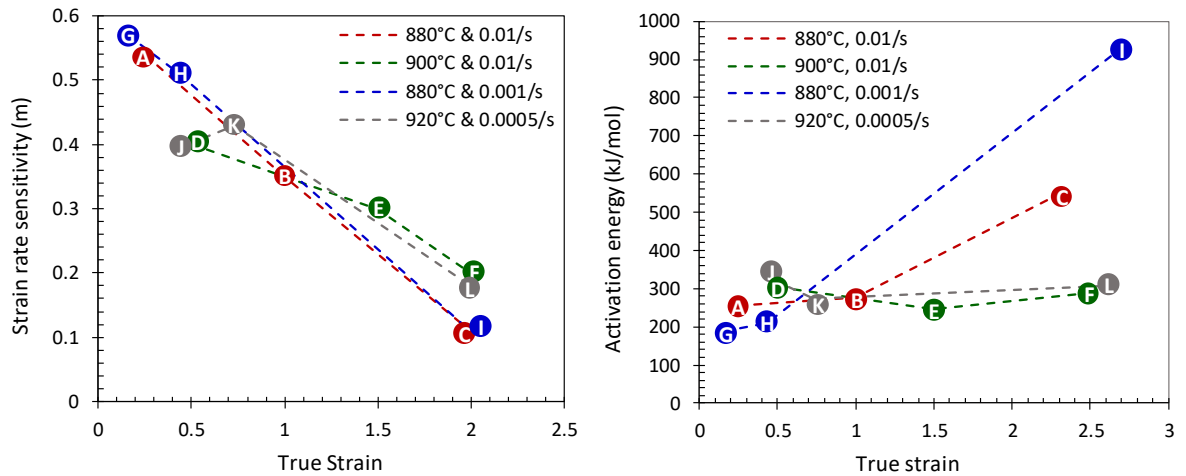


Figure 3. 4. (a) Strain rate sensitivity (b) activation energy

Further rationalizations of Figure 3.5 are discussed in section 3.4.

3.3.3. Evolution of Beta phase

The microstructure of undeformed material mainly consists of the α -phase (HCP) with 6% retained β -phase (BCC). All tensile tests are performed in the temperature range from 880 to 920 °C, hence phase transformation, from the α to β , may occur. After water quenching, the β -phase will be entirely or partially transformed into a martensitic type α , called as secondary α_s . This secondary α_s is used to quantify the β -phase transformation during thermomechanical tests. Figure 3.6 shows the evolution of β -phase using SEM-BSE micrographs for different strain rates and deformation temperatures. For the temperature 880 °C and strain rate of 0.01s^{-1} , it can be observed that at the early stage of the deformation, the β -phase increases by 10% from the stage A to the stage B. At stage B, the quantitative fraction of the β -phase is 17% and at fractured point C it is quantified as 20%, so the increase is just 3%. At the same time, the corresponding flow curve shows a sharp drop of stress value from 70 MPa to 10 MPa, and this drop alone can't be attributed to the increase of β -phase.

For the deformation conditions of 900 °C and 0.01s^{-1} , it is revealed that at stage D, E and F, the β -phase fraction is quantified approximately as 19%, 25%, and 49% respectively. From the initial stage to stage D, the fraction of β -phase increases by ~13%.

For the strain rate of 0.001s^{-1} and temperature 880 °C condition, the fraction of β -phase at stage G, H, and I is quantified as 18%, 25%, and 32%, respectively. There is an increase ~7 % in the fraction of β -phase in the transition from stage G to stage H, the stages which lie relatively close to each other, as shown by the morphology of the corresponding flow curve. While on the other hand, the increase in β -phase is also 7% between the stages H and I, the stages which are relatively highly departed in contrast to the stages G and H. Similarly from the flow curve it is evident that there is continuous strain hardening beyond the stage G. Keeping in the view of the softness of β -phase and the relatively low transformation of β -phase from the stage G to I, this strain hardening can be potentially due to the grain growth. For this flow stress curve, the maximum elongation has been

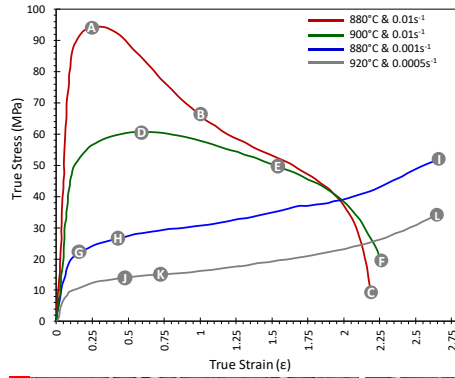
reported. The literature ([Seshacharyulu et al, 2000](#)) suggests the optimum α/β ratio of 80:20 for grain boundary sliding (GBS) to be the dominant mechanisms for superplastic deformation, as the sliding resistance analysed for the different boundaries is lowest in the α/β boundary, and increases on the order of $\alpha/\beta \ll \alpha/\alpha \sim \beta/\beta$ ([Kim et al, 1999](#)). Therefore, the features of these results (α/β ratio 75:25) found in this study suggest that the optimal GBS was achieved and, as a result, the maximum elongation.

Finally, for the strain rate of 0.0005s^{-1} and temperature 920°C case, the fraction of β -phase at the stage J, K and L are quantified as 15%, 15%, and 40%, respectively. Like the flow curve of 880°C and 0.001s^{-1} , the stage J and K on the curves are relatively close to each other, and the fraction of β -phase remains approximately constant, while from stage K to the stage L, the fraction of β -phase is increased by 25%. Moreover, for the said flow curve, the second maximum elongation has been achieved, which can be attributed to the high fraction of β -phase, 15% at stage K, and almost evolved to 40% at the point of fracture.

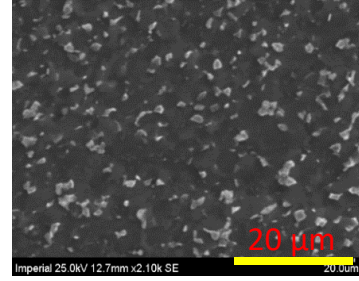
By comparing the phase transformation for $880^\circ\text{C}/0.01\text{s}^{-1}$ to $900^\circ\text{C}/0.01\text{s}^{-1}$, it can be observed that in the final fractured samples, the phase transformation considerably increased from 20% to 49% which means that for a specific strain rate, the increase in the deformation temperature can significantly accelerate the evolution of β -phase.

Referring to the results at $880^\circ\text{C}/0.001\text{s}^{-1}$, the evolution of β -phases changes from 20% to 30%. At a constant operating temperature of 880°C , as the strain rate varies from 0.01s^{-1} to 0.001s^{-1} , the microstructure of the specimen changes in two aspects. One is that the grains of α and β phases grow with decreasing strain rate, and the other is that the volume fraction of the β -phase increases with decreasing strain rate. The phenomena of α and β phases grains growth are the main possible cause for the increase in stress level for the flow stress curve representing the superplastic deformation at 880°C and strain rate of 0.001s^{-1} . Being having BCC structure, the β -phase requires relatively low flow stress values at higher temperatures due to its abundant slip system in contrast to α phase (HCP structure) ([Meier et al, 1991](#)). Thus, the increase of β phase fraction is of benefit to the decrease of the flow stress. The elongation of the specimen increases with the increase of the β phase fraction at a strain rate of 0.001s^{-1} (880°C), which shows that the β phase fraction increase improves the superplasticity of TA15 alloy. It is generally believed that the α/β and β/β

boundaries increase and α/α boundary decreases with the β phase fraction increase; thus the superplastic formability increases owing to the contribution of α/β boundary sliding (Kim et al, 1999). When the fraction of β -phase reaches about 30% (at 880 °C/0.001s⁻¹& 920°C/0.0005s⁻¹), the superplasticity of TA15 reaches the highest value. However, the increase of β volume fraction can also induce microstructural changes associated with static and dynamic grain over-growth, which leads to the deterioration of superplasticity (Guo et al, 2017). When the temperature is 900 °C, 49% β -phase is attained. This high β -phase volume fraction leads to a decrease in the elongation. In all of the deformation stages, the morphology of the phases following very high elongation remained equiaxed, which is a typical characteristic of superplasticity (Neih et al, 2005, Kaibyshev, 2012).



Initial Undeformed Sample



α : 84% β : 16%

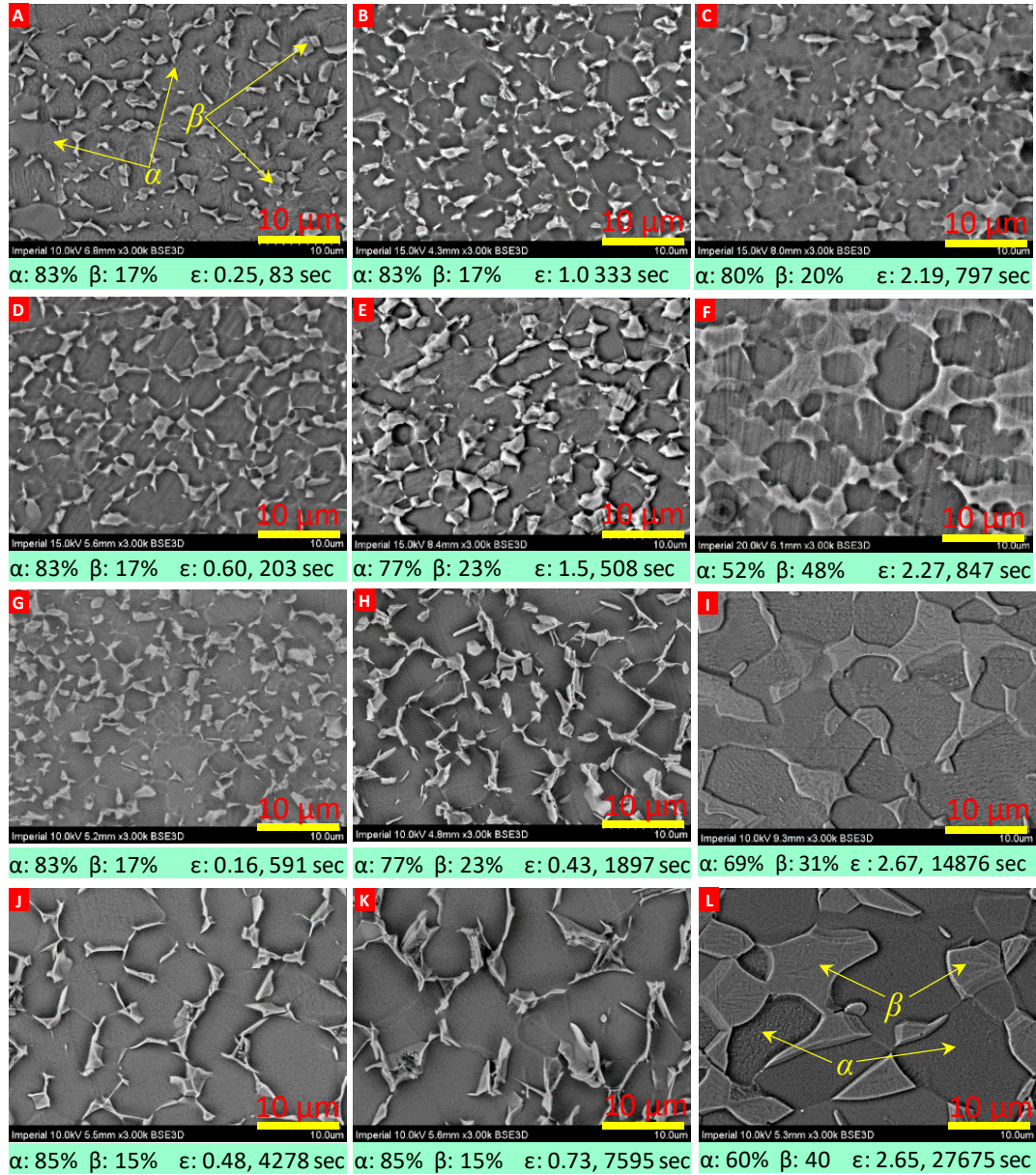


Figure 3. 5. BSE images of phase structure evolutions during deformations at stages A, B, C, D, E, F, G, H, I, J, K & L of stress-strain curves.

3.3.4. Grain size evolution

A quantitative description of grain size distributions and the average grain sizes under the studied conditions are shown in [Figure 3.7](#). It can be observed that moving forward from stage A to C for the deformation conditions of $880^{\circ}\text{C}/0.01\text{s}^{-1}$, the average grain sizes decrease from $7.92\mu\text{m}$ to $3.90\mu\text{m}$, which is in good agreement with the flow softening behaviour of the corresponding stress-strain curve. At the stages, A, B, and C, the major area fraction of grains are covered by the sizes of $3.7\mu\text{m}$, $3.5\mu\text{m}$, and $4\mu\text{m}$, respectively. Overall as the deformation continues from stage A to stage C, the volume fraction of the larger grain size increases while the volume fraction of smaller grain size decreases. Similarly, moving from point D to F for the deformation condition of $900^{\circ}\text{C}/0.01\text{s}^{-1}$, the average grain size varies from $6.2\mu\text{m}$ to $4.91\mu\text{m}$, which again is in agreement with the flow softening behaviour for the corresponding stress-strain curve. No apparent grain growth (compared to the grain size in the initial microstructure) is observed for the specimens deformed with a strain rate of 0.01s^{-1} and at temperature 880°C and 900°C .

On the other hand, among the specimens tested at the lower strain rates of 0.001s^{-1} and 0.0005s^{-1} , exhibit significant grain growth estimated as $\sim 116\%$ and $\sim 301\%$ to the initial as-received microstructure, respectively. Significant dynamic grain growth is found for points G, H, I and J, K, L which corresponds to the deformation condition of $880^{\circ}\text{C} \& 0.001\text{s}^{-1}$, $920^{\circ}\text{C} \& 0.0005\text{s}^{-1}$, respectively. It can be seen from [Figure 3.7](#) that the grain growth for these conditions is initiated at the beginning of the superplastic deformation and the grain size increases with an increase of the strain. Further rationalization of the grain size evolution is discussed in detail with the correspondence of GND evolution in section 3.4.

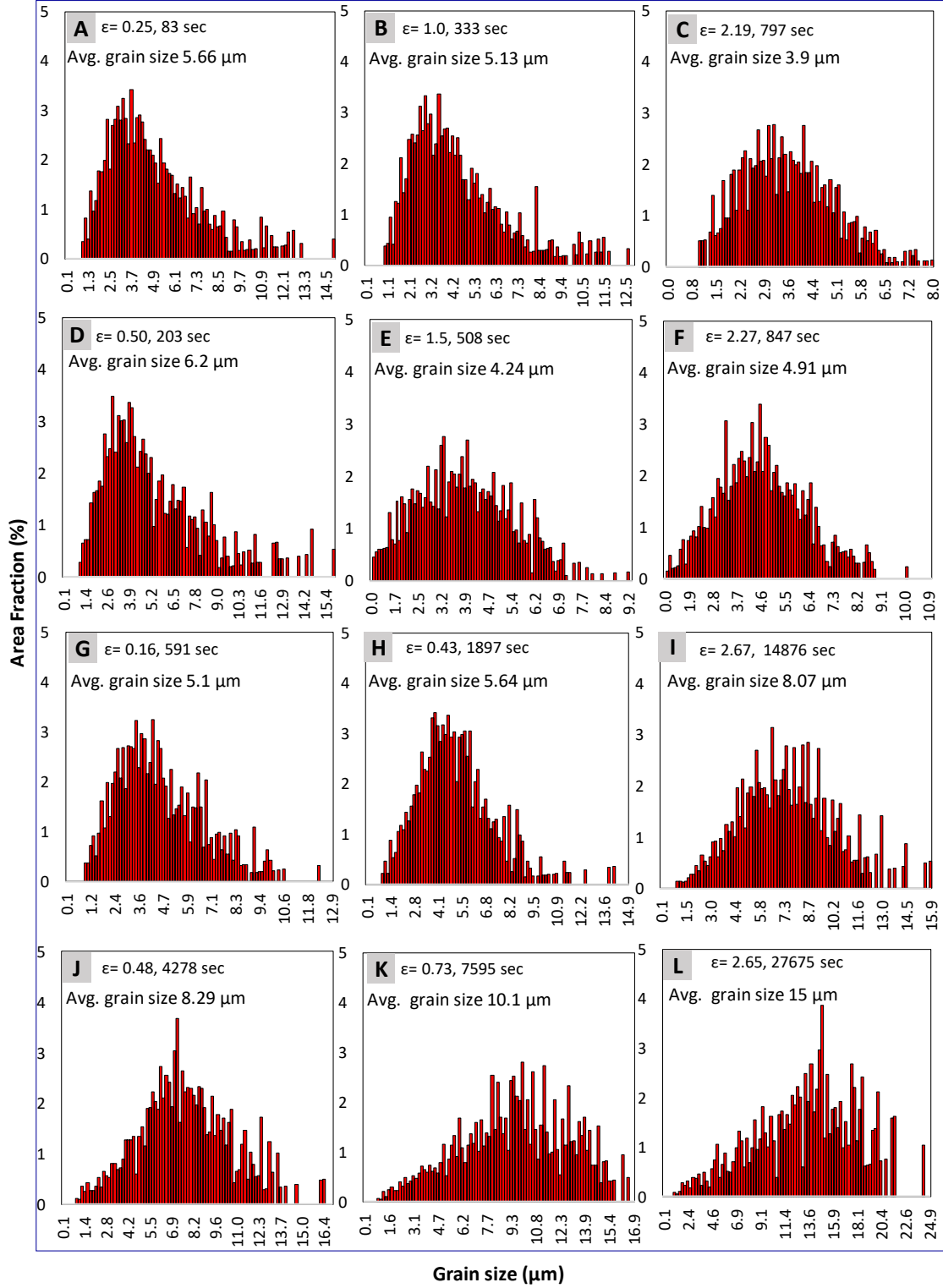


Figure 3. 6. Evolution of grain size in TA15 during deformation stages (A, B, C, D, E, F, G, H, I, J, K & L) illustrated in Stress-Strain Curves.

3.3.5. Texture Evolution

During the superplastic deformation process, the sliding surface and sliding direction of the grains gradually shift in the main deformation direction and progressively change the original crystal orientations. Such a phenomenon is termed as texture evolution. It is well known that the anisotropic mechanical properties of a material are significantly affected by its texture (Chen et al, 2014) and the texture evolution also reflects the occurrence of grain boundary sliding mechanism. Therefore, studying the texture evolution and distribution during the superplastic deformation is of value.

Texture analysis has been used to study the mechanisms of superplasticity in various alloys (Panicker et al, 2009; Korla et al, 2010; Watanabe et al, 2013; Liu et al, 2017b; Jia et, 2019). Figure 3.8 shows the grain orientation maps, and the corresponding inverse pole figures and pole figures with different deformation levels are presented in Figure 3.9. The colours in Figure 3.8 represent the crystal orientations of the alpha phase, where α colour key is provided. The pole figures, as given in Figure 3.9 of $\langle 0001 \rangle$ contain both the maximum and the minimum texture intensities under all conditions. The texture intensities from the colour bars are expressed in multiples of random distribution (MRD) and multiples of the uniform density (MUD) for the inverse pole figures and the pole figures, respectively.

Considering the texture evolution of $880^\circ\text{C}/0.01\text{s}^{-1}$ (*i.e.* stages A, B and C), it can be observed from Figure 3.8 that at a low strain value of 0.25 (*i.e.* stage A), the microstructures retained sharp textures, which is dominated by $[0001]$ basal textures (Figure 3.9), and the maximum texture intensity is estimated as 6.7 (MUD). Further deformation slightly weakened the texture (*i.e.* stage B), where the maximum texture intensity to 5.8. At the fractured stage C, the grain orientation distribution significantly randomized with a maximum texture intensity of 2.4, as shown in Figure 3.9. Considering the grain sizes, as shown in Figure 3.8, the initial coarse grains at the stage A are refined from $5.66\ \mu\text{m}$ to $3.9\ \mu\text{m}$ at stage C. The concurrently refined grain sizes may facilitate the grain rotation and sliding and lead to a weaker final texture. Moreover, the retention of the basal plane $[0001]$ for all three deformations, the stages, *i.e.*, from stage A to fractured stage C, can be

attributed to the slow grain rotation during superplastic deformation. These results provide evidence to support that the GBS is active, and the grain rotation acts as a strain accommodation mechanism.

For the deformation conditions of $900^{\circ}\text{C}/0.01\text{s}^{-1}$ (*i.e.* stage D, E and F), it can be observed that the maximum texture intensity monotonously decreases from 4.2 to 1.4 (MRD), which leads to the randomization of the initial texture, as shown in Figure 3.9, indicating the occurrence of the grain boundary sliding mechanism. As shown in Figure 3.9, the strong basal plane textures [0001] at stage D is completely disappeared at the stage F. From the grain orientation map in Figure 3.8, the average grain sizes reduced from $6.2\text{ }\mu\text{m}$ (*i.e.* stage D) to $4.24\text{ }\mu\text{m}$ (*i.e.* stage E), and then $4.91\text{ }\mu\text{m}$ (*i.e.* stage F). The smaller grain sizes during the deformation may contribute to the grain rotation and leads to weakened textures. It is worth mentioning that the texture intensity at stage E closely matches that at stage C (the fractured stage). This suggests that GBS and grain rotation can be considered as a prime deformation mechanism at the deformation conditions of $900^{\circ}\text{C}/0.01\text{s}^{-1}$.

For the deformation condition of $880^{\circ}\text{C}/0.001\text{s}^{-1}$, the initial stage G has an average grain size of $5.1\text{ }\mu\text{m}$, and the texture intensity decreases from the initial undeformed 11 to 6.2, which indicates the phenomena of GBS/grain rotation. The drop in the texture intensity to 4.5 at the next stage H is much bigger for a corresponding small variation in true strain from 0.16 to 0.43, which suggests that at this stage, the GBS/grain rotation is the dominant mechanism. The average grain size at stages H and I are $5.64\text{ }\mu\text{m}$ and $8.07\text{ }\mu\text{m}$, respectively, which can still be considered as favourable for the GBS/grain rotation to occur at the deformations of $880^{\circ}\text{C}/0.001\text{s}^{-1}$ so consequently the texture is randomized at fracture stage I.

For the deformation conditions of $920^{\circ}\text{C}/0.0005\text{s}^{-1}$ (*i.e.* stage J, K and L), the maximum texture intensity significantly dropped at the early stage of the deformation from the 4.9 (the initial undeformed material) to 2.6 (stage J). As the deformation continues from stage J to L, a slightly drop in the maximum texture intensity was observed from 2.6 to 2.1. The evolution of the texture intensity values indicates the facilitated GBS at the early stage of the deformation (before stage J), while the contribution of the GBS reduces at the later stage of the deformation (stage J to L).

Referring to the corresponding stress-strain curve in [Figure 3.8](#), the phenomenon of continuous strain hardening was observed for the deformation conditions of $920^{\circ}\text{C}/0.0005\text{s}^{-1}$. In addition, referring to the evolution of grain size in [Figure 8](#), it can be inferred that there is a continuous grain growth. Thus, the relatively weak drop in textural intensity from stage J to stage L may be attributed to the grain growth, which limits the extents of GBS and grain rotation, hence a strain hardening behaviour. It is worth noting that the significant randomization of the texture is achieved at the very first the stage (before stage J) rather than at the fracture stage, which was observed in the other cases of the deformation in this study. The soaking time of 15 minutes at this high temperature is believed to be the reason for the observed behaviour. Exposing the as-received material to a temperature of 920°C for 15 minutes, the possibility of dynamic recovery (DRV), and dynamic recrystallization (DRX) can be very fast, and the ideal grain size required for GBS can be achieved during the soaking period. In addition, when the sample is pulled at 920°C with a relatively low strain rate of 0.0005s^{-1} , the GBS/grain rotation can work efficiently, and the initial texture of 4.9 is considerably reduced to 2.6 just at the beginning (the stage J) of the stress-strain curve. There is continuous grain growth, which will lead to slow grain rotation and GBS and increases with the flow stress. At a high annealing temperature of 920°C , the recrystallized grain growth occurs and results in a weakening of cold rolling texture. Therefore, at the start of the tensile deformation, the texture components intensities were dropped.

Moreover, the optimum strain rate for superplasticity increases with decreasing grain size when grain boundary sliding (GBS) is the dominant process ([Ma and Mishra, 2014](#)). If the grain size is small (as in the case of $880^{\circ}\text{C}/0.01\text{s}^{-1}$ and $900^{\circ}\text{C}/0.01\text{s}^{-1}$), then the corresponding superplastic region shifts to a relatively higher strain rate of 0.01s^{-1} . Similarly, if the grain size is large (as in the case of $880^{\circ}\text{C}/0.001\text{s}^{-1}$ and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$) then the corresponding superplastic region shifts to a relatively lower strain rate of 0.001s^{-1} and 0.0005s^{-1} . Consequently, the randomization of texture is observed for both insignificant and significant grain growth scenarios.

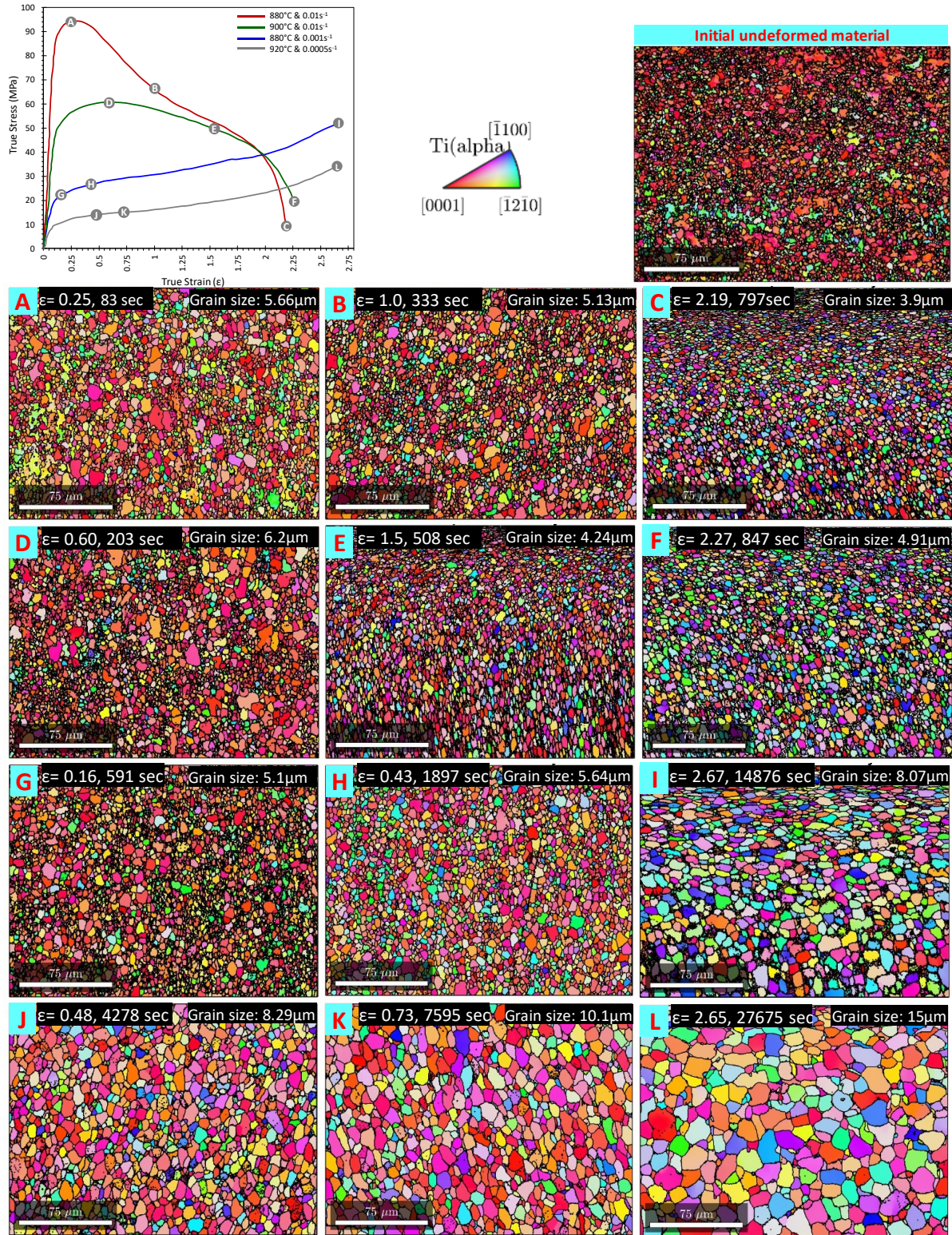


Figure 3. 7. Grain Orientation maps of TA15 during deformation stages (A, B, C, D, E, F, G, H, I, J, K & L) of stress-strain curves.

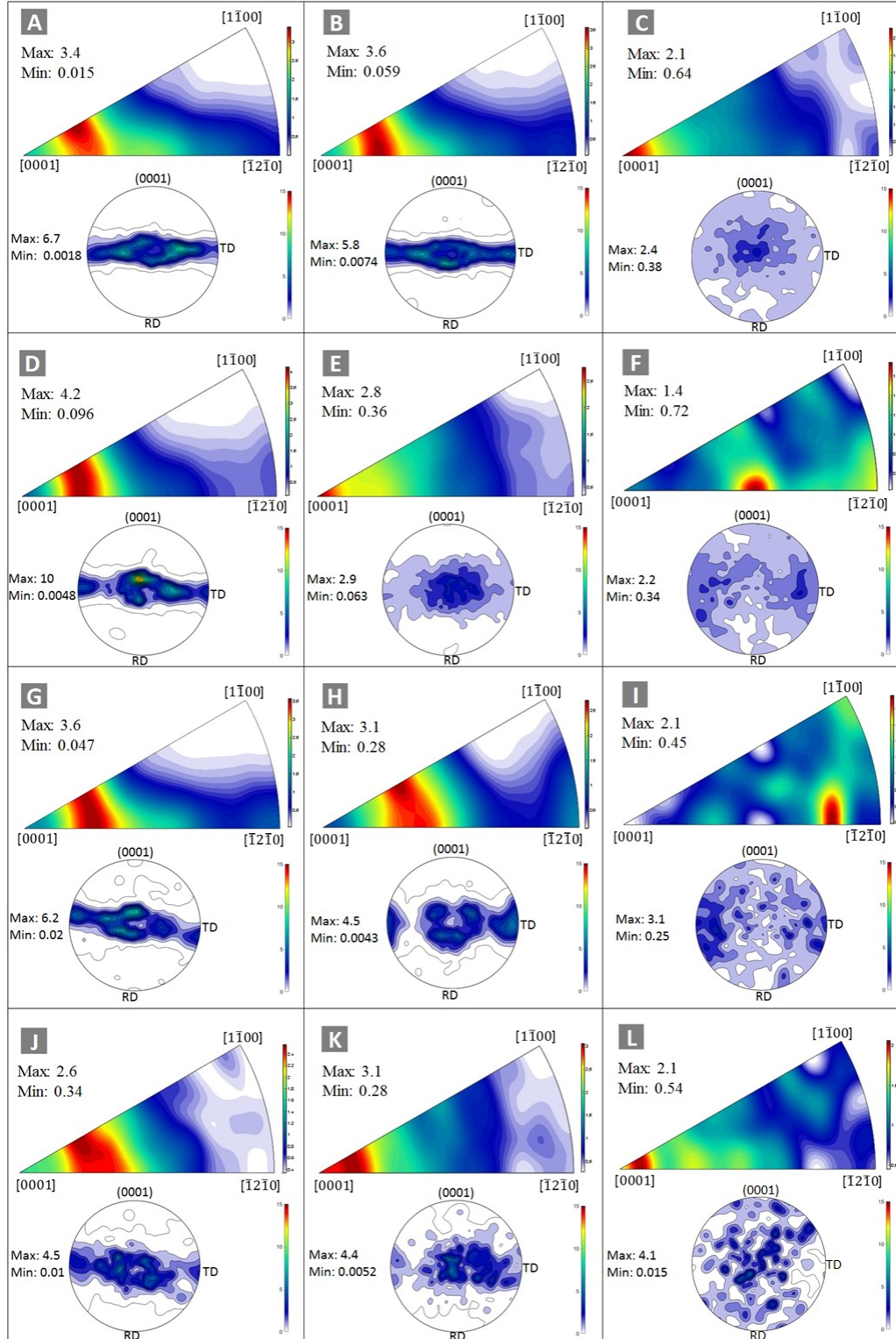


Figure 3. 8. Texture evolution of TA15 during deformation stages (A, B, C, D, E, F, G, H, I, J, K & L) illustrated in stress-strain curves.

3.3.6. GND Evolution

Geometrically necessary dislocation (GND) density is high in the as-received materials and the average GND value is quantified as 3.47×10^{14} , as shown in [Figure 3.10](#), which means that the initial material has high stored strain energy. Furthermore, it can be observed that the GND distribution of the as-received material has a channel-like structure which tends to form the cellular patterns. As described in [Section 3.2.1](#), prior to hot tensile deformation, the samples are soaked for 15 minutes at the corresponding deformation temperatures (e.g. 880 °C, 900 °C and 920 °C), where annealing will be triggered with significantly reduced dislocation density due to the static recovery process ([Sakai et al, 2014](#)).

For the deformation condition of 880°C/0.01s⁻¹, after annealing for 15 minutes, when the as-received TA15 alloy is pulled at a strain rate of 0.01s⁻¹ till the stage A, the average GND value is quantified as $8.89 \times 10^{13} \text{m}^{-2}$. As shown ([Figure 3.10](#)) the detailed observation of the GND map at stage A indicates that there are some large grains without dislocations, which is signified by the blue colour. Also, some dislocation-free small grains can be observed which support the creation of new recrystallized grains. In addition, these channel-like GND distribution, can be seen in the form of cellular or sub-grain patterns/structures, which implies the occurring of continuous dynamic recrystallization (CDRX) process. These results are consistent with the literature which reveals that CDRX is a recovery dominated process and proceeds by continuous absorption of lattice dislocations, generated during the deformation process, into sub-grains boundaries (without a change in the sub-grain size) ([Tan and Tan, 2003](#); [Momeni et al, 2014](#); [Humphreys et al, 2017](#)). As the deformation continues to stage B, the average GND drops to $6.80 \times 10^{13} \text{m}^{-2}$, which leads to the dropped flow stress from stage A to stage B ([Figure 3.10](#)), where dislocation glide dominates the deformation with negligible GBS, as evidenced by the rarely changed texture in [Figure 9](#). While moving from stage A to stage B, the grain refinements are observed, which implies that the impingement of large dislocation-free grains and the onset of grain refinement. At the fracture stage C, the average GND further drops to 5.0×10^{13} and the GND distribution is different from the stages A and B, which implies that the deformation mechanism from the stage B to C differs from the stage A to B. This may explain that from the stage A to B, the process of

dynamic recovery and recrystallization occurs, and the grain refinement is attained somewhere between the stages B and C. These refined grains would accelerate the process of grain boundary sliding, which can be supported with the corresponding texture evolution shown in [Figure 3.8 and 3.9](#).

For the deformation condition of $900^{\circ}\text{C}/0.01\text{s}^{-1}$, after annealing for 15 minutes, when the as-received TA15 alloy is pulled till the stage D, the average GND value is quantified as $6.20 \times 10^{13}\text{m}^{-2}$. The trend and characteristics of GND evolution at stage D resemble to stage A. At stage E, the number of GND increases by 18% to $7.30 \times 10^{13}\text{m}^{-2}$. As the deformation continues to the fracture stage F, the average GND drops to $5.70 \times 10^{13}\text{m}^{-2}$, which shows a decrease of 22% and 8% compared to the corresponding value of GND at the stage E and D, respectively. It can be observed that at the fracture stage F, the distribution of GND is almost the same as at stage E. This can be explained as that from stage D to E there is a process of dynamic recovery.

For the deformation condition of $880^{\circ}\text{C}/0.001\text{s}^{-1}$, after annealing for 15 minutes, when the as-received TA15 alloy is pulled till the stage G, the average GND value is quantified as $5.60 \times 10^{13}\text{m}^{-2}$, which is considerably lower than that at stage A (deformed at the same temperature but at higher strain rate 0.01s^{-1}). This infers that the lower strain rate provides more incubation time for DRV and DRX. During the initial stages of deformation, there is an increase in the flow stress as dislocations interact and multiply. Moving to stage H the GND further drops to $4.20 \times 10^{13}\text{m}^{-2}$ and the dislocation distribution is almost similar to stage G but with some deformed grains. As the deformation continues to stage I, both the GND value and distribution remain practically constant.

For the deformation condition of $920^{\circ}\text{C}/0.0005\text{s}^{-1}$, after annealing for 15 minutes, when the as-received TA15 alloy is pulled till the stage J, the average GND value is quantified as $2.79 \times 10^{13}\text{m}^{-2}$ which is the lowest GND value among all the corresponding the stages (i.e. A, D, G). At the deformation continues to the stages K and L, the GND almost remain constant with a slight drop.

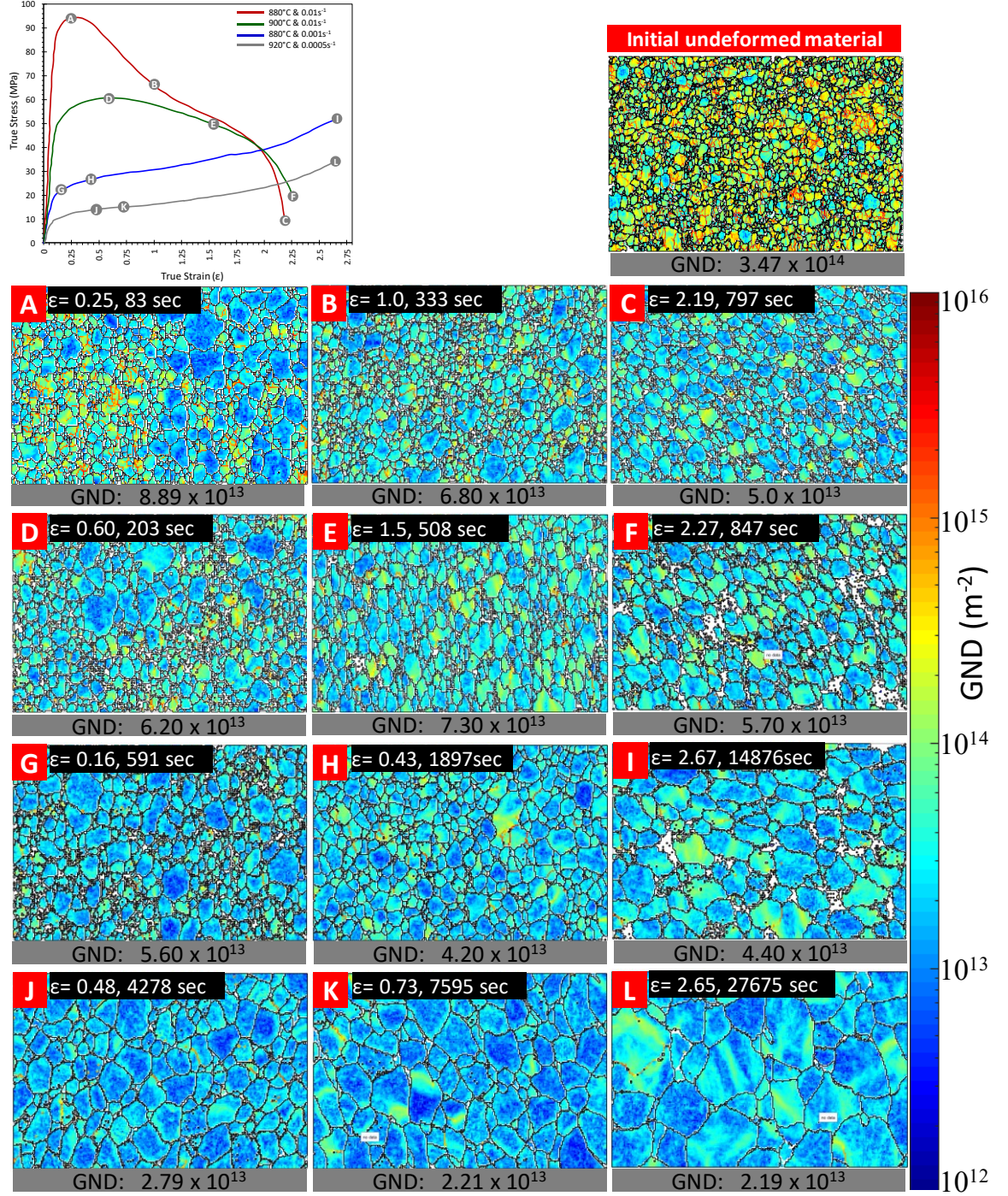


Figure 3. 9. GND evolution of TA15 during deformation stages (A, B, C, D, E, F, G, H, I, J, K & L) of Stress-Strain Curve

3.4. Discussion

In this section, the overall superplastic results presented in the above six subgroups (section 3.1 – 3.6) are integrated, as shown in [Figure 3.11](#) and discussed to provide a better understanding of the underlying deformation mechanism of TA15 alloy during superplastic deformation. Theoretically, different processes can occur such as (i) grain boundary sliding accommodated by dislocation slip/creep, diffusion creep and grain rotations ([Neih et al, 2005](#); [Langdon, 2009](#); [Alabort et al, 2016](#)) (ii) dynamic recovery and recrystallization ([Panicker et al, 2009](#); [Wang et al, 2010](#); [Zhang et al, 2010](#); [Zhang et al, 2014](#); [Matsumoto et al, 2015](#); [Alabort et al, 2016](#); [Liu, et al, 2017](#)), and (iii) dynamic grain growth ([Rabinovich and Trifonov, 1996](#); [Li et al, 1997](#); [Sotoudeh and Bate, 2010](#)). It is widely accepted that grain boundary sliding is the main mechanism for superplasticity of Ti alloys, and grain boundary sliding is accommodated by diffusion creep, and dislocation slip/creep or both ([Panicker, et al, 2009](#); [Jia et al, 2019](#); [Sotoudeh and Bate, 2010](#); [Rust and Todd, 2011](#); [Kotov et al, 2016](#); [Kamikawa et al, 2016](#); [Yakovtseva et al, 2016](#); [Mikhaylovskaya et al, 2018](#); [Tokunaga and Ohno, 2019](#); [Pozdniakov et al, 2019](#)).

The detailed microstructural analysis of the deformed TA15 alloy reveals that the predominating deformation mechanism varies as a function of true strain, strain rate, and deformation temperatures. Superplastic deformation mode of grain-boundary-sliding is also evolved in relation to β -phase transformation, the occurrence of continuous dynamic recrystallization process, and grain-growth during deformation.

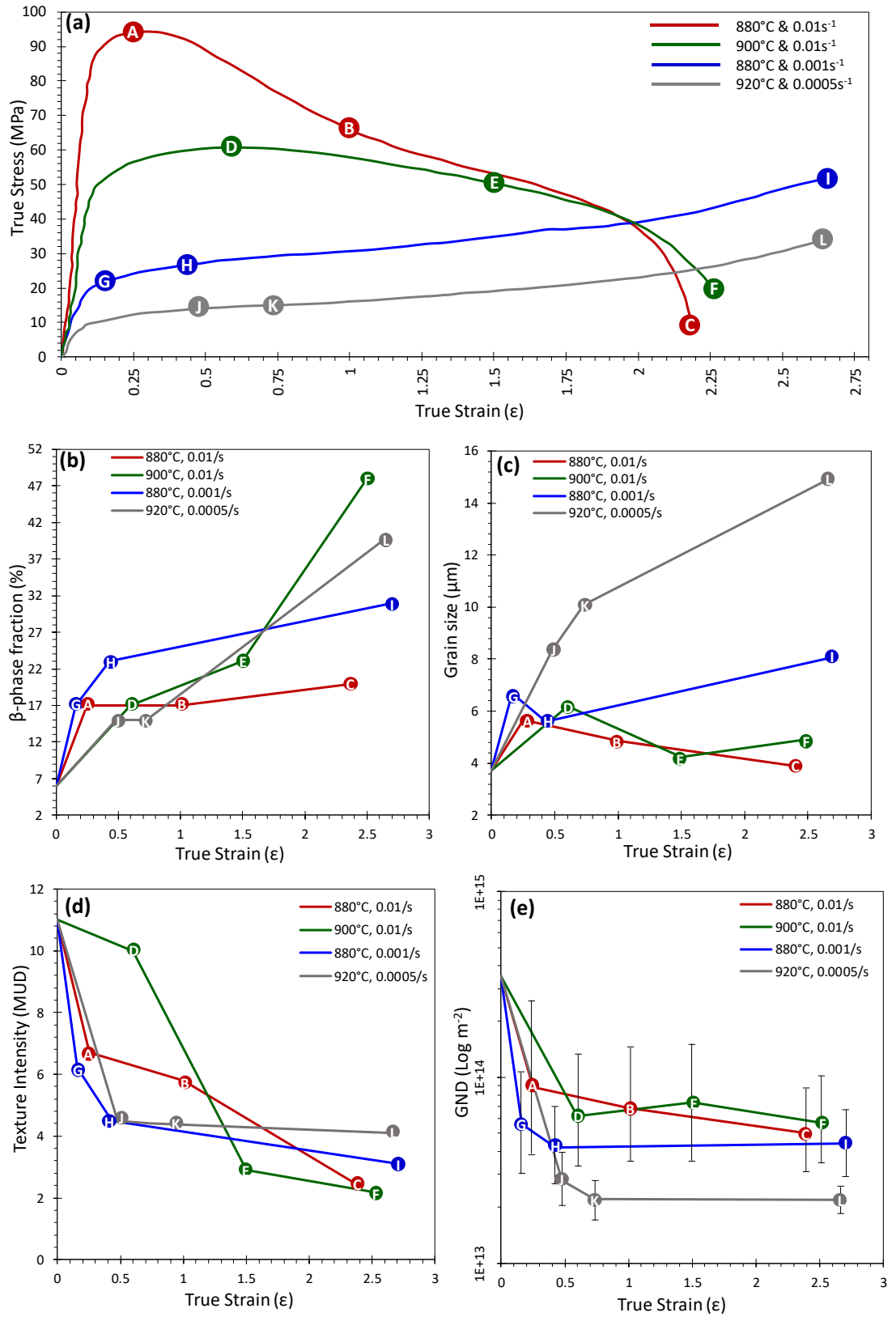


Figure 3. 10. Quantitative illustrations of the results presented in section 3: (a) true stress-strain curves (b) β -phase evolution (c) Grain size evolution (d) texture evolution, and (e) GND evolution

It is demonstrated from the experimental results that the deformation conditions of $880^{\circ}\text{C}/0.01\text{s}^{-1}$ and $900^{\circ}\text{C}/0.01\text{s}^{-1}$ show some similar deformation behaviour. It can be inferred that for the deformation behaviour from stage A to C and stage D to F; there is a continuous process of dynamic recovery (DRV), dynamic recrystallization (DRX), and grains refinement. It is because of initial fine grain size (which stores high store energy in the form of dislocation) and adiabatic heating, which locally increase the temperature at a high strain rate of 0.01s^{-1} . In addition, the high strain rate leads to cavity initiation at α/β grain boundaries (Roy and Suwas, 2013), rapid propagation and ultimate fracture so that the steady-state cannot be easily maintained (Shen et al, 2019). Another explanation for this cavity initiation is that at a temperature of 880 and a strain rate of 0.01s^{-1} , there is not enough β phase to accommodate the sliding of grains (Alabort et al, 2016); as a result, the cavities are left which leads to fracture. The presence of such cavities at a high strain-rate can be supported with the microstructure of the fracture stage C near the necking region (Figure 3.12).

At $900^{\circ}\text{C}/0.01\text{s}^{-1}$, the formation of cavities is minimal (Figure 3.12), which can be attributed to a relatively high-volume fraction of β -phase (49%) at given conditions. The soft β -phase accommodates the GBS to reduce the propensity for cavity nucleation at a triple-junction of grains and, thus, delays the fracture process (Nieh et al, 1999). Consequently, the drop in the flow stress at $900^{\circ}\text{C}/0.01\text{s}^{-1}$ can be attributed to the presence of higher β -phase.

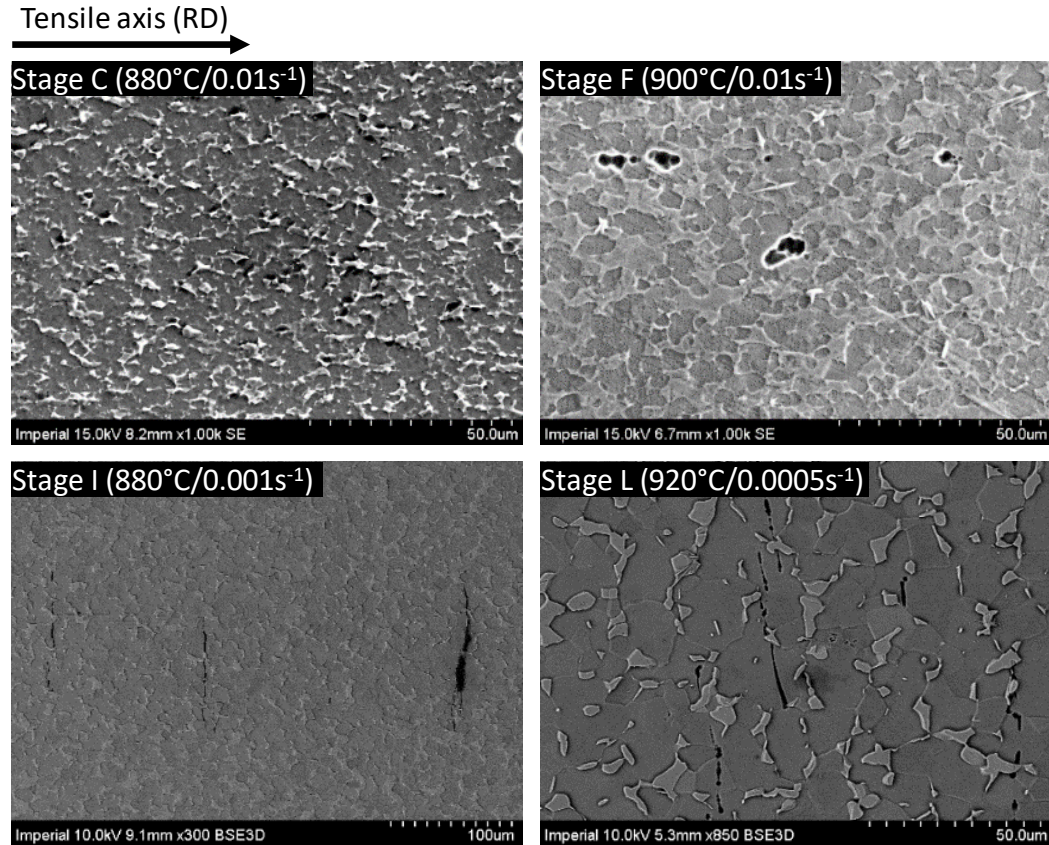


Figure 3. 11. SEM micrographs of void formation in the neck region of the fracture stage sample

As shown in [Figure 3.3](#), the initial average grain size of as-received material is $3.8\mu\text{m}$, and most of the grain distribution fraction is covered by grain size smaller than $1.7\mu\text{m}$. This fine grain size itself is very unstable. In order to lower its energy, it will try to grow in size. Similarly, the existence of pre-deformation dislocations and the high stacking fault energy of titanium alloys at such high temperatures will enhance the process of the dynamic recovery by dislocation glide and climb. At the stages A, B and D, there is a heterogeneous distribution of GND, implying that there are grains with and without accumulation of GNDs. The GND density is high in some of the small grains, and its structure is similar to the initial material. This characteristic implies that there is a process of DRV and DRX at the initial and intermediate stages of deformation where some GNDs are annihilated and recovered, which resulted in relatively larger grains without dislocations while some are still in the process of recovery and recrystallization. From the textural analysis ([Figure 3.8](#)

and Figure 3.9), it can be inferred that at the deformation stages A, B and D, the microstructures still exhibited strong textures, which is dominated by [0001] basal plane texture and the maximum texture intensity is estimated as 3.4, 3.6 and 4.2 (MRD) respectively, which implies that GBS/Grain rotation was minimal for the initial deformation the stages of A and B. In Figure 3.4, the sharp increase of the stress from the undeformed the stage to the stage A can be attributed to strain rate hardening as it can be seen that dislocation density is dropped from initial $3.50 \times 10^{14} \text{ m}^{-2}$ to $8.89 \times 10^{13} \text{ m}^{-2}$ due to the process of dynamic recovery (DRV) which means that work hardening is not a dominant mechanism for the increase in the yield stress and this increase can be attributed to the strain rate sensitivity. It is reported that the reduction in the GND density negates the occurrence of strain hardening, which is usually coupled with the increase in dislocation density (Li et al, 2018; Chu et al, 2018). At high strain rates, the dislocations do not have sufficient time to slide or climb (Zhang et al, 2019). Similarly, for the deformation stage D, it can be observed that the peak stress of the alloy decreased from 94 MPa (Point A) to 58 MPa (Point D) when the temperature increased from 880 °C to 900 °C at a strain rate of 0.01 s^{-1} . This might be because of the increased mobility of grain boundaries and the promoted annihilation of dislocations at higher temperatures (Tahreen et al, 2015).

From the grain size analysis (Figure 3.7), it can be seen that the initial average grain size is $3.8 \mu\text{m}$ increases to $5.66 \mu\text{m}$ and $6.2 \mu\text{m}$ at stage A and stage D, respectively. This increase can be explained as due to the adiabatic heating which will locally increase the temperature, and hence expedites the process of dynamic recovery and grain growth till the stage A and the stage D. Similarly, from the stage A to B there is a slight grain refinement $5.13 \mu\text{m}$, but a significant drop of stress from the stage A to the stage B. This drop in stress can be attributed to thermal softening caused by adiabatic heating which offsets the work hardening effect (Huang et al, 2010) by converting most of the plastic work into heat (Khan et al, 2007) and hence expedites the process of grain refinement. From stage B to stage C, grain refinement is achieved, which will make it easier for the GBS/grain rotation to occur and will complete the randomization of texture. In addition to grain refinement, the GND distribution at stage C (the fractured stage of the sample) is substantially different from that of stage A and stage B, which signifies that the deformation mechanism is changed from the stage B onward. The deformation mechanism change from stage B to stage C can also

be explained with the help of analysing the texture analysis as it can be seen that the texture intensity is dropped from 3.6 to the maximum of 2.1. This texture drop is associated with nearly random crystal orientations of the grains, indicating the presence of no or very weak texture. The observed changes of texture and intensity are closely related to grain boundary sliding (GBS) phenomenon (Martin et al, 2001; Pérez-Prado et al, 2001; Eddahbi et al, 2006; Watanabe and Fukusumi, 2008) because random sliding events give rise to a homogeneous decrease in texture intensity (Watanabe et al, 2012). In general, GBS, as such, does not need any change in the crystallographic orientation (Biswas et al, 2012). However, along with GBS grain, rearrangement, or grain rotation can occur during the superplastic deformation, which leads to a weakening of texture (Kandalam et al, 2017). Therefore, these results support that the texture analysis can also be used to investigate the deformation mechanism (Panicker et al, 2009; Watanabe et al, 2013). Moreover, from stage B to stage C, the stress-strain curve is dropping in two slopes *i.e.*, for stage B at a true strain of 1.0 to the true strain of 1.5, the slope is different from the slope available at the true strain of 1.5 to 2.5. It can be assumed the first slope from the stage B to the true strain of 1.5 is due to grain refinement and the ease of GBS/grain rotation due to fine grain size while the second slope is due to cavities as shown in the microstructure at the neck region (Figure 3.12).

As shown in Figure 10, the β phase at stage A, B, and C is quantified as 16%, 16%, and 20%, respectively, which implies that there is an average of 14% of β phase transformation from the initial 6% β phase. Therefore, it can be concluded that at the deformation conditions of 880 °C/0.01s⁻¹, there is a considerable change in the transformation of β -phase, and this β -phase evolution does contribute to the flow softening.

When grain boundary sliding is obstructed by an unfavourably orientated grain, the resultant stress concentration may be relaxed by dislocation motion in the blocking grain. There are two typical models for activating the boundary sliding: the Ball–Hutchison model (Ball and Hutchison, 1969) and Gifkins Core–Mantle model (Gifkins, 1982). Ball-Hutchinson model considers that grains slide in bigger groups, and the rate of accommodation is governed by grain boundary dislocations, which pile up at a triple point. While, in the Gifkins Core–Mantle model, it is proposed that groups of grains slide as a unit. The resulting stress concentration is relaxed by the dissociation of the leading

dislocation into dislocations capable of moving in the other boundaries which make up the triple point, and/or into lattice dislocations which accommodate sliding. It is reported (Kim et al, 1999, Semiatin and Sargent, 2010) that superplastic deformation-mechanism of titanium alloys with an average grain size of more than $3\mu\text{m}$ could be summarized in relation to the Gifkins Core–Mantle model. From the above discussion, it seems reasonable to believe Gifkins Core–Mantle model as dominant deformation mode from stage A to C due to the characteristic of heterogeneous GNDs distribution at stage C and that GNDs are strongly accumulated or accommodated mainly at grain boundaries. This is in good agreement with the superplastic deformation mechanism of two-phase titanium alloy with a fine grain size of $3\mu\text{m}$ (Kim et al, 1999) and an ultrafine grain size of $0.4\mu\text{m}$ (Matsumoto et al, 2015).

The aforementioned deformation mechanism can be further supported with the activation energy analysis (Figure 3.5b), where it can be observed that at the stage A and B the activation energy is 256 and 276 kJ/mol respectively which is somewhat higher than the self-diffusion activation energy values of both α -Ti (204 kJ/mol) and β -Ti (161 kJ/mol) (Meier et al, 1991). However, these values lie in the range of activation energy values for dislocation creep of α -Ti (200–360 kJ/mol) (McQueen and Bourell, 1987). Therefore, it may be inferred that the dominant mechanism at the stages A and B can be attributed to the GBS accommodated by dislocation glide/climb. At the fracture stage C the calculated activation energy (548 kJ/mol) is much higher not only than self-diffusion activation energy values of α and β phases but also from the activation energy of dislocation creep. Furthermore, the stress exponent value (9.1) at stage C is also much larger than those during deformation controlled by diffusion and dislocation (~ 1 -6) (Frost and Ashby, 1982). Such a high value of activation energy involving dynamic recrystallization (Apple et al, 2011) in the deformation can be observed as the flow softening at stage C.

Similarly, from the stage D to E there is a grain refinement 6.2 to $4.2\mu\text{m}$, and the grain size is uniformly distributed which implies that before the stage E, almost complete refinement of the grain is attained and causes the GBS/grain rotation process much easier to happen which can also be supported by the corresponding texture intensity drop from 4.2 to 2.8

MRD along with a relatively slow drop in stress from point D to E. Unlike the fracture stage C where grain refinement is observed, at the fracture stage F, slight grain growth of 4.24 to 4.9 μm occurred. The physics of this grain growth can be explained with the significant increase of β -phase at the fracture stage F, where it is quantified as 48%. It is known that an increase in β -phase stimulates grain growth during the superplastic deformation (Zhang et al, 2019). From Figure 3.6, it can be observed that as the deformation continues from stage D to stage E, the fraction of β -phase increases from 25% to 49%. Referring to the morphology of the corresponding flow curve (Figure 3.4) it can be observed that there is a sharp drop of stress from the stage E to the stage F. As β -phase has a BCC while α has HCP crystal structure due to which each phase exhibit different deformation behaviour and diffusion characteristics (Mikhaylovskaya et al, 2017). It is known that at the elevated temperatures like 900°C, BCC β is considerably softer than HCP α (Semiatin et al, 2002; Kim et al, 2005; Alabort et al, 2016). This effect is generally attributed to a higher diffusivity and more active slip systems of dislocations in β -phase than α -phase. The highly deformable β phase is mostly distributed along α phase grain boundaries, which results in weakening grain boundaries and improving grain boundary sliding (GBS) during deformation (Li et al, 2017). In addition to the increase in β -phase at stage E, it would be reasonable to assume that it is the volume fraction of β -phase, which plays a critical role in the grain growth from stage E to stage F. Moreover, from the microstructure of the fracture stage F at the neck region (Figure 3.12) cavities can be observed so together with the evolution of β -phase, the cavitation can be held responsible for the stress drop from the stage E to F. The large-size cavities formed during the superplastic forming are most harmful to the overall properties of the post-SPF materials, and the presence of these cavities may impose significant limitations on the commercial use of superplastically formed components.

To summarize the deformation behaviour of the TA15 alloy with these considerations, grain boundary sliding is the dominating mode, and the equilibrium β -phase is precipitated from the supersaturated α -phase, resulting in the contribution to the further enhancement of grain boundary sliding. Again, like points A, B, C, the Gifkins Core–Mantle model is supposed to be the dominant mode for the deformation stage D to F due to heterogeneous GNDs distribution and accumulation, especially at grain boundaries at stage F.

It is demonstrated from the experimental results that the deformation conditions of $880^{\circ}\text{C}/0.001\text{s}^{-1}$ and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$ shows some similar deformation behaviour. For the deformation conditions, $880^{\circ}\text{C}/0.001\text{s}^{-1}$ and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$, the evolution of GNDs at the stages G to I and J to K are lower than the stages A to C (at $880^{\circ}\text{C}/0.01\text{s}^{-1}$) and D to F (at $900^{\circ}\text{C}/0.01\text{s}^{-1}$) which means that at slower strain rate the deformation mechanism is changed. This is because a lower strain rate provides a longer time for the accumulation of deformation energy, which reduces the amount of plastic deformation per unit time and lengthens the corresponding steady-state the stage. Therefore, the additional time allows dynamic recovery to reduce dislocation density (Gong et al, 2017). At stage G with a true strain of 0.16, which is very much close to the initial stage, the average GND is reduced by 84% as compared to the as-received state and also lower than the GND average values at stages A, B, C, D, E, and F.

As compared to the higher strain rate 0.01s^{-1} , the GND distribution is relatively more uniform at lower strain rates of 0.001s^{-1} and 0.0005s^{-1} . The average grain size is $5.1\mu\text{m}$ and much uniform distribution, and the texture intensity is dropped to 3.6, which means that during the apparent elastic region (from initial to point G) the GBS/grain rotation is initiated and as much of the GND is accumulated along the grain boundary, that supports Gifkins Core Mantle model. Unlike the higher strain rate 0.01s^{-1} , where there is no considerable change in grain size, for deformation stages G to I, a significant (112%) grain growth is noted. The increase in grain growth is attributed to higher annealing duration at the elevated temperature and strain enhanced grain growth. Both of these factors *i.e.* annealing time and degree of deformation, are much high as compared to deformation the stages A to F where no significant change in grain size is observed. This can be further supported by the texture evolution (Figure 3.9), where it can be observed that as the deformation continues from stage G to I, the maximum texture intensity monotonously decreases from 3.6 to 2.1, which results in nearly no texture. The slightly high texture intensity at stage I (the fractured point at $880^{\circ}\text{C}/0.001\text{s}^{-1}$) compared to the texture intensity at the fracture stage C (2.1 MRD) and F (1.4 MRD) can be attributed to the grain growth,

which causes the growth of the grains. This is mainly due to the sampling size. However, a large sampling size will considerably increase the EBSD capturing time and make the characterization work become less feasible. The phenomenon of such grain growth can be supported by the findings of the orientation map (Figure 3.8). The grain growth kinetics for the deformation condition of $880^{\circ}\text{C}/0.001\text{s}^{-1}$ and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$ can also be explained due to, (a) the difference in the dislocation energy between two sides of the grain boundary. The large grains become larger, and the small grains become much smaller. As a result, the large grains consume the smaller grains (Liew et al, 2007), (b) Grains consumption at the expense of others, especially at the cost of smaller grains, and also (c) the curved border usually migrates to its centre of curvature. From the above discussion, the grain growth is believed to be responsible for the strain hardening in flow stress curves at the deformation the stages G to I and J to L, which is in agreement with the previous works (Mikhaylovskaya et al, 2017; Despax et al, 2018; Mosleh et al, 2019; Imai et al, 2019).

Moreover, at the fracture stage I and L, the GND is significantly dropped as compared to the GND value at the fracture stage C and F. The concentration of GND along the grain boundaries at stage I & L, supporting the Gifkin-Core-Mantle model. For both cases *i.e.*, deformation the stages G to I and J to L, there is a continuous increase in flow stress which is due to the continues grain growth which makes it difficult for the GBS/grain rotations to occur and ultimately the stress reaches the critical value where the intragranular deformation is initiated which leads to the fracture. This phenomenon can be supported by the SEM micrograph (Figure 3.12), and also by the deformation bands observed in the GNDs maps (Figure 3.10) at the stages I and L showing fracture of the alpha phase. Moreover, unlike the higher strain rate (0.01s^{-1}), where cavitation is observed at the fracture stage C, and F confirming the ductile failure, at lower strain rates (0.001s^{-1} and 0.0005s^{-1}) not cavitation is found at the fracture stage I and L instead cracks perpendicular to the tensile axis are observed which confirms the brittle failure.

Based on the above discussion, the following schematic (Figure 3.13) can be used to describe the various deformation phenomenon occurring during superplastic deformation TA15 alloy.

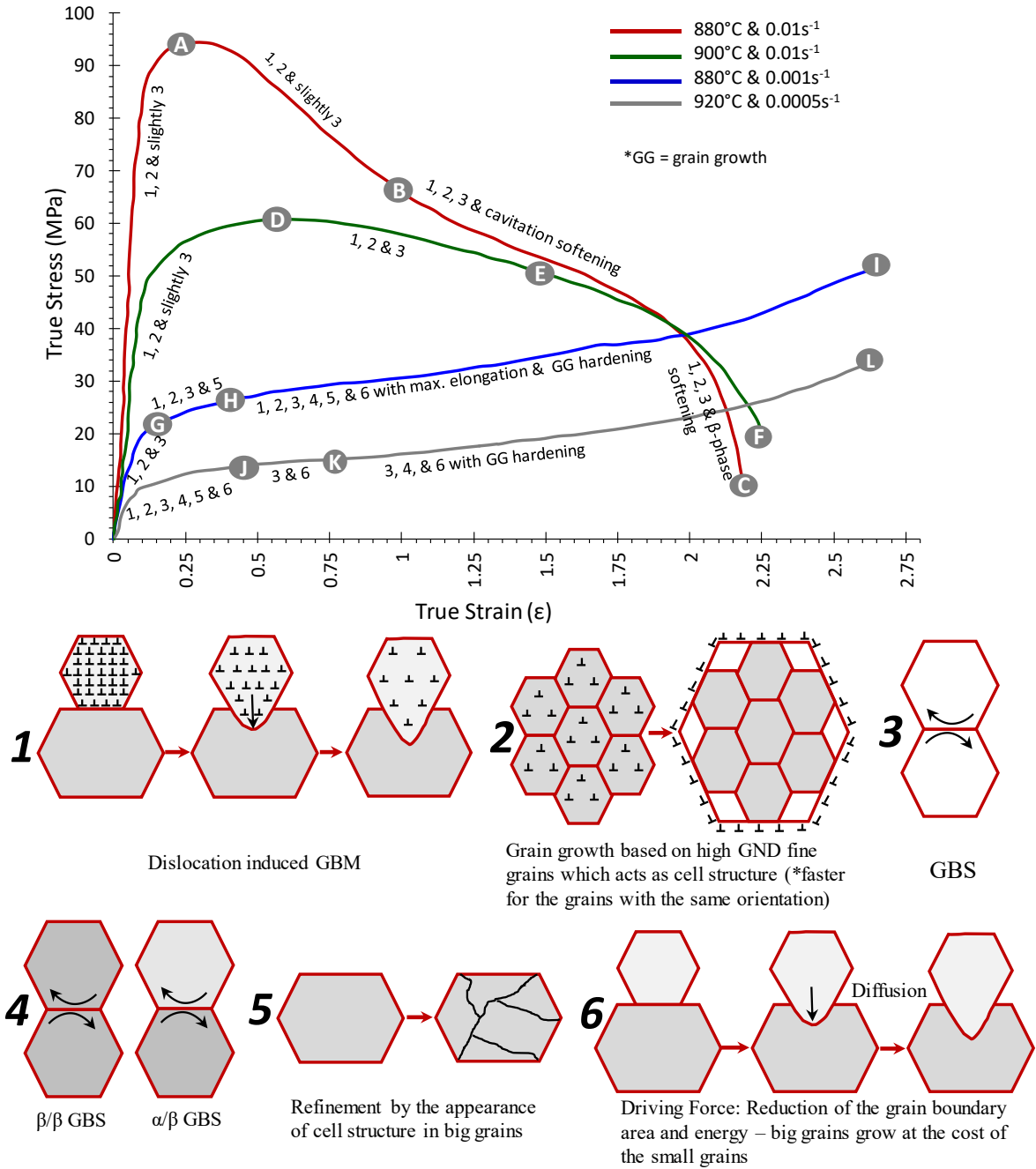


Figure 3. 12. Composite schematic of the possible deformation phenomenon in the present study

5. Conclusions

The superplastic behaviors of a near- α TA15 titanium alloy have been examined in terms of deformation kinetics, microstructure and texture evolutions. The following main conclusions could be drawn:

- i. TA15 alloy exhibit excellent superplastic behaviour in the temperature range of 880–920°C with strain rates of 0.0005 – 0.01s⁻¹, and all tensile elongations exceeded 700%. The optimum superplastic conditions are 880°C/0.001s⁻¹ with the maximum elongation of 1470% and optimized microstructure distributions (i.e. ~70% α and ~30% β).
- ii. The maximum elongation of 1450% achieved under the deformation conditions 880 °C/0.001s⁻¹ is attributed to the low dislocation density and the insignificant grain growth. At deformation conditions 880 °C/0.01s⁻¹ and 900 °C/0.01s⁻¹, although the grains are refined, the dislocation density is increased locally, which facilitates cracking at the triple-junctions of grain area, resulting in low elongation. At 920°C/0.0005s⁻¹, although the dislocation density is the lowest, the grain growth is the highest, resulting in high elongation, but lower than the deformation conditions 880 °C/0.001s⁻¹. Therefore, it can be concluded that the elongation depends on the dislocation density and grain growth, where the influence of dislocation density is greater.
- iii. The degree of the superplasticity (% elongation) and the hardening and softening of the stress-strain curves are predominantly depended on the strain rates, where strain-softening occurred at higher strain rates (*i.e.* 0.01s⁻¹) and strain-hardening occurred at lower strain rates (*i.e.* 0.001s⁻¹ and 0.0005s⁻¹).
- iv. Grain boundary sliding accommodated by the dislocation glide/climb is the dominant mechanism under all deformation conditions (*i.e.* 880°C/0.01s⁻¹, 900°C/0.01s⁻¹, 880°C/0.001s⁻¹, and 920°C/0.0005s⁻¹). This has been confirmed by the continuously weakened texture evolutions, the GND distributions, and the analysed activation energy values during the deformation,
- v. The strain hardening at lower strain rates is mainly attributed to the dynamic grain growth, which concurrently hinders the GBS process. While the strain-softening at

higher strain rates is attributed to a combined effect from grain refinement, dynamic dislocation recovery, and the high strain rate induced adiabatic heat generation.

- vi. The ductile fracture was observed for higher strain rate deformation (*i.e.* 0.01s^{-1}) with apparent cavities, while brittle fracture was observed for slow strain rate deformation with the observed intergranular fracture.

Chapter 4

Unravelling the effect of dynamic loading on microstructure evolution under superplastic deformation in a near alpha titanium alloy

4.1. Introduction

The structural integrity of the formed parts can be significantly affected by the material microstructure, particularly when there is a change in geometry *e.g.* change in wall thickness. Moreover, in recent years, due to the increasing demands of high-speed aircraft lightweight structure design, the near- α titanium alloys have been rapidly developed to improve specific properties to allow for the elevated service temperatures involved (at least 500°C).

No comprehensive study has been conducted to understand the relationships between dynamic loading on microstructure evolution as functions of strain, strain rate and temperature. Although there are some studies on microstructure evolution in titanium alloys during superplastic deformation, such as [Tan et al. \(2007\)](#); [Alabort et al. \(2016\)](#); [Zherebtsov et al. \(2016\)](#) and [Mikhaylovskaya et al. \(2017\)](#). The effects of dynamic loading on microstructure evolution are not clear, due to the mixed effects of static thermal annealing. In a study performed by [Motyka et al. \(2014a\)](#) shows that superplastic deformation of Ti-6Al-4V alloy at 875°C/0.001s⁻¹ led to decrease in the volume fraction of β -phase in both grip and gauge zones of the specimen, though the effect was focused on the gauge region. In the superplastic deformation of a Ti3Al based alloy investigated by [Yang et al. \(2003\)](#), the volume fractions of the β -phase in the grip sections for testing conditions 850°C/0.0005s⁻¹ was found to be higher than gauge region, but only limited data was obtained. In another study [Friedman and Ghosh, \(1996\)](#) found the grain size to grow faster during concurrent superplastic deformation than for static annealing of an aluminium alloy deformed at 550°C/0.0002s⁻¹. Similarly, [Kaibyshev et al. \(2003\)](#) found considerable

higher grain growth in gauge regions than grip regions for an aluminium alloy deformed at strain rate 0.0028s^{-1} in the temperature interval 500–580 °C. For a magnesium alloy [Cao et al. \(2015\)](#) reported that at the both grip and gauge regions grain size increases linearly with decreasing strain rate and increasing temperature, though the effect was much pronounced in grip regions. Similarly, the studies such as [Chai et al. \(2005\)](#); [Kim et al. \(2009\)](#); [Cepeda-Jiménez et al. 2013](#) and [Vijayananth et al. \(2019\)](#) reported smaller grain sizes in gauge regions as compared to grip regions. In summary, it is clear from the literature review that most of studies consider only one microstructure parameter i.e. grain size for comparative microstructure evolution in static annealing and superplastic forming. There is no comprehensive or simultaneous study to date which uses a consistent framework across multiple microstructural attributes (i.e. phase change, grain size, grain orientation, texture and geometrically necessary dislocations (GND) density) for comparative microstructural analysis in static annealing and superplastic deformation undergone the same thermal history.

In this context, the aim of this study is to clarify how dynamic loading affects microstructure evolution at superplastic forming conditions in a near- α titanium alloy by comparing the microstructure and GND density under static annealing and superplastic deformation (dynamic annealing) conditions. A better knowledge of these relative roles will improve the understanding of the super-plasticity phenomena involved in the SPF of titanium alloys.

The route chosen was to trace and examine two different regions of dog-bone style test specimens undergone the same thermal history in superplastic tensile testing. During superplastic deformation, the grip area is held by the crosshead and experiences little or no deformation as reported by [Motyka et al. \(2014a\)](#); [Du et al. \(2015\)](#) and [Fata et al. \(2016\)](#), whilst the specimen gauge area is exposed to significant strain. The grip and gauge regions are, however, exposed to the same temperature profile for the entire test duration as described in [Mishra et al. \(2018\)](#). As a result, the grip region of the tensile test samples experiences the thermal profile of the complete test duration but with little or no deformation as reported in [Zhang et al. \(2014\)](#) and [Du et al. \(2016\)](#). This thermal profile causes static annealing ([Liu et al. 2017a](#)). Therefore, the grip region exhibits the

microstructure associated with static isothermal annealing, whereas the gauge section exhibits the microstructure associated with superplastic deformation as observed in [Chai et al. \(2014\)](#). [Sakai et al. \(2014\)](#) reported that static annealing and superplastic deformation result in different microstructural changes under specific deformation conditions. Therefore, a comparison of microstructure and texture evolution in the two sections (i.e. grip & gauge) can reveal the superplastic deformation mechanisms in the material under investigation. The present study focuses on four important microstructure evolution: i) phases, ii) texture, iii) grain size, and iv) GND density.

4.2. Materials and Methods

The tensile specimens ([Figure 4.1b](#)) with a gauge length of 15 mm, the width of 6 mm, and a thickness of 2 mm were cut in parallel to the rolling direction of the TA15 alloy sheet. The β -transus temperature of TA15 alloy is 985°C, which are determined from the elemental composition of the alloy, as shown in [Table 4.1](#). The superplastic tensile tests were performed using LETRY-5kN electro-hydraulic servo mechanical testing machine ([Figure 4.1a](#)). To ensure the accuracy of the controlled temperatures, a conventional three-zone furnace was used to guarantee the uniformity of the temperatures. The temperature was monitored with the attached thermocouples, which were spot-welded on different positions of tested samples inside the upper and lower grip boxes. Additionally, the temperature was recorded and feedback-controlled to ensure that the testing temperature is within $\pm 3^\circ\text{C}$ of the target temperature. This enables a higher accuracy of the tests, where the ASTM standard for superplastic testing allows a tolerance of $\pm 6^\circ\text{C}$. The load cell was installed on the bottom fixed crosshead to avoid any heat input due to the radiation from the furnace and enable more accurate load records.

Table 4. 1. Chemical compositions of TA15 alloy.

Element	Al	Zr	Mo	V	Ti
Mass%	6.78	2.0	1.24	1.32	Bal.
Impurity	Fe	Si	C	N	
Mass%	0.050	0.01	0.0072	0.0038	

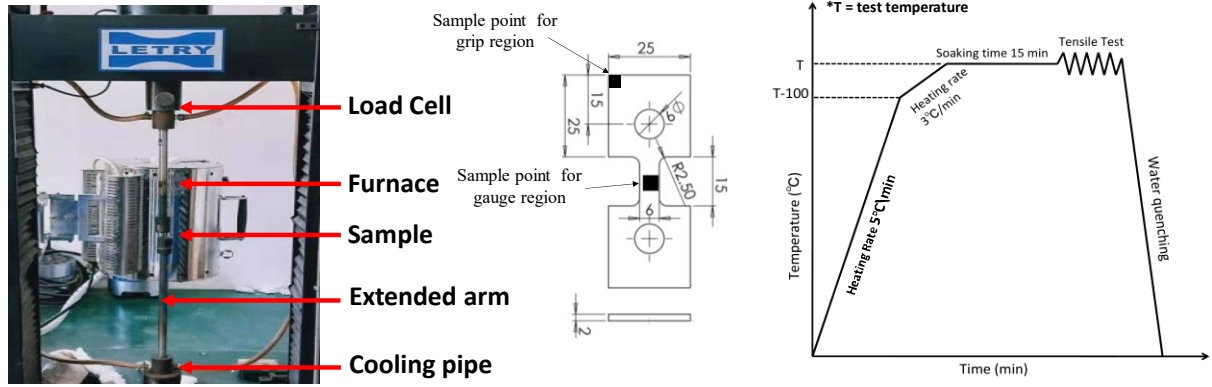


Figure 4. 1. (a) Hot tensile testing system used in this study (b) dimensions of uniaxial hot tensile specimen (c) schematic representation of thermo-mechanical processing route.

The detailed procedures of the superplastic tensile testing and heat treatment are presented in Figure 4.1c. The heating rate is determined based on the average heating rate of a conventional furnace heating, whilst avoiding any possible temperature overshooting issues. The specimens were firstly heated using 10 °C/min to a temperature that was 100 °C lower than the target temperature. Then a slow heating rate, 3°C/min, was applied until the specimens achieving the target deformation temperature, T . Subsequently, the specimens were held at T for 15 minutes to eliminate the thermal gradient of the sample and enable the furnace to attain an equilibrium temperature distribution. Finally, the specimens were superplastic deformed at the designed temperatures and strain rates to various strain levels, followed by water quenching to freeze the deformed microstructures. The deformation temperatures were 880°C , 900°C and 920°C and strain rates were 0.01s⁻¹, 0.001s⁻¹ and 0.005s⁻¹. The full matrix of the testing conditions (i.e. deformation

temperatures, strain rates and strain levels) are given in details in Figure 4.2. For clarity, the tests under each condition were firstly stretched to fracture to obtain the total elongation and the viscoplastic stress-strain curves. Then tests under identical conditions were interrupted at selected strain levels, as given in Figure 4.2, aiming to examine the microstructure evolutions and distributions.

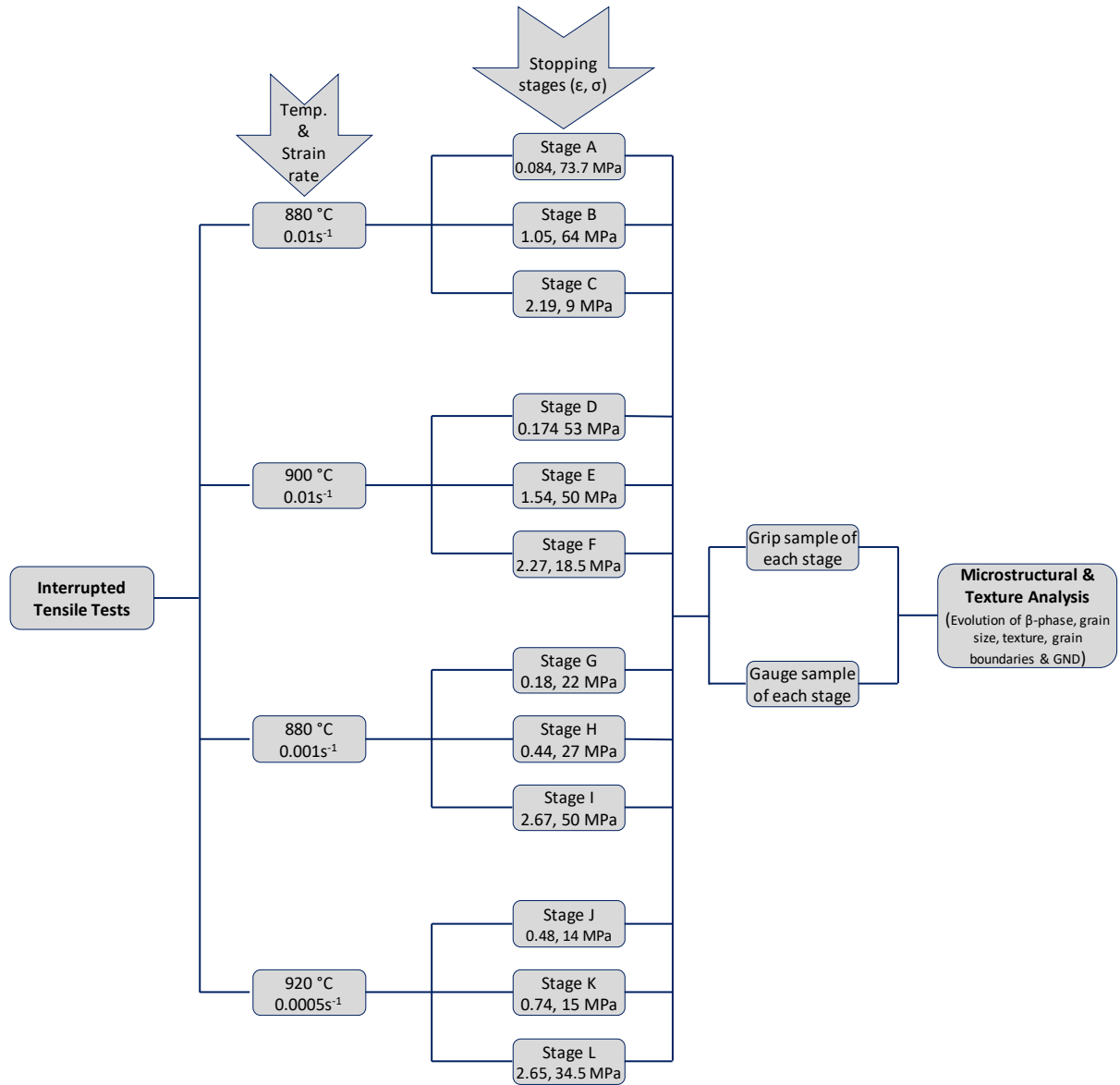


Figure 4. 2. The interrupted thermomechanical Testing conditions for microstructure examinations

The deformed and as-received samples were subjected to a scanning electron microscope (SEM), and electron backscatter diffraction (EBSD) characterization to reveal the superplastic deformation mechanism. For each deformation condition, the SEM and EBSD samples were taken from the gauge regions (deformed) and the corresponding grip regions (non-deformed) close to the fracture tips, where the heat treatment history is identical. The EBSD analysis was performed with a step size of $0.4\ \mu\text{m}$ and a scan area of $248.4 \times 186.9\ \mu\text{m}$. The α grain size, orientation maps, pole figures (PFs), inverse pole figures (IPFs), and geometrically necessary dislocations (GND) density were calculated. The EBSD scan surface is on the rolling free surface (RD-TD).

The GND densities were calculated from the EBSD measurements via the MTEX toolbox using a methodology outlined below. Due to the existence of hard and soft orientations in the predominant HCP α phase at a deforming temperature of $880\text{-}920^\circ\text{C}$, the local deforming mechanism can be complicated. The local deformation mechanism at the microstructural level can be investigated by calculating GND density and the distribution of it in individual grains (He et al. 2016). The state of dislocation represented by a second-order dislocation tensor proposed by Nye (1953) was resolved by studying the rotation gradient tensor, which can be measured from EBSD measurements. As the conventional 2D EBSD can only collect information on the specimen surface, only lattice rotation gradients along x_1 and x_2 directions are measurable. The GND calculation process is based on the method described by Pantleon (2008) using six available curvature tensors (five dislocation density tensors and one difference between two dislocation tensors).

The GND density was calculated using MATLAB™ toolbox MTEX with self-developed routines. A total of 33 potential dislocations are likely to present in α phase: 3 types of $\langle a \rangle$ edge on basal planes; 3 types of $\langle a \rangle$ edge on prismatic planes; 6 types of $\langle a \rangle$ edge on pyramidal planes; 3 types of $\langle a \rangle$ screw; 12 types of $\langle a+c \rangle$ edge on first-order pyramidal slip planes; 6 types of $\langle a+c \rangle$ screw. Different dislocation types have different line energies and contributions to the total energy of the dislocations in the material. The line energy of each kind of dislocations was used as weights; the objective function was to find the minimal weighted sum of all dislocation densities. According to Frank's rule, $E_{el} = \alpha G b^2$

(where α is a constant, G is the shear modulus, b is the magnitude of the Burgers vector), the energy of a $\langle a+c \rangle$ edge dislocation is bigger than a $\langle a \rangle$ edge dislocation by a factor of 3.51. The energy of a $\langle a \rangle$ edge dislocation shall be bigger than $\langle a \rangle$ screw dislocation by a factor of $1/(1-\nu)$ (Hull and Bacon, 2011), where ν is the Poisson's ratio

4.3. Results

4.3.1. Microstructure of the as-received material

According to the BSE micrograph (Figure 4.3a), the initial microstructure contains typical equiaxed grains and consists of a two-phase structure of α (dark region) and β (bright region). The volume fractions of α and β -phases are estimated to be 94% and 6%, respectively, with the β -phase randomly distributed in α -phase matrix. The histogram map of the grain size distribution shown in Figure 4.3b displays the microstructure as predominantly equiaxed in morphology with an average grain size of 3.74 μm . Figure 4.3(c, d & e) depicts the EBSD IPFZ results showing the microstructure and texture distribution of the alpha phase of the as-received material. The EBSD IPFZ results of the initial microstructures show fine equiaxed grains, but with preferred orientation (Figure 4.3c), which is attributed to the pre-rolling and subsequent insufficient annealing treatment. The IPF figure (Figure 4.3d) reveals that the microstructure of the as-received sheet exhibited strong textures, dominated by basal texture direction $[0001]$, and with maximum texture intensity of 4.9 (MRD). The GND distribution map of this cold-rolled TA15 alloy sheet is shown in Figure 4.3f, where average GND is calculated as $3.47 \times 10^{14} \text{ m}^{-2}$ with a high value of the standard deviation of $3.36 \times 10^{14} \text{ m}^{-2}$, which implies that the GND values for the as-received material have a widespread, where some of the grains contain rare dislocations while other contains much high values, as evidenced in Figure 4.3f. The EBSD Pattern quality map (Figure 4.3g) shows the fraction of LAGBs and HAGBs in the initial microstructure with f_{HAGBs} and f_{LAGBs} estimated as 83% and 17%, respectively.

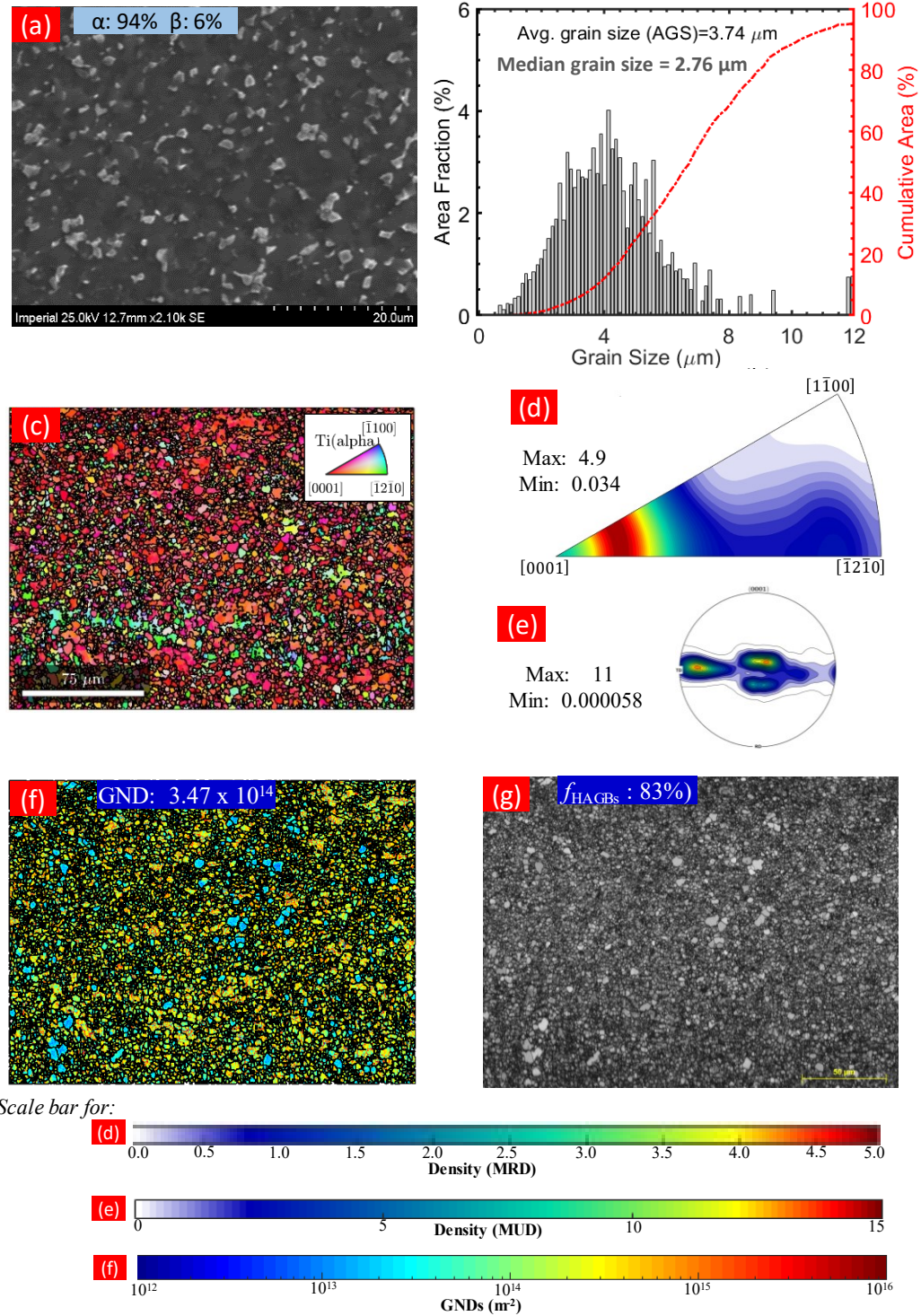


Figure 4.3. SEM and EBSD analyses of the microstructure of raw material: (a) BSE image, (b) grain size distribution, (c) inverse pole figure map, (d) & (e) IPF & pole figure, (f) GND distribution, (g) pattern quality map

4.3.2. Tensile test results

The true stress-strain curves for the hot tensile tests conducted at 880 °C / 0.01s^{-1} , 900 °C / 0.01s^{-1} , 880 °C / 0.001s^{-1} and 920 °C / 0.0005s^{-1} are presented in Figure 4.4. As generally accepted, recrystallization may result in apparent strain softening due to the grain refinement and the mitigation of the grain boundaries (Humphreys et al. 2017). Hence, it may be confirmed that, at higher strain rate (0.01s^{-1}), dynamic recrystallization occurs and hence the significant strain softening, while, at lower strain rates of 0.001s^{-1} and 0.0005s^{-1} , work hardening dominates. Detailed microstructure to further confirm the explanation are presented in the following sections.

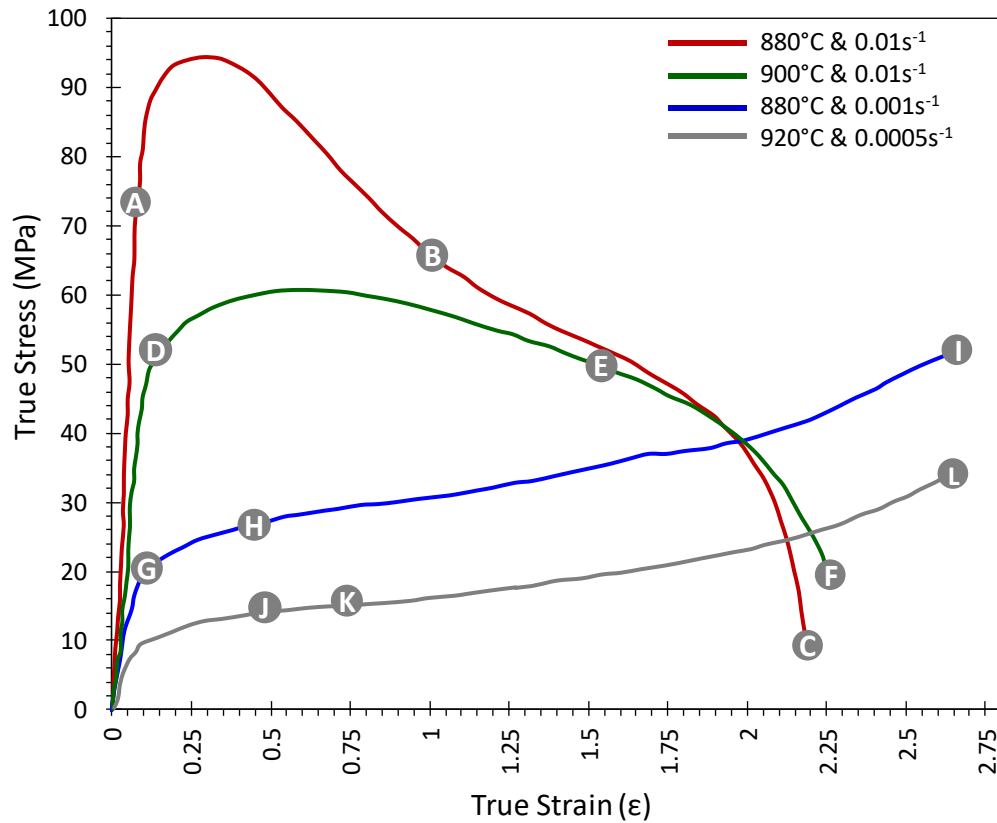


Figure 4. 4. Experimental stress-strain curves of the tested samples at deformation temperature of 880 °C, 900 °C and 920 °C

4.3.3. Evolution of Beta phase

The microstructure of undeformed material (Figure 4.3a) mainly consists of the α phase (HCP) with 6% retained β -phase (BCC). Since all tensile tests are performed in the temperature range from 880 – 920 °C, there is a chance of phase transformation from the α phase to β -phase. After water quenching, the transformed β -phase is completely or partially transformed into a martensitic type α , called as secondary α_s . This secondary α_s is used to quantify the β -phase transformation during thermomechanical tests.

Figure 4.5 presents the BSE-SEM images of the superplastic deformed samples both from the gauge region and from the grip region. The deformation history of each image corresponds to the annotated ‘A’ to ‘L’ in Figure 4.4. It can be observed that all testing conditions lead to a decrease in the volume fractions of the α -phase due to the β -phase formation at high temperatures. Such behaviour is found in both grip and gauge zones, although in the grip region, the increase in the volume fraction of β -phase is smaller than in the gauge region at all examined stages of deformation except at stages E, K, and L where the volume fraction of the β phase in the grip section is slightly larger than that in the gauge section. The volume fraction of the β -phase also increases with increasing deformation temperature or decreasing strain rate. It is worth noting that at lower strain rates of 0.001s^{-1} and 0.0005s^{-1} , where work hardening is dominant, there is no significant difference between volume fraction of β phase in grip and gauge regions, except at fracture stage L, where the β phase volume fraction in the gauge region is 167% higher than that of the grip region. Moreover, at the said lower strain rates, it is observed that as the strain increases (to 0.43 and 0.48 at 0.001s^{-1} and 0.0005s^{-1} respectively), the microstructure has undergone substantial evolution in terms of the size, shape, and distribution of phases. By comparing the β phase transformation for deformation conditions of 880 °C / 0.01s^{-1} to the β phase transformation for deformation conditions 900 °C / 0.01s^{-1} , it can be observed that for the final fractured sample that the volume fraction of the β phase considerably increased from 14.5% to 32% and 20% to 48% for grip and gauge regions, respectively. This means that for a specific strain rate, the increase in the deformation temperature can significantly increase the evolution of β -phase both in static annealing as well as in superplastic deformation. Overall, there is a relatively high modification of the microstructure in terms

of the distribution and shape of the phases in the gauge region as compared to the grip region. Moreover, for both grip and gauge regions under all testing conditions, the presence of curved grain/interphase boundary in microstructure features resembling “pinching off” of one phase by the other or wedging of one phase between the grains of the other phase is also observed. It appears that the softer β phase wedges between α grains to separate them. Similar characteristics have been observed in other types of titanium alloys ([Velay et al. 2016](#); [Mikhaylovskaya et al. 2017](#); [Mosleh et al. 2019](#)) where the softer β -phase appears as a concave interphase boundary while the harder α -phase appears as a convex one as the softer β -phase wedges between the grains of the harder α -phase.

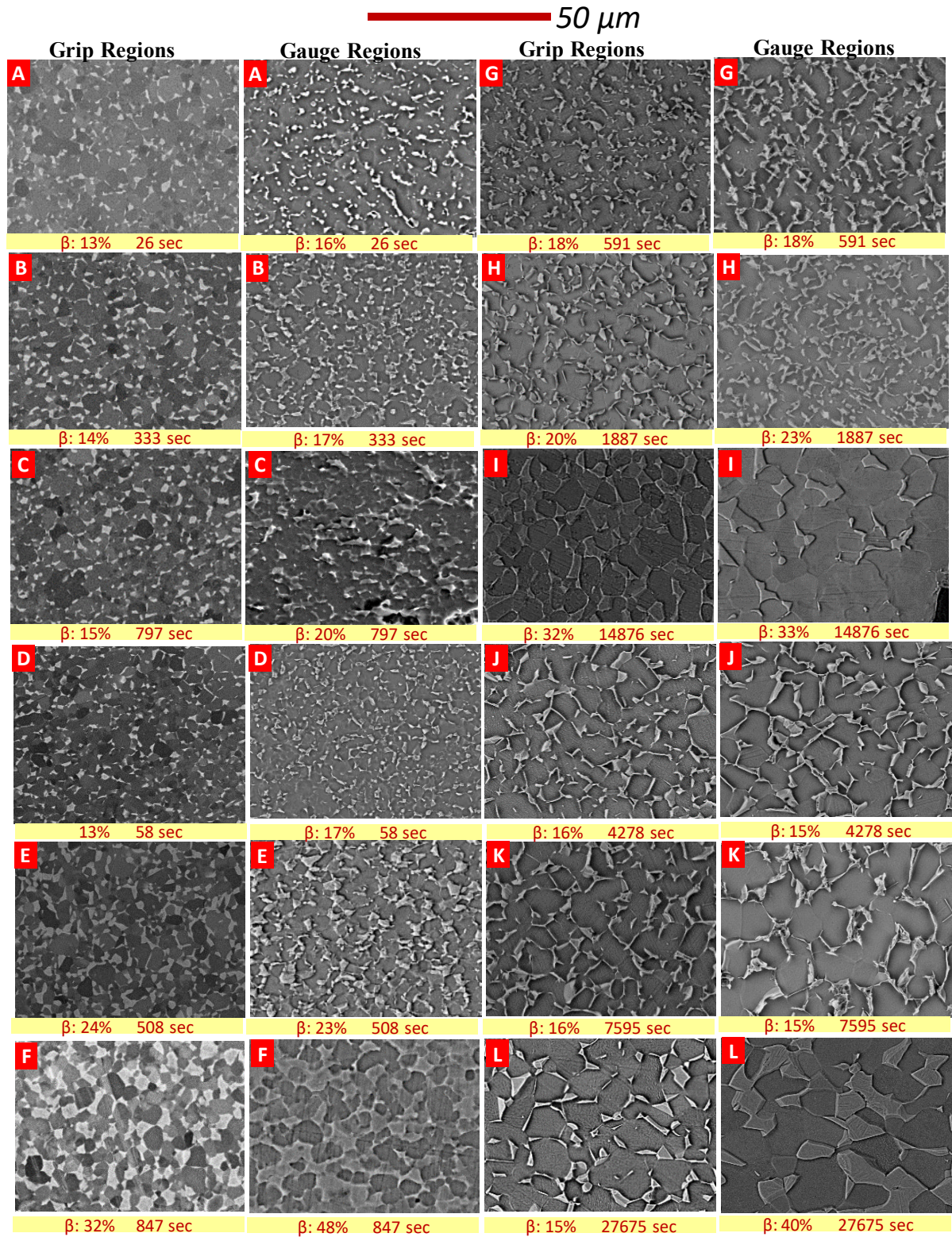


Figure 4. 5. Backscattered-electron micrographs (BSE-SEM) of microstructures evolution in TA15 during static annealing and superplastic deformation. (The labels A, B, ...L denote the interrupted stages of the tensile tests)

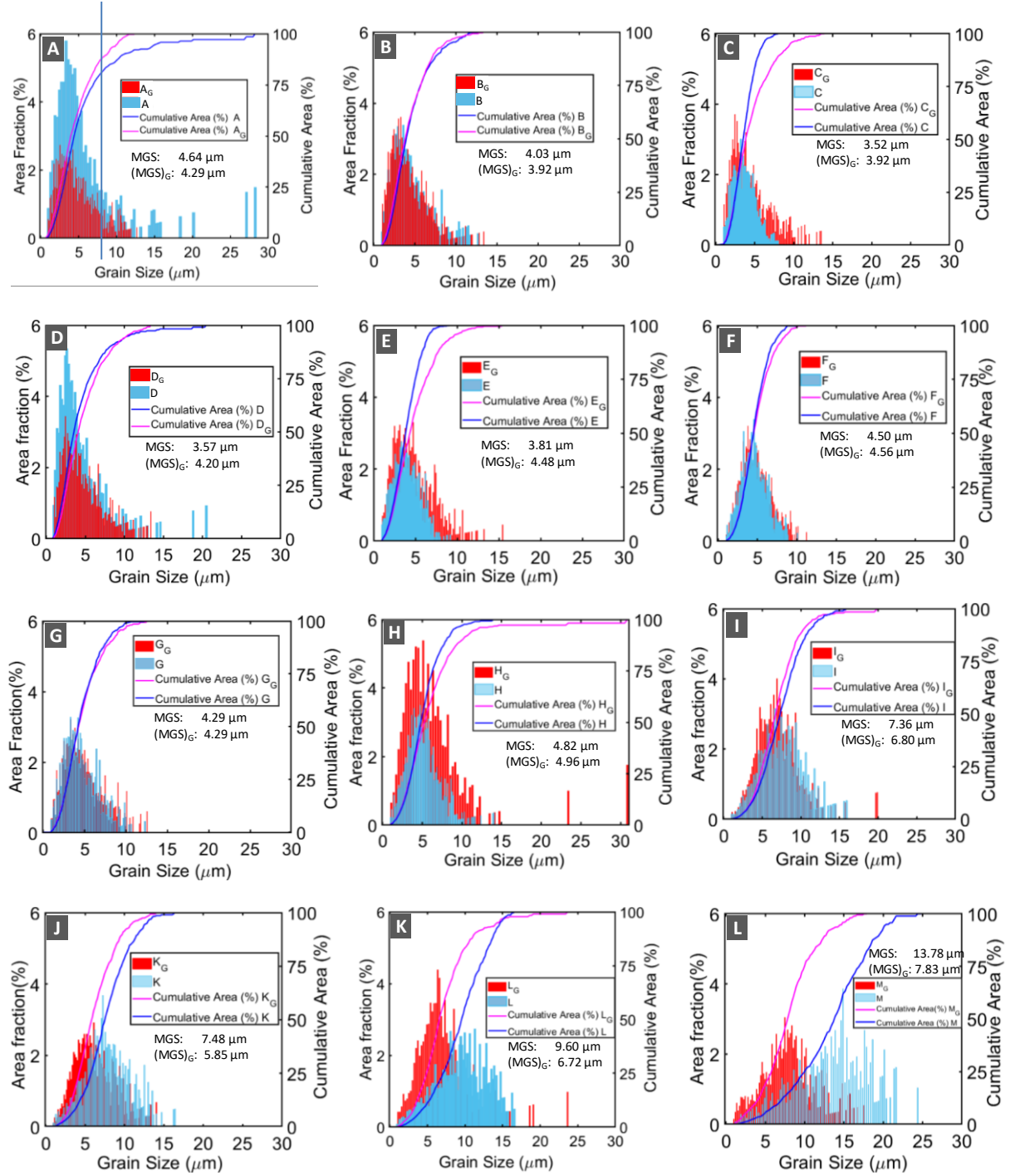
4.3.4. Grain size evolution

Figure 4.6 shows the comparative grain size evolution for the grip and gauge regions at different testing conditions. Overall, in both the static (grip) and dynamic (gauge) regions, grain growth is observed during the testing conditions. For the stages A to F, the morphology of grain size of the gauge specimens deformed at 0.01s^{-1} , changes from skewed grain size distribution to almost normal distribution. On the other hand, the distribution of grain sizes for the corresponding grip regions retains the degree of skewness from stages A to C. while for stages the D to F, the morphology of grain sizes changes from a skewed to normal distribution. It is observed that for the deformation stages C, E, F, G, H, and J, the average grain sizes in gauge regions are smaller than those in the grip regions. In contrast, for the rest of the deformation stages, the grain sizes of grip regions are smaller than those in gauge regions. It is noted that significant grain growth does not take place at a higher strain rate of 0.01s^{-1} . It can be observed that moving forward from stage A to C for deformation conditions of $880\text{ }^{\circ}\text{C}/0.01\text{s}^{-1}$, the average grain sizes in the gauge region decrease from $7.92\mu\text{m}$ to $3.90\mu\text{m}$, which is in well agreement with the flow softening behaviour of the corresponding stress-strain curve. On the other hand, the grain size in the grip region at deformation stages A to C, exhibits nearly steady behaviour, with only a slight decrease in grain size from $5.56\mu\text{m}$ to $5.23\mu\text{m}$. A possible interpretation for the larger grain size in the grip section than the gauge section under given deformation conditions can be related to dynamic recrystallization during superplastic deformation. As shown in Figure 4.4, for the higher strain rate (0.01s^{-1}), the material in the gauge region shows apparent strain-softening behaviour at a true strain >0.33 and >0.74 for 880°C and 900°C respectively, suggesting that DRX appears during the superplastic deformation. On the other hand, microstructures in the grip region are only governed by grain growth (Jain and Mishra, 2013) since DRX cannot take place in static conditions. Therefore, microstructures in the grip regions are coarser than those in the gauge regions. Due to the competition between DRX and the concurrent grain growth processes, the grain size in the gauge region does not change significantly.

By comparing the evolution of grain size in the grip region with the grain size evolution of the gauge region, it is clearly found that the final grain size in the gauge region increases

with increasing deformation temperature and/or decreasing strain rate. In the grip region, however, for a specific strain rate of 0.01S^{-1} the grain size shows a similar slightly increasing trend at both high (900°C) and low (880°C) deformation temperatures. However, at the lower strain rates, 0.001s^{-1} and 0.0005s^{-1} , a considerable increase in grain size is observed at all deformation conditions for both grip and gauge regions, although in the grip region, the increase in grain size is smaller than that in the gauge region for the same deformation conditions. However, the grains remain essentially equiaxed in both grip and gauge regions. At these low strain rates, both grip and gauge sections are exposed to the high temperature for an extended period, and so the grains have enough time to grow through thermally initiated mechanisms. It can be seen from [Figure 4.6](#) that for the deformation stages G, H, ...L (associated with low strain rates 0.001s^{-1} and 0.0005s^{-1}), grain growth in both grip and gauge region is initiated at the beginning of the superplastic deformation and grain size increases with increasing strain. The superplastic deformation behaviour at these low strain rates is believed to be controlled by the competition between dynamic recrystallization and dynamic grain growth. The strain hardening characteristic is as a result of the higher impact of dynamic grain growth rather than through grain refinement due to dynamic recrystallization.

The presence of larger final grain size in the gauge region than in the grip region indicates that dynamic grain growth, rather than grain refinement by dynamic recrystallization (DRX), took place under a given deformation condition ([Jeong et al. 2009](#)). Moreover, the presence of larger final grain size in gauge region, as compared to that in the grip region, contradicts studies of other alloys ([Cepeda-Jiménez et al. 2013](#); [Chai et al. 2014](#); [Cao et al. 2015](#); [Vijayananth et al. 2019](#)). These studies reported smaller grain sizes in gauge regions as compared to grip regions. The possible reason for this contradiction is the apparent absence of strain hardening in the stress-strain curves shown for the alloys used in these studies



*Subscript *G* denotes grip region *MGS denotes median grain size

*The labels A, B, ... L denote the interrupted stages of the tensile tests

Figure 4. 6. Evolution of grain size in TA15 during deformation stages A, B, ... L of the stress-strain curves.

4.3.5. Texture Evolution and Recrystallization

In the superplastic deformation, the texture evolution is completely different from that under non-superplastic conditions. Texture is a good indicator for investigating the superplastic deformation mechanisms of a material because, on one hand, the texture weakening at superplastic deformation is a result of random grain rotations (Duan et al. 2016) and is generally accompanied by a grain boundary sliding (GBS) phenomenon (Alabort et al. 2016). On the other hand, GBS is generally accommodated by diffusion or by slip, and the latter may cause some texture changes Watanabe et al. 2013. Figure 4.7 and Fig 4.8 shows the grain orientation maps and the corresponding inverse pole figures respectively for grip and gauge regions under different deformation condition. The colour codes, red for $\{0\ 0\ 0\ 1\}$, green for $\{\bar{1}2\bar{1}0\}$ and blue for $\{\bar{1}100\}$ are shown in the standard stereographic triangle and represent the crystal orientations of the alpha grains. The texture intensities from the colour bars are expressed in multiples of random distribution (MRD) and multiples of the uniform density (MUD) for the inverse pole figures and the pole figures respectively. It is noted that the texture present in the grip region is quite different from that found in the gauge regions under all testing conditions. Nevertheless, the texture in grip regions is only slightly changed from that of as-received material.

For the deformation $880^{\circ}\text{C}/0.01\text{s}^{-1}$, it is noted for gauge regions (images A, B & C of gauge regions in Figure 4.7) that DRX has been initiated at stage A and its fraction increases with increasing deformation so that the final fracture sample C represents a fully recrystallized structure. This recrystallization phenomenon is also clearly evident from the pole figures where strong weakening of the texture occurs through stages A to C and results in almost randomize texture. On the other hand, no apparent recrystallization is observed for grip regions (i.e. grip region images A, B, & C in Figure 4.7), where the final stage C_G still shows an inhomogeneous microstructure. Keeping the strain rate constant at 0.01s^{-1} and increasing the temperature from 880°C to 900°C , a significant increase in DRX of the gauge area is evident at the higher deformation temperature 900°C , as shown in Figure 4.7 (images D, E & F of gauge regions). This is evident from the dominant presence of fine recrystallized equiaxed grains replacing the coarse deformed grains. Similarly, to 880°C , at 900°C the degree of SRX is also slightly increased in grip region (grip region images

D, E, & F in Figure 4.7). The presence of SRX in grip regions at 900 °C, as evidenced by the randomized textures from D to F (Figure 4.8), is attributed to the thermal energy available during the static annealing. It can be noted that as the deformation continues from stage D to stage F, the maximum texture intensity of gauge regions monotonously decreases from 5.2 to 1.4, and leads to weakening and full randomization of the initial texture. The strong basal plane textures $[0\ 0\ 0\ 1]$ at stage D has completely disappeared at stage F. At the same time, there is little weakening of texture corresponding to grip regions of D, E & F in Figure 4.8.

For the deformation conditions of 920°C/0.0005s⁻¹ (Figure 4.8), the texture randomization of the gauge region is achieved at a very early stage of deformation, *i.e.* the maximum texture intensity at stage J drops to 2.6 which indicates randomization of the initial texture. Onward from stage J there is a relatively weak drop in textural intensity until the final fracture stage L. This can be attributed to dynamic grain growth, limiting the phenomenon of GBS/grain rotation. The grain orientation map of gauge regions (images G, H, & I in Figure 4.7) at given deformation conditions also supports the phenomenon of slow texture weakening beyond stage J. It can be observed that recrystallization in the gauge region has been completed at stage J, and in the later stages there is only an increase in grain size rather than further recrystallization. The maximized DRX at this lowest strain rate, and the highest temperature is mainly attributed to the synergistic effects of higher deformation temperature and the relatively longer deformation time, which is 4278 sec for 0.48 strain. The same high exposure time under a high-temperature environment also causes significant recrystallization in the grip regions (grip region images J, K, & L in Figure 4.7). However, the grains are more inhomogeneous relative to those in the corresponding gauge regions.

It is also interesting to note that the larger the strain is, the less elongated the grains become. Also, under all testing conditions, the grain orientation maps of the gauge regions show the grains slightly elongate along the tensile direction at initial and intermediate stages of deformation but regain fully equiaxed shape at the final fracture stage. Meanwhile, it is also evident that the grains oriented in the $\{0001\}$ direction symbolized by the red region, given in the grain orientation maps of gauge regions, narrow as the strain increases. Once a severe strain is performed, it is invariable that $\{\bar{1}100\}$ and even $\{0\ 0\ 0\ 1\}$ tend to rotate

in the other crystallographic directions (Wang et al. 2014). The preferential orientation weakens or even disappears in the microstructure of gauge regions (after superplastic deformation) in contrast to the microstructure of the corresponding grip regions.



Figure 4. 7. Grain orientation maps in TA15 during static annealing and superplastic deformation. (The labels A, B, ...L denote the interrupted stages of the tensile tests)

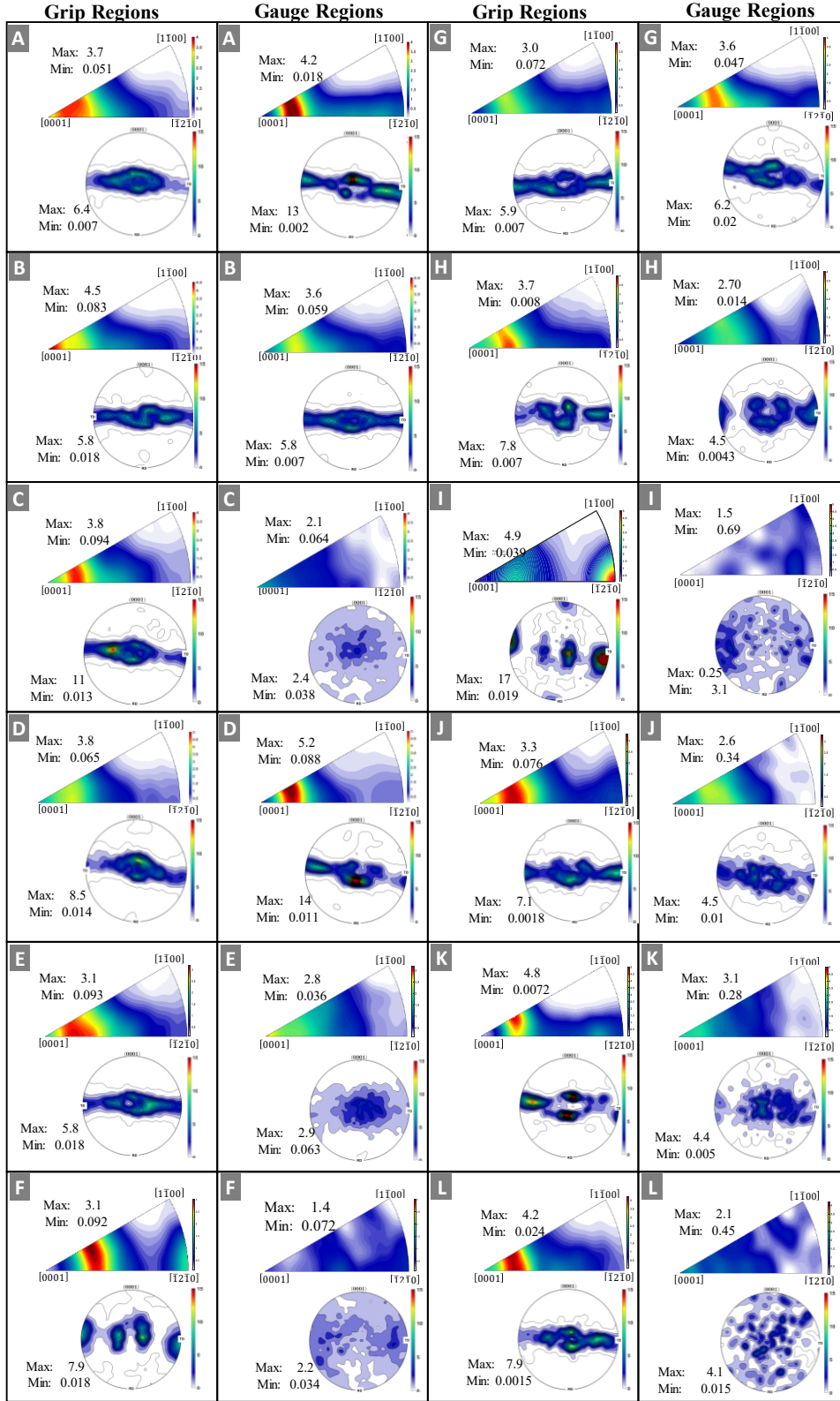


Figure 4. 8. Texture evolution in TA15 during static annealing and superplastic deformation. (The

labels A, B, ...L denote the interrupted stages of the tensile tests

4.3.6. GND Evolution

Comparative quantitative descriptions of GND evolution in the grip and gauge regions under different conditions of strain rate and deformation temperature are shown in [Figure 4.9](#). It can be observed that the GND values of gauge regions are smaller than that of the grip regions for all testing conditions except for deformation stages A and D, where the dislocation density of the gauge region is higher than the corresponding grip regions by 200 and 135% respectively. A possible explanation for this higher GND in the gauge region rather than the grip region can be described as following. In the initial stage of deformation at testing conditions of $880^{\circ}\text{C}/0.01\text{s}^{-1}$, there is an increase in the flow stress ([Figure 4.4](#)) due to the interaction and multiplication of dislocations. As shown in [Figure 4.8](#), the detailed observation of the GND map at stage A of the gauge region indicates that there are some large grains without dislocations, as signified by the blue colour. Also, some dislocation free small grains can be observed, which is evidence of the creation of new recrystallized grains. In addition, channels like GND distributions can be found in the form of cellular or subgrain patterns/structures, which implies the presence of the continuous dynamic recrystallization (CDRX) process. These results are consistent with the literature ([Qiu et al. 2020](#) and [Sun et al. 2020](#)), which reported that CDRX is a recovery dominated process and proceeded by continuous absorption of lattice dislocations, generated during the deformation process, into sub-grains boundaries (without change in the sub-grain size). This results in an increase in the misorientation angle between adjacent sub-grains ([Momeni et al. 2014](#) and [Humphrey et al. 2017](#)). Generally, it is revealed that for both grip and gauge regions, the decrease in dislocation density is greatly affected by strain rate (annealing time), while the variation of deformation temperature has only a trivial impact on the evolution of GND.

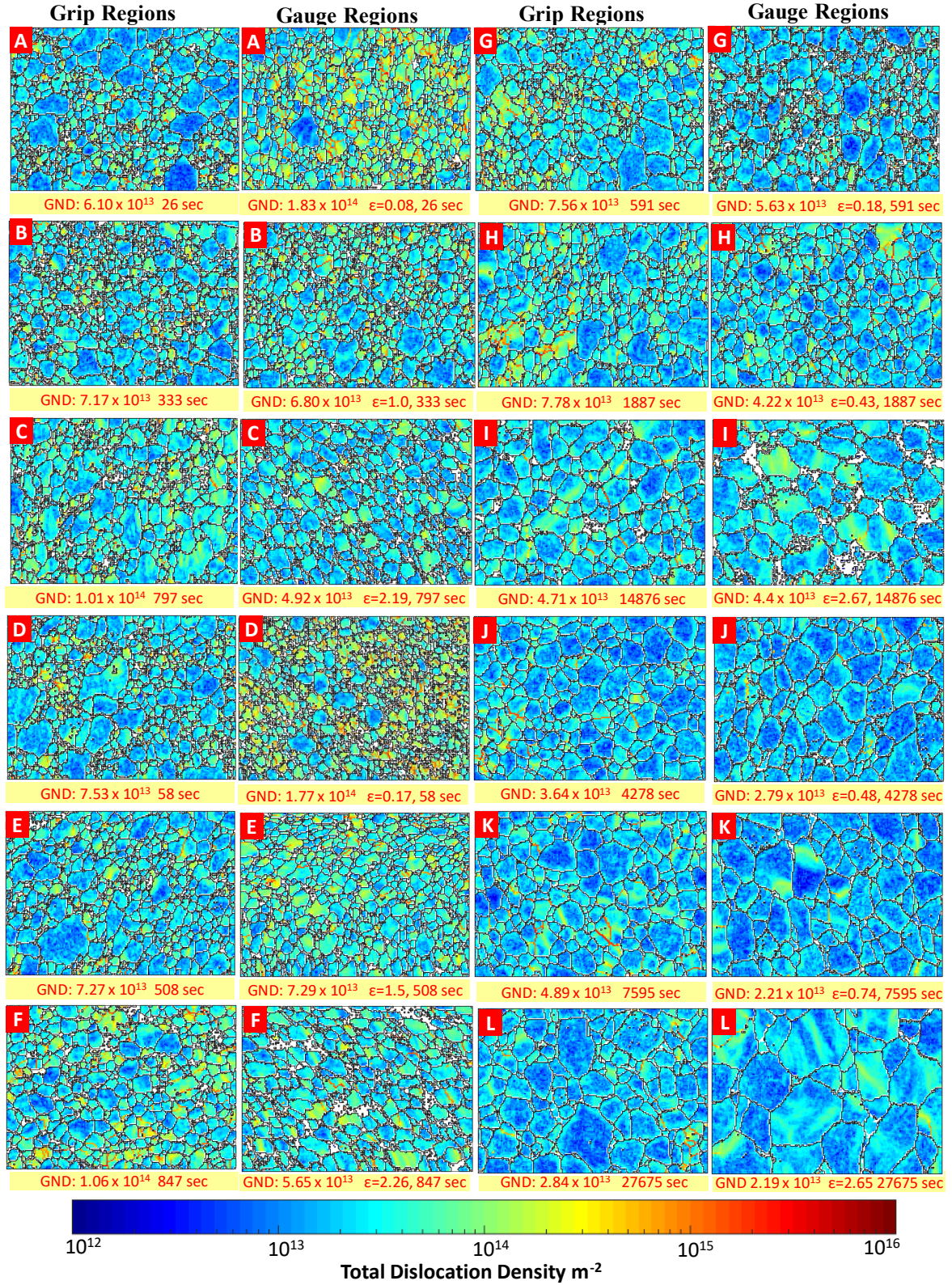


Figure 4. 9. GND evolution in TA15 during static annealing and superplastic deformation. (The labels A, B, ...L denote the interrupted stages of the tensile tests)

In addition to the evolution of GND, the percentage of high angle grain boundaries (HAGBs) is another important microstructural parameter used to differentiate between static annealing and superplastic deformation. In order to obtain ideal superplasticity, it is necessary to produce fine-grained alloys with both reduced grain size and an increased percentage of HAGBs. [Figure 4.10](#) shows the EBSD Pattern quality maps describing the evolution of the fraction of high angle grain boundaries (HAGBs) in the grip and gauge regions under different testing conditions. In this study, HAGBs correspond to misorientation angles higher than 15° while LAGBs correspond to $1-15^\circ$. Compared to the 83% fraction of HAGBs in the as-received material ([Figure 4.3g](#)), both the grip region and the gauge region have experienced an increase in the fraction of HAGBs under the testing conditions. Under all testing conditions, the evolution of the fraction of HAGBs in gauge regions is quite different from that in the corresponding grip regions, indicating that the microstructure evolution mechanisms are different. In case of gauge regions, the fraction of HAGBs monotonously increases with the increasing strain of the testing condition, while in case of grip regions, the fraction of HAGBs decreases with increasing strain, with the exception to the deformation condition $920^\circ\text{C}/0.0005\text{s}^{-1}$, where it shows an almost steady behaviour. In addition, it is well known that the HAGBs have higher boundary energy than the LAGBs, and thus the GBS occurs more easily along HAGBs than along LAGBs ([Park et al. 2008](#) and [Zhang et al. 2018](#)). The increase in HAGBs with increasing the strain at the lower strain rates of 0.001s^{-1} and 0.0005s^{-1} may indicate that grain boundary sliding (GBS) is the dominant deformation mechanism during the superplastic deformation process ([Mikhaylovskaya et al. 2017](#) and [Hémery et al. 2019](#)). On the other hand, an increase (from initial 83% to final 92%) in the fraction of HAGBs in the grip region at $920^\circ\text{C}/0.0005\text{s}^{-1}$, where grip material is exposed to high temperatures for a long duration of time, shows that the increase in the fraction of HAGBs during superplastic deformation is not only the result of the strain but is also enhanced by annealing time at the test temperature. In the meanwhile, it is evident from the grip samples exposed to deformation conditions of $880^\circ\text{C}/0.01\text{s}^{-1}$, $900^\circ\text{C}/0.01\text{s}^{-1}$ and $880^\circ\text{C}/0.001\text{s}^{-1}$, that the conversion of the high density of LAGBs in the interior of fine equiaxed grains into more homogenous HAGBs is greatly affected by annealing time and temperature.

As compared to the fraction HAGBs at stages A & B to stages D & E (which are deformed at same temperature but different strain rates), the proportion of LAGBs is relatively high in the latter case. This is mainly because the subgrain boundaries have insufficient time at higher strain rate to absorb dislocations for transformation.

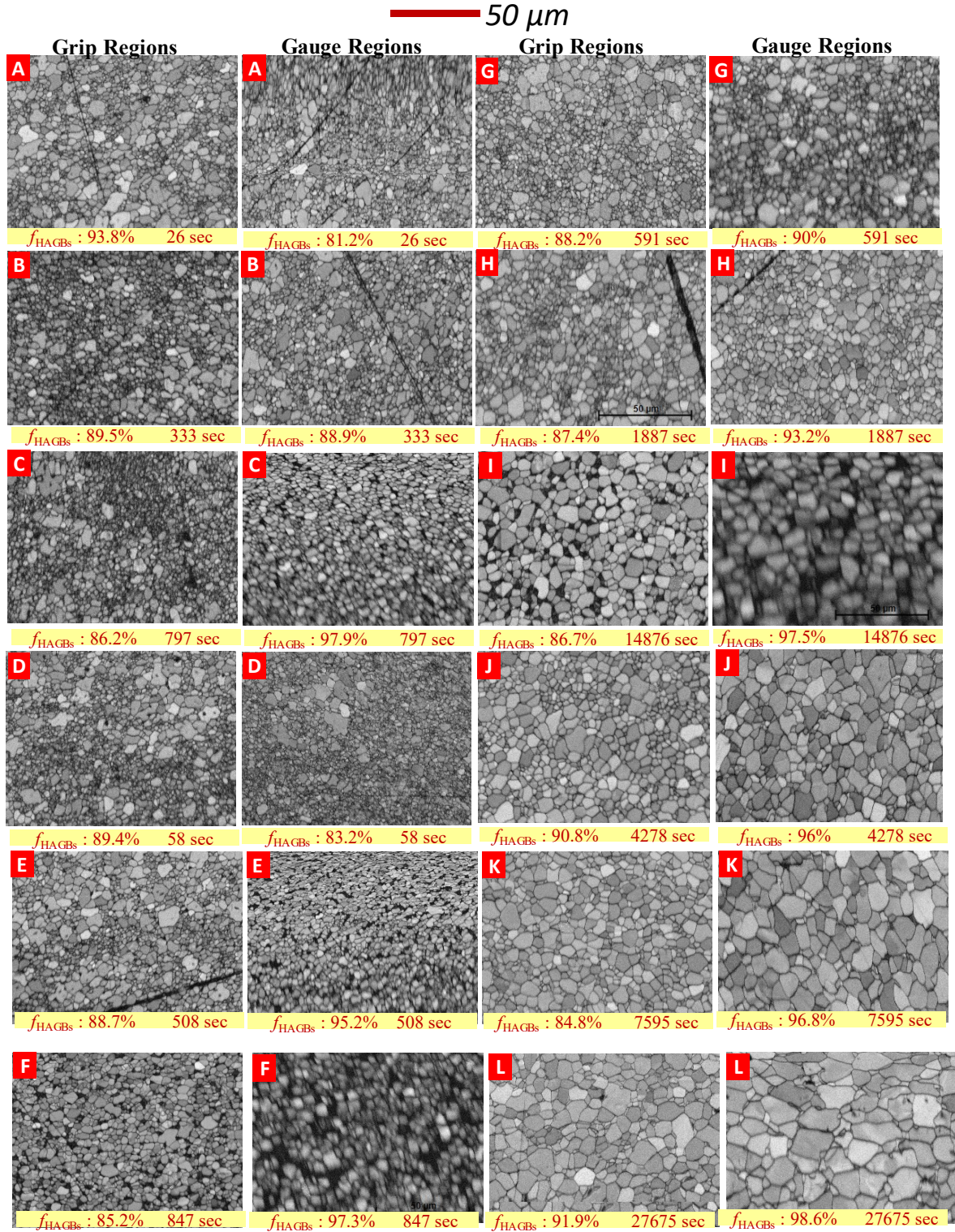


Figure 4. 10. EBSD pattern quality maps with fraction of HAGBs of TA15 during static annealing and superplastic deformation. (The labels A, B, ...L denote the interrupted stages of the tensile tests)

4.4. Discussion

Results summarizing the microstructure and texture evolution in static annealing (grip region) and superplastic deformation (gauge region) of TA15 alloy are quantified in Figure 11.

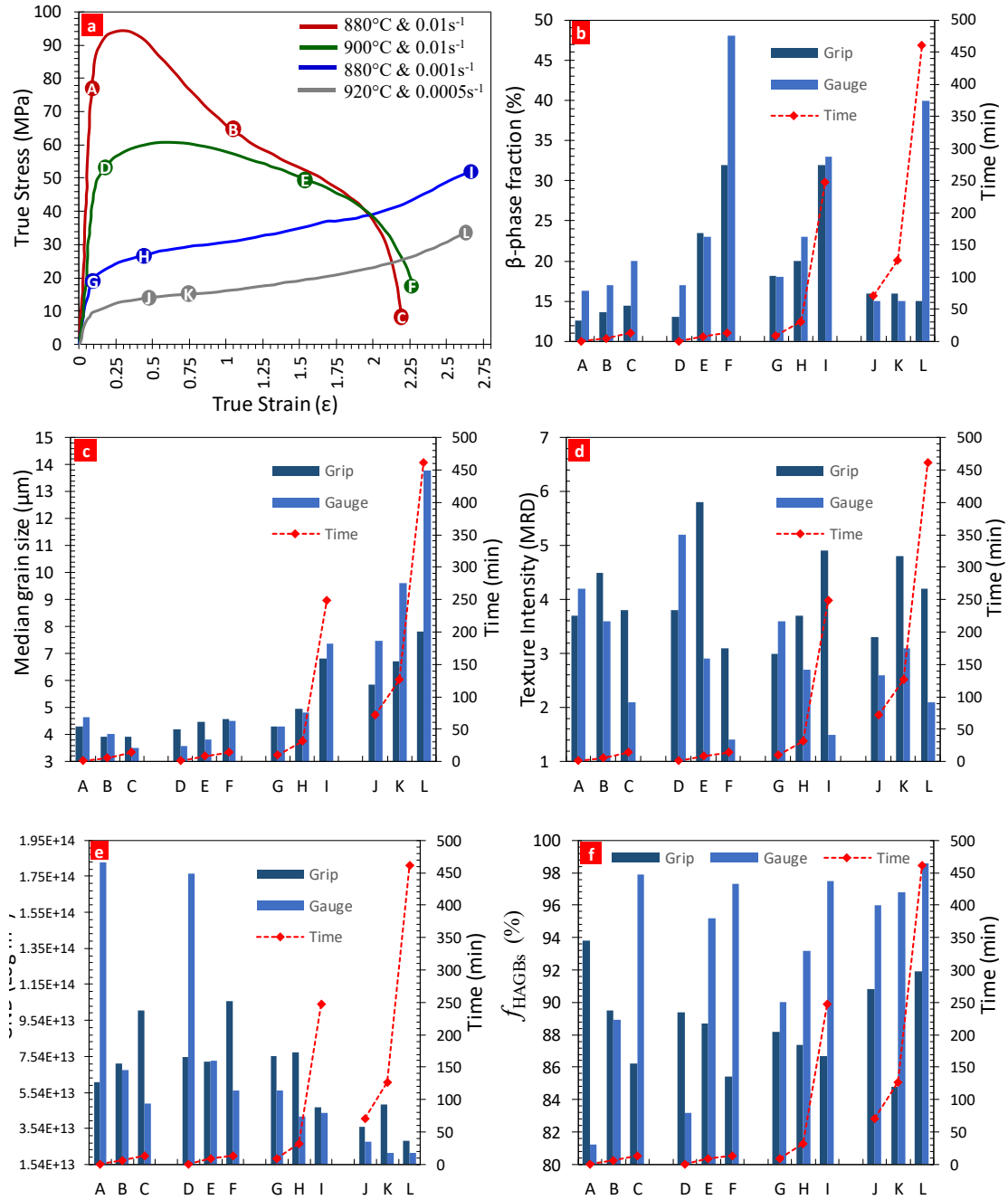


Figure 4. 11. Quantitative illustration of the results presented in section 3: (a) True stress-strain curves (b) β -phase evolution (c) Grain size evolution (d) texture evolution, and (e) GND evolution (f) fraction of high angle boundaries

4.4.1. β -phase evolution

Generally, in titanium alloys, the α to β -phase Transformation is controlled by diffusion transfer (Motyka et al. 2014b) of slow β -stabilizers (*e.g.* V or Mo atoms) from α to β . This is a thermally activated process, and so any increase in temperature will accelerate the rate of diffusion. From Fig 4.11b it can be observed that for all testing conditions except 920°C/0.0005s⁻¹, the increase in deformation temperature causes an increase in the volume fraction of β -phase. Stages A, B and C of the grip region show an increase in the β -phase fraction from initial 6% to the final 15%. The increase in β -phase fraction for the corresponding gauge regions also show a similar tendency, although in the gauge region, the increase in the volume fraction of β -phase for the fractured stage C is 5% higher than in the corresponding grip region. This is believed to be attributed to the presence of fine equiaxed α and β -grains which allows the fine β -grains to grow within the grain boundaries of α -phase. For testing condition 900°C/0.01s⁻¹, stage D shows a similar trend to stage A, while at stage E the fraction of β -phase at grip and gauge region are comparable. Grip region F which is exposed to an almost same aging time as grip region C. Here, however the volume fraction of β -phase is 113% higher than stage C which implies a strong dependency of α to β -phase transformation on temperature. Similarly, at gauge region F, the fraction of β -phase is $\approx 56\%$ higher than the corresponding grip region, this can be attributed to strain induced transformations. For stages G, H, and I, the volume fraction of β -phase at grip and gauge regions are almost identical. The possible reason for this similarity is that at these stages there is only a very low contribution made by strain induced transformation in the β -phase evolution, and overall for these stages the phase transformation is dominated by diffusion process within both grip and gauge regions. By comparing the fraction of β -phase at stages A, B, C with stages G, H, I, it is evident that a lower strain rate (0.001s⁻¹ as opposed to 0.01s⁻¹) provides longer exposure time at the same temperature (880°C). It can be seen that effect of a long exposure time at this lower temperature is not as strong as it is in stages D, E, F which are at higher temperature (900°C) but a same strain rate (0.01s⁻¹).

For stages J, K & L, due to the higher temperature (920°C, close to the β -transus temperature) and the longer exposure time resulting from the low strain rate (0.0005s⁻¹), an unusual trend for β -phase evolution can be observed. Unlike under other test conditions, the gauge region at J and K has a slightly lower β -phase fraction than the corresponding gauge regions. This unusual trend of β -phase in gauge regions J and K can be explained in terms of a reverse transformation of meta-stable β phase to α phase. The low strain and longer thermal exposure time (4278 & 7595 seconds for J & K respectively) at the temperature 920°C is believed to promote the $\beta \rightarrow \alpha$ reverse phase transformation as reported by [Guo et al. \(2018\)](#) and [Ji et al. \(2020\)](#). At final stage L where an elongation of 1357% was achieved, the fraction of β -phase is increased possibly of because of high strain-induced phase transformation.

A certain level of cavitation can be also observed in the fracture gauge regions deformed at 880°C/0.01s⁻¹ and 880°C/0.001s⁻¹ (gauge regions C and I in [Figure 4.5](#)) During superplastic deformation, the grains slide along the surface of their grain boundaries (known as grain boundary sliding GBS), causing stress concentration at the grain boundaries triple junctions ([Kaibyshev, 2012](#)). The stress concentration should be relieved by accommodation processes to avoid the formation of voids at the grain boundaries ([Ridley, 2011](#)). If the stress concentration is not relieved sufficiently rapidly due to insufficient accommodation, cavities will be nucleated at the interface.

In this study, it is found that the voids and cavities mainly tend to nucleate and grow at the grain boundaries adjacent to the α -phase, suggesting that the α -grains lacked the accommodative ability to successfully release the stress concentration during GBS ([Liu et al. 2013](#)).

4.4.2. Grain size evolution

The grain size results are discussed in the light of four phases of recrystallization which are similar for both dynamic and static recrystallization. The four phases include: (i) dislocation annihilation by DRV, (ii) nucleation of new grains, (iii) grains refinement by the absorption of the small grains by large grains or the by appearance of cell structures in large grains, and (iv) grain growth to further reduce the grain boundary energy.

Figure 4.11c shows that the final recrystallization phase (*Phase-IV*) of grain growth is achieved for both static and dynamic conditions at strain rates of 0.001s^{-1} and 0.0005s^{-1} whilst for the specimens tested at 0.01s^{-1} , grains refinement (*Phase-I*) is achieved for the gauge regions while the grip regions are still in simultaneous operation of Stage 1, Stage 2 & stage 3.

Table 4.2 shows the median grain size, mean grain size and the number of grains for grip and gauge regions. It can be seen that for all deformation stages the median is smaller than the average grain size which is also supported by the positively skewed spread for almost all the grip and gauge regions. The type of spread, corresponding to stages A to H (Figure 6) implies that there are very few large grains and a higher percentage of small grains. For the gauge regions I, J, K & L, the median is slightly smaller than the average grain size and the distributions are somewhat closer to normal. As shown in Table 4.2, the number of grains for the same scan area for the initial stages A & D (for both grip and gauge regions) drops to half of the 11468 grains of the as-received material. This decrease can be explained by the process of dynamic recovery during which the initial dislocation cell structure will readily convert into small grains, and large grains will absorb these small grains. This whole process will reduce the number of grains.

Moving to stage B and C, a continuous decrease in grain numbers is evident for the grip regions, indicating that Phases-I and II of recrystallization are operative. This is supported by the GND distribution in Figure 4.9 where it can be observed that the GND distribution in the grip regions is not uniform, and resembles the GND distribution of as-received material in which the small grains have more dislocations whilst larger grains are dislocation free. For the corresponding gauge region B, the number of grains drops further, and the GND distribution is similar to the gauge region A. At gauge region C, however, the number of the grains is increased, and this increase is due to the process of refinement. This refinement also corresponds to the median and mean grain sizes which are very close together with a more even grain size distribution. Similar explanations hold for the grip regions for D, E and F.

In the gauge regions at stages D, E, F, however, the grain refinement phase (Phase-III) is achieved somewhere between gauge regions E and F. The drop in the grain size at gauge region F can be attributed to the initiation of Phase-IV *i.e.* grain growth. This is supported by the corresponding grain size spread having less peaks, and the median and mean values being very close.

For the grip regions in the initial stages G and H at testing conditions $880^{\circ}\text{C}/0.001\text{s}^{-1}$ and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$, the numbers of grains drop to less than half. The distribution of the grain size at grip and gauge region G looks similar to that of grip regions C and D. This indicates that due to the longer exposure time of the grip and gauge region G, Phase-I, II & III of recrystallization is already achieved. Hence, recrystallization Phase-IV (grain growth) initiates at stage G and continues to stage H and I. The rate of grain growth is higher for the gauge regions compared to the grip regions. This is attributed to the additional strain energy supplied above that of temperature alone. For stages J, K, & L, the numbers of grains at grip region J (*i.e.* 2849) is less than half that of the as-received material. A similar explanation holds for this as for stage G, H and I, *i.e.* both grip and gauge regions of stage J achieve the Phase-IV of the recrystallization due to high thermal energy (high temperature 920°C) input and longer aging time (low strain rate 0.0005s^{-1}). The rate of grain growth is much higher for the gauge region, as can be seen by the number of grains at the final gauge region L dropping to 759.

In summary, for specimens tested at the lower strain rates of 0.001s^{-1} and 0.0005s^{-1} , data from the grip regions reveals the occurrence of limited time-induced grain growth. On the other hand, the microstructure of the corresponding gauge regions exhibits deformation-enhanced grain growth under these conditions. As expected, at these lower strain rates, a comparison of grain coarsening rates exhibited that the dynamic value (gauge region) was greater than the static one (grip region). This coarsening rate can be attributed to enhanced diffusion associated with the generation of dislocation substructure in the β -phase through which solutes are being transported (Sargent et al. 2008). In superplastic deformation the phenomenon of deformation-enhanced grain growth is usually associated with grain

boundary sliding where as in conventional plastic deformation, straining impedes the grain growth. The grip area shows general grain growth and a levelling of size distribution (less peaks). This is thermally driven by the static annealing process. For the grip region the general growth of grains and reduction in the histogram peak is attributed to the smaller grains being absorbed into larger growing grains. The gauge area has the same thermal input, but also has a deformation input. This results in more grain displacement and rotation, and greater diffusion of atoms in the grain boundary. It is possible that adjacent grains having low angle boundaries may merge, giving rise to the grain size anomalies shown in [Figure 4.6](#). At the same time, the superplastic deformation and thermal processes (dynamic annealing) creates multiple new grain initiation sites, and these will work to bring down average grain size. As in the grip region, thermal grain growth will occur, and may dominate at low strain rates and long timescales.

The observation of lower grain growth in the gauge regions A to F than for the corresponding grip regions (static annealing) can be ascribed to GBS as a dominant deformation mechanism. For the deformation condition $880^{\circ}\text{C}/0/0.001\text{s}^{-1}$, the lower gauge regions average grain size values at the initial stages G and H then the corresponding grip regions and a higher grain size value of the fracture gauge region I then grip region I, suggests dual deformation mechanism for the specimen. The initial deformation is controlled by GBS, while the subsequent one is dominated by slip creep accommodated by dislocation movement ([Li, 2013](#)). The higher grain growth for all gauge regions at testing condition $920^{\circ}\text{C}/0/0.0005\text{s}^{-1}$ suggests diffusion creep as a dominant deformation mechanism at all stages. Overall, the grains retained almost an equiaxed shape in all gauge regions, which indicates a high contribution of GBS controlled by the grain boundary diffusion of the β -phase and low contribution of dislocation glide to the total deformation ([Ma et al. 2009](#)).

Table 4. 2. Grain parameters for grip vs gauge regions

Stage	Grip Region			Gauge Region		
	No. of grains	Median grain size (μm)	Avg. grain size (μm)	No. of grains	Median grain size (μm)	Avg. grain size (μm)
A	5262	4.29	5.6	5002	4.64	7.9
B	5371	3.92	5	5056	4.03	5.1
C	5288	3.92	5.2	6278	3.52	3.9
D	5200	4.2	5.5	6285	3.57	5.6
E	5038	4.48	5.5	5629	3.81	4.2
F	3826	4.56	5.2	3466	4.5	4.9
G	4950	4.29	5.3	4308	4.29	5.1
H	3531	4.96	7.8	3921	4.82	5.6
I	1849	6.8	7.7	1588	7.36	8.1
J	2849	5.85	6.4	1858	7.48	8.3
K	2485	6.72	8.2	1373	9.6	10.1
L	2243	7.83	10.1	759	13.78	14.3
Initial material	11468	2.76	3.74			

4.4.3. Texture evolution

From [Figure 4.11d](#) it can be noticed that the texture intensity in the gauge regions follows a decreasing trend with increasing strain, while in case of grip regions the texture intensity remains similar to that of the initial material. For all grip regions except stage *I*, the initial texture experienced less significant orientation changes, and most grains orientations are still distributed around [0001] while the initial textural orientation distribution in the gauge section has been changed significantly, and the initial texture is almost randomized. This indicates that grain rotation has occurred in the gauge regions, causing the weakening of the initial texture.

The observed changes in texture and intensity of the gauge regions are closely related to grain boundary sliding (GBS) phenomena ([Liu et al. 2017b](#)), as random sliding events give rise to a homogeneous decrease in intensity ([Watanabe et al. 2012](#)). This supports the hypothesis that texture analysis can also be used to investigate the deformation mechanism ([Watanabe et al. 2013](#)). These results provide evidence to support the idea that GBS is

active and that grain rotation acts as a strain accommodation mechanism in the gauge regions.

4.4.4. GND evolution

From Figure 11e, it can be observed that for all gauge regions the GND shows a decreasing trend, which implies the deformation is governed by grain boundary sliding and grain rotation where diffusion creep/dislocation creep is the accommodation mechanism which reduces the overall GND. The dislocation density is controlled by the rate of dislocation absorption by grain boundaries (Kaibyshev, 2012). As shown in Figure 4.11e, a decrease in the strain rate enhances the capacity of grain boundaries to absorb dislocations, and this subsequently leads to a relatively lower level of GNDs in the gauge regions as compared to the grip areas. The mobile lattice dislocations can be rapidly absorbed by the boundaries of fine grains during grain boundary sliding (Rajabzadeh, 2013). In this study, this is supported as follows: It is generally accepted (Kaibyshev, 2012) that an increase in the deformation temperature for a specific strain rate increases the ability of grain boundaries to absorb dislocations. However, from Figure 4.11e, it is evident that the resulting GND value for the gauge region at 880°C is lower than the corresponding GND value at 900 °C. This can be attributed to the finer grain size at 880 °C.

For the higher strain rate of 0.01s^{-1} , the initial increase in GND in the gauge regions at points A and D can be explained by work hardening, as these regions are at the end of their elastic limit, and the dislocations are generated in the elastic range. These dislocations are subsequently absorbed at the later (plastic) stages. For the lower strain rates of 0.001s^{-1} and 0.0001s^{-1} , in the gauge regions at initial points G and J the low stress values cause low work hardening and a low generation of new dislocations. However, these newly generated dislocations are annihilated due to the long thermal exposure time.

For the subsequent gauge regions at points H, I, K and L, the GND drops further, supporting the idea that GBS/grain rotation is the main deformation mechanism while GND acts as the accommodation mechanism. The GND trend for the grip region at points A, B & C is similar to that of the grip regions at points D, E & F. As compared to the GND value

of the as-received material, the GND in the grip region initially drops, points A and D, but then gradually increases as the fractured stage at points C and F is approached.

This abnormal trend of GND evolution can be explained in three parts. First as the grip regions are held in clamps which will expand on heating, and so there is a possibility this will cause deformation of the sample in the grip region, thus increasing GND. Second, one of the major problems in high temperature superplastic tensile testing is the unavoidable material flow from the grip region into the gauge region ([Abu-Farha et al. 2011](#)), making the grip region thinner. The third reason is that the nature of the interrupted test necessitates the use of a separate test sample for each measurement point. This introduces the possibility of there being some difference in the initial GND distributions due to inhomogeneity in the original sheet microstructure. These above three reasons are believed to account for the increasing trend of GND for the grip regions at points $A \rightarrow C$ and $D \rightarrow E$. The GND for the grip regions at points $G \rightarrow I$ and $J \rightarrow L$ shows a decreasing trend, possibly as a result of deformation generated dislocations recovering due to the long thermal exposure time.

4.4.5. Microstructural evolution mechanism in static and dynamic annealing

The overall mechanism of microstructural evolution in static and dynamic annealing processes during superplastic deformation of TA15 alloy is illustrated in [Figure 4.12 \(a & b\)](#). These results cover the strain rate range of $0.01 - 0.0005 \text{ s}^{-1}$ and temperatures $880 - 920^\circ\text{C}$. During deformation through the slip process, the crystal lattice rotates so that the active slip direction moves towards alignment with the direction of the applied stresses. Consequently, the orientation of the crystal changes during deformation. In the perfect plastic deformation of a polycrystalline material, although the grains are initially orientated at random, after sufficient deformation, most of the grains realign into a preferred orientation, hence the development of texture. Based on this it can be argued that during the initial stages of deformation, for points A and D at 880°C and 900°C and strain rate 0.01 s^{-1} , plastic deformation is achieved by slip. This is shown by the rolling texture intensity only increasing from 3.7 to 3.8 from A to D in the grip region, but increasing from 4.2 to 5.2 from A to D in the gauge regions. There is also a corresponding GND

increase in the gauge region compared to the grip region. The reason for this increase in GND in the gauge region at strain rate 0.01s^{-1} is the existence of a competitive process between work hardening and dynamic recovery. In the grip region, however, there is only the process of dynamic recovery. At later stages of deformation *i.e.* from B to C and E to F, there is a relative drop in the texture intensity in the gauge region as compared to the grip regions, which suggests that GBS/grain rotation is initiated at these later stages. This possible explanation is also supported by the fall in the gauge region GND values as compared to the corresponding grip values. This is because superplastic deformation is generally associated with a decrease in dislocation activity. For grip regions, points A to C the initial rolling texture of the undeformed material is maintained.

For testing condition $880^{\circ}\text{C} / 0.01\text{s}^{-1}$, it can be seen that from A to C (grip regions) the microstructure is very heterogeneous implying that the initial stages of the static recrystallization /static recovery and grain nucleation are initiated, but to complete the next stage of grain refinement more annealing time is required. In the gauge region, it can be seen that at stage C the grains are refined, which implies that under these superplastic conditions the process of dynamic recrystallization is faster, and can be assessed by observing the similarities in microstructure at stage C (grip region) and stage B (gauge region). The refined microstructure is achieved after a dynamic annealing time of 333 sec, whereas for static annealing an annealing time of 797 sec is required, *i.e.* over twice as long.

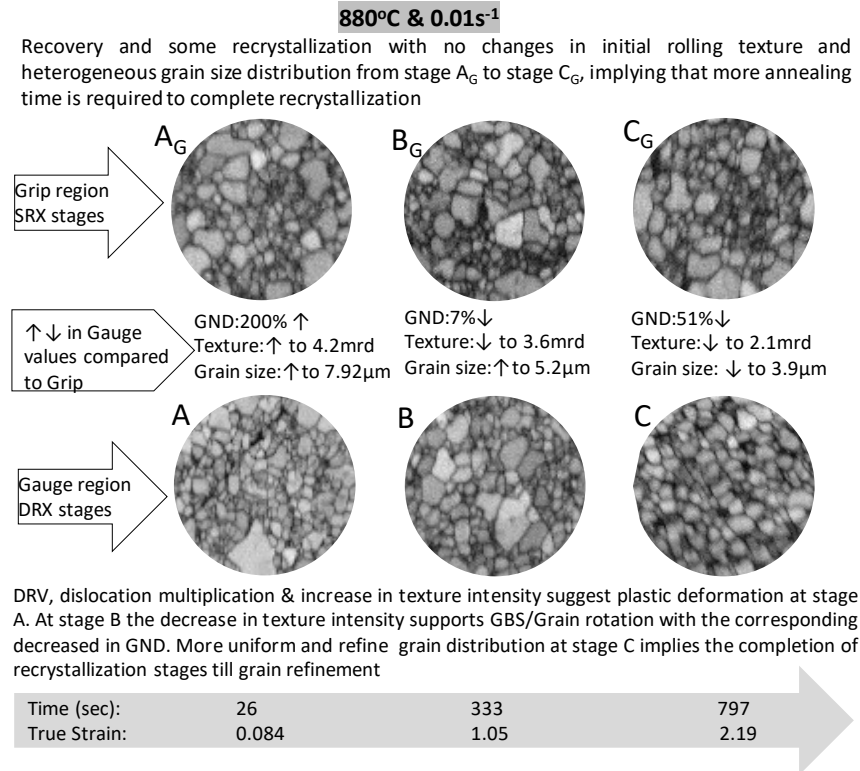
The above explanation also holds for the testing condition of $900^{\circ}\text{C} / 0.01\text{s}^{-1}$, where slight additional grain growth occurs with the increased in deformation temperature.

Considering the grip regions at the deformation temperature 880°C , when decreasing the strain rate from 0.01s^{-1} to 0.001s^{-1} , it can be observed that this decrease in strain rate provides more annealing time. Hence, all the stages of static recrystallization, including recovery, refinement, and grain growth, are achieved. It should be noted that with the decrease in the strain rate, the initial rolling texture of the alloy is maintained even after exposure to a long annealing time (14876 seconds). In the gauge region, however, the texture is entirely randomized, supporting the suggestion of the involvement of GBS/grain rotation. Comparing the grip region to gauge region, it can be seen that, for grip sample at

state G and corresponding gauge sample, the process of recovery and nucleation of new grains is initiated at the very start of tensile testing. As a result, grain growth is observed in these early stages of tensile testing. Here the growth in small grains is observed at the expense of the large grains. At the same time, some of the large grains merge with smaller grains, helping the material to attain an equiaxed and homogenous microstructure. The grain growth observed during superplastic deformation is the result of the simultaneous action of both static annealing and dynamic annealing (deformation). Based on the microstructural observation for the deformation condition of $880^{\circ}\text{C} / 0.001\text{s}^{-1}$, the effect of temperature is dominant, as the corresponding microstructures, and grain size of the gauge and grip regions are almost the same. This may be due to the fact that at the lower strain rate of 0.001s^{-1} the generated stresses are small, and this causes a decrease in the rate of dynamic recrystallization. This is also supported by the microstructural observation of grip and corresponding gauge regions for testing condition $880^{\circ}\text{C} / 0.01\text{s}^{-1}$, where the rate of dynamic recrystallization at the gauge region is fast compared to the grip region, possibly because of the higher deformation stresses at this higher strain rate. It can be deduced from these observations that a higher strain rate, resulting in higher stresses, subsequently enhances the rate of dynamic recrystallization.

At the testing condition of $920^{\circ}\text{C} / 0.0005\text{s}^{-1}$, the high thermal energy (at 920°C) causes the recrystallization process to be completed during the soak time of 15 minutes, and such recrystallization is static in nature. As a result, during the rest of the test stages, only grain refinement and grain growth are observed for both grip and gauge regions. Like the grip regions of all tests, the initial undeformed rolling texture is retained for the grip area. For the gauge regions, however, the texture intensity decreases at the initial stages, but texture develops at the later stages, due to grain growth. The grain growth is further enhanced by the process of grain rotation. Because of the importance of grain boundary sliding and grain rotation in superplasticity, there is the possibility of grain “coalescence” to occur during deformation ([Humphreys et al. 2017](#)). If the neighbouring grains rotate such that the misorientation is eliminated, then the boundary itself will be eliminated, and the two original grains will coalesce into one. After this coalescence, some local migration of the surrounding borders is likely to occur. This mechanism is capable of producing an increase

in grain size with strain and an increase in texture intensity, different from the texture of the original rolled plate.



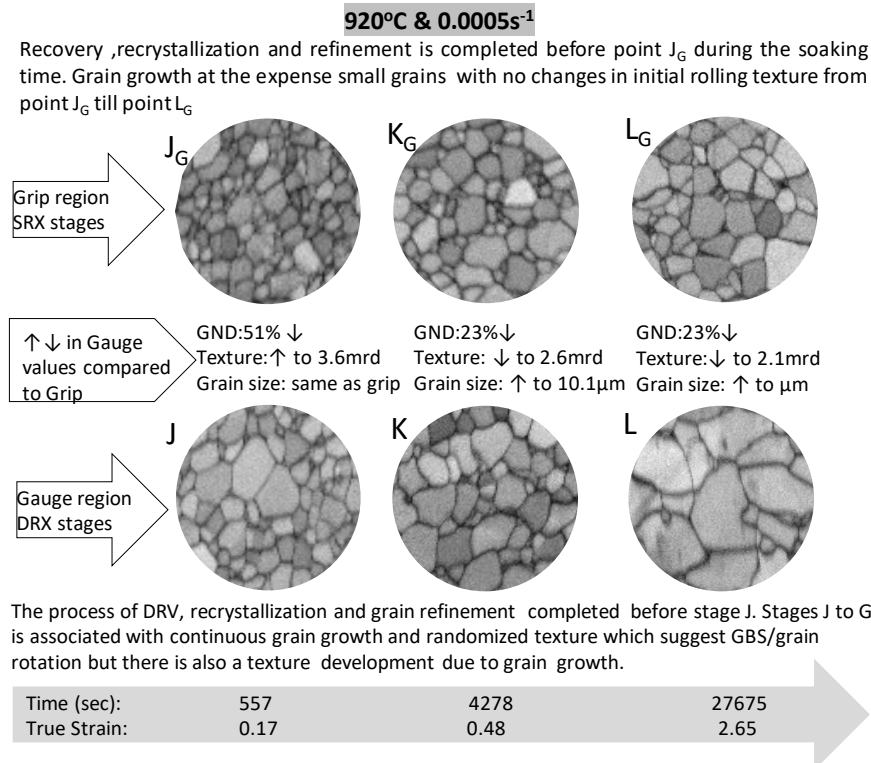
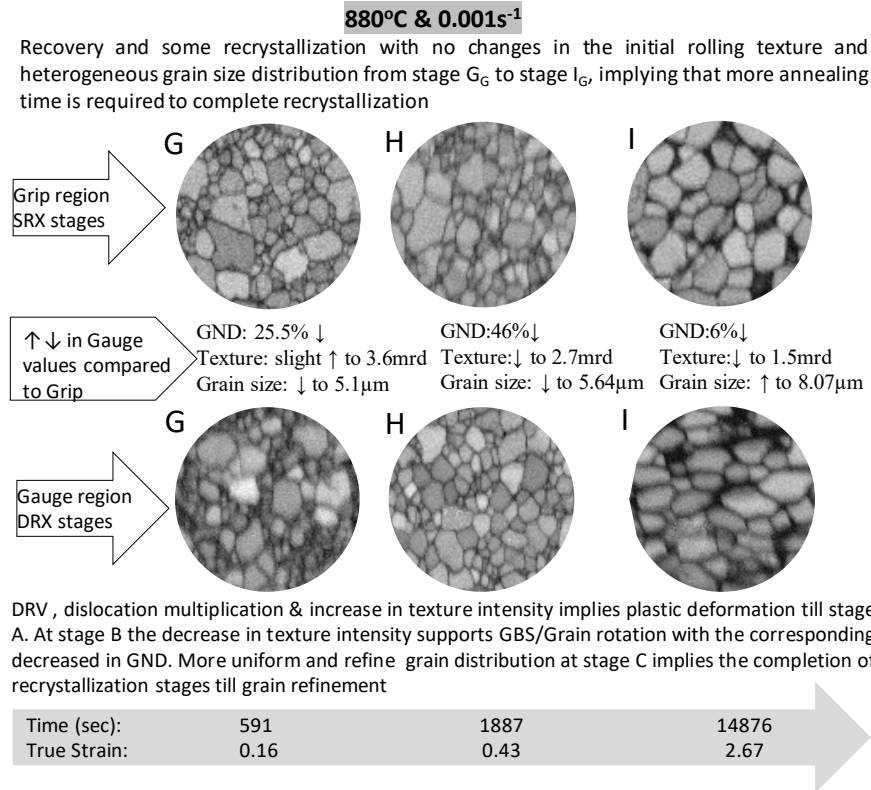


Figure 4.12b. Mechanism the microstructural evolution in static and dynamic annealing process during superplastic deformation of TA15 alloy at temperature 880°C and 920°C with strain rate of 0.001s⁻¹ and 0.0005s⁻¹, respectively.

4.5. Conclusion

The microstructure and GND density evolution of a near- α titanium alloy (TA15) was examined under conditions of static annealing and superplastic loading in the temperature range 880-920°C and at strain rates of $0.0005\text{s}^{-1} - 0.01\text{s}^{-1}$. The key conclusions can be drawn as:

- i. Overall, the grains retained almost an equiaxed shape in all gauge regions, which indicates a high contribution of GBS controlled by the grain boundary diffusion of the β -phase and low contribution of dislocation glide to the total deformation.
- ii. Superplastic loading accelerated the phase transformation process. An increase in the volume fraction of β -phase formation though in the case of static annealing, the increase in the volume fraction of β -phase was lower than in dynamic annealing. Similarly, in both cases, an increase in the volume fraction of β -phase was observed with increasing deformation temperature or decreasing strain rate.
- iii. Superplastic loading significantly reduced the initial rolling texture, which demonstrated the characteristic grain rotation process. Under all testing conditions, the texture observed in the grip regions was quite different from that found in the gauge regions. Nevertheless, the maximum intensity of the texture observed in the grip regions was slightly changed from that of as-received material, although the initial rolling texture was still maintained
- iv. GND density values in the superplastic loading regions seemed generally agree well with stress-strain behaviors for hardening and softening parts. GND and texture generally increase with lowering temperature and higher strain rates at the early stage of deformation. GND dropped after this, which indicates that the deformation mechanism changed from plastic to superplastic.
- v. The superplastic loading accelerated the dynamic recrystallization and resulted in faster grain growth compared to the static recrystallization occurred in the grip region. Grain size is considerably larger in gauge region compared to grip region. The superplastic loading led to difference in the evolution of the HAGBs fraction compared to the corresponding grip regions, indicating that the microstructure evolution mechanisms were different. The fraction of HAGBs in both grip and gauge regions increased under all testing conditions. The rate of dynamic

recrystallization increased at higher stress levels, as shown by a comparison of the results for $880^{\circ}\text{C} / 0.01\text{s}^{-1}$ and $880^{\circ}\text{C} / 0.001\text{s}^{-1}$.

- vi. Grain growth in superplastic deformation process resulted in less grain boundary sliding and thus hardening in stress strain behaviour.

Chapter 5

Establishing a mechanism-based constitutive model for simulating superplastic forming of the near- α titanium alloy

5.1. Introduction

The titanium plates commonly used on aerospace parts (*e.g.*, aircraft fuselages are mainly skins, with a sheet thickness of about 1-2 mm. However, the main issue restricting the broader adoption of these alloys for producing the complex geometry of thin-walled components is the difficulty of their formability at room temperature. Superplastic forming (SPF) offers a viable approach for forming titanium alloys thin-walled components and complex geometrical shapes. However, SPF is characterized by complexity in both the material flow behaviors and the hot deformation mechanisms. Amongst various SPF process for sheet metals (*e.g.* blow forming, thermoforming and vacuum forming), blow forming process is one of the most important and widely used SPF methods in recent years. During the blow forming process ([Figure 5.1](#)), the die and sheet material are normally maintained at the forming temperature and the gas (mostly argon) pressure (typically 690 to 3400 kPa) is imposed on one side of the sheet to make it flow into a die of the predetermined shape and geometry located on the other side of the sheet. The outer side of the sheet is held in a fixed position and does not draw-in as would be the case in typical deep-drawing processes. To reproduce the maximum ductility in the SPF process, it is desirable to adjust the forming pressure to keep the strain rate within an optimum range throughout the whole forming process. Friction between the die and the sheet is found to significantly affect the thinning of the component during the blow forming process (Chen et al, 2001). Severe thinning will lead to the failure of the component. Therefore, to successfully manufacture of SPF products, it is essential to have a good understanding of

the superplastic forming process. To predict the SPF of industrial parts, finite element (FE) tools are widely used for the process analysis and to optimize process parameters. However, FE simulation of an SPF process remains complex because of the highly non-linear aspects such as large deformation, history-dependent material behaviour, and long forming duration. The constitutive model is established as an input to the finite element program and gives an accurate prediction of material deformation behaviour under certain processing conditions. It is, however, important to note that FE simulation, based on the material's constitutive model description of structural behaviour, is, ultimately, an approximate solution. Therefore, the FE solution can never be more accurate than the mathematical models used. In other words, when considering the SPF process, the accuracy of the simulation code used to forecast the deformation mechanism of the sheet material is the major factor in optimizing the gas pressure cycles necessary to produce a workpiece with uniform thickness, and in the shortest possible time. The penalty of applying an inaccurate pressure/time cycle can range from sub-optimum forming to excessive thinning or rupture of the sheet.

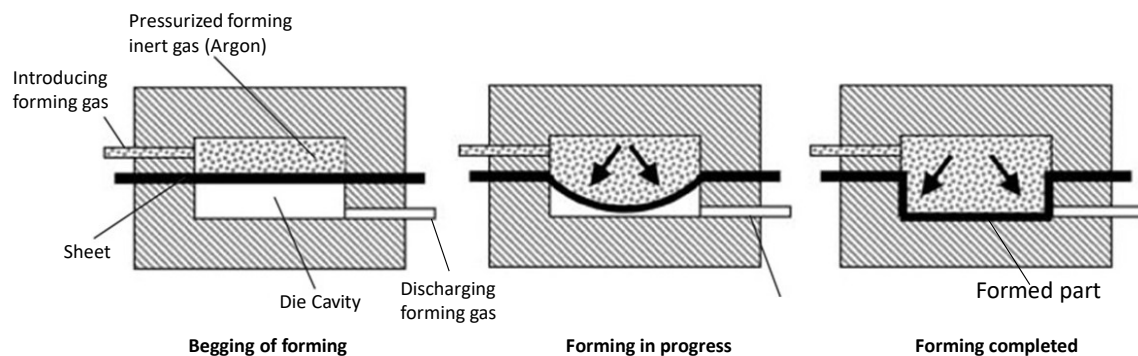


Figure 5. 1. Illustration of the blow-forming technique for superplastic forming. (Modified from: Verlinden, et al, 2007)

It is essential, therefore, to establish a valid constitutive model to link and analyse various process parameters of the material in such a manner that the flow stress and deformation can be accurately predicted. There are a number of studies available in the literature (Zhou et al, 2015; Sathish et al, 2017; Giuliano, 2019; Aleyari et al, 2020) devoted to the study of the industrial production of parts by SPF processes using FEA. However, most of these

FEA models are based on traditional constitutive models such as Power-law, Johnson-Cook and Arrhenius type model (Alabort et al, 2015; Fan et al, 2018), as available in the built-in materials libraries of commonly employed commercial software packages (ABAQUS, ANSYS, MARC, and DEFORM-3D), and which do not reliably simulate the forming behaviour for complex parts. The literature review (tabulated in Table 5.1) shows that only a limited number of studies are available considering the constitutive modelling of superplastic deformation based on the hot tensile test behaviour of titanium alloys. As illustrated in Table 5.1, most constitutive modelling work can be categorized into three types *i.e.* phenomenological (based Johnson-Cook and Arrhenius type model), physical (based on internal state variables), and advanced statistical models (based on artificial neural networks). The literature review presented in Table 1 shows that among these three types of models, the phenomenological model is currently the most widely used one due to the few parameters required and the simplicity in determining the parameters by regression analysis. However, the model presents poor predictability in determining deformation parameters outside the experimental range. The advanced statistical model is the most accurate in predicting the flow behaviour for a wide range of deformation conditions. However, the model does not have an intrinsic physical meaning, and cannot reflect the effects of microstructure evolution during the deformation.

In contrast, however, over the past ten years, the physical constitutive model, based on internal state variables, has become more important in the modelling of hot deformation and microstructure evolution. This ISV (internal state variable) based physical model is capable of capturing inherent microstructural complexity during the working process and achieving a reasonable accuracy, and so can be used in the computational simulation and process optimization of superplastic blow forming. Therefore, the main aim of this work is to construct a simulation capability for the superplastic forming process of TA15 sheet through developing mechanism-based constitutive equations derived from a series of physical parameters and phenomena such as phase volume fraction, grain size, dislocation density, recrystallization fraction and damage.

Table 5.1. SPF constitutive work published in the last twenty years (2001-2020)

Study	Constitutive Model	Alloy	Alloy Class	β transus temp °C	Initial microstructure	Grain size - μ m	Temp. °C	St. rate s ⁻¹
Li et al. (2019a)	Physical-based	Ti-5.4Al-3.7Sn-3.3Zr-0.5Mo-0.4Si	near α		lamellar	1, 6 & 10	930 – 1020	10 – 0.01
Wanget et al. (2019)	Phenomenological (Johnson-Cook)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed		600 - 750	0.1 – 0.005
Mosleh et al. (2019)	Phenomenological (Johnson-Cook)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed	3	800 - 900	0.003 - 0.0003
Maa et al. (2019)	Phenomenological (Arrhenius type)	Ti-15Mo-3Al-2.7Nb-0.2Si	β		Mainly beta phase		750 - 950	0.01 - 0.00005
Jiang et al. (2018)	Phenomenological (Arrhenius type)	Ti-5Al-5Mo-5V-1Cr-1Fe	near β	875			890 - 980	0.05 - 0.001
Gao et al. (2017)	Phenomenological (Johnson-Cook)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed	6.4	890 - 950	0.01 - 0.0005
dos Santos et al. (2017)	Physical-based (ISV)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed	4.9, 3 & 0.5	650 - 950	0.01 – 0.0001
Mosleh et al. (2017)	Artificial Neural Network (ANN)	Ti-2.5Al-1.8Mn	near α	906			840 - 890	0.0002 – 0.0008
Velayet et al. (2016)	Physical-based (ISV)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed	3	650 - 870	0.01 - 0.0004
Yang et al. (2016)	Physical-based (ISV)	Ti-5Al-2Sn-2Zr-4Mo-4Cr	$\alpha + \beta$	990	equiaxed		780- 820	0.1 – 0.001
Alabort et al. (2016)	Physical-based (ISV)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed	10	850 - 900	0.001 – 0.0001
Yang et al. (2015)	Physical-based (ISV)	Ti-6Al-2Zr-1Mo-1V	near α	990	biomodal		750- 850	0.1 – 0.001
Xiao et al. (2012)	Phenomenological (Arrhenius type)	Ti-6Al-4V	$\alpha + \beta$	995	equiaxed		650 - 750	0.05 - 0.0005
Yu et al. (2012)	ANN	Ti-6Al-2Zr-1Mo-1V	near α	990			800 - 1050	10 – 0.01
Kim et al. (2003)	Physical-based	Ti-6Al-4V	$\alpha + \beta$	995	lamellar	1, 4 & 8	715 - 900	0.1 – 0.0001

5.2. Experimental procedure

The experimental work comprised high-temperature tensile testing of dogbone style specimens (see [Figure 5.2](#)) and analysis of microstructure using SEM and EBSD techniques. The current study uses a rolled TA15 alloy sheet with a thickness of 1 mm. This thickness corresponds to that used in practical industrial applications of SPF. This sheet is produced by large deformation forging and subsequent cladding pack rolling. The colony microstructure is changed into equiaxed microstructure via necessary primary hot working (Fan et al, 2018). The β -transus temperature of the alloy is 985 °C. It is generally agreed that in superplasticity, the forming temperature must be in excess of about $0.5T_m$, where T_m is the absolute melting point (Courtney, 2005). The temperature at which

superplasticity is found in titanium alloys is usually between about $0.47 T_m$ (about $800\text{ }^{\circ}\text{C}$) to $0.61 T_m$ (about $1030\text{ }^{\circ}\text{C}$) (Nieh et al, 2005). As the β -transus temperature of TA15 alloy is $985\text{ }^{\circ}\text{C}$, so superplastic deformation of TA15 was investigated in the temperature range $880 - 940\text{ }^{\circ}\text{C}$. The specimens for hot tensile tests were cut using wire-electrode along with two directions i.e. the rolling and transverse direction, to check their anisotropy response. The 800# Silicon Carbide grinding paper was used to polish the specimens to minimize the influence of rough cutting. The isothermal tensile tests were carried out in the temperature range of $880 - 940\text{ }^{\circ}\text{C}$ and strain rate range of $0.0005 - 0.01\text{s}^{-1}$ using a computer-controlled universal testing machine. Prior to the tests, the tensile samples were coated with the layer of antioxidants for the prevention of oxidation at a high temperature. During these tests each specimen was held for 15 minutes at a corresponding temperature for uniform temperature distribution and then deformed under constant strain rate. The specimen was water quenched immediately after deformation.

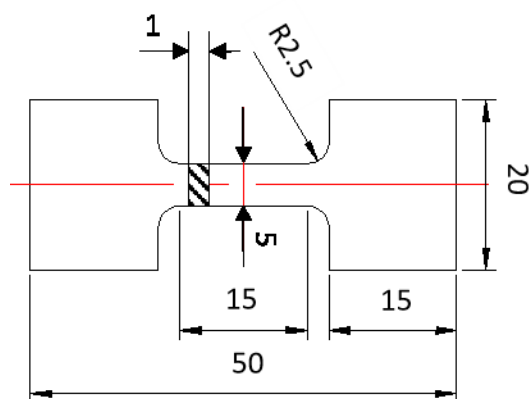


Figure 5. 2. Geometry of the tensile specimen (unit: mm)

5.3. Results and Discussion

5.3.1. Initial microstructure of as-received material

Figure 5.3a demonstrates the initial microstructure, which contains a small amount of ductile β phase grain dispersed in the intergranular region of a near α -phase structure. The

equiaxed microstructure is one of the requirements for the SPF applications due to its ability to enable the alloy to acquire a good proportion of strength and ductility. A histogram (Figure 5.3b) of the grain size of the as-received material (average grain size 3.74 μm) revealed a typical distribution in terms of shape and the ratio of the maximum to mean grain size (i.e., ~ 2.7), often found for single-phase alloys which have undergone discontinuous static (or dynamic) recrystallization (Zherebtsov et al, 2016). The grain orientation map (Figure 5.3c) of the initial microstructures shows fine equiaxed grains but with preferred orientation contrast, which can be attributed to the pre-rolling and subsequent insufficient annealing treatment.

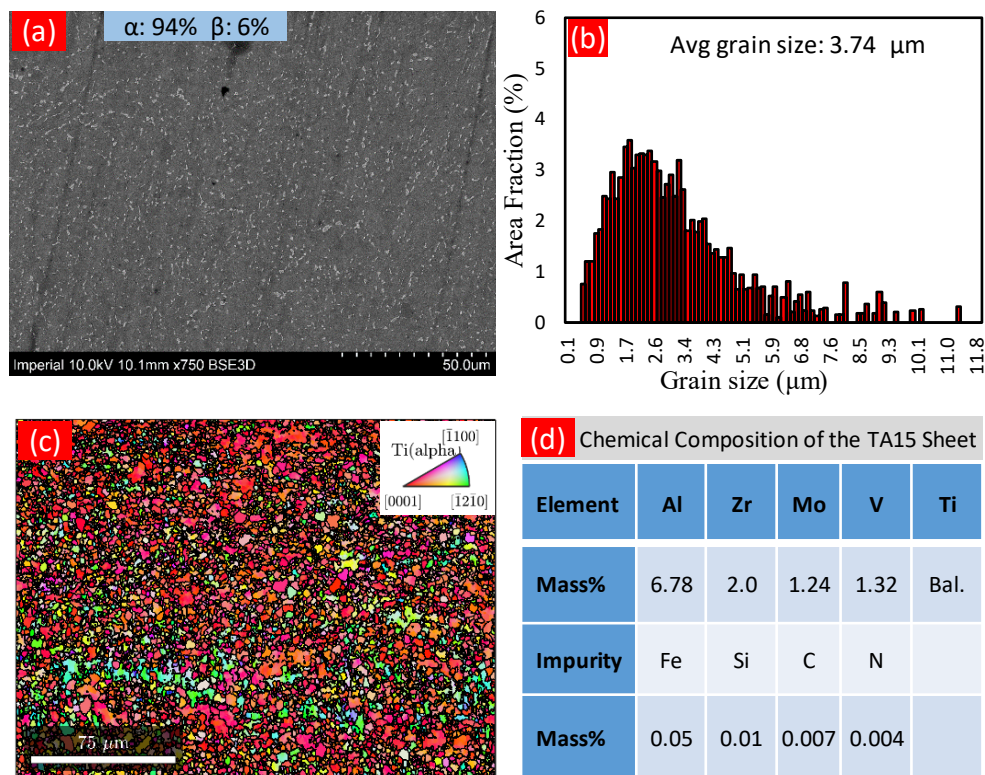


Figure 5. 3. Initial microstructure of the TA15 titanium alloy (a) SEM micrograph (b) grain size distribution (c) grain orientation map (d) chemical composition

5.3.2. Determination of the superplastic domain

Figure 5.4a shows the full range of tested samples after the superplastic deformation, while Figure 5.4b summarizes the mechanical characteristics of TA15 as a function of temperature, strain rate, and total elongation. The resulted stress-strain curves for the strain rates in the range of $0.0005\text{--}0.01\text{s}^{-1}$ and temperature $880\text{--}940\text{ }^{\circ}\text{C}$ are presented in Figure 5.5.

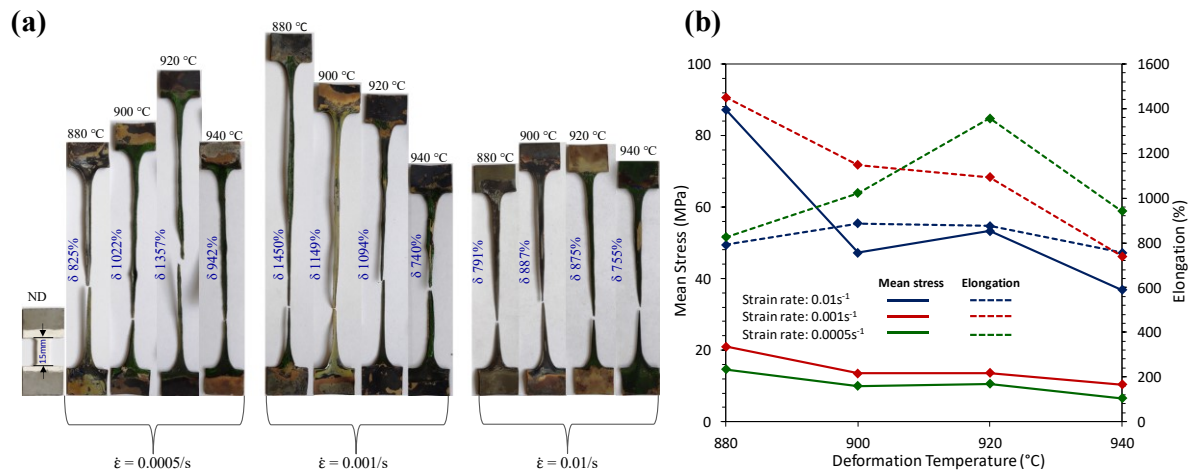


Figure 5. 4. (a) Appearance of TA15 SP specimens after the tensile tests at different temperatures and strain rates. Each testpiece shows elongation-to-failure in terms of engineering strain. (b) Elongation and mean flow stress of TA15 tested at different temperatures and strain rates.

In general, this Titanium alloy exhibits excellent superplastic behaviour over the range of temperatures and strain rates, with the elongations ranging between 740 and 1450%. One can immediately observe that the elongation achieved is highly dependent on processing conditions, *i.e.* strain rate and temperature. However, the increase/decrease in the elongation follows an uneven trend for all testing conditions. For the lowest strain rate (0.0005s^{-1}), up to $920\text{ }^{\circ}\text{C}$ the elongation first increases but then shows a significant decrease at $940\text{ }^{\circ}\text{C}$. For the intermediate strain rate (0.001s^{-1}), an interesting phenomenon can be overserved where elongation decreases with increasing temperature. On the other hand, for the highest strain rate (0.01s^{-1}), elongation remains practically constant with increasing temperature. The decreasing trend in elongation at the highest temperature ($940\text{ }^{\circ}\text{C}$) can

likely be attributed to oxidation and the formation of an alpha case. The uneven changes in the total percentage elongation imply that strain rate and temperature have interdependent impacts on the overall formability of the alloy. The maximum tensile elongation of 1450% is achieved at testing conditions 880 °C/0.001s⁻¹, while the lowest elongation of 740% is recorded for 940 °C/0.001s⁻¹. This provides an important basis for the guidance of subsequent SPF simulations and impacts on the actual process parameters in industrial applications.

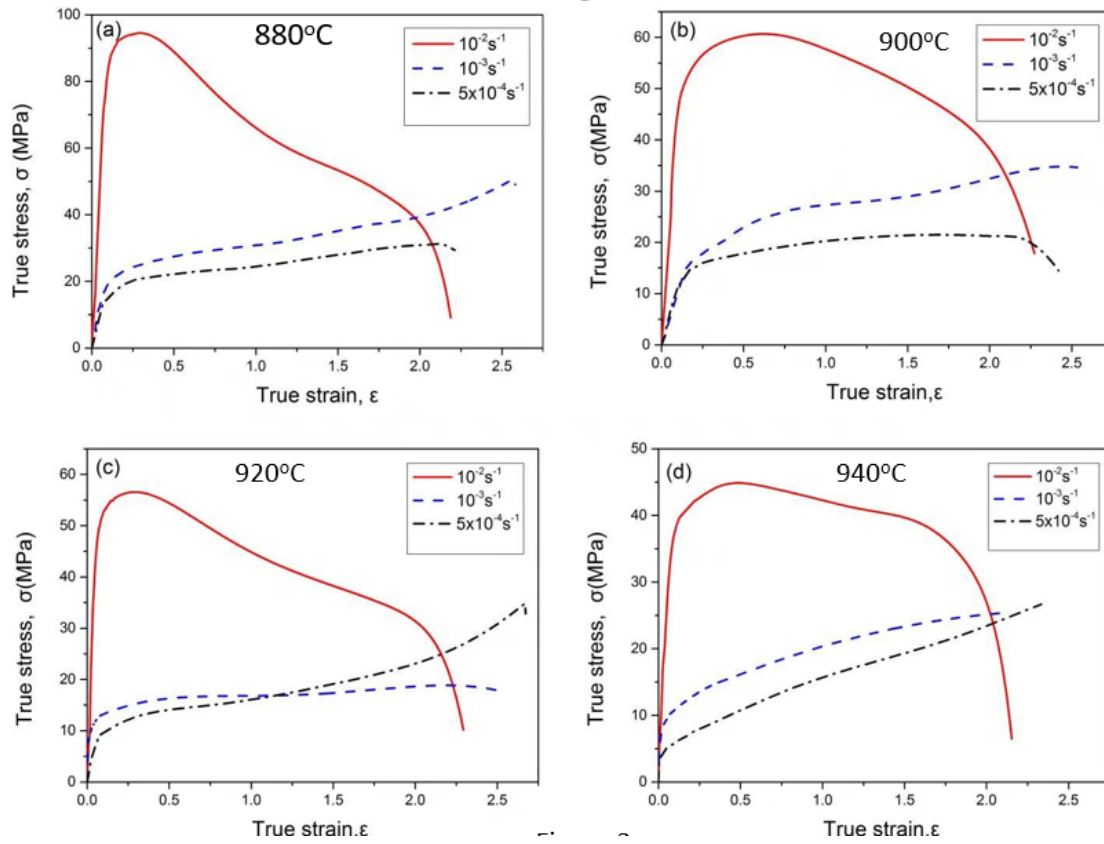


Figure 5.5. Flow stress curves of TA15 alloy at deformation temperature of (a) 880 °C, (b) 900 °C, (c) 920 °C and (d) 940 °C

5.3.3. Microstructure after superplastic deformation

Figure 5.6 and 5.7 show the BSE-SEM micrographs and grain orientation maps of the deformed specimens taken near to the necking regions, respectively, and shown for the various strain rates and temperatures. From Figure 5.6, it can be observed that with increasing deformation temperature and/or decreasing strain rate, the fraction of α -phase

decreased significantly, and conversely, new β -phase emerged, and β -grains grew together to form larger grains. A possible explanation for this is that the α -phase transforms into β -phase during superplastic deformation at temperatures close to the transformation point. It can be observed in the SEM micrographs for the lower strain rates, [Figure 5.6 \(b, c, e, f, h, I, k & l\)](#) that, instead of damage and cavity nucleation, cracks are seen, which suggests that the deformation at such lower strain rates provides enough time for the accommodation mechanism operating to be either as dislocation or diffusion. At the same time, grain growth over the longer testing time causes high internal stresses, which can result in a fracture, as observed in the SEM micrographs. From [Figure 5.7](#), it can be observed that the small α grains begin to grow together and form larger grains with a decrease in strain rate and/or an increase in deformation temperature. The refining and equiax effect of dynamic recrystallization creates favourable conditions for the superplasticity of titanium alloys (Zhang et al, 2013). At the testing condition $940^{\circ}\text{C}/0.0005\text{s}^{-1}$, the grain growth is much more pronounced, and the α -phase is significantly reduced, as indicated in [Figure 5.7l](#). With the strain rate decreasing to 0.001s^{-1} , the average grain size increases to $8.2\text{ }\mu\text{m}$ ([Figure 5.7i](#)). When the tensile specimen reaches fracture at 0.0005s^{-1} , the grains have grown significantly, reaching $18\text{ }\mu\text{m}$ ([Figure 5.7l](#)), almost 4.5 times greater than the grain size of the as-received material.

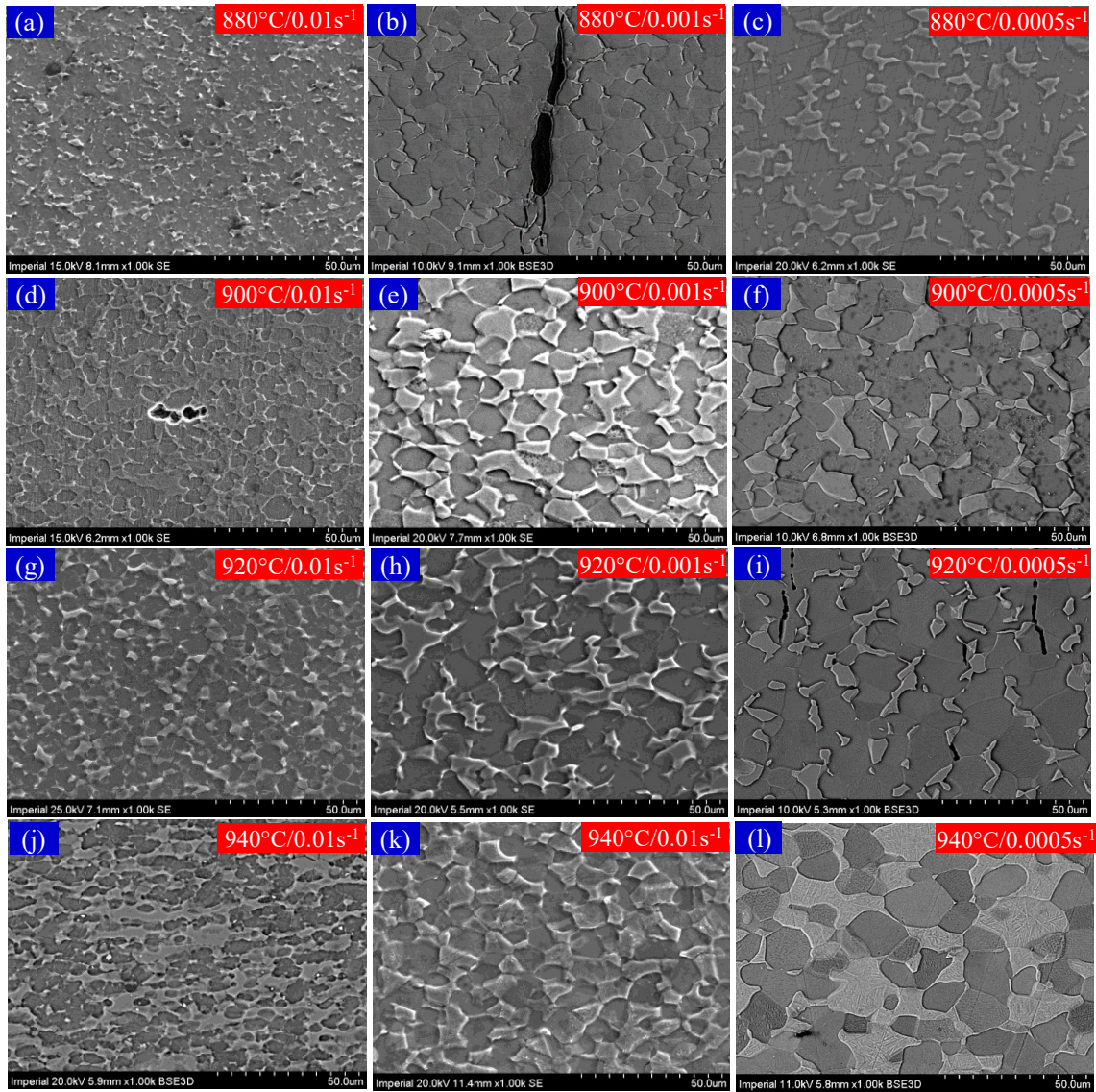


Figure 5. 6. BSE-SEM micrograph of TA15 at different deformation condition



Figure 5. 7. Grain orientation maps of TA15 at final fracture points under given deformation condition

From the GND distribution (Figure 5.8), it can be seen that for the higher strain rate of 0.01s^{-1} , the quantity and distribution of GND almost remain same for the entire temperature range. The possible reason for this phenomenon can be explained as follows. At high strain rates, such as 0.01s^{-1} , the rate of dislocation recovery and grain growth remains low. However, a significant effect on the GND distribution can be observed (Figure 5.8) with decrease in the strain rate at a certain temperature. A low strain rate at high temperature provides sufficient time for the diffusion based accommodation mechanisms to be operative and , consequently, leads to a significant reduction in GND. It is worth noting that the lowest GND values can be observed under deformation condition $900^{\circ}\text{C}/0.0005\text{s}^{-1}$ and $900^{\circ}\text{C}/0.001\text{s}^{-1}$. This can be attributed to the optimal α/β ratio of 80:20 for grain boundary sliding (GBS) to be the dominant mechanisms for superplastic deformation (Seshacharyulu et. al, 2000), as under optimum α/β ratio, the β -phase accommodate the deformation and causes a significant drop in GND in α -grains (Alabort et al, 2016). Therefore, the observed α/β ratio 75:25 and 65:35 at deformation conditions $900^{\circ}\text{C}/0.0005\text{s}^{-1}$ and $900^{\circ}\text{C}/0.001\text{s}^{-1}$, respectively suggest that the optimal GBS was achieved which resulted the highest reduction in GND.

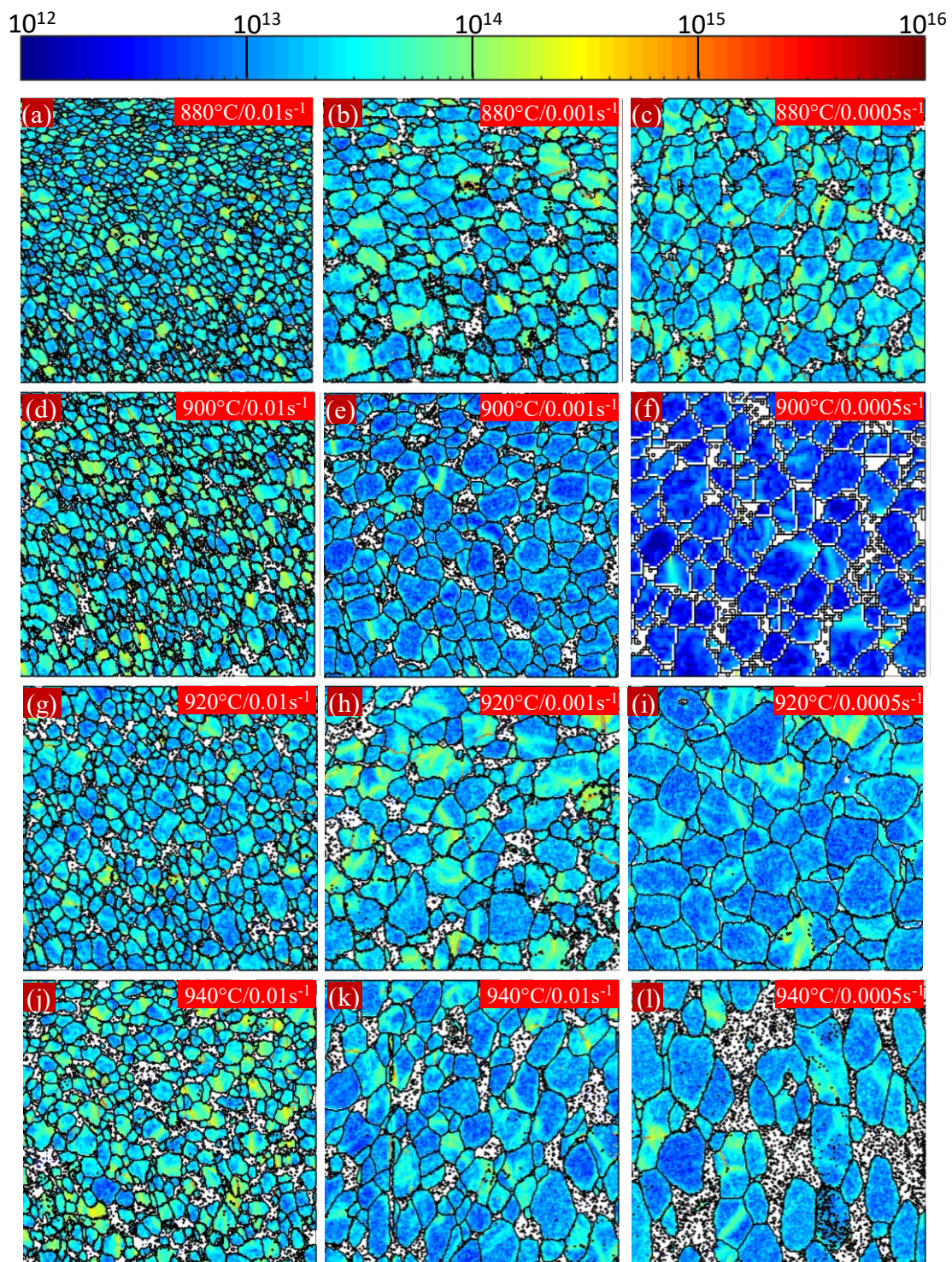


Figure 5. 8. GND maps of TA15 at final fracture points under given deformation condition

5.3.4. Flow stress behaviour

From the stress-strain curves presented in Figure 5.5, it can be seen that the morphology of stress-strain curves is strongly dependent on the strain rate and deformation temperature. The physics of the resulting flow stress curves for all testing conditions can be categorized into three distinctive types, as illustrated in Figure 5.9., each type being made up of distinct phases.

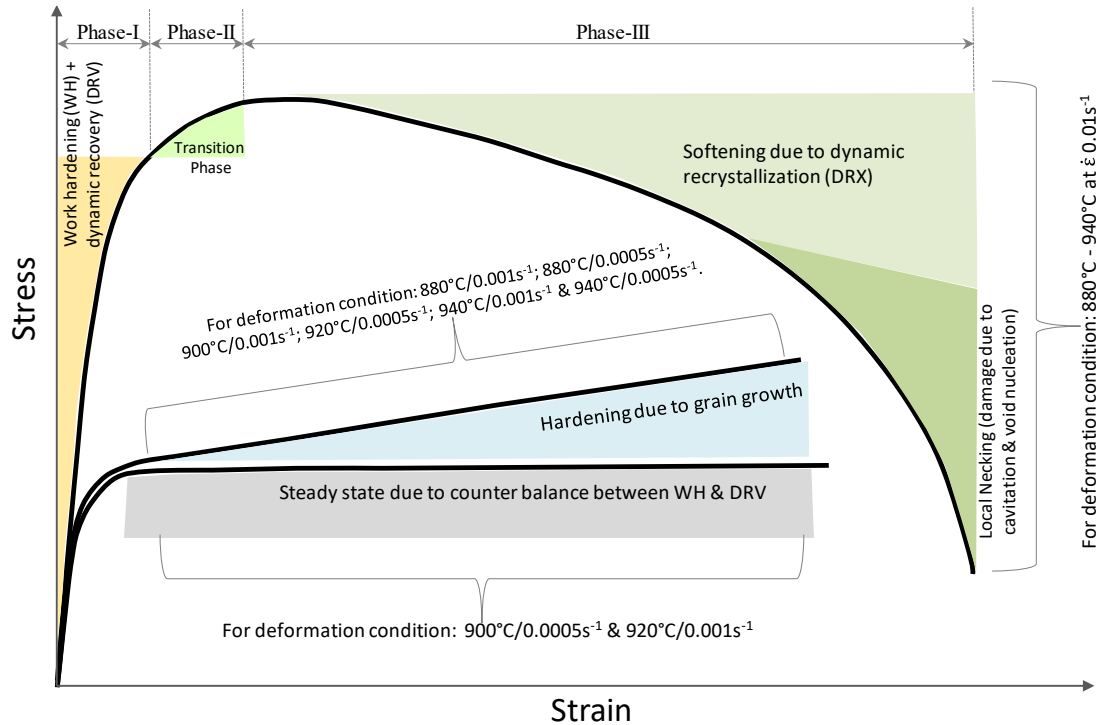


Figure 5. 9. Schematic representation of flow curves during superplastic deformation of TA15 alloy

The morphology of the curves at the higher strain rate of 0.01s⁻¹ for all testing temperatures can be explained with the help of three distinctive phases. In phase-I, it can be seen that the flow stress rises rapidly with the strain, which is attributed to the significant strain hardening (Khomei and Dehghani, 2010) due to a rapid increase in dislocation density (Chu et al, 2018; Li et al, 2018). Because of the high strain rates, the dislocations do not have sufficient time to slide or climb (Zhang et al, 2019). Meanwhile, as the deformation occurs at high temperatures ($>0.5T_m$), dynamic recovery (DRV) (dislocation annihilation) is also

expected (Lin et al, 2012). In Phase-I, these two competing mechanisms are activated at the same time, but the hardening mechanism predominates (Ghosh, 1977). In Phase-II, there is a slow increase in flow stress until the peak value is reached. This indicates that the underlying deformation hardening or softening mechanism starts to alter. The dislocation density (the stored plasticity energy) may reach a critical value, such that dynamic recrystallization (DRX) occurs. At the beginning of dynamic recrystallization, the dislocation density will be reduced by the formation of new grains or by grain boundary migration. This reduction in dislocation density is one of the main reasons for the considerable softening observed in phase-III. However, for the highest strain rate (0.01s^{-1}), the curve morphology at all testing temperatures can be divided into four distinct phases, including: (a) elastic phase, (b) transition phase (c) flow softening phase, and (d) localized necking phase. At this strain rate, in addition to the contribution of dislocation density reduction, the flow softening observed in Phase III region can be attributed to the following three causes. First, the smaller grain size facilitates the GBS/grain rotation and needs lower stresses values to be operative. Second, the formation of voids and cavities, as observed in SEM micrographs [Figure 5.6 \(a & b\)](#), cause macroscopic softening (Alabort et al, 2016). Third, the phase transformation from the hard α -phase to the soft β -phase, as is shown in [Figure 5.6 \(b, c & d\)](#) for the deformation conditions of 900°C , 920°C and 940°C with strain rate of 0.01s^{-1} . A further drop in the flow stress at the higher strains might be associated with continuous grain refinement and the formation of cavitation (Alabort et al, 2015; Tahreen et al, 2015).

Although at all deformation conditions, the flow stress is not characterized by a fully steady flow, the curve morphology shows nearly steady flow characteristics for the testing conditions of $900^{\circ}\text{C}/0.0005\text{s}^{-1}$ and $920^{\circ}\text{C}/0.001\text{s}^{-1}$, and can be divided into two distinct phases. In Phase I, similar to the high strain rate (0.01s^{-1}), the flow stress increases sharply with increasing strain until the peak stress is achieved. In Phase II, the rate of strain hardening gradually decreases and approaches zero, resulting in a near steady-state flow stress (Sakai et al, 2014). Such behaviour is attributed to the operation of dynamic recovery (DRV) as the main softening process (Rollett et al, 2017). As at these lower strain rates 0.0005s^{-1} and 0.001s^{-1} the material is exposed to high temperature for longer times as

compared to 0.01s^{-1} , this allows DRV to reduce the dislocation density (Gong et al, 2017), and this leads to a significant reduction in peak stress (Zhou et al, 2014). As Phase II progresses, the flow stress becomes steady with the increasing strain until the fracture occurs. The steady-state flow stress indicates that a dynamic balance between the hardening and softening process is achieved, such as between grain refinement and grain growth or between dislocation multiplication and annihilation (Zhou et al, 2014).

The curve's morphology for the testing conditions $880^{\circ}\text{C}/0.001\text{s}^{-1}$, $880^{\circ}\text{C}/0.0005\text{s}^{-1}$, $900^{\circ}\text{C}/0.001\text{s}^{-1}$, $920^{\circ}\text{C}/0.0005\text{s}^{-1}$, $940^{\circ}\text{C}/0.001\text{s}^{-1}$ and $940^{\circ}\text{C}/0.0005\text{s}^{-1}$, can be also split again into two distinct phases. In Phase I, initially, the flow stress increases rapidly with increasing strain, but then this rate decreases to a steady lower value, indicating the start of Phase II. In Phase II, there is only work hardening. Such behaviour is believed to be caused by high grain growth due to the long exposure time at high temperature. In these testing conditions, the rate of grain growth is fast enough to overcome the rate of dynamic recrystallization. Although the exposure to high deformation temperature for a long duration (*i.e.* low strain rate) can result in relatively full dynamic recrystallization (Figure 5.5i & 5.5l), it also stimulates the rapid growth of the recrystallized grains. Under such low strain rates, the effect of grain growth on the viscoplastic behaviour of material becomes more predominant with increasing temperature (Huang et al, 2014). The grain growth leads to the relative reduction in grain boundary length, and thus the increasing difficulty in grain rotation or grain boundary sliding. All such mechanisms result in hardening.

Overall it can be observed that flow stress decreases significantly with decreasing strain rate and increasing deformation temperature. The reduction in flow stress is due to dynamic softening, including dynamic recovery (DRV) and dynamic recrystallization (DRX), both of which are more active at high temperatures and low strain rates (Sakai et al, 2014; Zhang et al, 2019). However, the peak flow stress increases as the strain rate increases at a certain temperature. This is due to there being insufficient time for DRV or DRX to act at a high strain rate, and this results in the presence of higher dislocation densities, and hence a higher strain hardening effect (Xia et al, 2015). At the highest strain rate, 0.01s^{-1} , the flow curve exhibited a peak flow stress during the initial stages of deformation followed by pronounced flow softening. It is reported that during hot deformation, the decrease in

strain rate to the optimal value (where maximum elongation can be achieved) changes the viscoelastic behaviour into a marked strain-hardening response, which may attribute to the dynamic coarsening (Semiatin et al, 2010), and in general such behaviors are typical of superplastic flow (Zherebtsov et al, 2016).

The flow curve results based on tensile deformation tests of various titanium alloys under compatible testing conditions are presented in [Figure 5.10](#). It can be observed that peak flow stress of the TA15 alloy tested in this study is either comparable to, or below, the peak stress level of other titanium alloys. There are, however, significant differences in the degrees of elongation achieved. In all cases, the elongation achieved in this study is considerably higher than that of other titanium alloys tested in similar conditions.

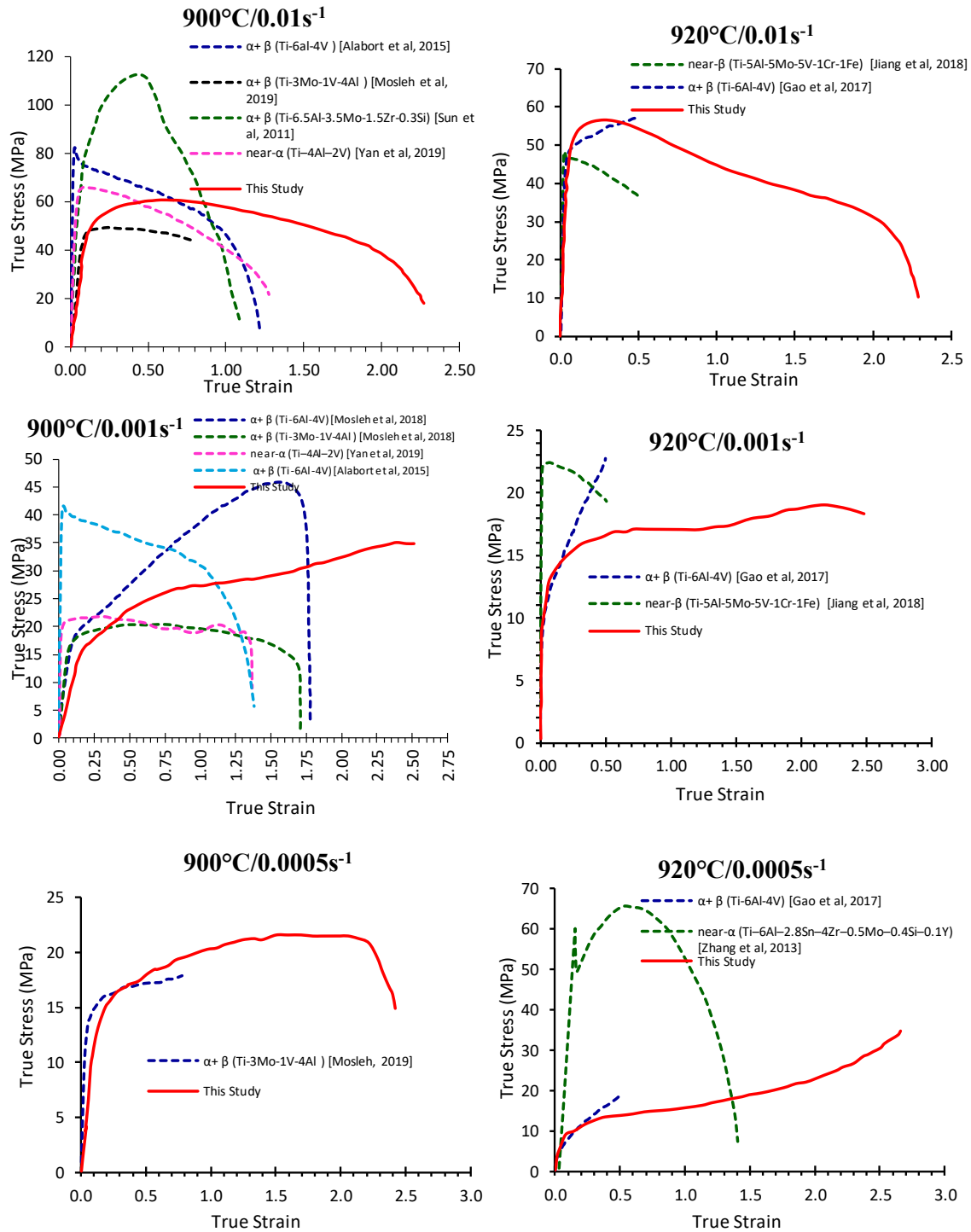


Figure 5. 10. Comparative illustration of thermo-mechanical tensile test flow curves of TA15 alloy in this study to the flow curves of other titanium alloys at compatible testing condition

5.4. Constitutive modelling using state variable

To evaluate the advantages of using a material-based model, and to offer a point of comparison, in this chapter the model is developed in two independent phases. In the first phase, original thermo-mechanical tensile test data of the same alloy class *i.e.* TA15 was provided by an industrial collaborator. A mechanism-based constitutive model (hereinafter referred to as '*Initial Model*') was developed, based on the said data without taking into consideration information about the microstructure and its deformation path. The *Initial Model* was implemented in an FEA code to generate a gas pressure profile for an industrial die, and the results were compared to actual SPF parts produced using a gas profile generated on the bases of power-law' material expression. The *Initial Model* was able to predict the thickness distribution in these experimentally formed parts with higher accuracy than the power-law stress-strain model. However, the *Initial Model* was unable to predict the flow stress behaviour at lower strain rates and higher deformation temperatures. The results of the *Initial Model* were presented in a journal paper (Tabassam et al, 2019), available at Annexure-A, and the said *Initial Model* is reproduced in Eq. 5.1 below:

$$\left\{ \begin{array}{l} \dot{\epsilon}_p = \left(\frac{\sigma - H - k}{K} \right)^n \cdot \frac{1}{(1 - S)} \\ H = B \bar{\rho}^\gamma \\ \dot{\bar{\rho}} = A_1 (1 - \bar{\rho}) |\dot{\epsilon}_p| - A_2 \bar{\rho}^{c_1} \\ \dot{S} = \eta \dot{\epsilon}_p^{\eta_1} (\bar{\rho} - \bar{\rho}_c)^{\eta_2} (\bar{\rho} - S) \\ \sigma = E (\epsilon_T - \epsilon_p) \end{array} \right. \quad (5.1)$$

In the second phase, a sheet of TA15 was prepared with a similar initial microstructure (*i.e.* equiaxed) to that of former testing TA15 test specimens. The thermo-mechanical tensile tests were performed with testing parameters/scope compatible with the thermo-mechanical tensile tests of the first phase. In the second phase, the *Initial Model* was modified to incorporating microstructure evolution parameters such as grain size, dynamic recrystallization, and damage. This model is hereinafter referred to as the '*Revised Model*'. The formulation of the *Revised Model* is explained below.

5.4.1. Modelling grain growth

In superplastic deformation of titanium alloys, grain growth by the applied plastic strain will occur subject to the availability of sufficient time and stored energy. The grain growth can be broadly classified into two categories: static grain growth and dynamic grain growth. The static and dynamic grain growth work independently and play a significant role in viscoplastic deformation at low strain rates and high temperatures, such as in superplastic deformation. On the other hand, during the recrystallization, new grains nucleate, and the total number of grains increases. Consequently, the average grain diameter' decreases. Considering these phenomena, a kinematic grain size model to account for the effect of static and dynamic grain growth can be expressed as:

$$\dot{d} = \dot{d}_{static} + \dot{d}_{dynamic} - \dot{d}_{DRX} \quad (5.2)$$

Considering the interfacial energy associated with grain boundaries Shewmon (1969) proposed a constitutive relation for the rate of change of grain size during static grain growth which takes the form

$$\dot{d}_{static} = \frac{G}{d} \quad (5.3)$$

where d is the average grain size (μm), and G is a material constant. The Eq. 5.3 was found to be useful only over limited ranges of temperature and strain rates.

It is reported (Shewmon, 1969; Sandström and Lagneborg, 1975) that static grain growth is an atomic diffusion process, affected by temperature and relates to grain boundary mobility (M) and grain boundary energy density effects. Therefore, Eq. 5.3 has been modified to the following form (Callister, 1991):

$$\dot{d}_{static} = \frac{\alpha_1}{d^{\gamma_1}} \quad (5.4)$$

Where α_1 and γ_1 are temperature-dependent material constants.

The dynamic grain growth in the second term of Eq. 5.2 can be expressed as (Zhou and Dunne, 1996; Dunne, 1998)

$$\dot{d}_{dynamic} = \frac{\alpha_2 \dot{\epsilon}_p}{d^{\gamma_2}} \quad (5.5)$$

Where γ_1 is a material constant. The parameter α_2 is a strain rate dependent material constant, reflecting the dependence of grain growth on diffusion processes (Zhou and Dunne, 1996).

To consider the grain refinement due to dynamic recrystallization, the grain size evolution equation can be expressed as (Lin, 2015):

$$\dot{d}_{DRX} = -\alpha_3 \dot{S}^{\gamma_4} d^{\gamma_3} \quad (5.6)$$

where α_3 , γ_3 and γ_4 are material constants while $\dot{S}$ is the volume fraction of recrystallization.

Combining Eq. 5.4, 5.5 and 5.6, the evolution of average grain size growth is defined as

$$\dot{d} = \frac{\alpha_1}{d^{\gamma_1}} + \frac{\alpha_2 \dot{\epsilon}_p}{d^{\gamma_2}} - \alpha_3 \dot{S}^{\gamma_4} d^{\gamma_3} \quad (5.7)$$

Referring to the morphology of the flow stress curves in the present study (Figure 5.5), strong work-hardening was observed for flow curves associated with the lower strain rates of $0.001s^{-1}$ and $0.0005s^{-1}$. This shows that the grain size evolution due to dynamic recrystallization is less critical in these conditions. Therefore, for the said deformation conditions, the third term of the Eq. 5.7 can be ignored.

5.4.2. Modelling cavity and void damage rate

Grain boundary sliding (GBS) is widely believed to be the dominant mechanism of the superplasticity in fine-grained materials. Such relative displacement of grains is accommodated by the distribution of matter within the mantle adjacent to grain boundaries (Zhang and Cai, 2010). If, when, due to the deformation rate, the accommodating

mechanism is not enough to power the sliding of the entire fleet of grain boundaries within the material, then stress concentrations are created at the triple junction of the grain boundaries which can nucleate cavities if the stress concentrations are not relieved sufficiently rapidly (Zherebtsov et al, 2016). During the hot deformation, the material behaviour at the later stages of deformation is dictated by damage accumulation in addition to the viscoplasticity (El Fakir et al, 2014). For the case of isotropic damage mechanics, the void fraction parameter ω is scalar and can be defined as:

$$\omega = \frac{A_v}{A} \quad (5.8)$$

where A_v is the area of voids A is the total area of interest.

In most cases, the evolution of damage (cavitation/voids) can be additively decomposed into nucleation and growth of voids (Perzyna, 1984):

$$\dot{\omega} = \dot{\omega}_{growth} + \dot{\omega}_{nucleation} \quad (5.9)$$

Based on the general expression for damage evolution proposed by Perzyna(1984), Khaleel et al. (2001) proposed a damage law for superplastic deformation based on void nucleation and growth around particles, either at grain boundaries or within grains and the improved damage evolution equation is given as:

$$\dot{\omega}_{growth} = \eta(1 - \omega)\dot{\epsilon}_p \quad (5.10)$$

Where the parameter η is considered as a constant material constant function to power of the strain rate ($\propto \dot{\epsilon}_p^{0.6}$). The rate of cavitation due to nucleation can be defined as

$$\dot{\omega}_{nucleation} = \frac{F \dot{\epsilon}_p^G}{(1 - \omega)} \sigma(\dot{\epsilon}_p)^{z2} \quad (5.11)$$

where the parameters F and G must be determined using experimental cavitation measurements. The parameter F is assumed proportional to a power of the strain rate ($\propto \dot{\epsilon}_p^{0.4}$).

Combining Eq. 5.10 and 5.11, the rate of change in the void fraction can be expressed as:

$$\dot{\omega} = \eta(1 - \omega)\dot{\varepsilon}_p + \frac{F\varepsilon^G}{(1 - \omega)}\sigma\dot{\varepsilon}_p \quad (5.12)$$

In the present work the Eq. 5.12 is modified to the following form

$$\dot{\omega} = \eta(1 - \omega)(\dot{\varepsilon}_p)^{z_1} + \frac{F\varepsilon^G}{(1 - \omega)}\sigma(\dot{\varepsilon}_p)^{z_2} \quad (5.13)$$

Where z_1 and z_2 are material constants.

5.4.3. Modelling Softening (recrystallization)

One of the important phenomena in high-temperature deformation is flow softening. This influences the macroscopic deformation behaviour of the material. Moreover, flow softening is an important reason for flow instability, which may result in superplastic forming defects. As shown in [Figure 5.5](#), at a strain rate of 0.01s^{-1} continuous flow softening was observed after the peak flow stress was reached. Many studies (Wu et al, 2011, Guo-Zheng, 2013; Roy and Suwas, 2013) indicate that the softening phenomenon in the hot deformation is closely related to dynamic recrystallization (DRX). The onset of the nucleation of DRX is usually associated with the accumulation of dislocations. During the hot deformation process, dislocations continuously multiply and accumulate with increasing strain during the work hardening stage. It is generally accepted that when the dislocation density increases to a degree where the accumulative stored energy is sufficient to trigger the nucleation of DRX, then such a dislocation density is known as the critical dislocation density (ρ_c). The strain associated with ρ_c is called critical strain (ε_c). At this stage, as the deformation proceeds, the DRX gradually intensifies, and its volume fraction increases as the strain increases. Under DRX, the nucleation of grains, and subsequent migration of grain boundaries, leaving new dislocation-free grains that then deform easily (Humphreys et al, 2017). This causes the slope of the curve to decrease with the increasing strain, and the flow stresses decrease after peak stress.

Mathematically, the critical dislocation density necessary for the onset of recrystallization can be described by (Roberts and Abloom, 1978):

$$\rho_c = \left(\frac{10\Omega\dot{\epsilon}}{3blM\tau^2} \right)^{1/3} \quad (5.14)$$

Where Ω is the grain boundary energy, M the grain boundary mobility, l the dislocation mean free path and τ is the dislocation line energy. l and τ can be determined from the following relations:

$$l = \frac{q_1\mu b}{\sigma} \quad (5.15)$$

$$\tau = q_2\mu b^2 \quad (5.16)$$

Where σ is the flow stress, b the Burger vector, μ the shear modulus, q_1 and q_2 are the material constants.

Considering the effects of dislocation density on recrystallization during hot deformation, the evolution of recrystallized volume fraction which can be expressed as (Sandström and Lagneborg, 1975)

$$\dot{S} = \frac{v\delta}{d} \lambda \rho \quad (5.17)$$

Where λ is a material constant and v is the moving grain boundary velocity expressed as (Alabort et al, 2015)

$$v = M\tau\rho \quad (5.18)$$

Considering the normalized dislocation density concept (explained in *Section 5.4.4*), the Eq. 5.17 can be expressed as

$$\dot{S} = \frac{C_1\delta\bar{\rho}}{d} \quad (5.19)$$

where C_1 is a material constant while the parameter δ is related to the fraction of mobile boundaries and can be expressed following the work of Roucoules et al (2003)

$$\delta = (1 - S)(0.1 + S)^{C_2} \bar{\rho} \quad (5.20)$$

where C_2 is a material constant while S represents the fraction of recrystallized volume varying in value from 0 to 1 and which depends on the normalized dislocation density $\bar{\rho}$.

Combining Eq. 5.19 and Eq. 5.20, the evolution of recrystallized volume is defined as

$$\dot{S} = \frac{C_1(1 - S)(0.1 + S)^{C_2} \bar{\rho}^2}{d} \quad (5.21)$$

From the flow stress curves (Figure 5.5), it can be observed that the softening phenomenon increases with increasing plastic strain rate ($\dot{\epsilon}_p$), therefore, to make it sensitive to the effect of the plastic strain rate ($\dot{\epsilon}_p$), the Eq. 5.21 is modified as below:

$$\dot{S} = \dot{\epsilon}_p \left[\frac{C_1(1 - S)(0.1 + S)^{C_2} \bar{\rho}^2}{d} \right] \quad (5.22)$$

5.4.4. Modelling dislocation density

It is well known that the dislocation structure developed in plastic deformation constitutes a driving force for microstructure evolution (Lin et al, 2005), such as recrystallization and grain growth. The evolution of dislocation density is due to three mechanisms: the first is the creation and accumulation of dislocations due to work hardening induced by plastic deformation, the second is annihilation resulting from static and dynamic recovery, and the third is annihilation caused by microstructure evolution, such as recrystallization (Xiao et al, 2018). The generation of new dislocations and their climbing with existing dislocations causes working hardening. Meanwhile, the stored energy of the material enhances and drives recovery and microstructure change. Conversely, the recovery, including the static and dynamic recovery, leads to the absorption of tangled dislocations, causing strain softening. In this work, the dislocation density rate was treated as an internal variable.

Mecking and Kocks (1981)) developed a phenomenological model (the KM model) to characterize the rate of dislocation density change as a function of dislocation storage and annihilation. A number of dislocation density rate models have arisen since then (Estrin et al, 1998; Ding and Guo, 2001; Lin and Liu, 2003; Lin et al, 2005). The evolution of dislocation density ρ can be expressed as (Estrin, 1998):

$$\frac{d\rho}{d\varepsilon_p} = k_1\sqrt{\rho} - k_2\rho \quad (5.23)$$

where ε_p is plastic strain; k_1 material constant; k_2 thermally activated process of dynamic recovery by dislocation cross-slip and recombination or dislocation climb, a function of temperature and strain rate. The first term in Eq. 5.23 is related to the dislocation storage as a result of the plastic straining. The second term models the reduction of dislocation density due to the dynamic recovery. A third term is introduced to consider the change in dislocation density caused by static recovery as:

$$\dot{\rho} = -2M\tau\rho^2 \quad (5.24)$$

where M is the grain boundary mobility, and τ is the energy per unit length of dislocation. Eq. 5.24 can be described by a phenomenological form as (Lin et al, 2005):

$$\dot{\rho} = -A_2\rho^{A_3} \quad (5.25)$$

where A_2 and A_3 are material constants.

Because the absolute density of dislocations in a material is difficult to determine, the normalized dislocation density concept introduced by Lin and Liu (2003) is considered for the modelling the dislocation density which can be formulated as:

$$\bar{\rho} = 1 - \rho_0/\rho \quad (5.26)$$

where $\bar{\rho}$ is normalized dislocation density, ρ_0 is the initial dislocation density, and ρ is the dislocation density during the deformation process. The normalized dislocation density $\bar{\rho}$

has a range from 0 (initial state) to 1 (saturated state) means that $\bar{\rho}$ equals zero in the fully annealed material and in case of considerably large plastic deformation (where $\rho \gg \rho_0$) $\bar{\rho} \rightarrow 1$, corresponding to the dislocation saturated state.

To consider softening behaviour during the hot deformation process, a fourth term accounting for the reduction of dislocation density due to recrystallization is incorporated in Eq. 5.23. Recrystallization annihilates dislocation networks when new dislocation-free grains are created. This phenomenon translates into a variation of the dislocation density expressed:

$$\dot{\rho} = -A_4 \rho \frac{\dot{S}}{1-S} \quad (5.27)$$

where A_4 is a temperature-dependent material constant, and S is the fraction of recrystallization.

Combining Eq. 5.23, 5.24, 5.25, 5.26, and 5.27, a constitutive equation to model the evolution of the normalized dislocation density evolution is defined as:

$$\dot{\bar{\rho}} = A_1(1 - \bar{\rho})|\dot{\epsilon}_p| - A_2\bar{\rho}^{A_3} - A_4\bar{\rho} \frac{\dot{S}}{1-S} \quad (5.28)$$

Where A_1 , A_2 , A_3 , and A_4 are material constants.

Considering the effect of grain size on dislocation density, the normalized dislocation density evolution of Eq. 5.28 is modified as:

$$\dot{\bar{\rho}} = A_1(1 - \bar{\rho})(|\dot{\epsilon}_p|)^{g_2} - A_2\bar{\rho}^{A_3} - A_4\bar{\rho} \left(\frac{\dot{S}}{1-S} \right) d^\beta \quad (5.29)$$

Where β is a material constant

5.4.5. Modelling isotropic hardening

Isotropic hardening is another factor that could influence the superplastic deformation of titanium alloys. Isotropic hardening is closely related to the accumulated dislocation density and expands the yield surface of a material under plastic deformation (Skrzypek, 2000). The evolution of isotropic hardening H can be expressed as (Chaboche and Rousselier, 1983)

$$\dot{H} = h(\dot{Q} - H)\dot{\epsilon}_p \quad (5.30)$$

where h is a material constant and is related to the speed for H to saturate and its value is normally between 0.5 and 1 (Lin and Yang, 1999). Similarly, Q is also a material parameter and represents the level of saturation (Cheong et al, 2000). After integrating the Eq. 5.30, a direct relationship between H and $\dot{\epsilon}_p$ can be expressed as:

$$H = Q(1 - e^{-b\dot{\epsilon}_p}) \quad (5.31)$$

Although Eq. 5.31 does not take into account the static recovery of dislocations during hot deformation; it has, however, been used in many studies in the literature e.g. Zhou and Dunne (1996), Lin and Yang (1999), and Cheong et al. (2000). To consider the static recovery of dislocations during low strain rate superplastic deformation, Lin et al (2011) proposed a new normalized dislocation density-based relation for the evolution of isotropic hardening, which takes the form:

$$\dot{H} = 0.5B\bar{\rho}^{-0.5}\dot{\bar{\rho}} \quad (5.32)$$

where B is a constant.

By integrating Eq. 5.32, can take the form:

$$H = B\bar{\rho}^{0.5} \quad (5.33)$$

Eq. 5.33 is modified by Tabassam et al (2019) to the following form

$$H = B\bar{\rho}^\varphi \quad (5.34)$$

Where φ is a material constant which generally varies from 0.3 to 1.0.

From the flow stress curves (Figure 5.5), it can be observed that the hardness phenomenon increases with decreasing plastic strain rate ($\dot{\epsilon}_p$), therefore to incorporate the effect of plastic strain rate ($\dot{\epsilon}_p$), the Eq. 5.34 is modified as below:

$$H = B\bar{\rho}^\varphi \dot{\epsilon}_p \quad (5.35)$$

5.4.6. Viscoplastic flow rule

The flow rule describes the stress-strain relationship of a material when it is deformed, and factors, such as initial yield stress, material hardening, and dislocations, are incorporated. The total strain ϵ_T can be additively decomposed into two parts, *i.e.* elastic strain ϵ_e and plastic strain ϵ_p . The relationship between the ϵ_e and stress σ for an ideal elastic-viscoplastic material can be expressed by the well-known Hooke's Law:

$$\sigma = E(\epsilon_T - \epsilon_p) \quad (5.36)$$

Where E represents Young's modulus, ϵ_T is total strain and ϵ_p is plastic strain.

Because the material exhibits viscoplastic flow during the SPF process, the overall flow stress can be decomposed into three parts: initial yield stress, viscoplastic stress, and hardening stress (Lin et al, 2005).

$$\sigma = k + \sigma_v + H \quad (5.37)$$

Where k is the initial yield stress, σ_v the viscoplastic stress, and H the isotropic hardening stress.

The potential stress σ that initiates viscoplastic flow must exceed the initial yield stress k , *i.e.*, the value of $(\sigma - k)$ should be positive (Lin et al, 2005). After sufficient plastic straining occurs, dislocation entanglement, pile-ups, etc., harden the material by an amount H and the stress that leads to viscoplastic flow stress σ_v is reduced to $(\sigma - R - k)$. The stress should overcome H to continue the viscoplastic flow, which is expressed as $\langle \sigma - R - k \rangle_+$. On the basis of this information Lin and Liu (2003) modified the flow rule, considering its dependence to be directly on the isotropic hardening (H) and initial yield stress (k) and can be expressed as:

$$\dot{\epsilon}_p = \left\langle \frac{\sigma - H - k}{K} \right\rangle^n \quad (5.38)$$

where $\dot{\epsilon}_p$ is the plastic strain rate, σ is the flow stress, H is the hardening due to material isotropic work hardening, n is the viscosity exponent. The term $\left\langle \frac{\sigma - H - k}{K} \right\rangle$ within Macaulay brackets imply that this equation is not applicable until plastic deformation occurs. The parameter k is the initial yield stress, and K a temperature-dependent parameter.

Considering the effect of grain size and damage on flow stress, the flow rule of Eq. 5.38 is modified as:

$$\dot{\epsilon}_p = \left(\frac{\frac{\sigma}{1 - n_1 \omega^{n_2}} - H - k}{K} \right)^n \cdot d^{-\mu} \quad (5.39)$$

Where n_1 and n_2 are material constants while n and μ are the stress and grain size exponent, respectively.

5.4.7. Formulation of unified viscoplastic constitutive equations

A set of unified viscoelastic constitutive equations consists of the plastic (rate-independent) and creep (rate-dependent) parts. This enables the metal flow and microstructure evolution, such as strain hardening, recovery, recrystallization, grain size, and dislocation density, to be well captured and described. To represent the superplastic behaviour of a TA15 alloy having an equiaxed microstructure, the set of mechanism-based unified viscoplastic constitutive equations takes the form of:

$$\left\{ \begin{array}{l} \dot{\epsilon}_p = \left(\frac{\frac{\sigma}{1 - n_1 \omega^{n_2}} - H - k}{K} \right)^n \cdot d^{-\mu} \\ H = \dot{\epsilon}_p B \bar{\rho}^\varphi \\ \dot{\omega} = \eta(1 - \omega)(\dot{\epsilon}_p)^{z_1} + \frac{F \varepsilon^G}{(1 - \omega)} \sigma (\dot{\epsilon}_p)^{z_2} \\ \dot{d} = \frac{\alpha_1}{d^{\gamma_1}} + \frac{\alpha_2 \dot{\epsilon}_p}{d^{\gamma_2}} - \alpha_3 \dot{S}^{\gamma_4} d^{\gamma_3} \\ \dot{S} = \frac{\dot{\epsilon}_p (C_1 (1 - S) (0.1 + S)^{C_2} \bar{\rho}^2)}{d} \\ \dot{\bar{\rho}} = A_1 (1 - \bar{\rho}) (|\dot{\epsilon}_p|)^{g_2} - A_2 \bar{\rho}^{A_3} - A_4 \bar{\rho} \left(\frac{\dot{S}}{1 - S} \right) d^\beta \\ \sigma = E(\epsilon_T - \epsilon_p) \end{array} \right. \quad (5.40)$$

There are total of 29 parameters in the above set of equations, out of which 19 are temperature dependents. These temperature dependent parameters can be formulated using the classic Arrhenius relationships, as defined below:

$$(C_i)_j = c_i \exp \left(\frac{Q_i}{RT_j} \right) \quad i = 1, 2, \dots, I \quad j = 1, 2, \dots, J \quad (5.41)$$

where C_i are the temperature compensated constants, c_i is a temperature-independent constant, Q_i is the i th activation energy (J/mol), I is the number of constants to be determined, R is the universal gas constant ($8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$) and T_j is the j th temperature in Kelvin. This trend is plotted in the [Figure 5.13](#).

In order to avoid the overuse of material parameters, an optimization route is proposed (Figure 5.11), in which a series of equations are activated or otherwise based on the fitting quality of the global model. This approach allows for the elimination of redundant parameters but, more importantly, has provided physical insight into the deformation modes, which are operative.

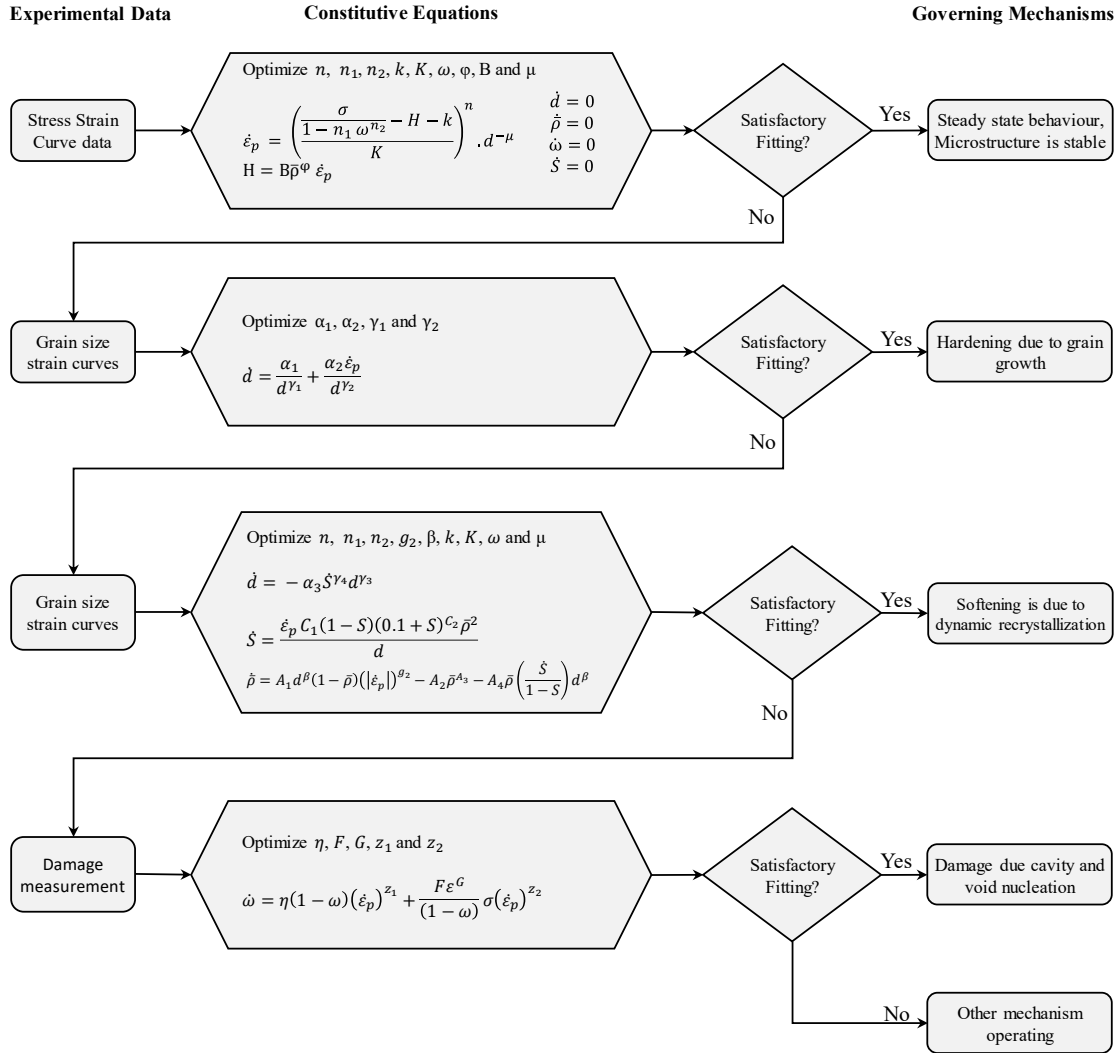


Figure 5. 11. Optimization route for the proposed constitutive model.

5.5. Determination of material constants

One of the most challenging tasks encountered in the development of viscoplastic constitutive equations is to accurately determine the associated material constants of the equations, valid over a range of strain rates and temperatures, from experimental data (Lin et al, 2002; Mohamed et al, 2012). The equation set has a total of 28 material parameters. The tensile data obtained at different temperatures and strain rates (Figure 5.7) were used to determine the material constants arising in the constitutive equations.

The determination of material constants for unified constitutive equations is challenging without using an effective optimization technique. Considerable studies have been conducted over the years (Cao and Lin, 2008; Igual and Xu, 2015; Christopher and Choudhary, 2018) to formulate optimization methods. To determine the material constants for the proposed constitutive equations, a gradient-based optimization technique is used in this study. The gradient-based optimization is achieved using the ‘Trust region reflective least square algorithm’ from the MATLAB optimization tools. The objective function of the model is evaluated by determining the square of the difference between the experimental and the computed data for the flow stress, grain size & geometrical necessary dislocation (GND) as normalized dislocation density ($\bar{\rho}$) in the following form:

$$f(X) = \frac{1}{M} \sum_{j=1}^M \left[\frac{1}{N_j} \sum_{i=1}^{N_j} \{ (\sigma_{ij}^e - \sigma_{ij}^c) + (d_{ij}^e - d_{ij}^c) + (\bar{\rho}_{ij}^e - \bar{\rho}_{ij}^c) \}^2 \right] \quad (5.42)$$

Where $f(X)$ is the objective function evaluating the error between experimental and computed values of flow stress (σ), grain size (d) & dislocation density ($\bar{\rho}$), X represents number of material constants in unified constitutive equations, M represent the number of experimental flow stress-strain curves, N_j is the data points on the j th curve, superscript ‘ c ’ symbolizes computed values from the material model and superscript ‘ e ’ corresponds to experimental values.

Based on the square of the difference between the experimental and the computed data for the flow stress and grain size, the Trust Region Reflective Least Square Algorithm is programmed to determine the material constants. The objective function is based on

“LSQNONLIN” which solves nonlinear least-squares problems, including nonlinear data-fitting problems. Rather than computing the value $f(X)$ (the "sum of squares"), LSQNONLIN requires the user-defined function to compute the vector-valued function. Hence the objective function $f(X)$ returns a vector (or array) of values, and not the sum of squares of the values (The algorithm implicitly computes the sum of squares of the components of $f(X)$). [Figure 5.12](#) shows a schematic flow flowchart of the iterative procedure used to determine the material constants. It comprises numerical integration of differential equations (*i.e.* unified constitutive Eq. set 38), which are solved by the forward Euler method. As for gradient-based optimization, initial trial values of unknown constants are necessary inputs; therefore the lower and upper bounds for these 13-material constant set were estimated on the basis of information available in the literature for different types of titanium alloys ([Yang et al, 2016](#), [Luo et al, 2010](#); [Cai et al, 2011](#); [Xiao et al, 2012](#); [Fan et al, 2013](#); [Yang et al, 2015](#)). To ensure that the derived set of material constants converges to a local minimum, the iterative procedure is repeated for different initial values. The determined model material constants are listed in [Figure 5.13](#).

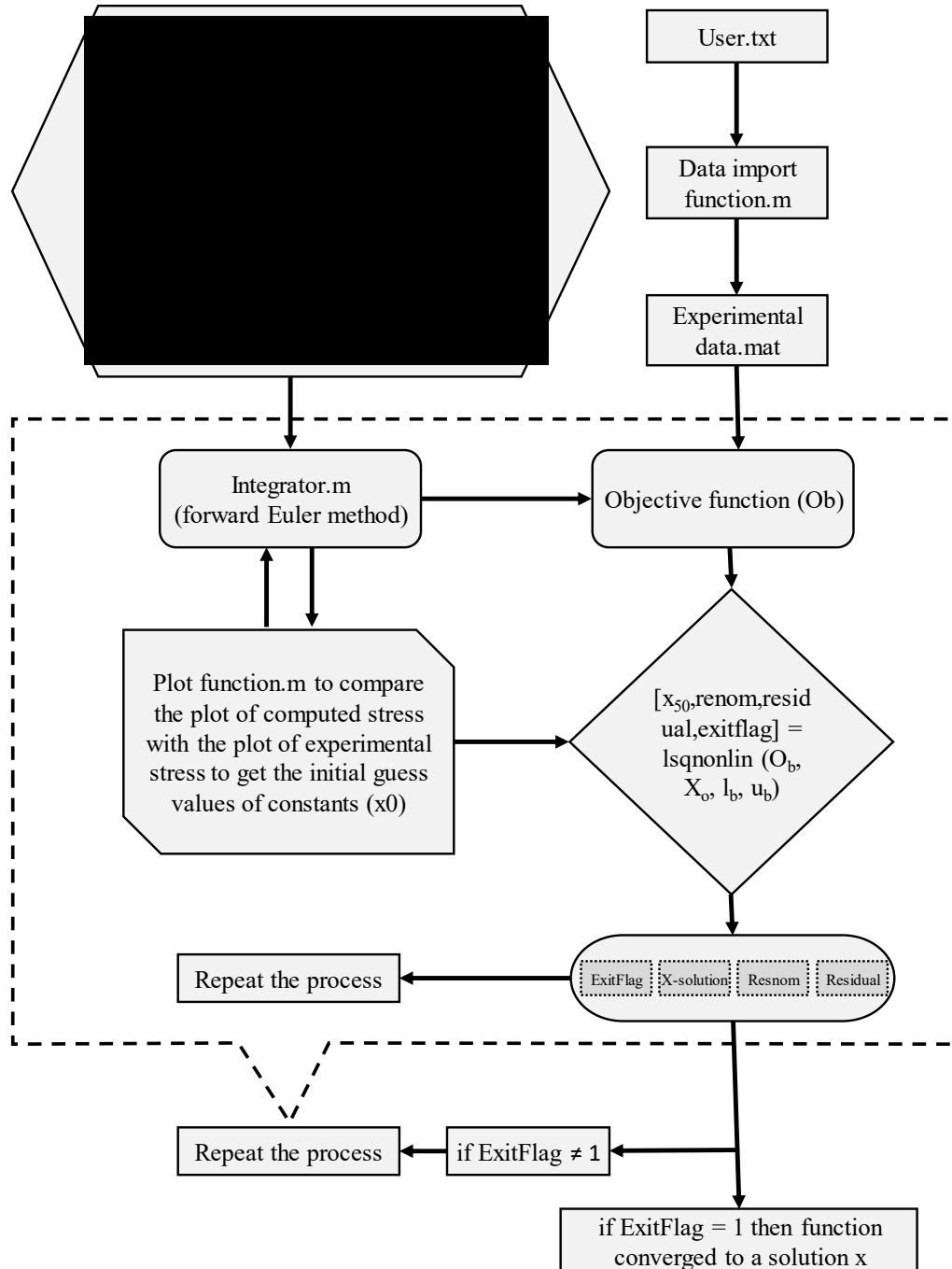


Figure 5. 12. Schematic flow diagram describing numerical algorithm for optimization of material constants

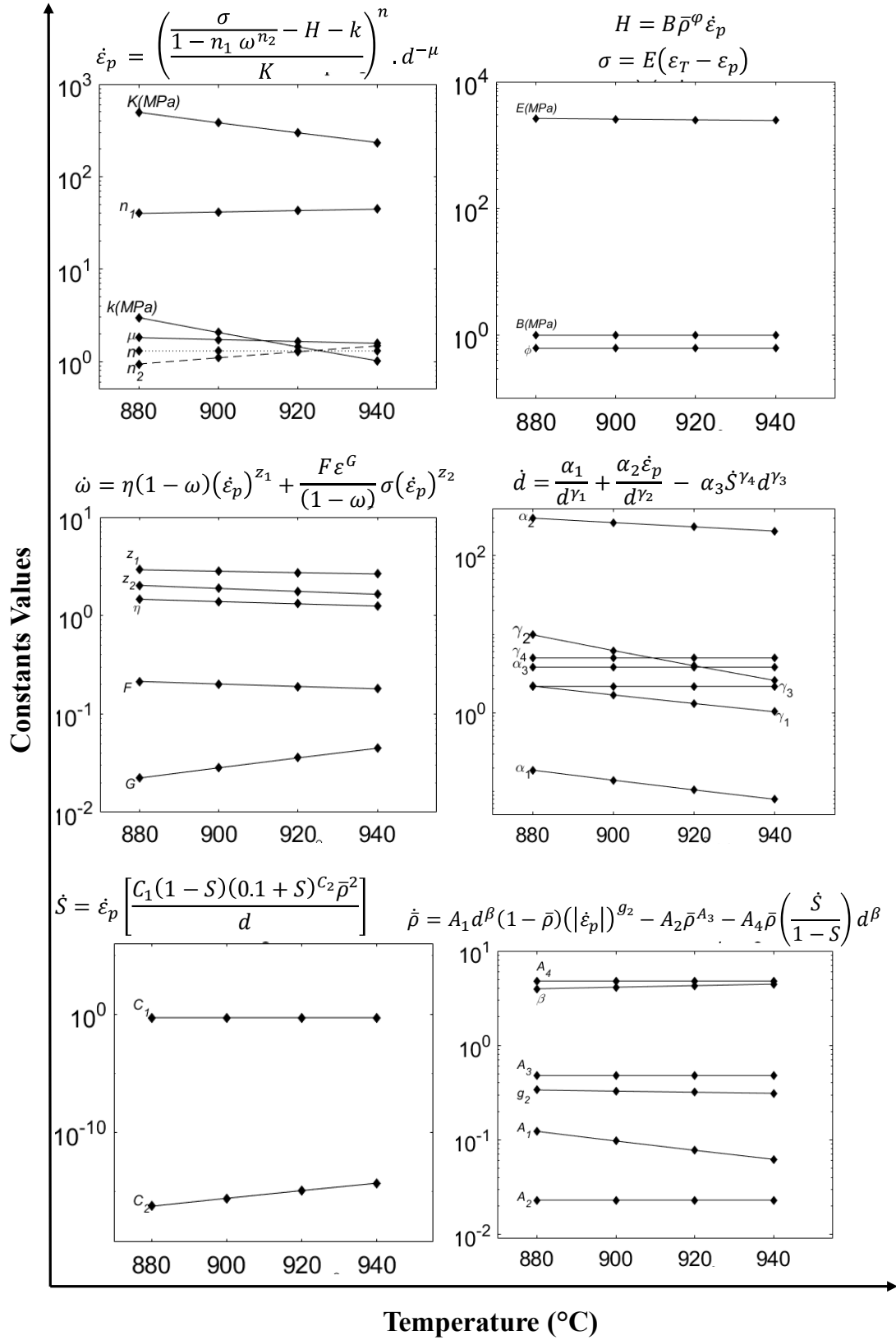


Figure 5.14 (a, b, e & f) shows the comparison between experimental (symbols) and predicted flow stress (solid lines) of TA15 alloy for the given range of strain rate and temperature. In general, it can be concluded that the proposed constitutive model is reasonably efficient to predict the deformation behaviour in the given temperature and strain rates. As shown in Figure 5.14 (a, d, g & h) the experimental flow stress curves show a work hardening region followed by softening, for the strain rate 0.01s^{-1} and the proposed constitutive equations successfully captured this phenomenon. On the other hand, for the lower strain rates, *i.e.* 0.001s^{-1} and 0.0005s^{-1} , there is no softening and for all such conditions of grain growth hardening, the proposed model can capture the flow stress pattern quite effectively.

Figure 5.14 (b, e, h & k) shows the computed and experimental evolutions of the average grain size at a given range of testing conditions. The initial undeformed microstructure is cold rolled with the non-uniform distribution of grain size showing the average grain size of $3.74\mu\text{m}$. The *Revised Model* provides a good fit for the variation of the experimental grain size and is in good agreement with the corresponding Flow stress curves. For the lower strain rates where grain growth is observed, it is evident that the computed grain size is compatible with the grain size of experimental data.

Figure 5.14 (c, d, g & h) shows the computed and experimental evolutions of the normalized dislocation density at a given range of testing conditions. The initial undeformed microstructure is cold rolled with the maximum average dislocation density of $3.47 \times 10^{14} \text{m}^{-2}$ which was normalized to 1. The *Revised Model* provides a good fit for the variation of the experimental dislocation density which is decreasing from the maximum of 1. This decrease in dislocation density is in good agreement with the corresponding flow stress curves (Figure 5.5) for superplastic phenomena where the dislocation at high temperature are usually annihilated.

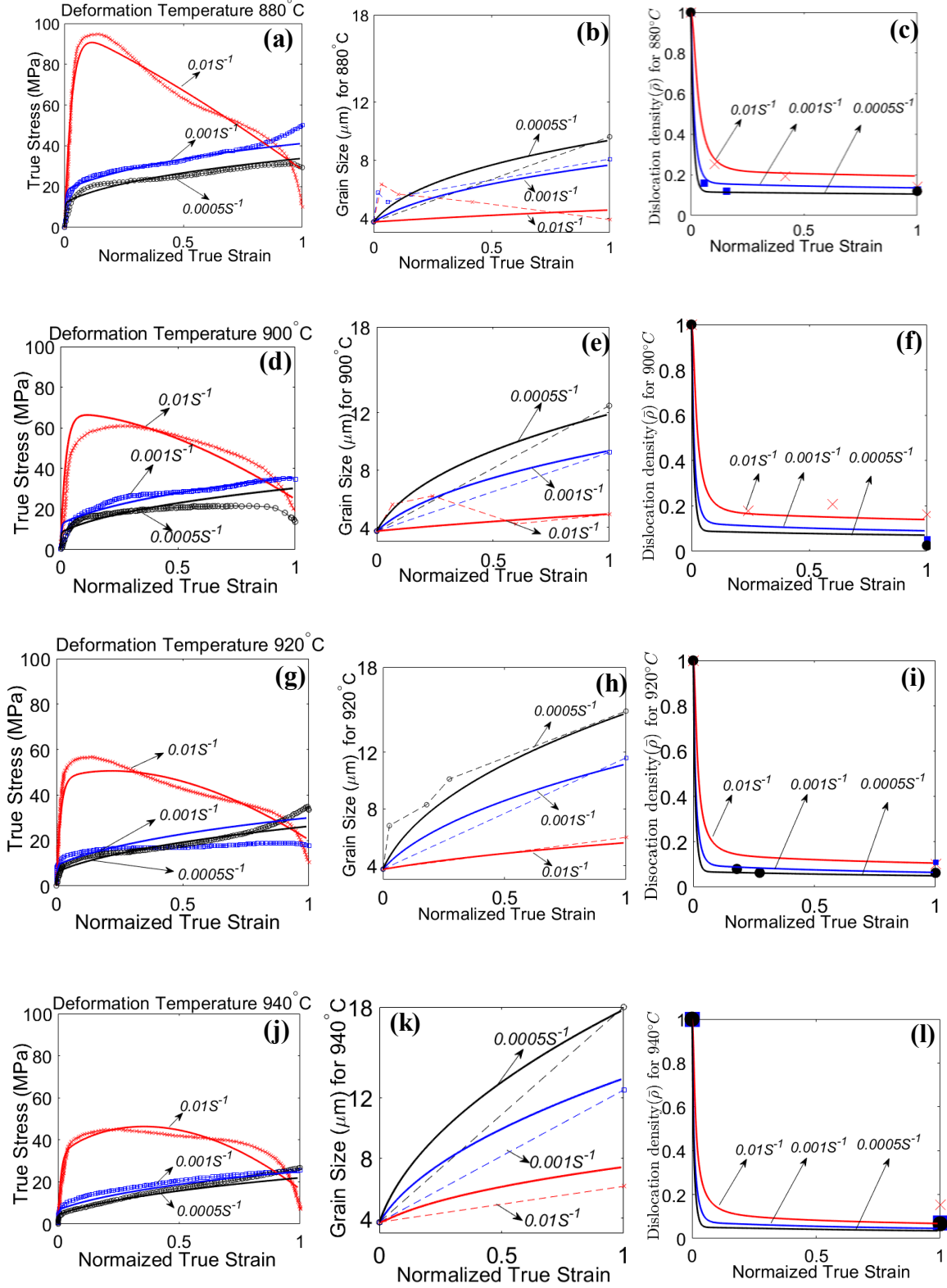


Figure 5. 14. Comparison of the experimental (symbols) and computed (solid lines) flow stress , grain size & dislocation density of TA15 at different deformation temperatures and strain rates

To evaluate the advantages of the *Revised Model* for the purpose of offering a point of comparison, [Figure 5.15](#) shows the prediction accuracy of the *Initial Model* and *Revised Model* at deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$, the case where maximum elongation is achieved. From the morphology of the flow curve, it can be concluded that the *Revised Model* provides reasonably best fit for capturing the flow hardening behaviour of the stress-strain curve while the *Initial Model* is unable to predict this behaviour very well.

The *Revised Model* is based on the experimental deformation mechanism, where it can be seen that the normalized dislocation density measured on the basis of GND in the initial un-deformed material is 1, which decreases to 0.2, while in the *Initial Model* the dislocation density increases and attains a saturation value of 0.5. In the *Initial Model*, the grain growth was not taken into account, while the *Revised Model* considers the effect of grain growth and this is the main reason for the increase in flow stress as larger grain size hinders the GBS/grain rotation, an inherent characteristic of superplasticity. Similarly, the *Initial Model* predicts a zero-recrystallization fraction, while the *Revised Model* shows a small fraction of recrystallization, which is reasonable, as at deformation $880^{\circ}\text{C} / 0.001\text{s}^{-1}$, there will be recrystallisation, as explained in Chapter 3 & 4 and grain growth. So in summary the *Revised Model* can relatively better capture the deformation mechanism including microstructure variable e.g. grain growth, dislocation density of the component during FEA simulation.

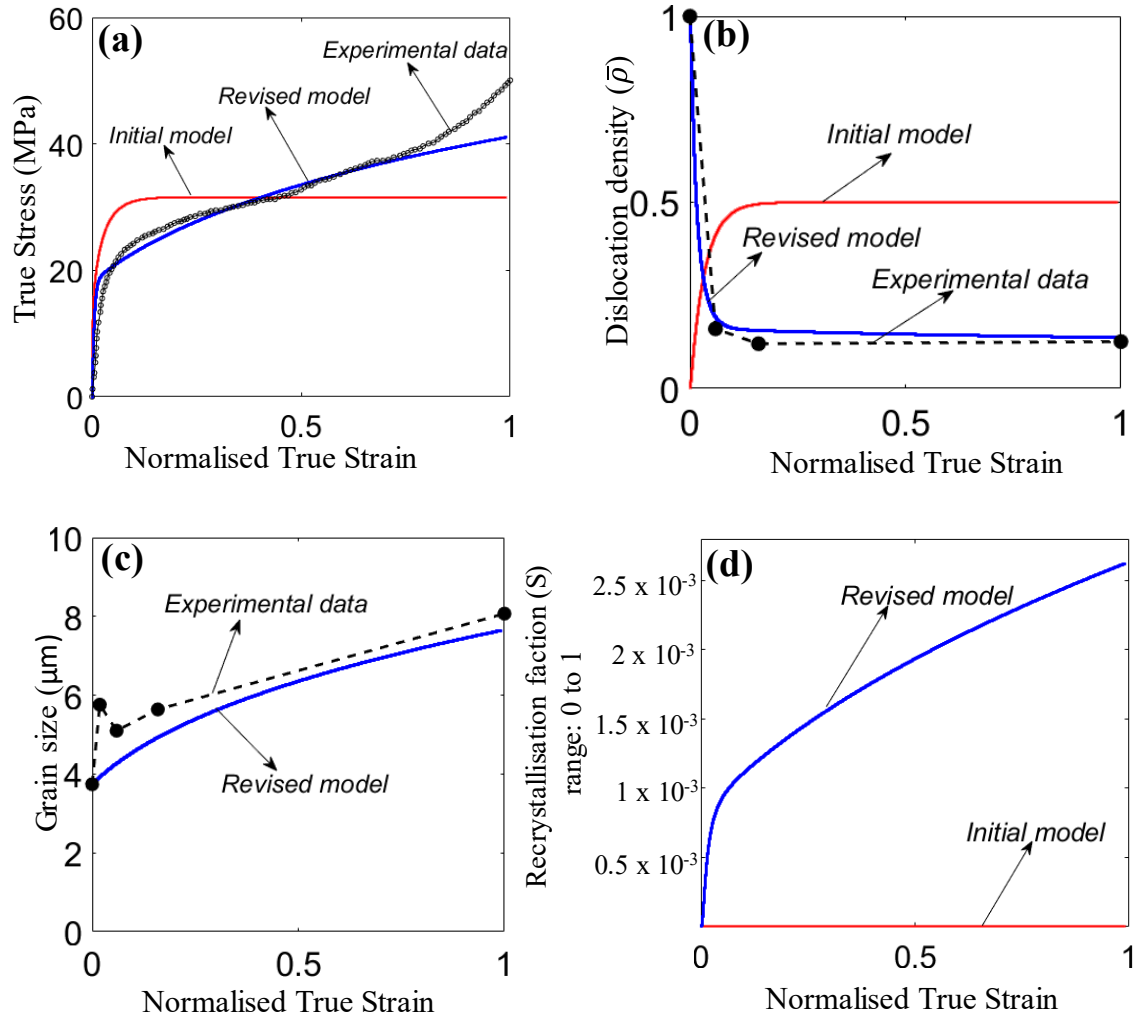


Figure 5. 15. Flow stress prediction accuracy of *Initial Model* and *Revised Model* at deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$.

5.6. Finite element model of a superplastic forming process

To illustrate the advantages and power of the proposed constitutive relationship, it has been incorporated into a numerical treatment of the deformation of a superplastic forming structure using finite element analysis (FEA) and the results compared for the same FEA procedure but using the *Initial Model*.

5.6.1. Numerical Method

To perform the numerical analysis, a commercial finite element implicit solver, ABAQUS, is employed. In quasi-static processes - which superplastic forming is usually considered to be as the kinematic energy is a minimal fraction of the total deformation energy, hence an implicit finite element analysis (FEA) code is preferred for modelling the SPF process over an explicit FEA code (Ng et al, 2011). The implicit analysis uses an iterative process that enforces equilibrium between the internal structure forces and the externally applied forces at the end of each increment, and reconstructs the stiffness matrix accordingly; this improves computational accuracy. However, as the analysis needs to update and reconstruct the stiffness matrix for each iteration, this can cause a very high computational cost if a large time increment cannot be maintained (Ng et al, 2011). On the other hand, an explicit analysis uses a small time step to compute the incremental displacement and associated changes in loading instead of checking for equilibrium at the end of each increment.

5.6.2. Constitutive Equations Implementation in the FE Code

The proposed model was implemented for large strain formulation using a user-defined CREEP subroutine compiled in FORTRAN. In the compiled subroutine, the defined strain-rate potential was written as a function of the equivalent stress and a series of solution-dependent variables or evolutionary equations, solved using the forward Euler method. To implement the proposed constitutive equation into ABAQUS, the following quantities need to be updated within ABAQUS using a CREEP subroutine.

A common approach which ABAQUS makes use of - is the additive decomposition of the rate of deformation tensor D as

$$D = D^e + D^p \quad (5.43)$$

Where D^e and D^p are elastic and plastic components, respectively. This is a valid approach provided that elastic stretches are small ($V^e \approx 1$). If so, one can determine the Jaumann (co-rotational) stress rate using the tensorial form of Hooke's law as

$$\hat{\sigma} = 2GD^e + \lambda T_r(D^e)I \quad (5.44)$$

where $\hat{\sigma}$ denote Jaumann rate of Cauchy stress tensor, G is the shear modulus, and λ is the Lamé elasticity constant. This can be used to derive the material rate of stress as following

$$\hat{\sigma} = \dot{\sigma} + W\sigma - \sigma W \quad (5.45)$$

where $\dot{\sigma}$ is the material time derivative of Cauchy stress tensor σ

W is the continuum spin tensor. The Cauchy stress rate can be integrated in order to obtain the stress within a reference frame.

In order to implement the proposed constitutive equations for superplasticity, it is necessary to generalize the constitutive laws for multiaxial conditions. Using the steady-state creep rate equations and by assuming von Mises behaviour and the normality hypothesis, the multiaxial viscoelastic strain rate can be defined for each individual mechanism as (Dunne, 1998):

$$D^{p, gbs} = \frac{3}{2} \frac{\sigma'}{\sigma_e} \dot{p}_{gbs} \quad (5.46)$$

where σ_e is the effective stress, σ' is the deviatoric stress and $\dot{p}_{gbs}$ is the effective plastic strain rates for grain boundary sliding, dislocation creep.

$$\dot{p}_{gbs} = \frac{A_{gbs} f_{\beta}^q D_{gbs} \mu b}{kT} \left(\frac{b}{d} \right)^{p_{gbs}} \left(\frac{\sigma_e}{\mu} \right)^{n_{gbs}} \quad (5.47)$$

Additionally, this set of constitutive equations is a function of the internal state variable track changes in the microstructure as introduced in Eq. 5.40.

Using the stress rate tensor as defined in Eq. 5.45, it is possible to define the necessary stress update after a given increment of time as

$$\Delta\sigma = \Delta\sigma^0 + (W\sigma - \sigma W)\Delta t = \Delta\sigma^0 + R\sigma_t R^T \quad (5.48)$$

where $\Delta\sigma^0$ is the co-rotational stress increment assuming that $D^e = D - D^{p, gbs}$.

As creep is a time-dependent phenomenon, when implementing the material model in ABAQUS via CREEP subroutine, it is, therefore, more relevant to ensure that the solution of the problem is not varying too rapidly from one time step to another. In this case, the imposed strain rate can be additively decomposed into an elastic and viscoplastic part: $\dot{\epsilon} = \dot{\epsilon}_{el} + \dot{\epsilon}_{vp}$. The strain increment during one-time step (which is known since the displacement is imposed) is then $\Delta\epsilon = \dot{\epsilon}\Delta t = \Delta\epsilon_{el} + \Delta\epsilon_{vp}$. The stress response is obtained from the elastic strain and elasticity constants: $\Delta\sigma = E\Delta\epsilon_{el}$. If the viscoplastic strain rate is constant, $\Delta\epsilon_{vp}$ is calculated accurately, and the right stress may be obtained for any time step. In real problems, $\dot{\epsilon}_{vp}$ changes with time and its variation during each time step should be kept to an acceptably low value. This procedure controls the accuracy of the creep analysis, and it is implemented in ABAQUS as the CETOL parameter, which measures the variation in time step (time increment) as follows:

$$CETOL = (\dot{\epsilon}_{vp}|_{t+\Delta t} - \dot{\epsilon}_{vp}|_t)\Delta t \quad (5.49)$$

The value of CETOL is usually taken to be constant, and Δt thus varies throughout the calculation. The first objective was to determine how small CETOL should be to obtain stable solutions. The size of the time increment is set automatically by the ABAQUS during the FE analysis, but it is constrained by a user-specified accuracy tolerance parameter which limits the maximum inelastic strain rate change allowed over an increment. In this study the value of CETOL was set to 0.001 and the Creep subroutine was called at each material point for the beginning and end of each time increment.

5.6.3. Model geometry and boundary conditions

The finite element model must be adequately restrained by displacement constraints to eliminate rigid-body motion and, more importantly, to accurately capture physical conditions (Chang, 2016). The geometry and boundary conditions constructed in the FE model for simulating this superplastic blow forming experiment are schematized in [Figure 5.16](#), and [Figure 5.17](#) respectively. The initial dimensions of the sheet were $374 \times 259 \times 2$ mm. The sheet was placed over the die, and its periphery was firmly clamped along with the rigid die with the boundary conditions as follows: Edge 1 & Edge 3 are restrained in Y and Z direction while Edge 2 & Edge 4 are restrained in X and Z direction. The loading is applied to the top surface of the sheet. As the model geometry exhibits biaxial mechanical symmetry, so to minimize the computation time, only a quarter of the assembly was considered in this FE model.

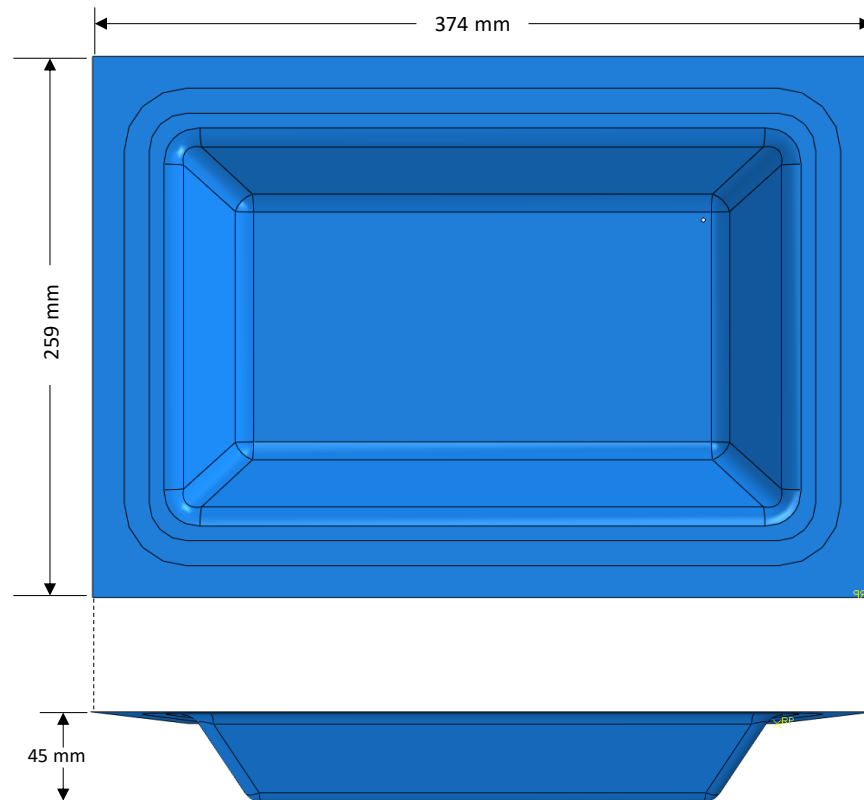


Figure 5. 16. Geometry of 3D FE model for superplastic blow forming

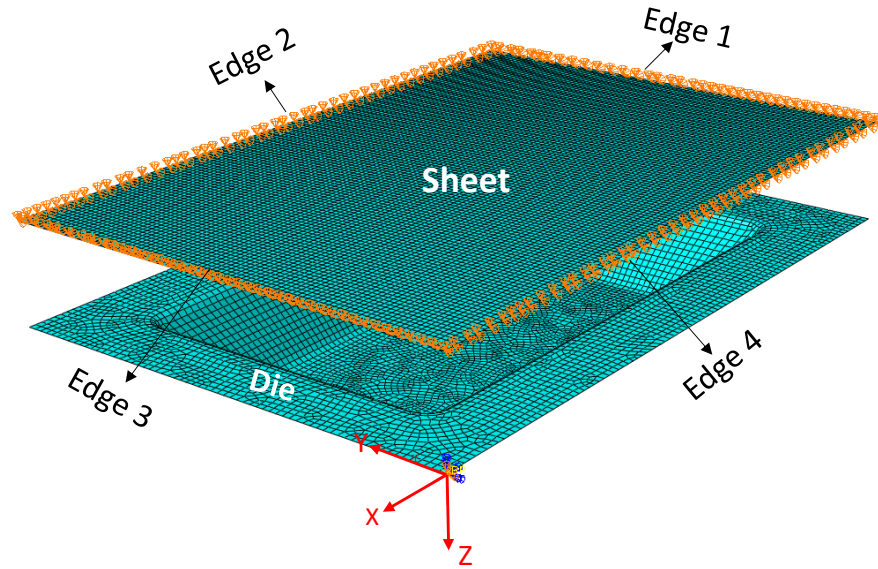


Figure 5. 17. Boundary conditions for 3D FE model for superplastic blow forming.

5.6.4. Surface and mesh generation

Simulating the manufacture of industrial parts made through the SPF process is typically complex shaped structural components. It is essential to accurately define the intricate shaped die surface and generate a quality finite element mesh over it, into which the flat sheet metal is deformed. A 3D model of the die and sheet was developed by using a CAD tool Solid Works, considering the sheet as a deformable body and die as a discrete rigid body. The selection of the element type for the FEA meshing process is very critical as it ultimately determines the accuracy of the simulation. Numerical analysis of the superplastic blow forming process can be carried out using membrane element (Nazzal et al, 2007; Li and Soo, 2011; Balasubramanian et al, 2013; Kumaresan, et al, 2016), shell elements (Ganesh et al, 2015; Tang et al, 2015; Du et al, 2016; Carunchio et al, 2017; Rollin et al, 2018; Nazzal et al, 2019) and solid (continuum) elements (Jarrar et al, 2010; Giuliano, 2011; Hu et al, 2016; Hosseini et al, 2018). Naturally, the question of which element will be the most suitable arises. Simulations of sheet forming are performed dominantly using a membrane or shell element because of the reduced computational cost.

However, a membrane element formulation is based on the assumption that the plane stress state is dominant and in-plane strains can be approximately distributed linearly through the sheet thickness (Li and Fu, 2019) without considering moments and bending stiffness (Chen et al, 2001, Guo and Haiki, 2017). Therefore, the membrane element is not appropriate for problems that involve considerable bending effects (Jung et al, 2000). Normally a shell element possesses five degrees of freedom (two rotations, two in-plane displacements, and a transverse displacement) compared to a membrane two degrees of freedom (a transverse and a rotational). Moreover, both membrane and shell elements assume that the thickness strain can be computed as a function of the in-surface deformation due to material incompressibility (Rebelo et al, 1990). This simplified assumption does not allow for the development of stress in the thickness direction of the sheet. On the other hand, as compared to the membrane and shell elements, the solid continuum element formulation can produce more precise results, but as continuum analysis involves large computation time and enormous memory requirements, they are very seldom used. In this study, the shell element is selected as it considers bending stresses, thus getting more accurate results, and at the same time involve less computational cost as compared to solid continuum elements.

The die was meshed with a total number of 35372 quadrilateral R3D4 rigid elements while the blank was modelled with total 96866 four-node 3D shell-type S4 elements with five integration points in the thickness direction. The average initial element sizes adopted for meshing the die and the blank were 2.0 mm and 1.0 mm, respectively. In order to obtain reliable contact convergence, enough elements must be used for the master surface to assure a smooth surface but its elements density should be below that of the slave surface.

5.6.5. Contact and friction between die and sheet

During a superplastic blow forming process, the contact between the sheet and the die results in friction there. Friction is widely recognized as an important factor affecting the thinning of SPF components (Chen et al, 2001). An important consideration in modelling the process is how to simulate this friction effect. In practice, superplastic forming involves complex contact conditions as the metal blank is forced by gas pressure to deform into the shape of the die. The contact between the tooling and the component is highly non-linear,

the surfaces are dissimilar, curved, asymmetric, and the contact region is difficult to predict. ABAQUS defines the contact between two bodies in terms of two surfaces that may interact; these surfaces are called a contact pair. For each node on the first surface (the “slave” surface) ABAQUS attempts to find the closest point on the second surface (the “master” surface) of the contact pair where the master surface’s normal passes through the node on the slave surface (Figure 5.18).

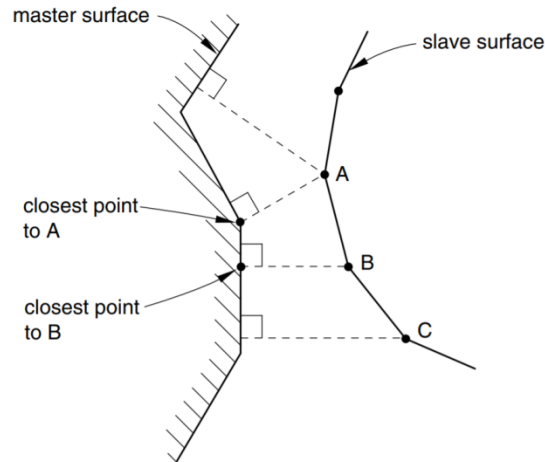


Figure 5. 18. Node-to-surface contact discretization

During the contact analysis, it is essential that the rigid surface must always be the master surface, and the deformable bodies must be the slave surface. For the contact discretization, the surface-to-surface technique offers better accuracy and convergence (Simulia, 2014). For the contact constraint enforcement; the penalty method is used. This technique allows certain penetration between the slave and the master surfaces as a function of a penalty stiffness value k (Figure 5.19). This approach offers a good compromise between accuracy and convergence rate. The numerical treatment of contact often leads to highly non-linear and even discontinuous behaviour, which causes convergence difficulties in implicit finite element methods (Murali et al, 2004, Luckey et al, 2005). To address convergence problems in ABAQUS, it is necessary to apply "softened" contact to the pressure-overclosure relationship (Luckey et al, 2009). To avoid having points “fall off” the rigid surface during the analysis, it is always a good idea to extend the rigid surface far enough, so that the contacting nodes will not slide off the master surface. Frictional boundary conditions are used to determine when a point of the interface between the die and sheet

transitions from sticking-to-slipping or slipping-to-sticking. For this purpose, the general Coulomb friction model is used. This defines the minimum shear stress τ_{crit} at which sliding of the surface starts as a function of the contact pressure between the surfaces, thus $\tau_{crit} = \mu P$. The friction coefficient μ is approximated as 0.2 based on the experimental results reported in the author's published work (Tabassam et al, 2019).

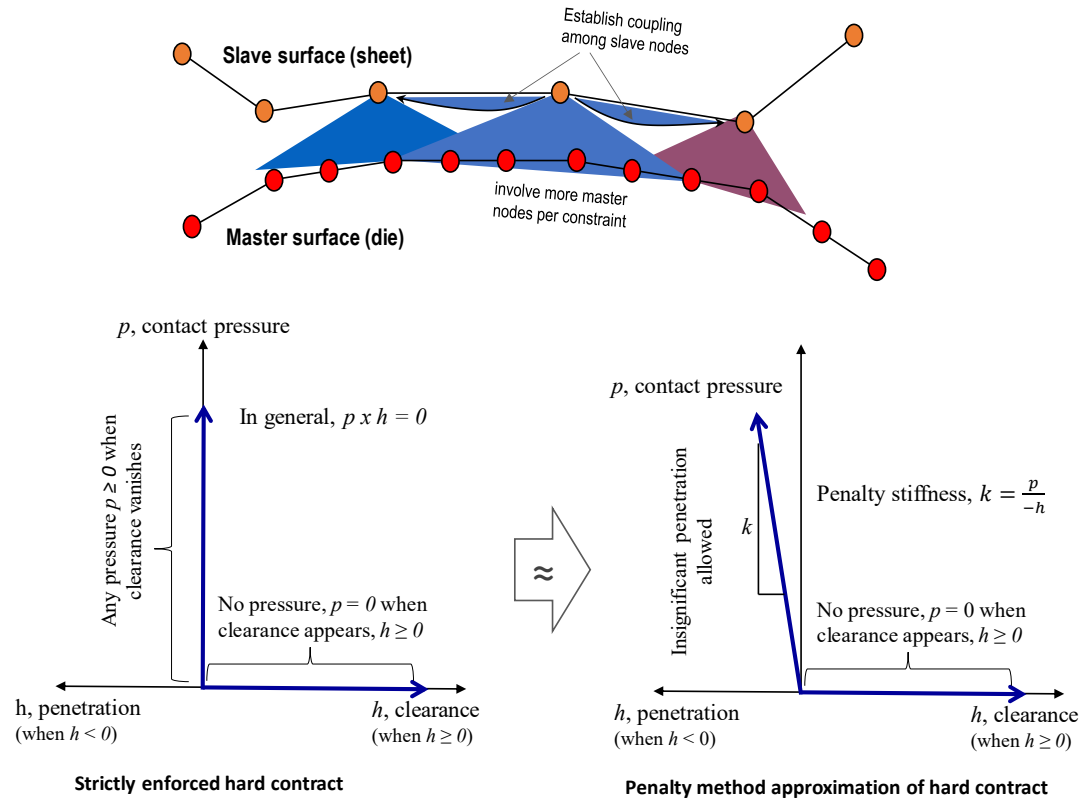


Figure 5. 19. Details of the surface-to-surface contact theory assuming penalty stiffness

5.6.6. Gas pressure profile

As discussed in Chapter 2, that the superplasticity of metals exhibits only over a narrow range of the optimum strain rate. This factor makes it essential to determine the pressure

loading history to maintain the maximum strain rate near the optimum value throughout the whole forming process. During the blow forming process, gas pressure is imposed over a sheet to make it flow into a die of the desired shape. To produce the maximum ductility in the SPF process, it is desirable to adjust the forming pressure to keep the strain rate within an optimum range throughout the whole forming process. Due to the geometry of the die cavity, the sheet undergoes different strain conditions according to the considered area and the forming phase. Thus, significantly different strain rates can be found along with the sheet, and the location of critical zones changes during the process. The ABAQUS contains a built-in algorithm (named *Creep Strain Rate Control*), which allows the maximum strain rate, in a given set of elements, to be controlled in order to have an almost constant value regulating the pressure load. This pressure algorithm internal to ABAQUS was interfaced with a Creep subroutine to compute the gas pressure profile by continuously control the pressure upon the sheet and to keep the maximum strain rate of the whole sheet a target strain rate of 0.001/sec. The algorithm computes the optimal pressure as follows. During each increment, the ratio γ of the maximum equivalent strain rate, $\dot{\epsilon}_{max}$ in the sheet to the target creep strain rate, $\dot{\epsilon}_{tar}$ (specified by the user), is calculated via

$$\gamma = \frac{\dot{\epsilon}_{max}}{\dot{\epsilon}_{tar}} \quad (5.50)$$

The pressure is then adjusted according to the following conditions.

If $0.2 \leq \gamma < 3.0$, the increment is accepted and the pressure is adjusted as follows

$$if \begin{cases} 0.2 \leq \gamma < 0.5 & \text{then } P_{new} = 1.5P_{old} \\ 0.5 \leq \gamma < 0.8 & \text{then } P_{new} = 1.2P_{old} \\ 0.8 \leq \gamma < 1.5 \text{ (target range)} & \text{then } P_{new} = P_{old} \\ 1.5 \leq \gamma < 3.0 & \text{then } P_{new} = 0.83P_{old} \end{cases}$$

Where P_{new} is the updated pressure and P_{old} is the previous increment pressure magnitude. However, if $\gamma < 0.2$ or $\gamma > 3.0$ in any given increment, the increment is abandoned and restarted according to the following logic:

$$if \begin{cases} \gamma < 0.2 \\ \gamma > 3.0 \end{cases} \quad then \begin{cases} P_{new} = 2P_{old} \\ P_{new} = 0.5P_{old} \end{cases}$$

The algorithm monitors the strain rate of each element during the deforming of the blank. For this work, the algorithm was programmed to neglect the elements with the highest 5% of strain rates and averaged the elements with the next highest 20% of strain rates. This average was used to adjust the pressure at predefined increments of time such that the average strain rate was maintained at a predefined target strain rate of $0.001s^{-1}$ throughout the forming cycle.

5.7. Simulation Results and Discussion

The resulting pressure profiles and corresponding creep strain rate variation history is shown in [Figure 5.20](#) and [Figure 5.21](#) respectively for the initial and revised models. The pressure profile of the *Revised Model* contained the following distinctive stages. In the first phase, a very gradual pressure increase is noted up to ~25% of the forming time where an inflection in the slope of the curve appears when the sheet material first touches the die bottom, and sticking friction occurs. During this initial forming period, the sheet is slowly warping and stretching around the die entry radii. The ABAQUS built-in pressure algorithm controls the target strain rate in the corresponding regions of the sheet which can also be observed in the corresponding strain rate variation history ([Figure 5.21](#)). The rise in pressure is due to the rapid increase in the stresses, mainly due to isotropic hardening within the sheet material (Cheong et al, 2000). As the sheet now touches the bottom and sides of the die, more pressure will be required up to the termination of forming. Hence an increase in pressure is observed at 50% of the forming time. After $T/T_f=0.6$, the deformation is restricted locally, and the effective area on which the gas pressure is applied becomes smaller.

The pressure profile of the *Initial Model* (Figure 5.20) also has three distinctive stages. However there are relatively high oscillations in the pressure profile generated with *Initial Model*. The possible explanation for this is that the *ABAQUS Built-in Pressure Control Algorithm* is based on the maximum creep strain, so to maintain the creep strain rate close to the target strain rate the pressure is trying to adjust. In other words, as the *Initial Model* is not capturing the actual hardness so due to lower stresses, the slight increase in pressure resulted in high strain rate so to adjust it to the target strain rate, ABAQUS algorithm lowers the pressure almost instantly which subsequently produce oscillation in the pressure values.

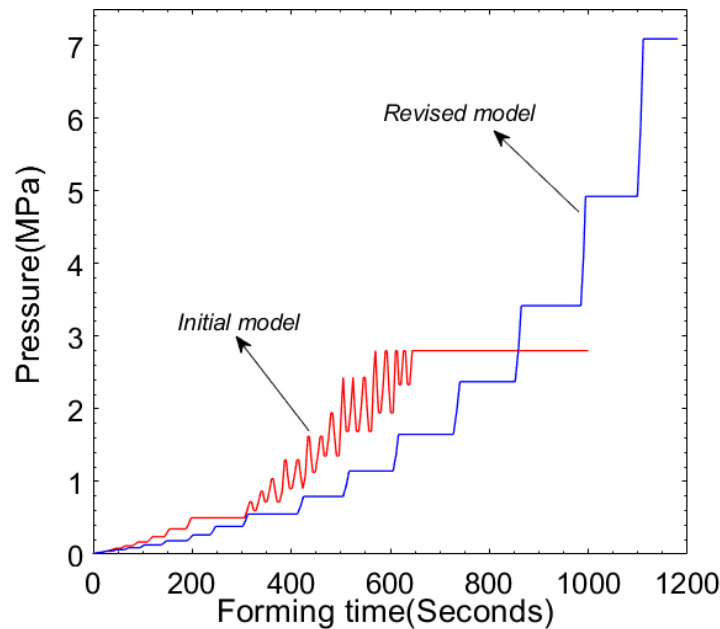


Figure 5. 20. Pressure profile for superplastic blow forming

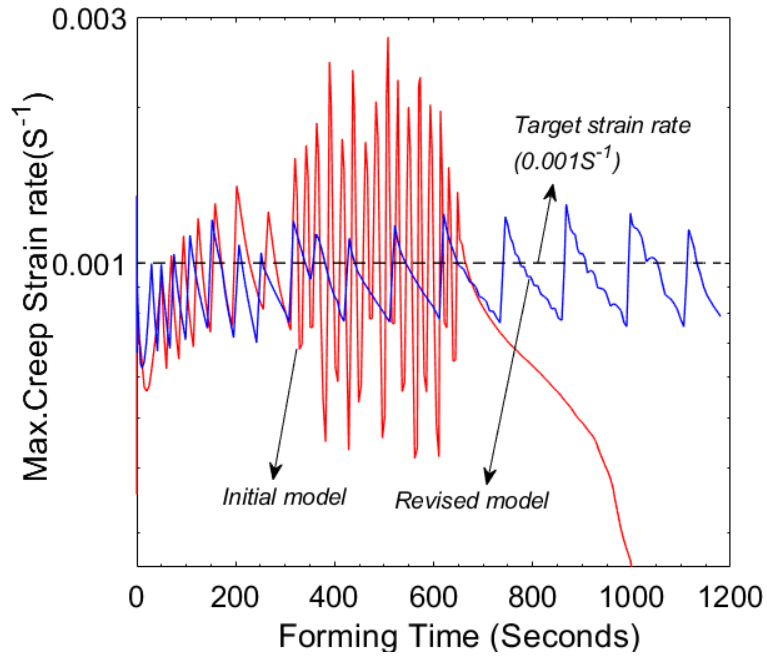


Figure 5. 21. Strain rate variation history

Further explanation of the two models are illustrated by comparing the corresponding ISVs (Internal State Variables) of both models. The resulted contours for von-Mises stresses, creep equivalent strain (CEEQ), dislocation density and softening are presented in Figure 5.22, Figure 5.23, Figure 5.24 and Figure 5.25 respectively.

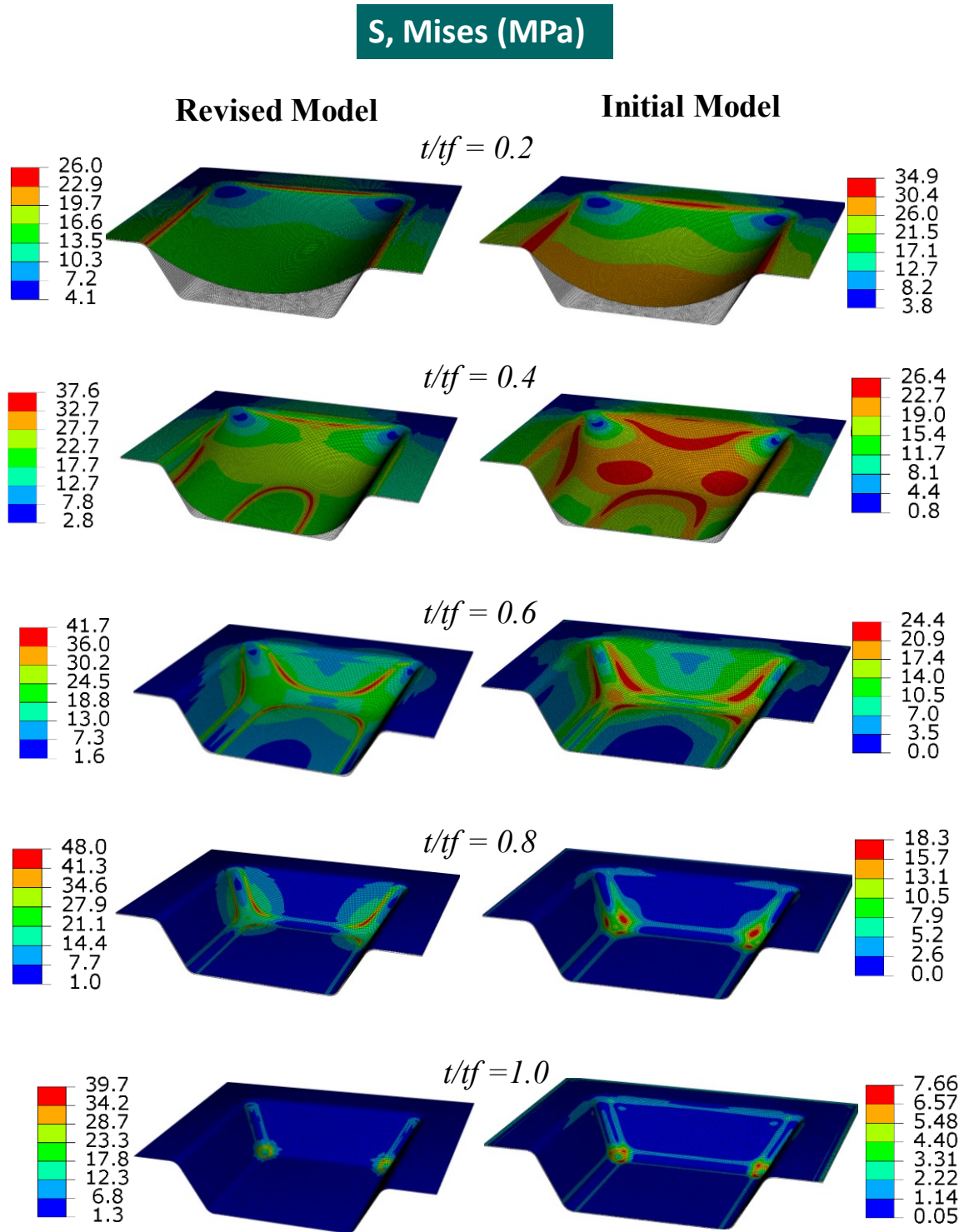


Figure 5. 22. Variation of von-Mises stress for stress for deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$.

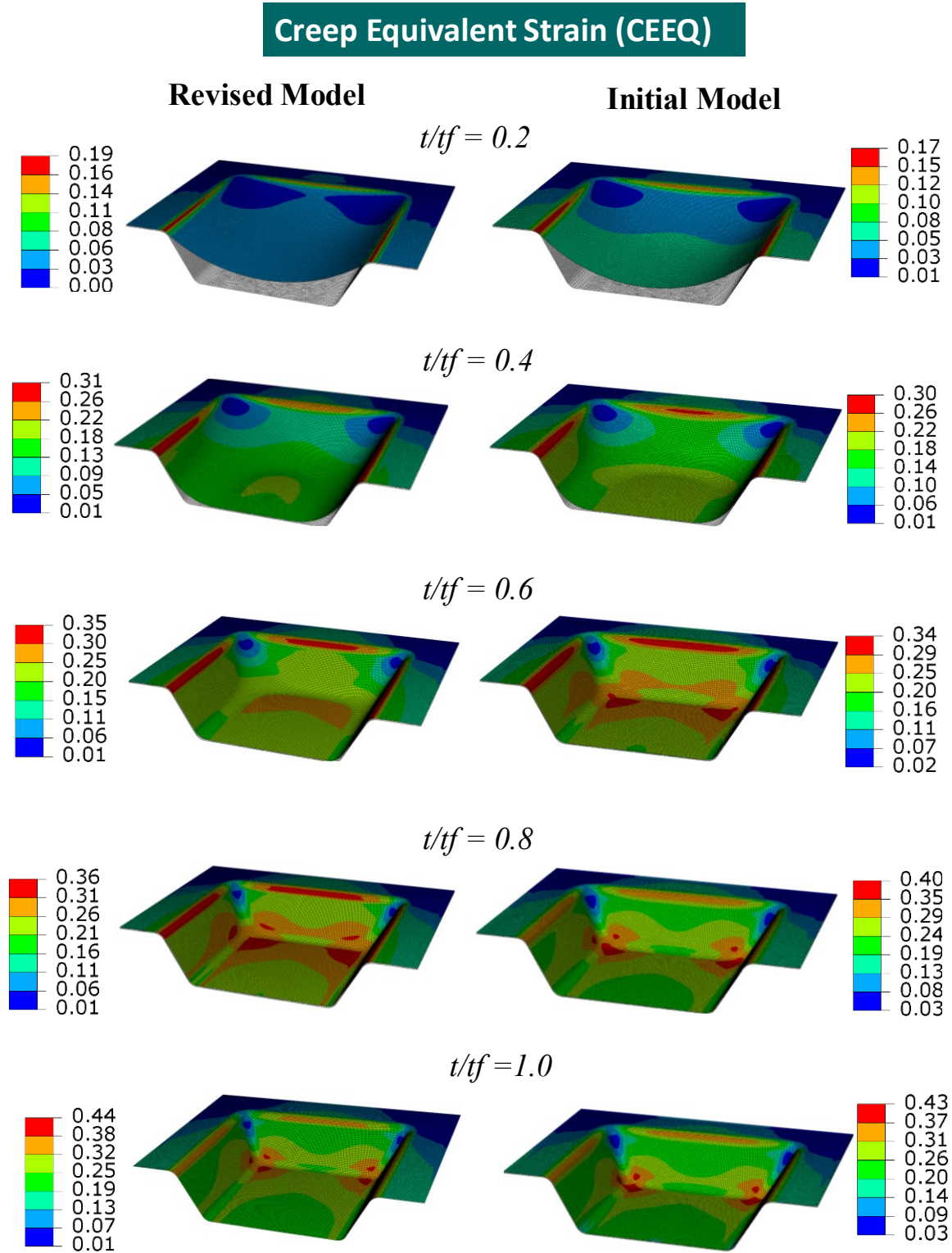


Figure 5. 23. Variation of creep equivalent strain (CEEQ) for deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$.

$\bar{\rho}$, Dislocation Density (SDV2)

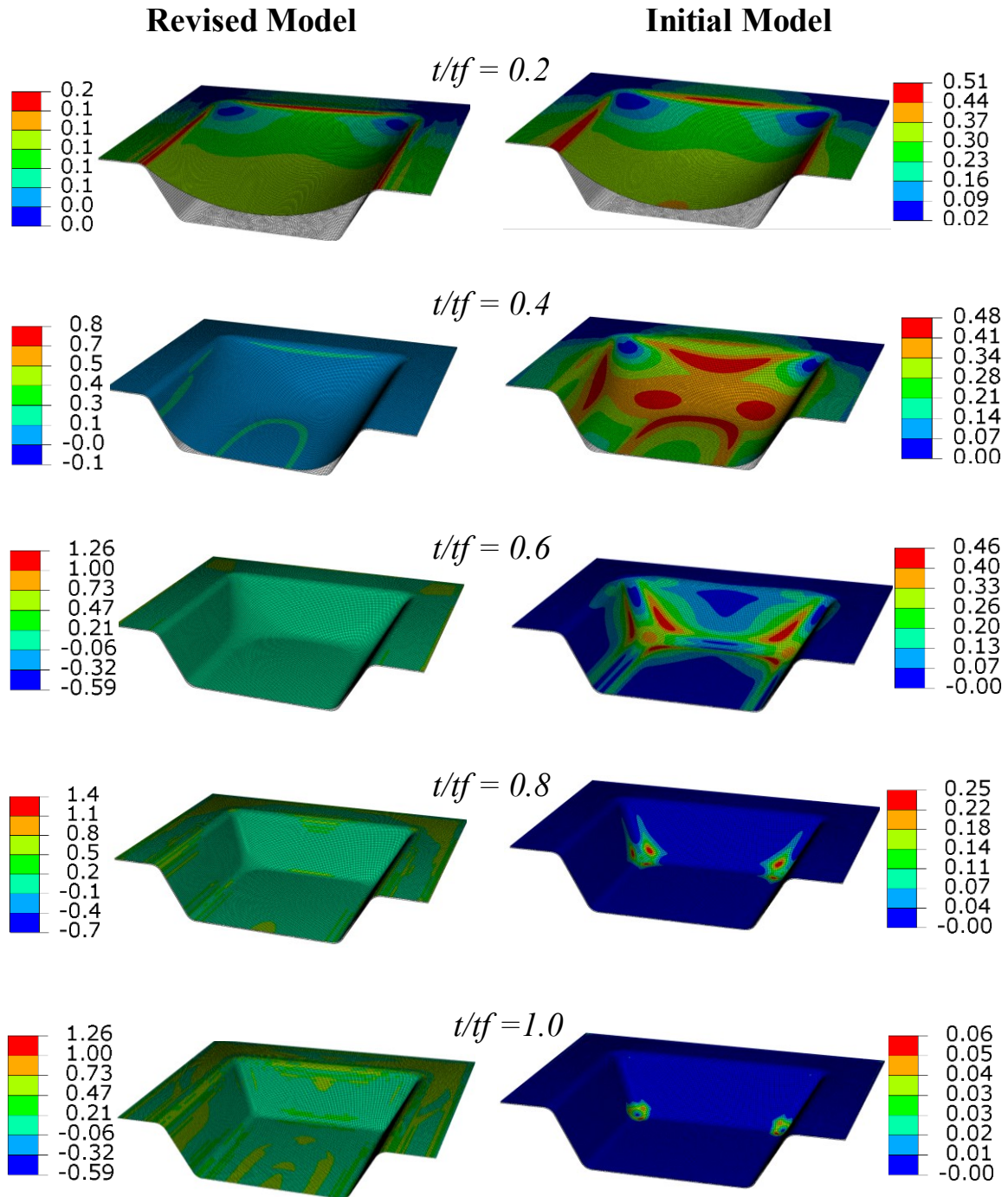


Figure 5. 24. Variation of dislocation density for deformation condition 880°C/0.001s⁻¹

S, Softening (SDV3)

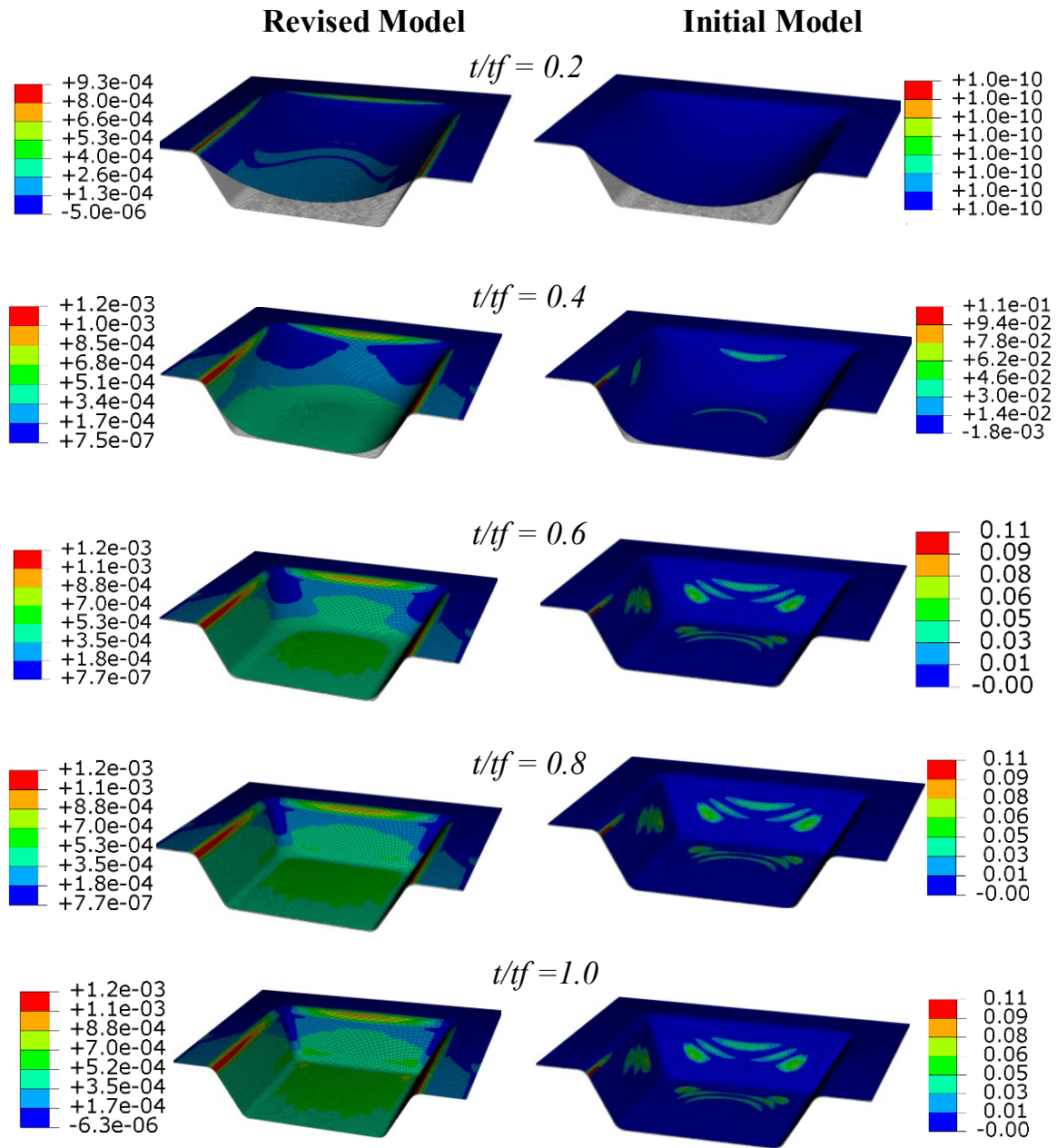


Figure 5. 25. Variation of softening for deformation condition 880°C/0.001s⁻¹.

The dislocation density for the *Revised Model* is based on the GND calculation as explained in Chapter 3 and 4. From tensile tests flow curve (Figure 5.5), it can be observed that at deformation conditions $880^{\circ}\text{C}/0.001\text{s}^{-1}$, the dislocation are reduced at very early stage of deformation and such dislocation variation phenomena is well predicted by the *Revised Model* (Figure 5.24). At the early stage of forming ($t/t_f = 0.2$), the maximum dislocation density is reduced to 0.2. These results are rationale as during the whole forming period at deformation conditions $880^{\circ}\text{C}/0.001\text{s}^{-1}$, there will diffusion-based activities which help to reduce the dislocation density. In case of the *Initial model*, the dislocation density variation is based on the stresses which will be explained in the upcoming section

As predicted by the tensile test results it was expected to have grain growth at deformation conditions $880^{\circ}\text{C}/0.001\text{s}^{-1}$, the same can also be observed in the evolution of grain size as predicted by the Revised Model. The grain size contours (Figure 5.26) are compatible with the corresponding stress, strain and softening contours (Figure 5.22, 5.23 & 5.25 respectively).

d, Grain size - μm (SDV5)

Revised Model

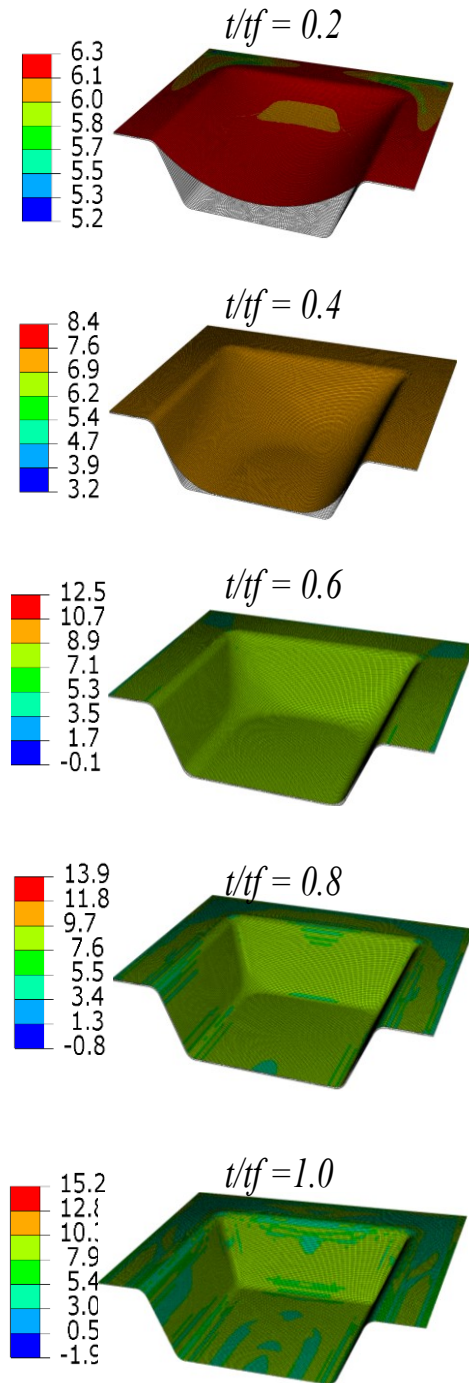


Figure 5. 26. Variation of grain size for deformation condition $880^\circ\text{C}/0.001\text{s}^{-1}$.

The variation of stress for the *Initial Model* and *Revised Model* in the light of the ISVs are presented in Figure 5.27 and 5.28, respectively. For the *Initial Model*, it can be seen that the stress contours are almost similar to dislocation contours which means that the stress state is maximum when the dislocation density is maximum. The high dislocation density of 0.51 at $t/t_f = 0.2$ corresponds to maximum stress of 34.9 MPa while in case of *Revised Model* the initial contour at $t/t_f = 0.2$ stress contours are similar to dislocation density but as the forming time proceeds and the grain size grows, the stress increases which means the stress increase is now related to the grain size. The same kind of observation is made in the uniaxial tensile tests for deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$, so in the light of these observations it can be concluded that the *Revised Model* can accurately capture the deformation mechanism of superplasticity at given condition.

Initial Model

$\bar{\rho}$, Dislocation Density (SDV2)

S, Mises (MPa)

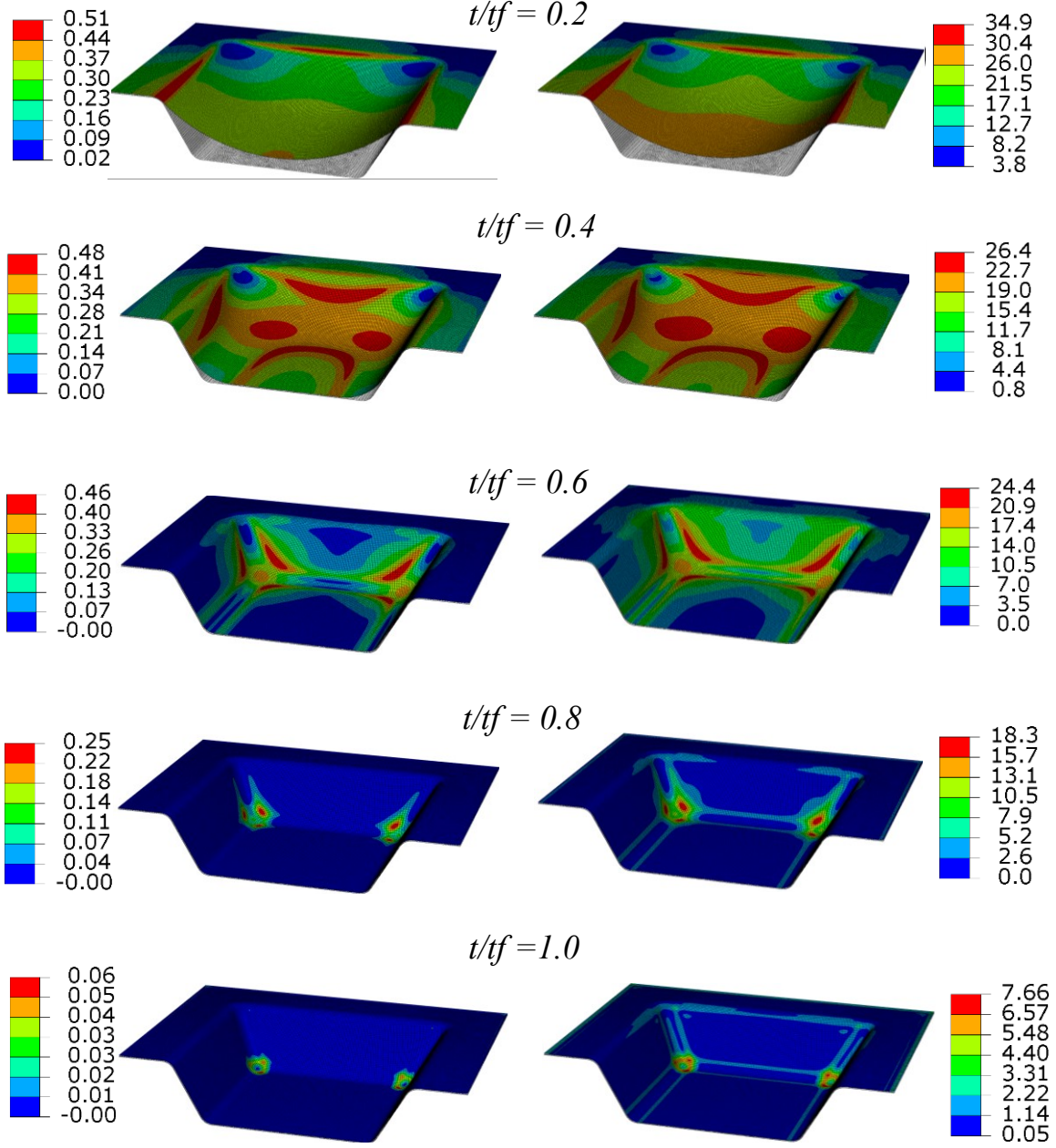


Figure 5. 27. Variation of stress with respect to dislocation density for *Initial Model* at deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$.

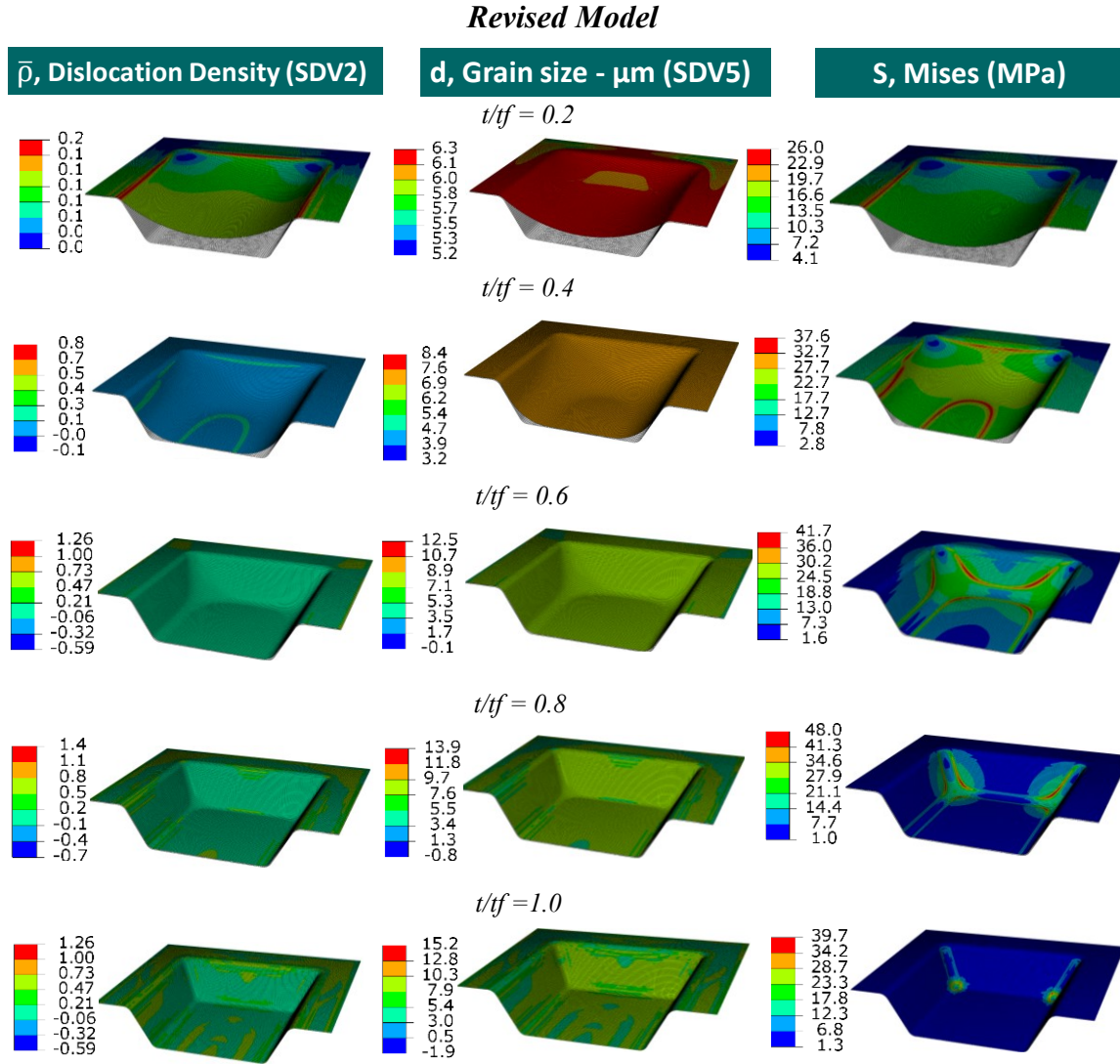


Figure 5. 28. Variation of stress with respect to dislocation density & grain size for *Revised Model* at deformation condition $880^{\circ}\text{C}/0.001\text{s}^{-1}$.

The thickness variation for both models are presented in Figure 5.29. The predicted thickness variation results are almost compatible for both models. However *Initial Model* is not providing microstructure details of the formed component while the *Revised Model* provides the can predict the microstructure details as it is calibrated against the experimentally validated ISVs data for grain size and dislocation density so it can be concluded that the formed components based on *Revised Model* is more reliable. Based on the *Revised Model*, the generated pressure profile predicts the average grain size of 9 μm and almost negligible dislocation density for the given simulated component.

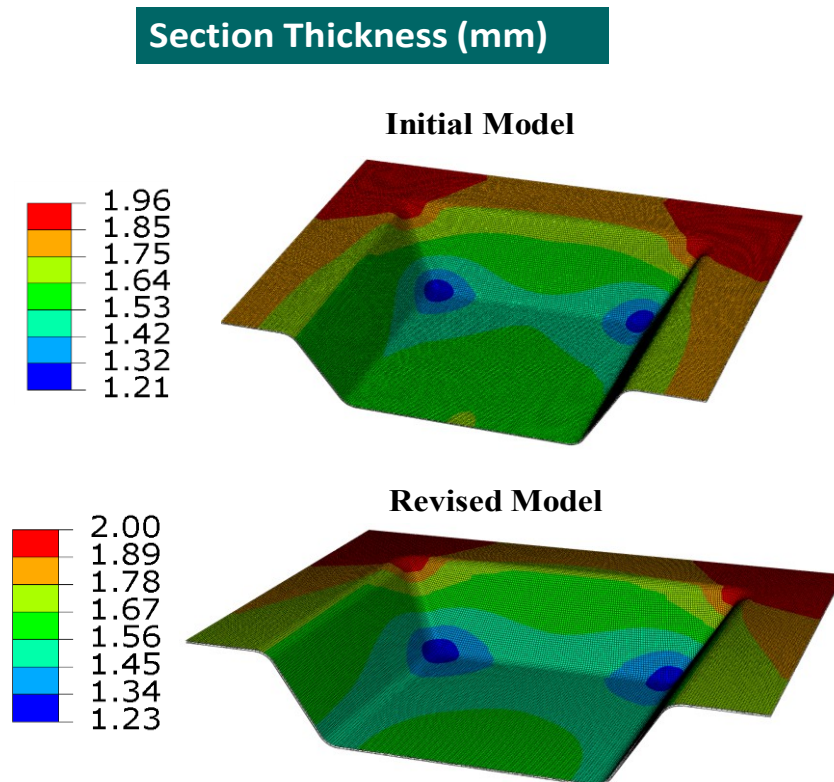


Figure 5. 29. Thickness distribution in the deformed simulated part predicted by Initial Model and Revised Model

5.8. Conclusion

The objective of this chapter is to develop a new viscoplastic constitutive model to accurately simulate the superplastic behaviour of TA15 alloys. A range of thermo-mechanical tensile tests was conducted to characterize the superplastic behaviour in the temperature range of 880– 940 °C and strain-rates of $0.0005 - 0.01\text{s}^{-1}$. Following conclusion can be drawn:

- i. The alloy demonstrated excellent superplasticity with percentage elongation above 700% at all testing conditions. The maximum elongation of 1470% is achieved for the deformation condition 880 °C/ 0.001s^{-1} .
- ii. The microstructure remains equiaxed even after large deformations; moreover, the texture remains random. It is suggested that grain boundary sliding is responsible for deformation in the superplastic regime.
- iii. At a higher strain rate of 0.01s^{-1} for all the deformation temperatures, there is grain refinement, while for the lower strain rates (0.001s^{-1} & 0.0005s^{-1}) there is grain growth.
- iv. At a higher strain rate of 0.01s^{-1} for all the deformation temperatures, the formation of voids and cavities are observed, so keeping in view the integrity of the SPF part under real-world service conditions, this strain rate with an initial grain size of $3.74\text{ }\mu\text{m}$ is not recommended for the industrial application. Nevertheless, at lower strain rates, fracture failures with no cavities nucleation are observed. Based on these observations, the fatigue life of an SPF part and industrial constrains of the SPF process; the deformation 880 °C/ 0.001s^{-1} is recommended for the industry to produce an SPF part with a grain size of $10\text{ }\mu\text{m}$.
- v. The understanding of microstructure evolution is translated into a set of viscoplastic constitutive equations that describe the observed phenomena. A mechanism-based internal state variable (ISVs) model is proposed to simulate the macroscopic behaviour of the material. The model involves a number of dynamic equations that represent the underlying deformation mechanisms and are based on the micromechanical deformation modes, which have been shown to be operating *e.g.* softening, grain size, and void evolution.

- vi. A gradient-based optimization method is applied to determine the material constants of the proposed equations. The determined material constants can reasonably predict the morphology of the flow curves at the given range of temperature and strain rate.
- vii. To validate the accuracy of the established constitutive model, the finite element simulation of the superplastic gas-blow forming of TA15 sheet of the industrially relevant problem is performed. The simulation results of the *Revised Model* are compared with the *Initial Model* (which does not involve microstructural parameters), and pressure profile is generated using both models to explain the importance of the Revised Model, which keeps the microstructure stable. It is demonstrated that the pressure profile generated based on the *Revised Model* maintains the microstructure stable and keeps the variation in the strain rate closer to the target strain rate, which results in a superplastically formed part with a more uniform thickness distribution.

Chapter 6

Conclusion and Future Work

The work presented in this thesis involves a systematic combination of experimental and computational techniques for investigating the superplastic deformation in a near- α titanium alloy. First, the superplastic phenomenon has been measured in the TA15 (Ti–6Al–2Zr–1Mo–1V) titanium alloy by carrying out uniaxial interrupted tensile tests at testing conditions of $880^{\circ}\text{C}/0.01\text{s}^{-1}$, $900^{\circ}\text{C}/0.01\text{s}^{-1}$, $880^{\circ}\text{C}/0.001\text{s}^{-1}$, and $920^{\circ}\text{C}/0.0005\text{s}^{-1}$. The interrupted tensile test data has been analysed in terms of deformation kinetics, microstructure, and texture evolution. The results provided useful information for understanding the dominant deformation mechanism under given testing conditions. Second, to further understand the microstructure and texture evolution during superplastic deformation in terms of static and dynamic annealing, comparative SEM and EBSD analysis of the samples captured from the grip and gauge regions of interrupted tensile test specimens. The differing microstructure evolution mechanism in the grip and gauge regions provided useful insight to understand the microstructure evolution in superplastic deformation. Third, the understanding of microstructure evolution and flow behaviour is translated into a set of viscoplastic constitutive equations which represent the observed phenomena. Finally, to validate the accuracy of the established constitutive model, the finite element simulation of the superplastic gas-blow forming of TA15 sheet of the industrially relevant problem is performed.

In addition to the conclusions made at the end of each experimental and results chapter, the following major outcome of the study can be presented:

- i. TA15 alloy exhibits excellent superplastic behaviour in the temperature range of 880 – 940°C with strain rates of 0.0005 – 0.01s^{-1} , and all of the tensile elongations exceed 700% . The maximum elongation of 1470% is achieved at 880°C , with a strain rate of 0.001s^{-1} .

- ii. From the stress-strain curves, it is observed that as compared to the deformation temperature, the degree of superplasticity (% elongation) is predominantly depended on the strain rate.
- iii. At higher strain rate 0.01s^{-1} , there is no significant increase in the grain size, while at lower strain rates 0.0005s^{-1} and 0.001s^{-1} , significant grain growth is observed with increasing temperature, but the grains still keep equiaxed-shape.
- iv. The fraction of β -phase is accelerated during deformation and contributed to the enhancement of superplasticity.
- v. For the same temperature of 880°C at higher strain rate 0.01s^{-1} , the cavities are observed, and the type of failure is ductile, while for the slower strain rate, no cavities are observed, and the failure type is brittle *e.g.* intergranular fracture.
- vi. The extensive strain hardening at high temperatures and low strain rates is attributed to dynamic grain growth.
- vii. The continuous weakening of the initial rolling texture, the changes of crystal orientation suggest that the deformation mechanism is dominated by grain boundary sliding (GBS), during all deformation conditions of $880^\circ\text{C}/0.01\text{s}^{-1}$, $900^\circ\text{C}/0.01\text{s}^{-1}$, $880^\circ\text{C}/0.001\text{s}^{-1}$, and $920^\circ\text{C}/0.0005\text{s}^{-1}$.
- viii. The processing history of the initial material strongly affects the initiation of GBS. If the initial material is dislocation free then during the initial deformation stages, the GBS will be the dominated deformation mechanism, as confirmed by the deformation condition of $880^\circ\text{C}/0.01\text{s}^{-1}$ and $920^\circ\text{C}/0.01\text{s}^{-1}$ but at the later stages, there will be considerable grain growth.
- ix. GBS accommodated by dislocation glide/climb should be dominant at high temperature and low strain rate ($920^\circ\text{C} / 0.0005\text{s}^{-1}$), whereas for the optimum superplastic conditions ($880^\circ\text{C}/0.001\text{s}^{-1}$) with the maximum elongation of 1470%, the value of stress exponent and activation energy is closely related to GBS accommodated by grain boundary diffusion.
- x. For the higher strain rate of 0.01s^{-1} , the GND and the rolling texture was increased till a strain of 0.084 and 0.174 at 880°C , and 900°C respectively confirms that deformation mechanism changes with true strain from plastic to superplastic.

- xi. Both static and dynamic annealing caused an increase in the volume fraction of β -phase formation though, in the case of static annealing, the increase in the volume fraction of β -phase was lower than in dynamic annealing. Similarly, in both cases, an increase in the volume fraction of β -phase was observed with increasing deformation temperature or decreasing strain rate.
- xii. In all testing conditions, both the static (grip region) and dynamic (gauge region) grain growth were observed during the deformation. At the same time, a mixed trend of grain growth was observed in grip and gauge regions.
- xiii. Static annealing did not affect the grain shape and the original rolling texture but caused grain growth, indicating an absence of discontinuous recrystallization.
- xiv. The texture observed in the static annealing (grip regions) was quite different from that found in the dynamic annealing (gauge regions) under all testing conditions. Nevertheless, the maximum intensity of the texture observed in the grip regions was slightly changed from that of as-received material, though the initial rolling texture was maintained for all grip regions, while for the deformed regions, the texture was randomized, which is an attribute of superplastic deformation.
- xv. The GND values of gauge regions were generally smaller than that of the grip regions for all testing conditions. Generally, it was revealed that for both grip and gauge regions, the decrease in dislocation density was relatively greatly affected by strain rate (annealing time) while the effect of change in deformation temperature on the evolution of GND was trivial.
- xvi. Both grip and gauge regions experienced an increase of the fraction of HAGBs under all testing conditions. However, the evolution of the fraction of HAGBs in gauge regions was quite different from the corresponding grip regions indicating that the microstructure evolution mechanisms were different.
- xvii. At a higher strain rate of 0.01s^{-1} for all the deformation temperatures, the formation of voids and cavities are observed, so keeping in view the integrity of the SPF part in real-world application, this strain rate with an initial grain size of $3.74\text{ }\mu\text{m}$ is not recommended for the industrial use. Nevertheless, at lower strain rates, fracture failures with no cavities nucleation are observed. Based on these observations, the fatigue life

of an SPF part and industrial constraints of the SPF process; the deformation $880^{\circ}\text{C}/0.001\text{s}^{-1}$ is recommended for the industry to produce an SPF part with $10\text{ }\mu\text{m}$.

- xviii. Microstructural changes during deformation have been modelled using a rate-dependent internal state variable (ISV) model. Changes in dislocation density, grain size, and cavity/void formation are included explicitly. To validate the accuracy of the established constitutive model, the finite element simulation of the superplastic gas-blow forming of TA15 sheet of the industrially relevant problem is performed. The simulation results of the *Revised Model* are compared with the *Initial Model* (which does not involve microstructural parameters), and pressure profile is generated using both models to explain the importance of the Revised Model, which keeps the microstructure stable. It is demonstrated that the pressure profile generated based on the *Revised Model* maintains the microstructure stable and keeps the variation in the strain rate closer to the target strain rate, which results in a superplastically formed part with a more uniform thickness distribution.

Future work recommendations

The outcomes of this thesis have a number of implications for further research. Therefore, to extend this research, the following future works are recommended:

- As the maximum elongation was observed at 880°C which was the lowest deformation temperature considered in this work, therefore to reduce the cost and energy consumption and to enhance the die life, the temperature window of the current study should be extended to lower deformation temperature *e.g.* 750°C - 850°C . Given the initial grain size of $3.74\text{ }\mu\text{m}$ in this study, it is possible to have a lower window of deformation temperature to obtain superplastic characteristics (at least 400% elongation) of the alloy.
- Since varying the initial grain size of the material can significantly alter the optimal superplastic window of the material, it is, therefore, possible to further study the effect of grain size (in the range 1 to $10\text{ }\mu\text{m}$) on the superplasticity with the objective to achieve industrial acceptable superplastic elongation in TA15 alloy with a grain size higher than $3.74\text{ }\mu\text{m}$.

- A constitutive model based on experimental data generated in this study (*e.g.* GND, β -phase evaluation, and the fraction of DRX) can provide an explicit representation of the microstructure evolution during superplastic deformation and would offer a significant advantage for improving the internal state variable model. Therefore, the proposed constitutive model could be modified to include a description of said parameters. This would give a closer representation of the real microstructure state.

- One of the most important technological developments of the superplastic forming process was the introduction of diffusion bonding (DB), which is now become a well-established process for the manufacture of SPF components such as ducts, jet nozzles, turbine and compressor blades. However, usually undesired thickness variation of ribs, which leads to poor structural integrity, has been observed during the manufacturing of titanium alloy multilayer sandwich structure by SPF/DB. Hence, to optimize the forming window for the SPF of TA15 combined with diffusion bonding in an effective manner, more fundamental research is necessary.

- The predicted grain size of the simulated component under the recommended optimal SPF condition is 9 μm , and the grain size of up to 10 μm of the SPF component is considered good for the component fatigue life. However, there is no comprehensive study available to examine the effect of SPF condition on the component fatigue life. It is therefore required to investigate the fatigue life of the SPF components (as per real-world application) formed at different deformation conditions.

- Although it is claimed that TA15 alloy can retain its mechanical properties at high temperatures up to 550 °C, but there are no systematic scientific studies in the academic literature that examine the service temperature of TA15. Therefore, an in-depth investigation is needed to assess the microstructure stability of TA15 above 500 °C.

- In addition to various microstructure-related parameters, this study examines the role of GND in the TA15 superplastic deformation mechanism using EBSD data. However, the EBSD analysis does not offer enough detail about the mechanism of (i) the mobile dislocation density, (ii) the dislocation density stored inside the cells, and (iii) the dislocation density stored on the cell walls. Transmission electron microscopy (TEM) can provide more comprehensive detail on this phenomenon during superplastic deformation. However, one of the major limitations of TEM analyses is that it focuses on a tiny area (a spot in nanometer) of the test sample. Therefore, to represent the overall dislocation mechanism in a given sample, a TEM analysis of the deformed samples from this study can be conducted, and then coupling the TEM data with EBSD data of this study can provide useful insight for understanding the dislocation mechanism in TA15.

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