



Understanding effects of deformation parameters on dynamic recrystallization-dependent superplasticity in an Al-Cu-Li alloy

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ABSTRACT

Aluminum alloys with initial unrecrystallized structures generally exhibit better superplasticity and are produced more efficiently and cost-effectively than fully recrystallized ones. However, the underlying recrystallization and deformation mechanisms of dynamic recrystallization (DRX)-dependent superplastic aluminium alloys under varying deformation parameters are not yet fully understood. This study investigates the effects of deformation parameters, Al₃Zr dispersoids, and coarse secondary particles on DRX and superplasticity in an Al-Cu-Li alloy. The alloy achieves a maximum elongation of 780 % at 430 °C and 0.002 s⁻¹, primarily due to continuous dynamic recrystallization (CDRX) and grain boundary sliding (GBS). Under optimal conditions, deformed grains transform into equiaxed recrystallized grains through sub-grain rotation and coalescence, with GBS dominating subsequent deformation. Lower Zener-Hollomon parameter (lnZ) conditions promote dynamic recovery (DRV) and sub-grain growth, hindering grain refinement and superplastic deformation. Conversely, higher lnZ values inhibit recrystallization due to insufficient thermal driving force and lower DRV, resulting in retained banded grains and reduced elongation. Cu-rich secondary phases enhance CDRX but lose efficacy with their dissolution and coarsening at low lnZ conditions. This work provides insights into DRX-dependent superplastic mechanisms and offers guidance for optimizing deformation parameters to enhance the performance of aluminum alloys.

1. Introduction

Advanced aluminium (Al) alloys, due to their improved relationship of strength, ductility, and low cost, have been widely used in the aviation, transportation, and electronics industries [1,2]. Superplastic forming (SPF), which reduces deformation resistance and enhances forming quality, is ideal for producing thin-walled components with complex shapes [3,4]. The combination of SPF and Al alloys can reduce structural weight by over 20 % and cut costs by more than 60 %, exhibiting fascinating application prospects [5]. Therefore, continuous research has been devoted to the development of superplastic Al alloys and their superplastic forming technologies.

Superplasticity is typically achieved in fine-grained alloys at high temperatures (> 0.5 melting point) and low strain rates (10⁻⁴–10⁻¹ s⁻¹) [6,7]. The key to achieving excellent superplasticity lies in developing fine, equiaxed grains, which enable extensive grain boundary sliding (GBS) during the process with concurrent straining at high temperatures [8,9]. For Al alloy sheets, fine-grained microstructures can be obtained

through two different routes following thermomechanical processing (TMP): static recrystallization or dynamic recrystallization (DRX) during hot deformation [3,10]. In the first case, near-equiaxed grains are obtained after static annealing, and the recrystallized (RXed) grains undergo growth, rotation, and elongation during subsequent superplastic deformation [11,12]. In the second case, for alloys with initial unrecrystallized structures, the deformed grains transform into fine equiaxed grains through DRX during the early stages of superplastic deformation and subsequently achieve excellent superplasticity [10,13,14]. These superplastic Al alloys can be produced without final annealing, allowing for a shortened preparation process and reduced production costs. And it is worth noting that these Al alloys often exhibit better superplasticity, characterized by greater fracture elongation at higher strain rates and/or lower deformation temperatures [14–16].

The superplastic deformation process of Al alloys with initial unrecrystallized structures can be divided into two stages based on their grain structure. At the initial deformation stage, the alloys undergo complex microstructure evolution such as dynamic recovery (DRV),

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DRX, and dynamic grain growth, in which DRX plays a vital role in lateral grain structure and superplasticity [13,15,17]. DRX, a grain refinement method during hot deformation, can be classified into three types [18–20]: (a) discontinuous dynamic recrystallization (DDRX), (b) continuous dynamic recrystallization (CDRX), and (c) geometric dynamic recrystallization (GDRX). DRX can be promoted by increasing temperature but hindered by a higher strain rate [21], with its mechanism varying depending on deformation parameters [22,23]. DDRX is difficult to occur in Al alloys due to their high stacking fault energy (HFE) [19]. However, at low temperatures and high strain rates, DDRX is found to be the dominant DRX mechanism; with increasing temperatures, CDRX and GDRX tend to increase due to the enhanced capacity for DRV [22,23]. Some researchers [24,25] noticed that the occurrence of CDRX can be accelerated by increasing strain rate. Therefore, for a superplastic Al alloy with initial unrecrystallized structures, it is essential to manipulate the deformation parameters to obtain finer recrystallized grains, which is vital for subsequent superplastic deformation.

Due to the changes in grain structure, the deformation mechanism of Al alloys with initial unrecrystallized grains typically evolves throughout the superplastic deformation process [3,17]. In the initial deformation stage, the banded grains are not conducive to GBS, so intragranular dislocation sliding (IDS) [13,26] or diffusion creep (DC) [27,28] is founded to be the dominant deformation mechanism. In the second stage, finer equiaxed grains with relatively random misorientation are formed through DRX, significantly activating GBS and causing it to dominate the superplastic deformation process [13,26]. In addition to the grain structure, deformation parameters such as temperature and strain rate have a great impact on the superplastic deformation mechanisms. Normally, the GBS is enhanced with an increase in temperature [29], and a lower strain rate is beneficial for the GBS [24,30]. However, grain growth is more likely to occur at low strain rates or higher temperatures, which weakens GBS and may reduce elongation.

As summarized above, recrystallization and deformation mechanisms are complex and closely related to deformation parameters. These behaviours are interdependent and competitive, collectively influencing the superplasticity of Al alloys. This study aims to investigate the effects of temperature and strain rate on the deformation behaviour and underlying mechanisms of a novel superplastic Al-Cu-Li alloy, which exhibits dynamic recrystallization-dependent superplasticity while maintaining an optimal balance between high strength and low density [13,31]. The DRX mechanisms under various deformation parameters are investigated by using EBSD and TEM. Additionally, the role of Al_3Zr dispersoids and the evolution of coarse secondary phases, along with their impact on DRX and superplastic deformation, are analysed. Furthermore, the underlying superplastic deformation mechanisms are discussed. This work may provide valuable insights for selecting deformation parameters and designing new superplastic Al alloys.

2. Materials and methodology

2.1. Material preparation

The studied 2A97 Al-Cu-Li alloy (Al-3.4Cu-1.5Li-0.5Zn-0.4 Mg-0.14Zr-0.3Mn, wt.%) was received initially as a 20 mm thick commercial hot rolled plate. The superplastic sheet with a thickness of 2.5 mm was prepared via the following TMP [13]: Following the solution heat treatment at 520 °C for 4 h and water quench, the plate was then cold rolled with a reduction of 20 % (at room temperature). After that, a two-stage over-ageing (300 °C × 24 h + 400 °C × 24 h) was conducted to obtain coarse spherical secondary phases. Finally, two steps of warm rolling at 300 °C were performed, producing the superplastic sheet with a thickness of 2.5 mm.

2.2. High temperature tensile test

The samples for superplastic deformation with a gauge length of 10

mm and width of 6 mm were cut along the rolling direction (RD) from the sheet via electrical discharge machining. The uniaxial tensile was performed on the RWS50 tensile machine at 400–460 °C within the initial strain rate range of 0.0005–0.01 s⁻¹. Samples were kept at the target temperature for 15 mins before tensile to ensure uniform distribution of temperature. After being deformed to fracture or target strains, the sample was taken off within 6 s and then quenched in water immediately to preserve the microstructure.

2.3. Microstructure characterization

The samples for SEM observation were ground and then polished mechanically, the SEM scanning was conducted on a TESCAN MIRA4 LMH scanning electron microscope operated at 15 kV. Electro-polish was taken out for preparing EBSD samples, which was conducted in a 1:9 HClO₄/C₂H₅OH acid solution at 20 v for 6 s at room temperature. TESCAN MIRA4 LMH scanning electron microscope equipped with an Oxford Instrument EBSD detector was used for the EBSD test, with an operating voltage of 30 kV and a step size of 0.3 μm. The EBSD data was processed using Channel 5 software, the grain boundaries with misorientation between 2° and 15° were defined as low-angle grain boundaries (LAGBs), and that of larger than 15° were defined as high-angle grain boundaries (HAGBs). The grains were classified into recrystallized, substructure, and deformed grains according to their grain average misorientation (GAM) [32], corresponding to the GAM value less than 1.55°, between 1.55° to 7.5°, larger than 7.5°, respectively.

To quantitatively compare the relative dislocation density of different samples, the geometrically necessary dislocation (GND) was obtained based on the EBSD-KAM maps, by using the following equation [33]:

$$\rho_{\text{GND}} = 2KAM_{\text{av}}/(\mu b) \quad (1)$$

where ρ_{GND} is the geometrically necessary dislocation density, KAM_{av} is the average value of local misorientation angle, μ is the step size, and b is the Burgers vector (0.286 nm for aluminium alloy).

The average grain size was calculated by using the intercept length method [31,34] from the EBSD maps with the following equation:

$$d_{\text{AVR}} = (d_{\text{ND}} \times d_{\text{RD}}^2)^{1/3} \quad (2)$$

where d_{ND} is the average grain size along the nominal direction, d_{RD} is the average grain size along the rolling direction. By calculating different types of grain boundaries, such as LAGBs, RXed grain boundaries, and HAGBs, the average sub-grain size, average RXed grain size, and average grain size can be obtained.

The samples for TEM observation were punched into wafers with a diameter of 3 mm after mechanically thinning to ~ 60 μm, then the wafers were further processed via ion-beam thinning. The TEM was performed on a Thermo-Scientific Talos F200X transmission electron microscope with an accelerating voltage of 300 kV.

3. Results

3.1. Initial microstructures of 2A97 Al-Cu-Li alloy

Fig. 1 shows the initial microstructures of the tested 2A97 Al-Cu-Li alloy. After TMP and holding at 430 °C for 15 mins before superplastic tensile, the alloy still exhibits a deformed banded grain structure (Fig. 1a) with a strong rolling texture (Fig. 1b). The deformed grains contain a high fraction (45.0 %) of LAGBs (Fig. 1c). A small number of recrystallized grains can be seen, which are formed during the prior hot rolling process. As shown in Fig. 1d, micron-sized phases are found distributed evenly in the matrix, these Cu-rich phases (T_1 and T_2) were coarsening and nodulizing at the prior over-ageing process [31]. Furthermore, Al_3Zr dispersoids are observed and shown in Fig. 1f.

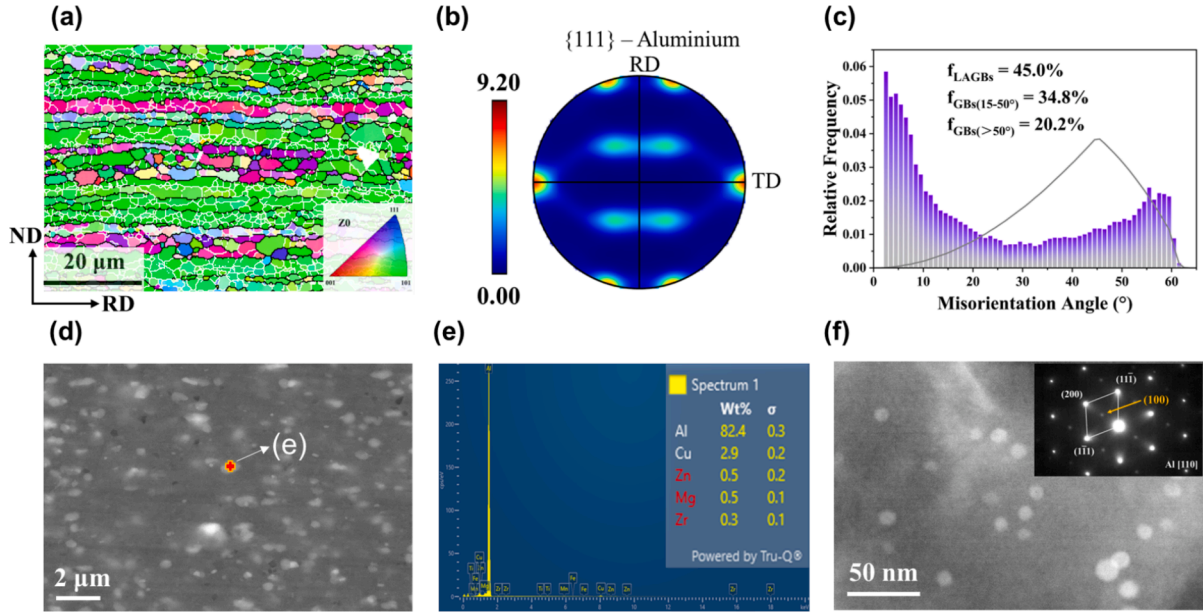


Fig. 1. Microstructures of the superplastic 2A97 Al-Cu-Li alloy: (a) EBSD inverse pole figure (IPF) map, (b) Pole figure, (c) Distribution of grain boundary misorientation, (d) Backscattered electron (BSE) image, (e) EDS pattern of coarse secondary phase, and (f) HAADF-STEM image of the dispersoids within the grain and corresponding selected area diffraction pattern.

3.2. Superplastic deformation behaviour of 2A97 Al-Cu-Li alloy

The true stress-true strain curves of the 2A97 Al-Cu-Li alloy deformed at the temperature range of 400 – 460 °C within the initial strain rate of 0.0005–0.01 s⁻¹ are shown in Fig. 2, the corresponding sample images are also given. It can be seen all the curves exhibit similar flow behaviours, that is, true stress increases rapidly at the initial stage and then decreases gradually until fracture. Temperature and strain rate have no obvious influence on the flow behaviour of the tested alloy except for its peak stress and elongation. Fig. 3 shows that peak stress and elongation are influenced by temperature and strain rate significantly. The variation in peak stress follows a simple trend: it decreases with increasing temperature and decreasing strain rate. In contrast, the effect of deformation parameters on elongation is more complex. Overall, the alloy exhibits superplasticity (elongation larger than 200 % [35]) under all the test conditions except for at 460 °C & 0.0005 s⁻¹. Larger superplastic elongation can be obtained when tested under 430 °C, with all the elongation higher than 450 %. Especially, the highest elongation (780 %) was obtained at 430 °C & 0.002 s⁻¹. When tested at the initial strain rate of 0.01 s⁻¹, the elongation at all selected temperatures exceeds 200 %, indicating that the alloy exhibits high strain rate superplasticity.

The strain rate sensitivity index (m) and activation energy (Q) are used here to analyse the superplastic deformation behaviour and mechanisms. The m was obtained from plots of the natural logarithmic relationship between stress and strain rate at a specific strain level, as given by the following equation [4,36]:

$$m = \partial \ln \sigma / \partial \ln \dot{\epsilon} \quad (3)$$

where σ is stress and $\dot{\epsilon}$ is strain rate.

The Q was obtained by using the following equation [3,4,37]:

$$Q = \frac{R}{m} \left. \frac{\partial \ln \sigma}{\partial (1/T)} \right|_{\dot{\epsilon}} \quad (4)$$

where R is the gas constant, m is the strain rate sensitivity index, σ is stress, T is deformation temperature, and $\dot{\epsilon}$ is strain rate.

In Fig. 4, the m values under different true strain levels and various temperatures are displayed. At the initial deformation stage (true strain

of 0.4), m_{avr} is 0.32; with increasing the true strain to 0.7 and 1.1, the m_{avr} increases to 0.36 and 0.41, respectively. In Fig. 4d, the change of m value with true strain at different deformation temperatures is provided. At 400 °C and 430 °C, the m value shows an increasing tendency with the deformation processes, whereas at 460 °C, the m value remains relatively stable. It is worth noting that, at 430 °C, the m value increases from ~ 0.3 to close to 0.5 at the true strain of ~ 1.3 , indicating that the deformation mechanism transforms into the GBS gradually.

As shown in Fig. 5a–c, the relationship of $\ln \sigma$ versus $(1/T)$ at the true strain of 0.4, 0.7, and 1.1 are plotted, respectively. The Q value can be calculated from the slope of corresponding data. The evolution of Q value with superplastic deformation at different initial strain rates is provided in Fig. 5(d). According to Fig. 5a–c, with increasing true strain from 0.4 to 1.1, the average Q value (Q_{avr}) decreases gradually from 144.8 KJ/mol to 85.4 KJ/mol. When it comes to every deformation strain, it can be found that the alloy tested at a lower initial strain rate typically shows a higher Q value. For instance, at the true strain of 1.1, the Q value increases from 63.2 KJ/mol at 0.01 s⁻¹ to 109.5 KJ/mol at 0.0005 s⁻¹ gradually. It can be noted that when tested at the initial strain rate of 0.002 s⁻¹, the Q value exhibits an increase after the true strain of about 1.1. Since the Q value is related to the deformation mechanism, its variation with different true strains and different deformation parameters reflects changes in the deformation mechanisms, which will be discussed in section 4.4.

3.3. Microstructure evolution at optimum deformation condition

To understand the microstructure evolution during superplastic deformation, the sample deformed at 430 °C and the initial strain rate of 0.002 s⁻¹ (with the largest superplastic elongation) was focused. Fig. 6 shows the EBSD IPF maps, recrystallization maps, and corresponding grain boundary misorientation maps for regions with different strains (from no. I to no. V, with strain increasing closer to the fracture end). The corresponding microstructural data are displayed in Table 1. Before deformation, hardness points are made at equal intervals on the gauge section of the sample, allowing for the calculation of strain in each region after deformation. This sampling method can be used to study the effect of strain on grain structure while maintaining consistent temperature and holding time.

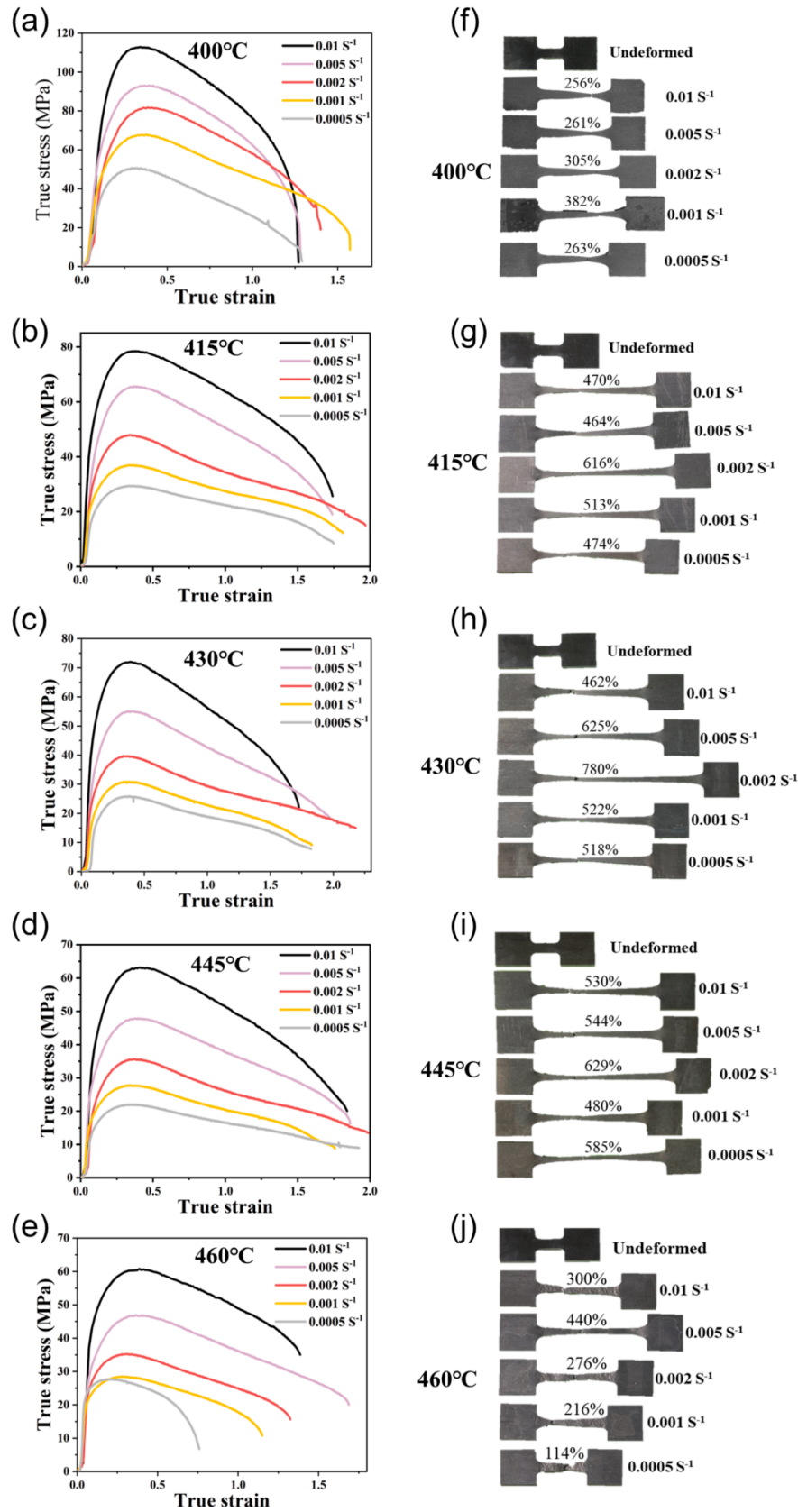


Fig. 2. (a–e) True stress – true strain curves of superplastic 2A97 Al-Cu-Li alloy under different deformation parameters; (f–j) the corresponding samples deformed to fracture.

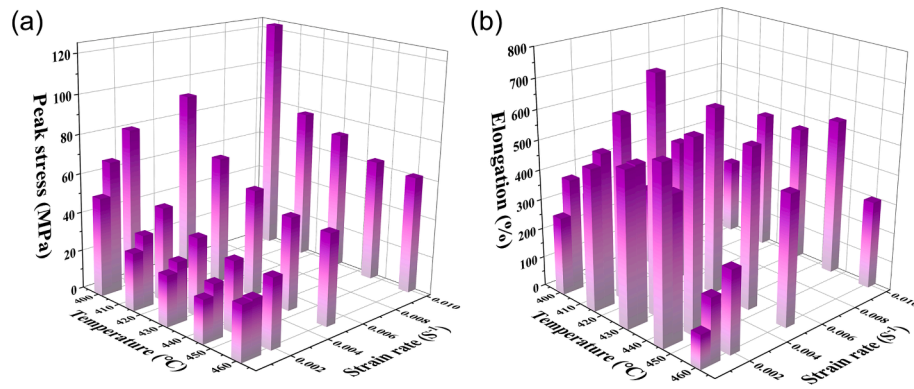


Fig. 3. (a) Peak stress and (b) elongation of superplastic 2A97 Al-Cu-Li alloy under various testing parameters.

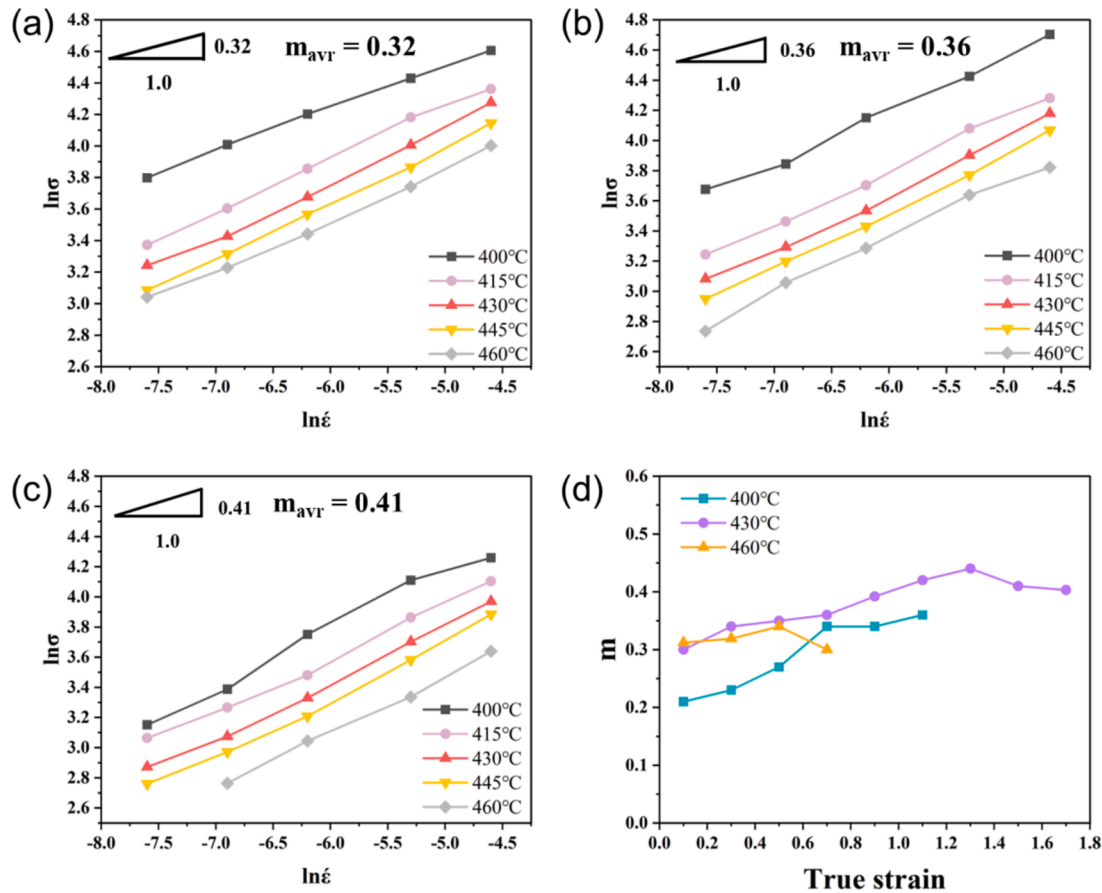


Fig. 4. The logarithmic relationship between stress and strain rate of superplastic 2A97 Al-Cu-Li alloy under different testing parameters at the true strain of (a) 0.4, (b) 0.7, and (c) 1.1; (d) the variation of strain rate sensitivity (m) with true strain at different temperatures.

At the undeformed part (region I), the microstructure remains composed of banded unrecrystallized grains, despite prolonged exposure to high temperatures. This reflects that the alloy is relatively stable under the influence of high temperature alone. The fraction of LAGBs increases slightly to 50.5 % compared to the initial state, which is caused by the recovery. As the stain increases, from region I to V, the grain structure evolves from the banded shape into an equiaxed shape gradually. Meanwhile, the fraction of RXed grains increases progressively from 4.6 % at the grip region to 89.5 % near the fracture end. Furthermore, the fraction of LAGBs decreases from 34.9 % in the undeformed region to 5.3 % in the fracture region, where the distribution becomes nearly random and dominated by HAGBs. A decrease in GND density, along with an increase in sub-grain size and recrystallized grain

size, can also be observed. By calculating the average grain size with HAGBs, the alloy exhibits a grain refinement during the superplastic deformation process. It should be noted that, in region V, the average grain size shows a slight increase, and the grains tend to elongate towards the tensile direction, which may be attributed to diffusion-induced grain growth.

A deformed grain from region II was chosen and shown in Fig. 7. Significant differences in IPF colour inside the grain indicate the presence of substructures with varying misorientations. Several internal boundaries can be seen inside the deformed grain in Fig. 7a, whose misorientation between 2 and 15° was set as purple (LAGBs) and that of larger than 15° (LAGBs) was set as black. As presented in the KAM map (Fig. 7b), the LAGBs have high KAM value, indicating a high dislocation

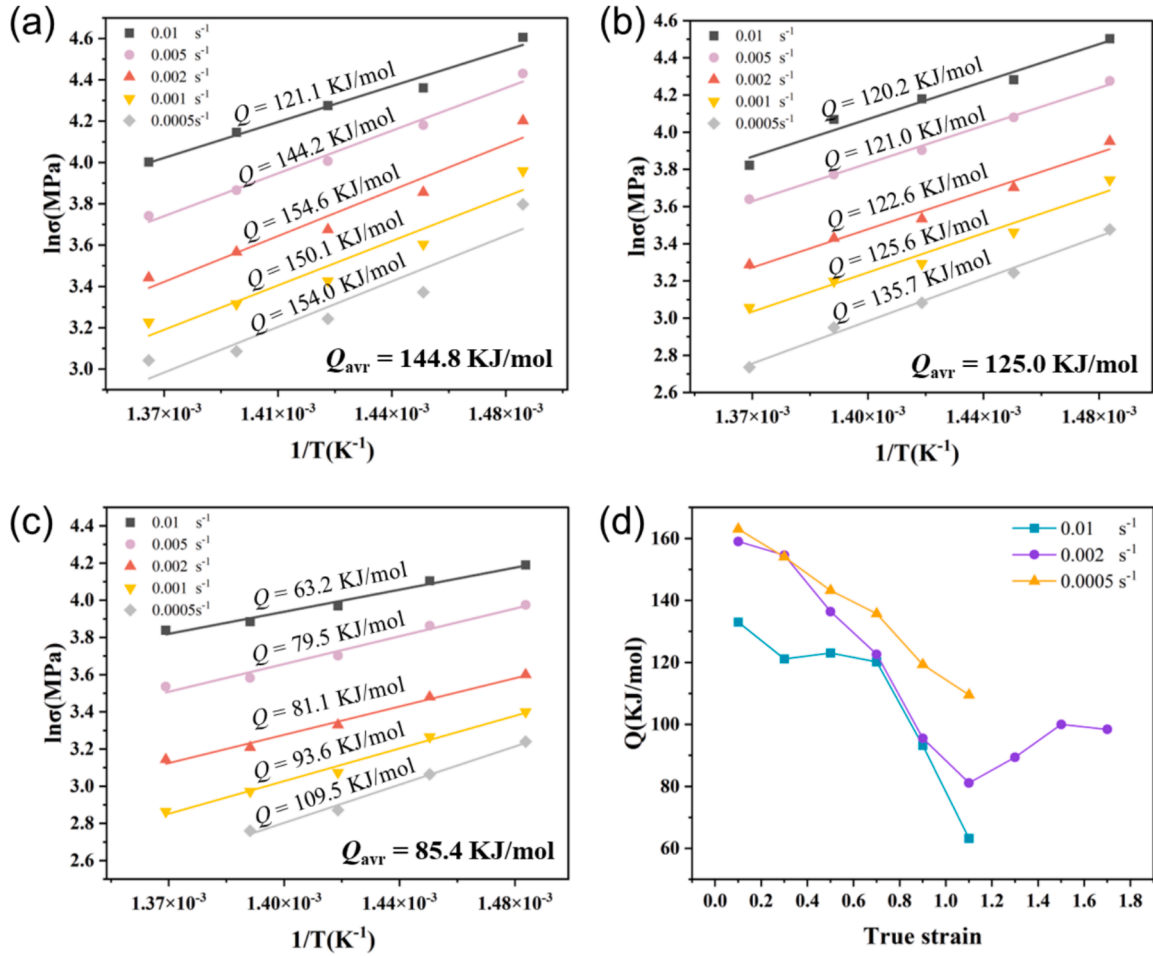


Fig. 5. The relationship between $\ln\sigma$ and $1/T$ of superplastic 2A97 Al-Cu-Li alloy under different deformation parameters at the true strain of (a) 0.4, (b) 0.7, and (c) 1.1; (d) the variation of activation energy (Q) with true strain at different strain rate.

density. To further verify the grain boundary type inside the grain, the misorientation distribution was measured with black lines as shown in Fig. 7c. Corresponding data are displayed in Fig. 7d–f. Some misorientations at the boundaries are less than 2°, which can be regarded as sub-grain boundaries (SGBs). These SGBs transform into LAGBs gradually via polygonization, as shown in Fig. 7d. With the superplastic deformation further processes, the LAGBs transform into HAGBs due to the increase of orientation difference, as confirmed in Fig. 7f.

Fig. 8 demonstrates the role of coarse secondary phases in dynamic recrystallization by using TEM. As can be seen in Fig. 8a, before superplastic deformation, a large quantity of coarse secondary phases can be found distributed in the grains. In the high-magnification image (Fig. 8b), dislocation tangle can be found around the coarse phases. As superplastic deformation goes on, in Fig. 8c, dislocation tangle and dislocation wall can be observed near the coarse phases, and LAGB forming can also be noticed. This represents the process of DRV. With the true strain further increasing to 1.1, as shown in Fig. 8d, no obvious dislocations are observed, and the size of the secondary phases decreases significantly. This can be considered as a fully recrystallized grain.

3.4. Effect of temperature on microstructure and fracture mechanism

Fig. 9 shows the EBSD IPF maps of the 2A97 alloy deformed to each stage of true strain at different temperatures, the initial strain rate was 0.002 s⁻¹. It can be seen that the grains transform from banded shape into equiaxed shape gradually with increasing true strain. This evolution can be also reflected in Fig. 9d that the fraction of recrystallization increases gradually with deformation going on. Especially, at 430 °C, the

recrystallization fraction is always the highest at each tested strain stage. As shown in Fig. 9e, from the true strain of 0 to 1.1, the grain aspect ratio decreases gradually. With further increasing true strain, the grain aspect ratio increases to different degrees at different deformation temperatures. According to the calculated average grain size (calculated with HAGBs) displayed in Fig. 9f, at 400 °C and 430 °C, the grain refinement occurs in the whole process of superplastic deformation. However, at 460 °C, the grain grows continuously with increasing true strain, despite the initial grain refinement. In Fig. 9c, abnormal grain growth can be observed at the fractured stage when tested at 460 °C, which may be attributed to the excessive temperature.

As demonstrated in section 3.3, coarse Cu-rich secondary phases play a positive role in DRX during the superplastic deformation process. To better unveil the evolution of these phases under different deformation temperatures and their contribution to DRX, the SEM-BSE images were recorded for the tested alloy. As shown in Fig. 10, the distribution and size of the coarse secondary phases show significant differences with the variation of testing temperature. In general, at lower deformation temperature (400 °C), these phases maintain uniform distribution and show little change in size. When the sample is deformed to fracture, some abnormal coarse phases begin to appear. With the increase of deformation temperature, the dissolution and coarsening of the secondary phases become more significant. At 460 °C, phases with initial size of ~1 μm are barely visible, even at a small true strain of 0.4. Instead, abnormal coarse phases larger than 5 μm dominate.

By conducting quantitative calculation, the area fraction and average size of the coarse secondary phases were obtained and drawn in Fig. 11. As can be observed, with an increase in strain, the area fraction of coarse

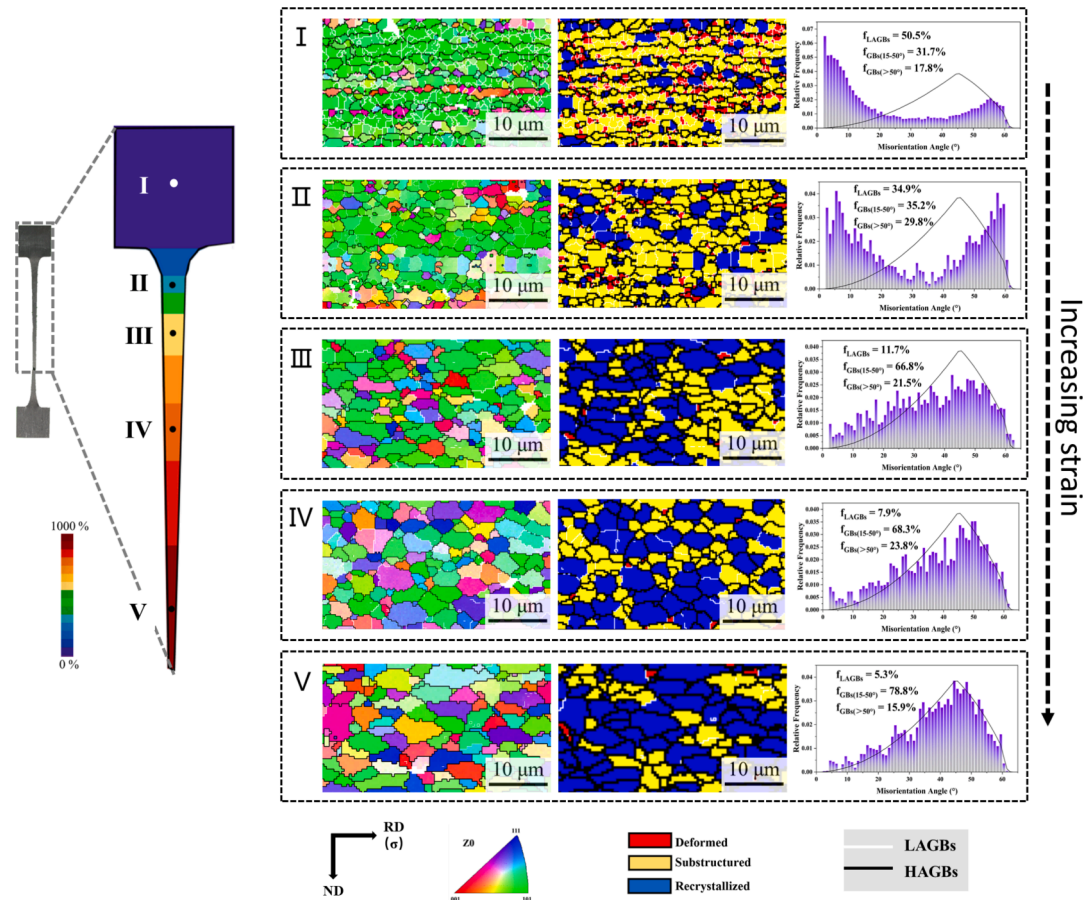


Fig. 6. EBSD IPF maps, recrystallization maps, and corresponding grain boundary misorientation distribution maps at various regions with different strains of the tensile sample, which was tested at 430°C and the initial strain rate of 0.002 s^{-1} .

Table 1

Microstructural data of the corresponding regions presented in Fig. 6.

	Before deformation	Region I (0 %)	Region II (24 %)	Region III (148 %)	Region IV (315 %)	Region V (886 %)
Fraction of recrystallization (%)	3.5	4.6	10.8	30.3	75.6	89.5
GND density (m^{-2})	14.7×10^{15}	11.9×10^{15}	5.7×10^{15}	1.31×10^{15}	1.17×10^{15}	1.02×10^{15}
Average sub-grain size (μm)	1.32 ± 0.1	1.43 ± 0.3	2.01 ± 0.2	2.51 ± 0.2	3.05 ± 0.4	3.32 ± 0.3
Average RXed grains size (μm)	2.16 ± 0.2	2.49 ± 0.2	2.72 ± 0.5	3.26 ± 0.3	3.55 ± 0.2	4.08 ± 0.3
Average grain size (μm)	5.13 ± 0.5	4.93 ± 0.4	3.66 ± 0.1	3.28 ± 0.3	2.96 ± 0.3	3.3 ± 0.4

secondary phases decreases, and the rate of decrease becomes more profound with higher deformation temperatures. As presented in Fig. 11b, the secondary phases show a continuous increase in average size as the deformation goes on. Especially at 460°C, the average size of the coarse secondary phases increases from $\sim 1 \mu\text{m}$ prior to deformation to $\sim 4 \mu\text{m}$ after fracture.

The fracture morphology of samples deformed to failure under different temperatures and the initial strain rates of 0.002 s^{-1} are displayed in Fig. 12. When deformed at a lower temperature (400 °C), as shown in Fig. 12a, the fracture surface is composed of tearing edges and small dimples, which is a typical ductile fracture. With the increase of deformation temperature, as can be seen in Fig. 12c and d, fewer tearing edges and larger dimples can be seen. When deformed to fracture at 460 °C, the surface is mainly characterized by intergranular fracture.

3.5. Effect of strain rate on microstructure and fracture mechanism

The EBSD IPF maps and corresponding microstructural data of the

2A97 alloy deformed to various true strains at 430 °C and different initial strain rates are given in Fig. 13. According to the IPF maps, the grains can be also noticed to transform from banded shape into equiaxed shape, exhibiting dynamic recrystallization. The fraction of recrystallization increases with increasing true strain (Fig. 13c). The measured grain aspect ratio shows a decreasing tendency with increasing true strain. Especially, the grain aspect ratio of the sample tested at the initial strain rate of 0.002 s^{-1} slightly increases after the true strain of 1.1, which can be attributed to the elongation of grains to the tensile direction. When tested at the initial strain rate of 0.005 s^{-1} , the average grain size increases firstly at the initial strain level (Fig. 13f). This may be due to the alloy staying too long at high temperature, causing the grain growth rate to surpass the rate of DRX.

As shown in Fig. 14, the fracture surface of samples stretched at the same temperature (430 °C) and different initial strain rates are displayed. When tested at a high strain rate (0.01 s^{-1}), the surface exhibits ductile fracture with many tearing edges. As the strain rate decreases, the number of tearing edges decreases, and the morphology of grains

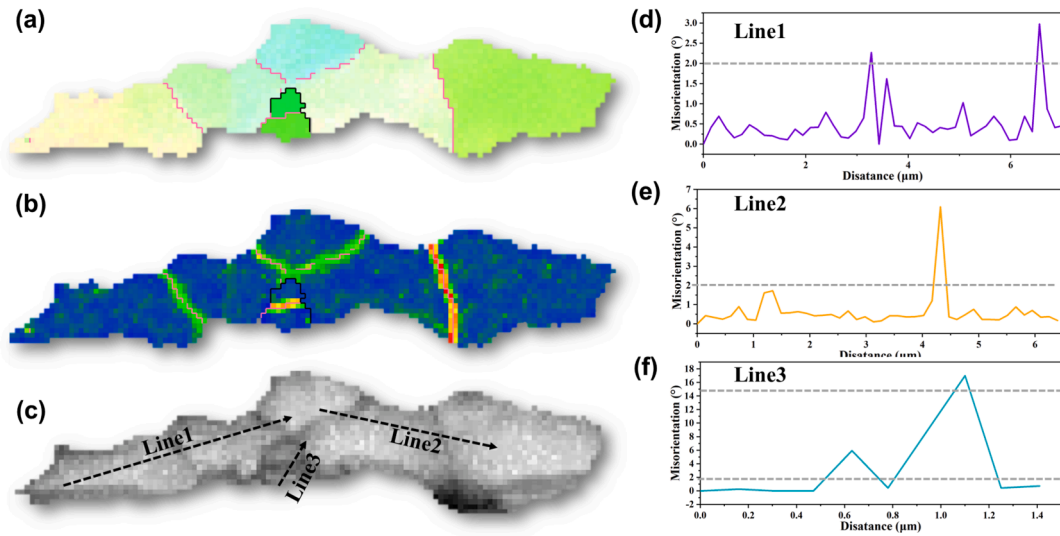


Fig. 7. (a) EBSD IPF map, (b) KAM map, and (c) Band contrast map of deformed grain for the 2A97 Al-Cu-Li alloy deformed to a strain of 24 % at 430°C & 0.002 s⁻¹; and the misorientation distribution for: (d) line 1, (e) line 2, and (f) line 3.

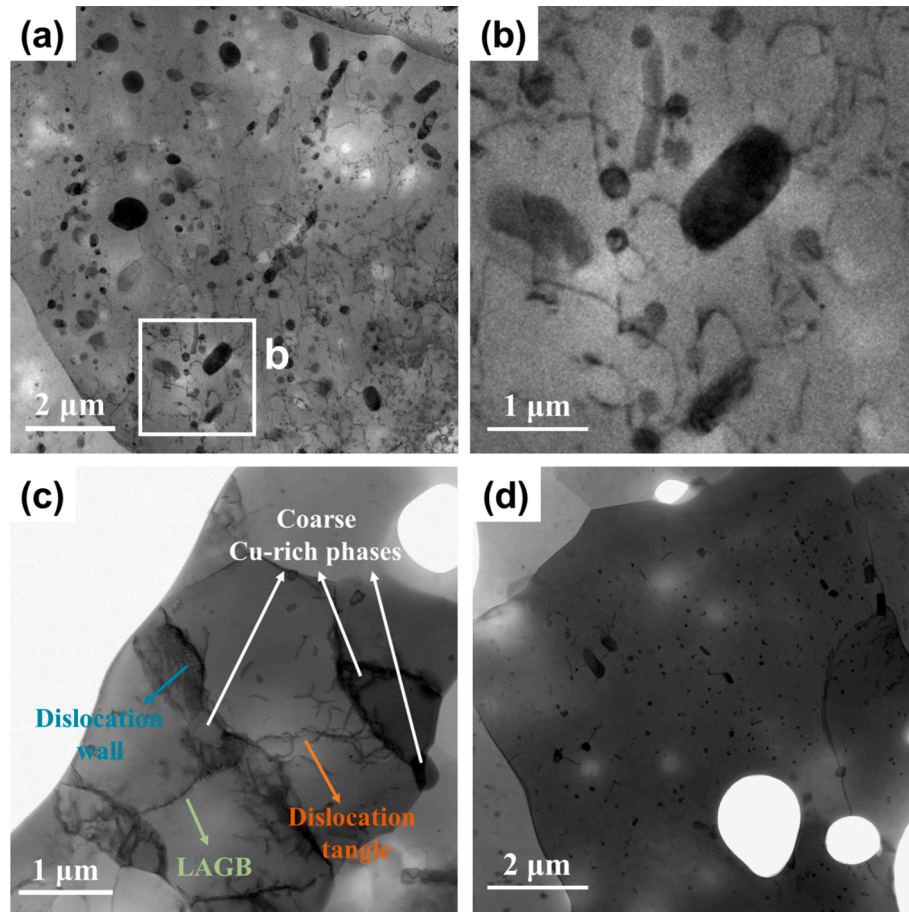


Fig. 8. The bright field TEM maps for 2A97 Al-Cu-Li alloy when deformed to the true strain of (a, b) 0, (c) 0.4, (d) 1.1 at 430°C & 0.002 s⁻¹.

begins to occur. More cavities can be observed, indicating a transition into intragranular fracture. Besides, when the strain rate decreases to 0.0005 s⁻¹, as can be noticed in the magnified image in Fig. 14c, the grain morphology is not that smooth, and some filament structures start to occur. This is because the eutectic with low melting point melted due to long exposure at elevated temperature, and the melted material was

drawn into filaments under stress.

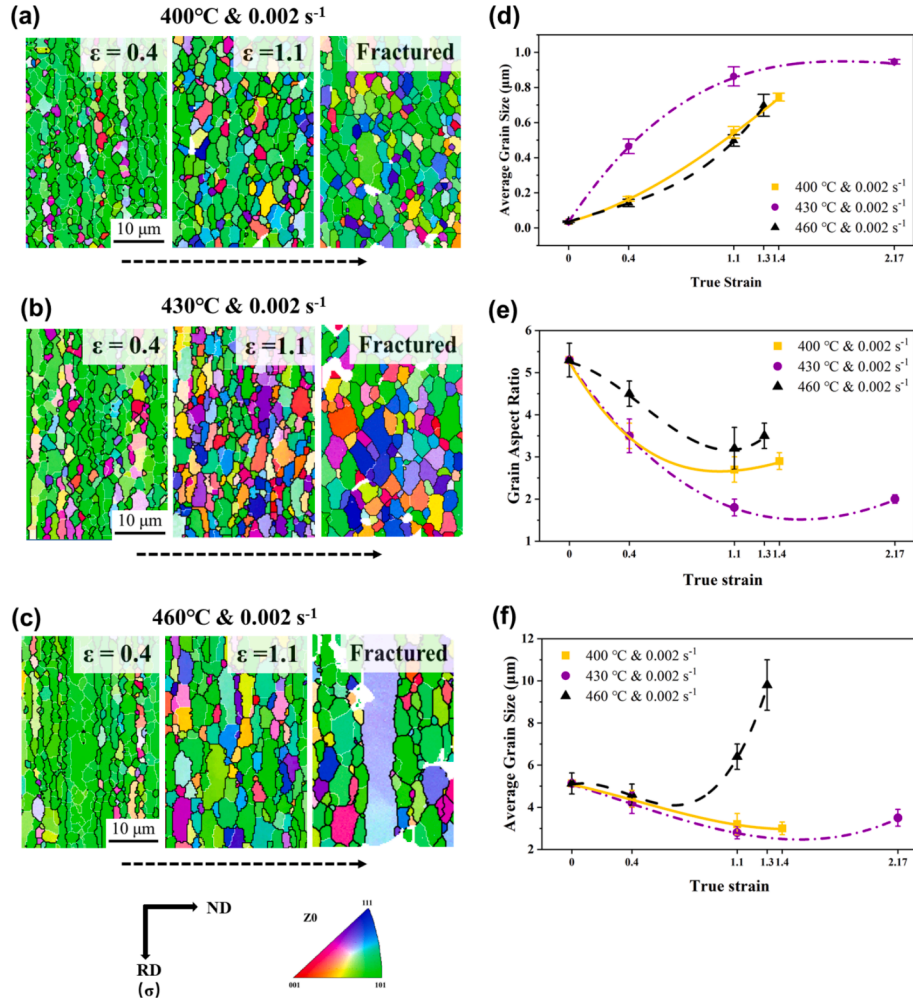


Fig. 9. EBSD IPF maps of the 2A97 Al-Cu-Li alloy when deformed to various strains at the initial strain rate of 0.002 s^{-1} under different temperatures: (a) 400°C , (b) 430°C , and (c) 460°C ; Evolution of (d) the fraction of recrystallization, (e) grain aspect ratio, and (f) average grain size.

4. Discussion

4.1. DRX mechanism analysis

Based on the above research results, DRX in the tested alloy led to the formation of fine equiaxed grains, facilitating significant GBS and thereby enabling good superplasticity. The main DRX mechanisms of 2A97 Al-Cu-Li alloy during superplastic tensile are discussed as follows. The mechanisms of DRX can be divided into three types: discontinuous dynamic recrystallization (DDRX), continuous dynamic recrystallization (CDRX), and geometric dynamic recrystallization (GDRX). In DDRX, new grains nucleate at pre-existed HAGBs through grain boundary bulging (migration), and their misorientation differs from the original grains [18,38]. Necklace-shaped grains are commonly observed in this process [39,40]. CDRX is a process that HAGBs form via sub-grain migration or sub-grain rotation, which is strongly associated with DRV [18,19]. The new grains formed via GDRX result from the pinch-off of serrated HAGBs when the grain thickness is reduced to twice the size of sub-grains [18,19]. Therefore, GDRX usually occurs during compression deformation when the grains reduce thickness. When undergoing superplastic tensile deformation, the grains tend to slide and rotate relatively, making their further thinning difficult. As a result, GDRX has little opportunity to occur.

DDRX is considered hard to occur in aluminium alloys due to their high SFE [18]. Here, the DDRX and CDRX can be analysed by comparing whether the deformation conditions favour grain boundary bulging,

using the following formula [41]:

$$\frac{\rho^3}{\dot{\epsilon}} > \frac{2\gamma_b}{K \cdot M \cdot L \cdot G \cdot b^5} \quad (5)$$

where ρ is dislocation density ahead of the moving grain boundaries, $\dot{\epsilon}$ is the strain rate, γ_b is the grain boundary energy per unit area, K is the fractional constant, M is the mobility of the grain boundaries, L is the mean slip distance of the dislocations, G is the shear modulus, and b is the Burgers vector. The term on the right of the above equation remains constant if the alloy is deformed at a certain temperature. Therefore, sufficiently high dislocation density is needed for grain boundary bulging nucleation during the deformation process. In this study, a certain degree of recovery happened in the alloy during the heating stage before hot tensile deformation. The subsequent superplastic tensile strain rates (ranging from $0.0005 - 0.01 \text{ s}^{-1}$) were too low to generate enough dislocations necessary for grain boundary bulging, as reflected in the continuous decrease in GND density (Table 1). Therefore, CDRX, rather than DDRX, predominantly governed the microstructural evolution of the tested alloy.

From the perspective of grain structure evolution, the as-rolled alloy was mainly composed of deformed grains. After static annealing at testing temperature (Fig. 1a), the microstructure remained in banded shape but with a large number of fine sub-grains inside. After superplastic deformation, at the grip section (Fig. 6I), the grains maintained a banded shape despite prolonged exposure to elevated temperatures, with no significant recrystallization or sub-grain growth occurring.

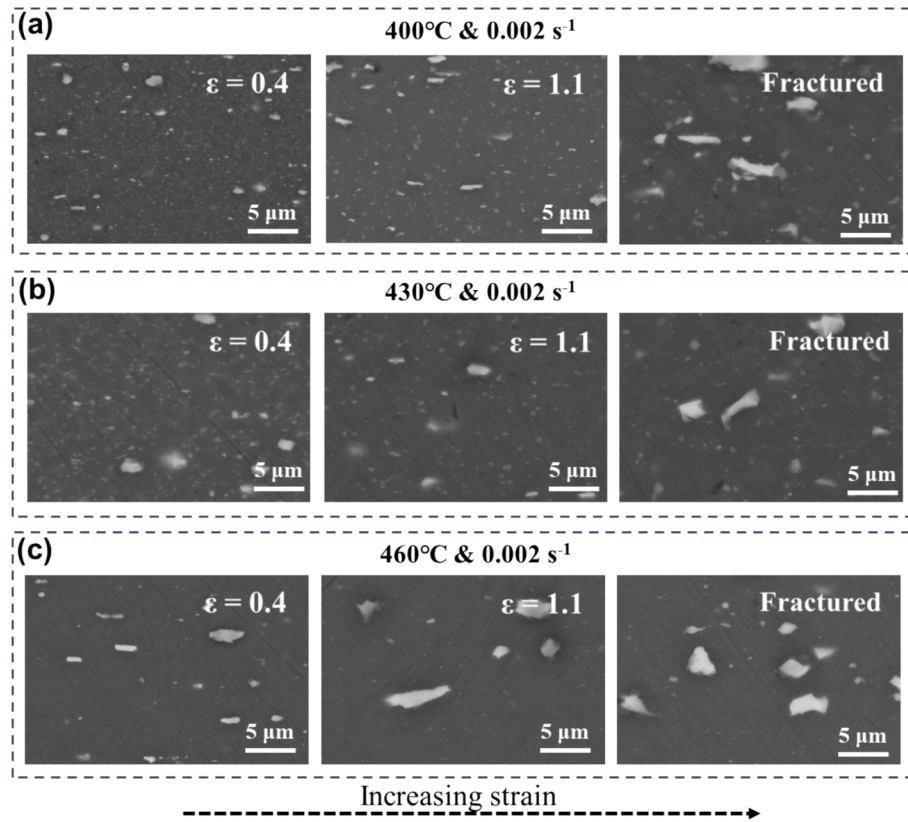


Fig. 10. SEM-BSE images showing the coarse Cu-rich secondary phases of samples deformed to different true strains at the initial strain rate of 0.002 s^{-1} and the temperature of: (a) $400 \text{ }^{\circ}\text{C}$, (b) $430 \text{ }^{\circ}\text{C}$, and (c) $460 \text{ }^{\circ}\text{C}$.

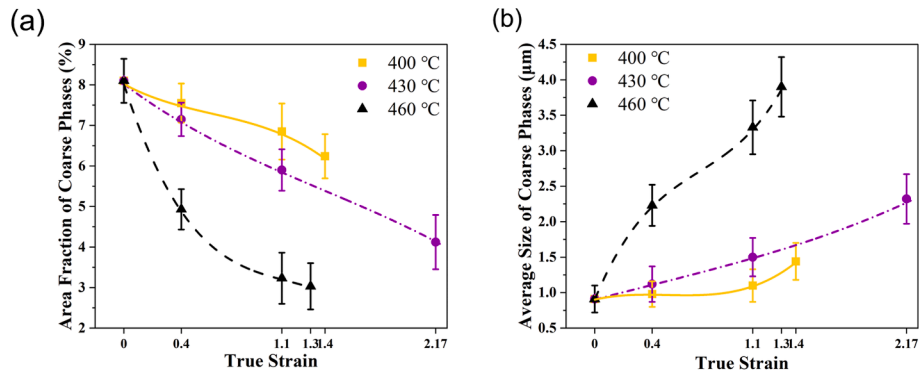


Fig. 11. Area fraction (a) and average size (b) of coarse Cu-rich secondary phases deformed to different true strains at the initial strain rate of 0.002 s^{-1} and various temperatures.

However, at the deformed section, with the increase of strain, significant RXed grains formed. The fraction of recrystallization grew with increasing strain, and the grain boundary misorientation increased rapidly. The evolution of grains from banded shape into equiaxed shape, the growth of sub-grains, and the transformation of LAGBs into HAGBs are typical characterizations of CDRX [18].

The stable microstructure at the undeformed section mainly benefited from the fine Al_3Zr dispersoids (Fig. 1f) [3,42]. By hindering the grain boundary motion via the Zener drag effect [42–44] of the fine dispersoids, the grain growth and recrystallization can be retarded. As a comparison, the significant evolution of grain structure at the deformed part of the sample can be attributed to the role of straining. As demonstrated in Fig. 6 and Table 1, the alloy experienced DRX with the increase of strain, accompanied by an obvious sub-grain growth. There are two potential mechanisms for the growth of sub-grains and their

evolution into RXed grains: one is sub-grain boundary migration; and the other one is sub-grain rotation and coalescence [17]. The sub-grain boundary migration causes sub-grain growth and a gradual increase in grain boundary misorientation, which can occur at static annealing. The sub-grain rotation is a process in which sub-grains move, rotate, and coalesce during concurrent straining and annealing, resulting in a rapid increase of grain boundary misorientation. In this study, the microstructure at the grip zone I shows little change, indicating that sub-grain boundary migration was inhibited due to the aforementioned Al_3Zr dispersoids. At the deformation zone II with strain, the fraction of LAGBs decreases rapidly into 34.9 %, while the fraction of HAGBs with misorientation greater than 50° increases to 29.5 %. There is no noticeable change in the fraction of HAGBs with misorientation between 15° and 50° . This is a clear manifestation of sub-grain rotation, which was rarely observed in the statically annealed samples [17]. Moreover,

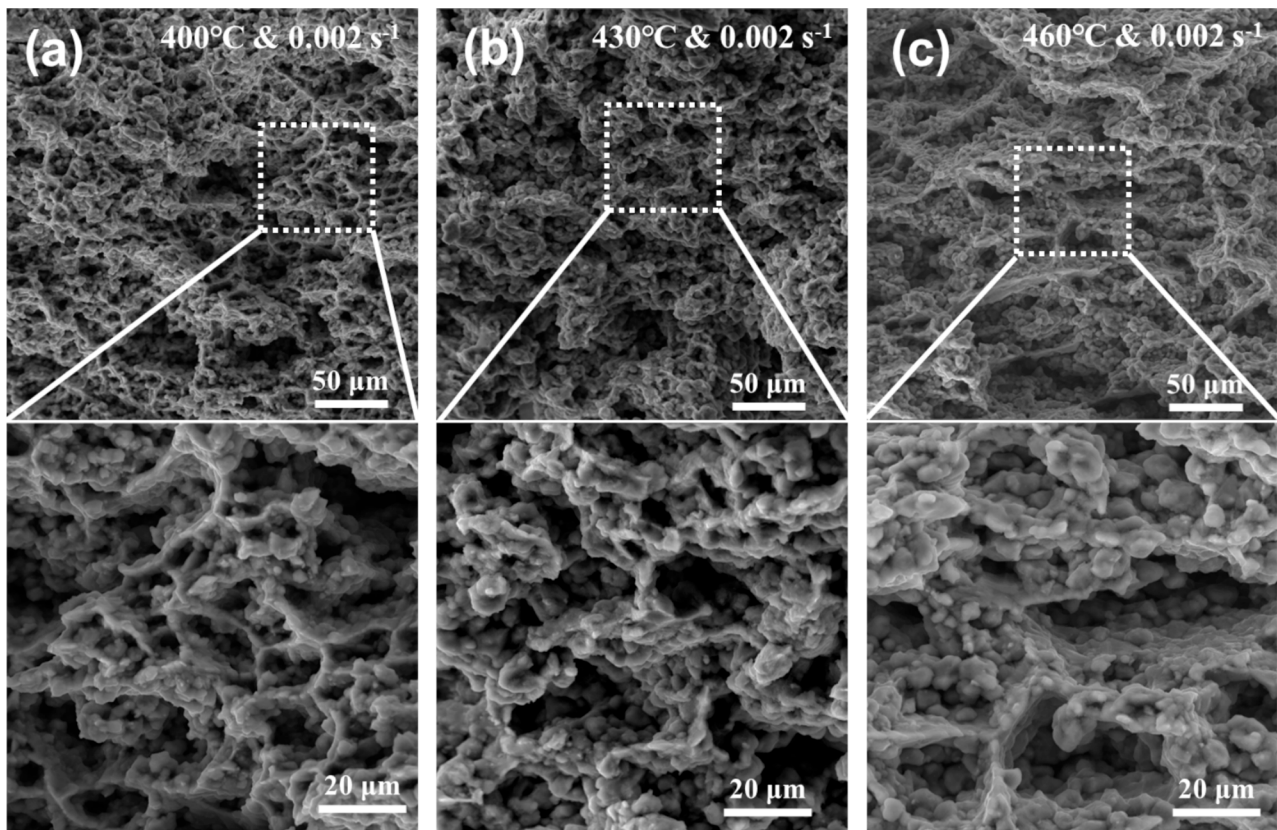


Fig. 12. The fracture surface of 2A97 Al-Cu-Li alloy deformed at the initial strain rate of 0.002 s^{-1} under different temperatures: (a) 400°C , (b) 430°C , and (c) 460°C .

the increase of grain boundary misorientation and formation of discontinuous HAGBs displayed in Fig. 7 is direct evidence of sub-grain rotation. The above phenomena confirm the role of sub-grain rotation and coalescence to the DRX during the initial stage of superplastic deformation.

In addition to the CDRX occurring through the rotation and coalescence of the sub-grains that formed during prior rolling and subsequent sample preheating, it is believed that the fine particles can promote the transformation of LAGBs to HAGBs at the early deformation stage [18]. As can be observed in Fig. 2, the strain hardening at the beginning of deformation is the indication of further dislocation multiply. The already formed sub-grains were pinned by particles (Al_3Zr and Cu-rich phases), causing dislocations to be further trapped at these boundaries due to straining, as observed in the TEM result in Fig. 8c. This intragranular deformation promoted the formation of sub-grains and led to their transformation into HAGBs.

According to the discussion above, DRX mechanisms of the tested alloy under the superplastic deformation process can be concluded as follows. The high density of dislocations was generated during the prior rolling process and the sub-grains inside the deformed grains quickly formed during the holding process before superplastic deformation. Furthermore, strain-induced dislocations at the early stage of deformation further boosted the DRV process. With the processing of hot tensile, the sub-grains began to rotate and coalesce under straining, resulting in a rapid increase of grain boundary misorientation. Thus, the transformation from LAGBs into HAGBs facilitated the forming of fine equiaxed grains with random misorientation.

4.2. Evolution of coarse secondary phases and their influence on DRX

As demonstrated in Fig. 8, from the TEM characterization, the coarse secondary phases played a vital role in accelerating the CDRX. The dislocation tangle, dislocation rearrangement, and initiation of LAGBs

can be observed directly near these phases. However, these phases formed and nodulized from T_1 precipitates under over-ageing process are not stable at the tested temperatures. The elevated temperature would cause dissolution and coarsening of these secondary phases [31,45], affecting the CDRX by promoting or inhibiting the mechanism of PSN.

As shown in Figs. 10 and 11, the micron-sized secondary phases experienced significant coarsening and dissolution during the superplastic deformation, with these phenomena becoming more pronounced as the temperature increased. At lower temperatures (400 and 430°C), massive secondary phases of $\sim 1 \mu\text{m}$ existed until fracture, with their content falling steadily. These evenly distributed phases provided nucleation sites for new grains at the initial deformation stage and boosted the CDRX significantly. However, when raising the temperature to 460°C , fast dissolution of secondary phases happened, with some particles coarsened severely (Fig. 11c). Under this situation, the PSN effect was remarkably weakened. This is one of the reasons for the low recrystallization fraction at 460°C . Since the evolution of coarse secondary phases is mainly influenced by the temperature, the strain rate has a slight effect on them. However, it is important to note that a very low strain rate allows the samples to remain at high temperatures for longer periods, which can negatively impact the distribution and size of these phases. This, in turn, may have an adverse effect on CDRX to some extent.

4.3. Effects of deformation parameters on DRX

Since the alloy showed a dynamic evolution of microstructures that significantly affected its superplasticity, it is important to further explore the influence of deformation temperature and strain rate on DRX of the studied alloy. To describe the effects of deformation parameters (temperature and strain rate) on DRX, Zener-Hollomon parameter Z [20,46,47] is used as follows:

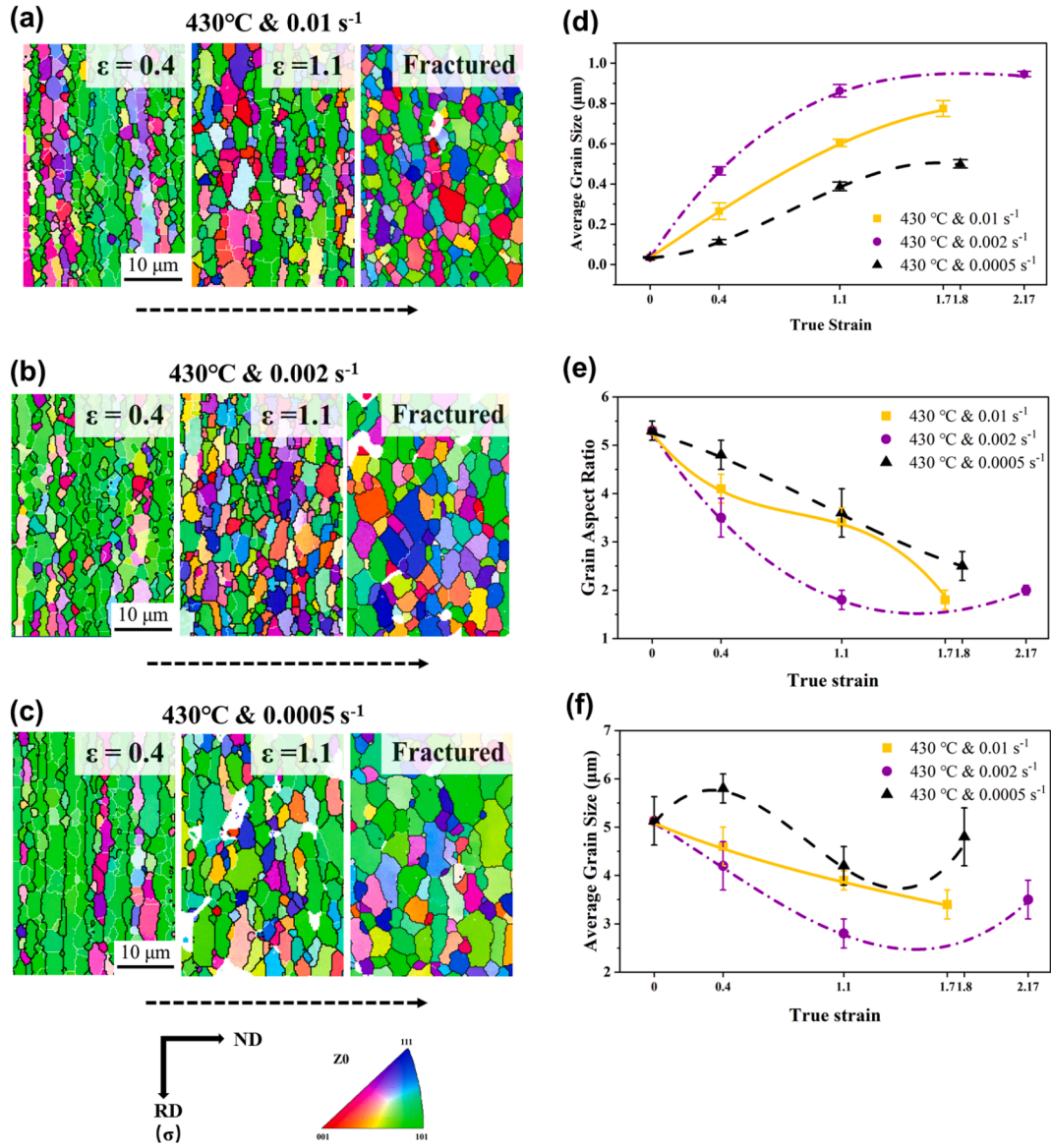


Fig. 13. EBSD IPF maps of the 2A97 Al-Cu-Li alloy when deformed to various strains at 430°C under different initial strain rate of: (a) 0.01 s⁻¹, (b) 0.002 s⁻¹, and (c) 0.0005 s⁻¹; Evolution of (d) the fraction of recrystallization, (e) grain aspect ratio, and (f) average grain size.

$$Z = \dot{\epsilon} \exp(Q/RT) \quad (6)$$

where $\dot{\epsilon}$ is the strain rate, Q is the activation energy, R is the gas constant, and T is temperature.

In this work, according to the results in section 3.2, the average Q value for the tested conditions is 112.3 KJ/mol, which is used here to calculate the Z parameter. Therefore, the $Z = \dot{\epsilon} \exp(112,300/RT)$ in this study. According to the equation, lower $\ln Z$ corresponds to the condition of higher temperature and lower strain rate. On the contrary, higher $\ln Z$ corresponds to lower temperature and higher strain rate. Based on the results of EBSD, the relationship between the fraction of recrystallization and $\ln Z$ for the alloy deformed into different true strains is given in Fig. 15. It can be seen that the highest fraction of recrystallization occurs at the $\ln Z$ of 13.0 (430°C & 0.002 s⁻¹). Increasing and decreasing $\ln Z$ both result in lower recrystallization. This indicates that within the range tested in the present study, an appropriate combination of strain rate and temperature helps to develop optimum sub-structure and facilitate CDRX.

At the preheating and initial deformation stage, the dislocation density plays a vital role in the DRV and CDRX process. The accumulated

dislocations at prior rolling would decrease at elevated high temperatures, and the dislocation exhaustion rate ($\dot{\rho}$) can be interpreted as the following equation [48]:

$$\dot{\rho} = \rho_{T0} - a e^{-bT} \quad (7)$$

where ρ_{T0} , a , and b are constants, which means the dislocation density would reduce faster at higher temperatures. Therefore, in the present study, under lower $\ln Z$ conditions, higher temperature or longer exposure to high temperature led to lower dislocation and higher DRV [19,49]. Furthermore, sub-grain boundary migration was enhanced [50], which increased the sub-grain size and impeded sub-grain rotation. These factors hindered the CDRX, resulting in larger grain size and a lower fraction of recrystallization. Additionally, as discussed in section 4.2, the micron-sized secondary phases underwent rapid dissolution and coarsening under lower $\ln Z$ conditions. The resulting decrease in particle density and the formation of excessively large particles were detrimental to PSN, further hindering CDRX. In contrast, when tested at higher $\ln Z$ conditions, lower temperature or higher strain rate resulted in insufficient thermal driving force, which slowed down the DRV rate and made it more difficult for sub-grains to form [38]. Furthermore, the

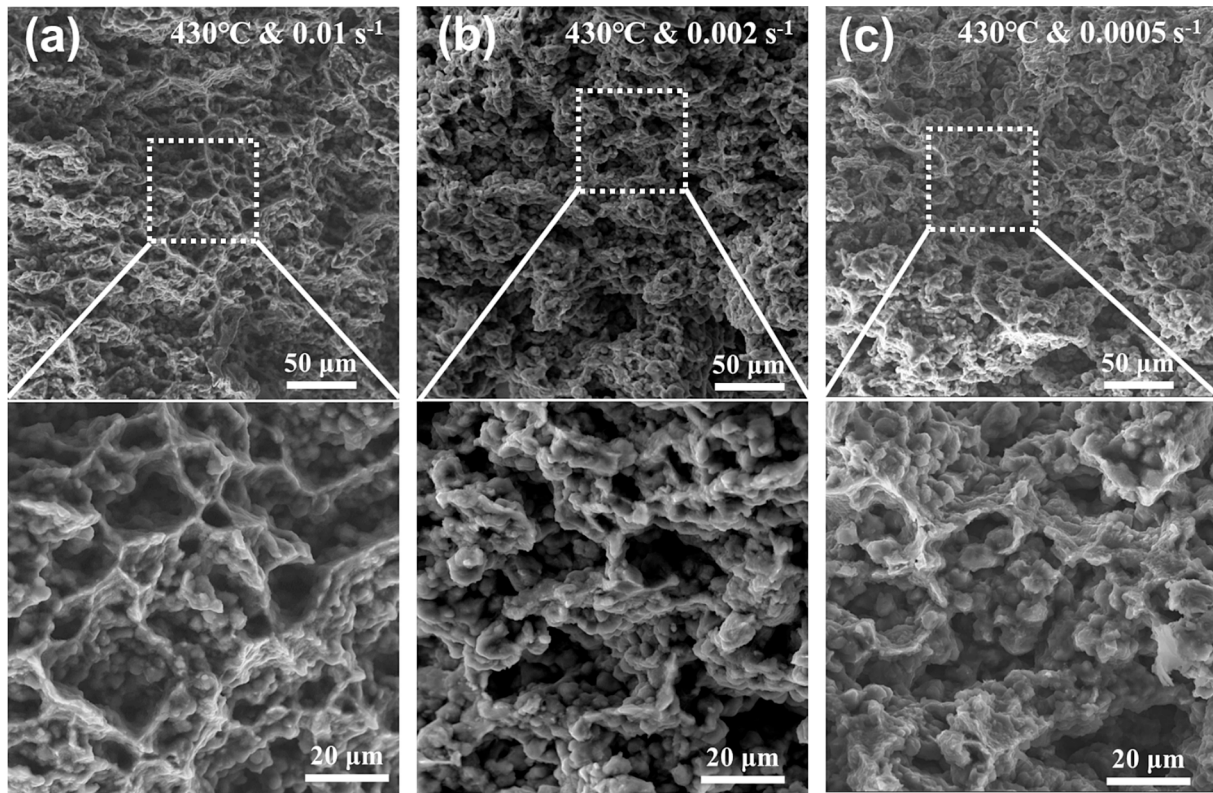


Fig. 14. The fracture surface of 2A97 Al-Cu-Li alloy deformed at 430°C under different initial strain rate of: (a) 0.01 s⁻¹, (b) 0.002 s⁻¹, and (c) 0.0005 s⁻¹.

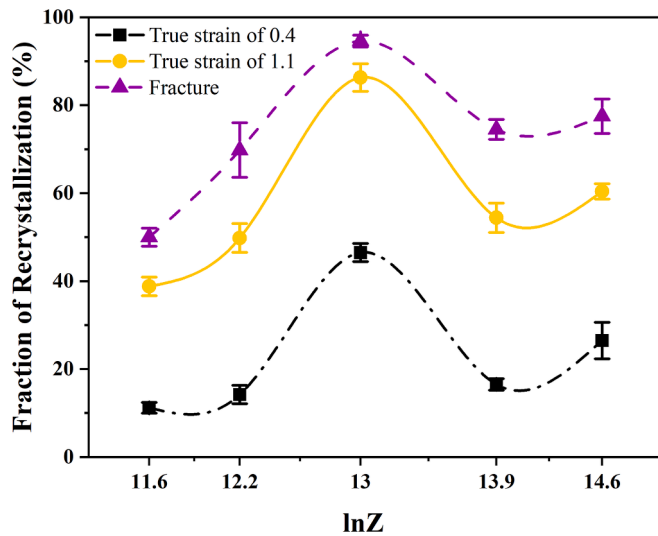


Fig. 15. Relationship between the fraction of recrystallization and $\ln Z$ at different true strains.

sub-grain rotation was hindered, which slowed down the CDRX process. These factors caused the low recrystallization fraction at higher $\ln Z$ conditions, and the resulting insufficient GBS led to the formation of tearing edges on the fracture surfaces (Figs. 12a and 14a).

4.4. Superplastic deformation behaviour and mechanism

It is well acknowledged that fine equiaxed grains are crucial for superplastic deformation, as they provide abundant randomly distributed grain boundaries with high angle misorientation, which can maximize GBS [3,17]. However, in this study, the as-received alloy exhibited a

banded grain structure dominated by LAGBs, which is not suitable for superplastic deformation [17]. This is supported by the m value as shown in Fig. 4(d). At the initial deformation stage, the m value is about 0.3, which is relatively low compared to the m value of 0.5 observed under the condition of sufficient GBS. With increasing the strain, as demonstrated in Figs. 6, 9, and 13, the banded grains evolve into equiaxed shape gradually, with the dominant grain boundaries transforming from LAGBs to HAGBs. The fine equiaxed RXed grains with random misorientation distribution provide suitable conditions for GBS, resulting the increase of m value with deformation under 400 and 430°C (Fig. 4d). When the deformation temperature increases to 460°C, it can be seen that the m shows a stable lower value of ~ 0.3 . The reason is that the grains grow significantly and hence resulted in the decrease of the sliding plane and the weakness of GBS.

In addition to the m value, the Q value, which represents the free energy barrier for dislocation slipping on slip planes, can also reflect deformation mechanisms to some extent. In Fig. 5, the calculated Q_{avr} varies from ~ 144.8 KJ/mol to ~ 85.4 KJ/mol, which is roughly between the grain boundary and lattice self-diffusion for aluminium alloy [51,52]. This indicates that GBS and IDS/DC act as superplastic deformation mechanisms, with their contributions changing following different deformation strains or deformation parameters. As shown in Fig. 5d, when the true strain increases from 0 to ~ 1.1 , the Q value decreases progressively under every testing initial strain rate, which indicates that the dominant deformation mechanism transforms from IDS/DC to GBS. This is because DRX results in more equiaxed finer grains and then promotes the GBS. When tested at 430 °C & 0.002 s⁻¹, the Q shows an increase after the true strain of 1.1, which can be attributed to the grain growth and the initiation of cavities.

To further analyse the deformation mechanisms under different deformation parameters, the pole figures of samples deformed to different true strains at different temperatures and initial strain rates are presented in Figs. 16 and 17, respectively. As shown in Fig. 16a, d, and g, when deformed to a true strain of 0.4, the alloy retains a rolling texture,

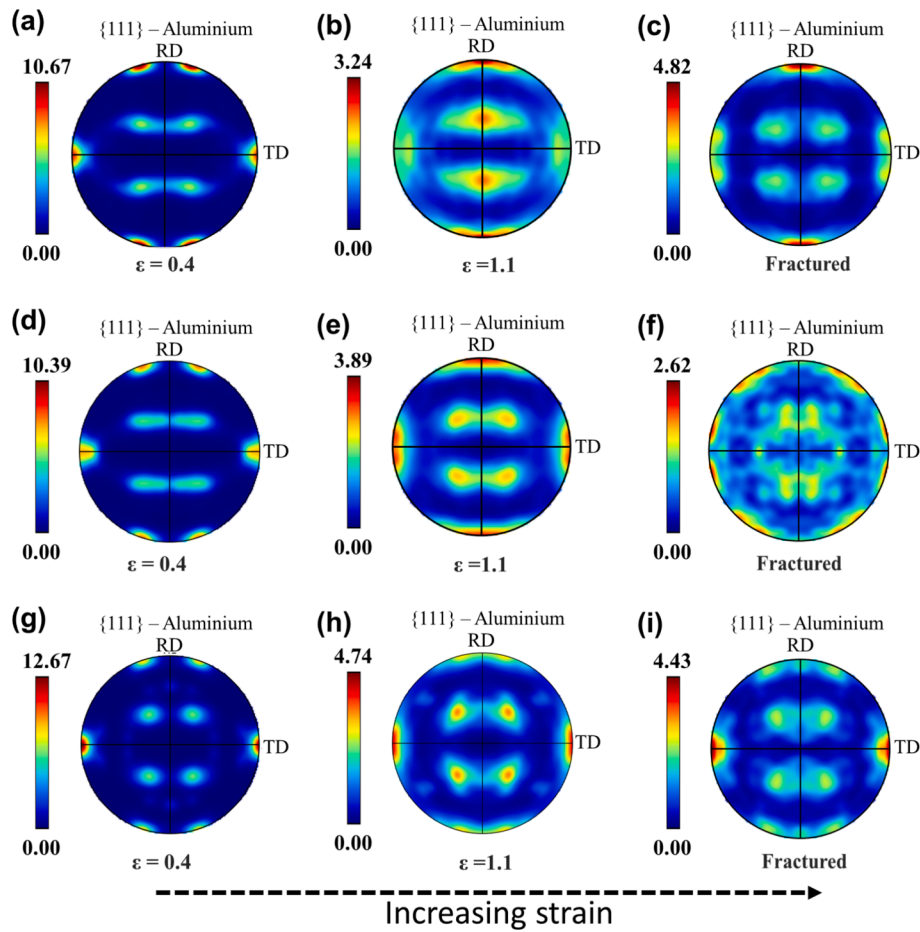


Fig. 16. Pole figures of the 2A97 Al-Cu-Li alloy at different true strains tested at the initial strain rate of 0.002 s^{-1} and the temperature of: (a), (b), (c) 400°C ; (d), (e), (f) 430°C ; (g), (h), (i) 460°C .

but with a higher peak density compared to its value before deformation (9.20). This indicates that IDS acts as the dominant deformation mechanism at this stage, as dislocation movement directly responds to the applied stress [24,53]. With further increasing strain, the texture peak density decreases, and the texture distribution starts to randomize. Especially at 430°C , the texture is almost completely randomized in the fracture stage. The randomization and weakening of texture result from GBS and grain rotation [10,24,54]. In return, the randomly distributed grains provide a suitable condition for GBS. This indicates that GBS plays a more important role at the second deformation stage.

When tested under different initial strain rates, as can be seen in Fig. 17, the evolution of texture exhibits a similar trend: the texture peak density increased during the initial deformation stage, followed by randomization and weakening with continued deformation. At higher initial strain rates (0.01 and 0.002 s^{-1}), the texture shows nearly completely randomized at fracture strain. However, when tested at a low initial strain rate (0.0005 s^{-1}), the rolling texture retains when deformed to fracture, albeit with a lower peak density. This is because a very low strain rate is not conducive for CDRX. As a result, the fraction of recrystallization is low until fracture (Fig. 13d), and the grain size is larger (Fig. 13f), which is unfavorable for superplastic deformation.

Based on the above discussion, the tested alloy underwent CDRX during deformation, with the fine RXed grains playing an important role in enabling superplastic deformation. When tested under the optimum deformation parameter (430°C & 0.002 s^{-1}), the grains transformed from banded shape into nearly equiaxed shape with increasing strains. The deformation mechanism was dominated by IDS during the initial deformation stage. After full recrystallization, the deformation mechanism changed to GBS. Increasing the deformation temperature and

decreasing the strain rate led to a lower recrystallization fraction and rapid grain growth, which resulted in poor superplastic elongation.

5. Conclusions

In the present work, the deformation behaviour, microstructure evolution, and mechanisms of dynamic recrystallization of a 2A97 Al-Cu-Li alloy under various superplastic deformation parameters were investigated. The main conclusions are summarized as follows:

- (1) Under the conditions tested in this study, the alloy exhibited the highest elongation of 780 % at 430°C and 0.002 s^{-1} . It also showed high strain rate superplasticity over a wide temperature range ($415\text{--}460^\circ\text{C}$). Both temperature and strain rate have a significant influence on the microstructure evolution process of the tested alloy, consequently leading to different superplastic behaviour.
- (2) With increasing superplastic strain, the initial banded grains transformed into fine equiaxed grains mainly through CDRX, achieved via sub-grain rotation and coarsening under the straining. The coarse Cu-rich secondary phases facilitated the CDRX process via PSN, while Al_3Zr dispersoids played a role in stabilizing the grain structure.
- (3) Under the optimal parameter (430°C & 0.002 s^{-1}), the recrystallization fraction was the highest. A decrease in $\ln Z$ led to higher DRV, lower dislocation density, and significant dissolution and coarsening of secondary phases, which hindered the process of CDRX. Increasing $\ln Z$ resulted in a lower recrystallization fraction due to insufficient thermal driving force, and the

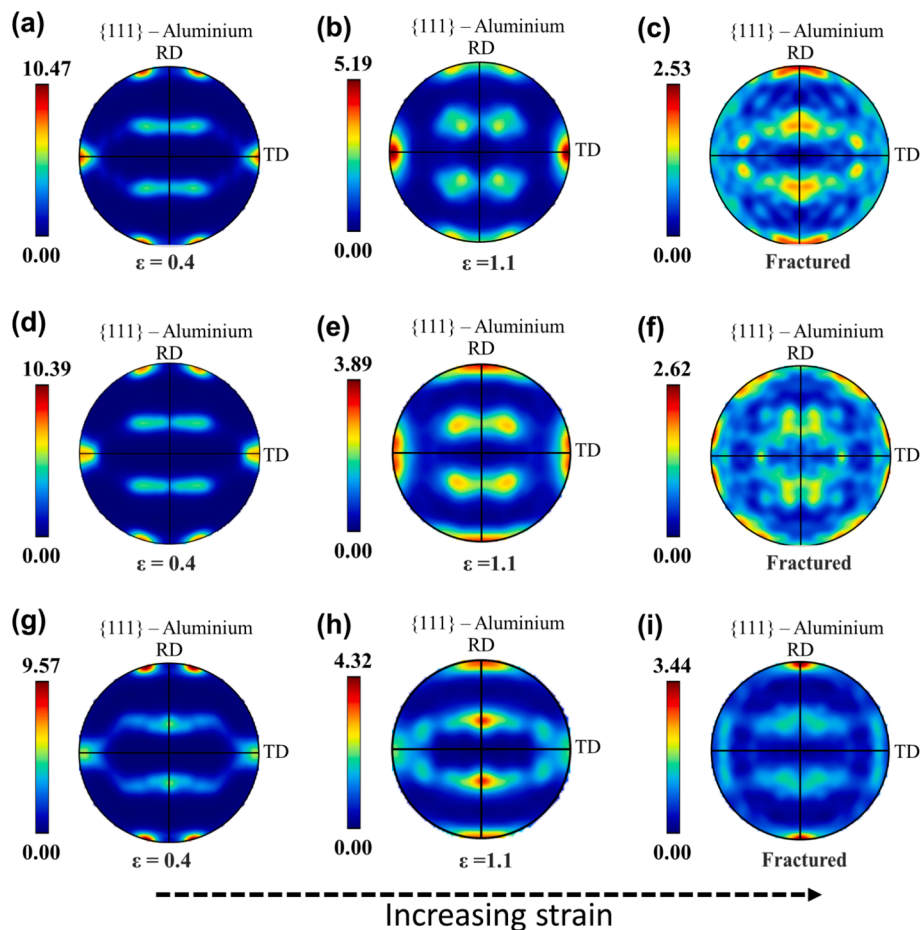


Fig. 17. Pole figures of the 2A97 Al-Cu-Li alloy at different strains tested at the temperature of 430°C and the initial strain rate of: (a), (b), (c) 0.01 s⁻¹, (d), (e), (f) 0.002 s⁻¹; (g), (h), (i) 0.0005 s⁻¹.

deformation conditions became less favourable for GBS, resulting in poor superplasticity.

- (4) According to the m value, Q value, and texture evolution, the dominant superplastic deformation mechanism changed from IDS into GBS with the increase of strain. Initially, the primary deformation mechanism was IDS due to the banded grain structure. After the grains evolved into an equiaxed shape through CDRX, GBS became the dominant deformation mechanism.

CRedit authorship contribution statement

Guotong Zou: Writing – original draft, Methodology, Investigation, Conceptualization. **Ruiqiang Zhang:** Writing – review & editing, Data curation. **Wei Wang:** Writing – review & editing, Data curation. **Jun Li:** Methodology, Investigation. **Lingying Ye:** Writing – review & editing, Supervision, Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Not applicable.

Data availability

Data will be made available on request.

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