Flow behavior and microstructure evolution of Al-3.65Cu-0.98Li (wt%) alloy during hot deformation

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Abstract
The phenomenological constitutive model, processing map and microstructure evolution of Al-3.65Cu-0.98Li (wt%) alloy were studied by means of isothermal compression tests conducted on a Gleeble-3500 isothermal simulator in the temperature range of 350 °C – 500 °C and strain rate range of 0.01 s⁻¹ – 10 s⁻¹ and EBSD. The strain compensated Arrhenius model describes the flow curves accurately with a relative error of 0.9898 and an average absolute relative error of 4.70%. The plastic capability was characterized by strain rate sensitivity index, and it has the positive relation with temperature and negative relation with strain rate. The processing map was constructed, the instable deformation window and optimal hot working window of this alloy was identified to be 350 – 455 °C & 0.37 ~ 10s⁻¹ and 440 – 500 °C & 0.01 ~ 0.368s⁻¹, respectively. Moreover, the dynamic recrystallization occurs more violently at lower strain rate, and most portion were transformed into substructure with increasing strain rate, which was nearly vanished at the strain rate of 10 s⁻¹. The geometric necessary dislocation distributions under different temperatures and strain rates were analyzed. More uniform distributed dislocation cell structures were observed at high temperature with low strain rate conditions and intensive dislocation and more pileups occurs at the contrary conditions.

1. Introduction

Comparing to traditionally commercial Al alloys, Al–Cu–Li alloys have advantages such as higher specific strength and lower density, exhibiting better properties in the structural components used in aircraft, aerospace and military applications [1–3]. As to Al–3.65Cu–0.98Li alloy, it has been seen as an alternative to traditional 7xxx series alloys such as AA7050 alloy and AA7075 alloy because of its higher SCC resistance and density reduction [4, 5]. However, different from the 7xxx series alloys which were extensively studied in terms of flow behavior and microstructure evolution analysis, relevant research for Al-3.65Cu-0.98Li (wt%) alloy is rarely reported, which limits the application extension of this alloy.

The study methods for investigating flow behaviors of alloys have become quite mature, including constitutive modeling [6, 7], dynamic recrystallization (DRX) analysis [8, 9], constructions of processing maps [10, 11] etc. The Arrhenius model, Johnson–Cook model, Fields-Backofen model etc have been extensively applied to study the flow behaviors of Al-Li alloys [12–14]. There models are the fundaments for deformation numerical simulation of relevant components. It is important to deeply study the constitutive modelling for Al-3.65Cu-0.98Li (wt%) alloy which is subject to hot deformation since it was rarely reported. Moreover, the deformation parameters (strain, strain rate and temperature) undoubtedly affect the microstructure of a hot deformed part. An efficient way to identify the microstructure with deformation parameters is processing map...
which contains an instability map and a power dissipation map. Instability map identifies the stable or unstable microstructure evolutions by the instability criterion calculated from flow stress. It has been proved to be efficient in characterizing titanium alloy, aluminum alloy and ultra-high strength steel, etc [15–17]. Power dissipation map also recognizes the potential microstructure evolutions by the range of power dissipation efficiency (PDE) value. Higher PDE value generally corresponds to positive microstructure evolution such as DRX and dynamic recovery (DRV) [18]. Of course, the construction of processing map cannot exclude the strain rate sensitivity index (m), which represents the plastic deformation capability of metals and alloys [19]. To reveal the effect of microstructure evolution on the plastic deformation capability, it is necessary to understand the relationship between them, i.e. higher m-value may correspond to more uniform deformation and lower especially negative m-value may be the result of cracking or some other plastic defects. To evaluate the homogeneity of deformation, the dislocation distribution analysis in a large-scale area is persuasive. Ashby [20] believe that geometrical necessary dislocation (GND) accommodates the deformation gradients to satisfy the compatible deformation. Based on that, according to the description from Zheng et al [21], the distribution and evolution of various deformation states can be quantified by characterizing GNDs in Al alloys. GNDs are generally estimated based on the rotation of grain orientations [22, 23], which can be measured by EBSD.

In this work, hot compression tests were conducted for the Al-3.65Cu–0.98Li (wt%) alloy and the flow curves were obtained. The flow behavior was characterized by strain compensated Arrhenius model. The strain rate sensitivity index was calculated and comparatively analyzed with microstructure. The processing map was constructed to identify the instable and optimum deformation areas. The GND distributions under different strain rates and temperatures were introduced to understand the deformation homogeneity.

2. Experimental procedures

2.1. Specimen preparation

The materials studied in this work is Al-3.65Cu–0.98Li (wt%) alloy after homogenized heat treatment. Its chemical compositions are shown in table 1. The specimens with size of $8 \times 12$ mm for following isothermal compression tests were roughly machined by wire-electrode cutting and the end faces of the specimens were ground by #800 sandpaper to ensure lower friction during compression.

2.2. Isothermal compression tests

The prepared specimens were isothermal compressed on a Gleeble-3500 isothermal simulator. On the simulator, the specimens were resistance heated to 350 °C, 400 °C, 450 °C and 500 °C with a heating speed of 5 °C/s and hold 3 min to eliminate internal temperature gradient. Graphite flakes were used to reduce the friction between anvils and the end faces of specimens. Thermocouples were weld on the cylindrical surfaces in the 1/2 height position of the specimens to monitor the temperature variation during hot compression. After heating and holding processes, the specimens were compressed to the height reduction of 60% under the strain rate of 0.01 s$^{-1}$, 0.1 s$^{-1}$, 1 s$^{-1}$ and 10 s$^{-1}$. The nominal stress and strain values were simultaneously collected and converted into true stress and true strain according to the following equations.

$$\sigma_T = \sigma_N(1 - \varepsilon_N)$$  \hspace{1cm} (1)

$$\varepsilon_T = \ln(1 - \varepsilon_N)$$  \hspace{1cm} (2)

where $\sigma_N$, $\sigma_T$, $\varepsilon_N$, $\varepsilon_T$ are nominal stress, true stress, nominal strain and true strain respectively [24].

2.3. Preparation for microstructure analysis

After compression, the specimens were immediately water quenched to retain the deformed microstructures. The quenched specimens were wire-electrode cut across the center of end faces. The cutting faces were then ground using #400–#4000 sandpapers successively. To prepare the specimens for EBSD characterization, the ground faces were electrochemically polished in the solution of HNO$_3$:CH$_3$OH = 30 ml:70 ml at the temperature of $-30 \degree$C for 30 s. The geometric center of the cutting face was then characterized by an EBSD-equipped JSM-7800F field emission scanning electron microscopy. The results were analyzed by the commercial software CHANNEL 5 and MTEX module in MATLAB.

| Table 1. Chemical composition of the Al-3.65Cu-0.98Li (wt%) alloy (wt%). |
|------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| Cu               | Li           | Ag           | Mn           | Mg           | Zr           | Fe           | Ti           | Si           | Al           |
| 3.65             | 0.98         | 0.40         | 0.39         | 0.38         | 0.12         | 0.06         | 0.03         | 0.02         | Bal          |

N, PDE, T are nominal stress, true stress, nominal strain and true strain respectively.
3. Results and discussion

3.1. Flow curves and softening behavior

The obtained flow curves are shown in figure 1. The flow stress has a negative correlation with temperature but a positive correlation with strain rate. Flow softening behavior of this alloy is much more evident than that of other Al-Li alloys (e.g. 2196 alloy [6] and 2055 alloy [25]) under the same conditions, especially under lower temperature and lower strain rate.

As shown in figure 2(a), the flow stress generally shows three-stage variation, namely the work-hardening (WH) stage (stage 1), peak stage (stage 2) and softening stage (stage 3). It has been a consensus that the decrease of flow stress after reaching peak value is predominated by the combined effect of DRV and DRX, which overcomes the effect of WH [26–28]. The relative softening (RS) was usually introduced to quantify the softening degree and it is described by a mathematically expression as equation (5).

\[
RS(\%) = \frac{\sigma_p - \sigma_{0.91}}{\sigma_p} \times 100\% 
\]

Where \( \sigma_p \) and \( \sigma_{0.91} \) are the peak stress and the stress at the strain of 0.91. The variation of RS-value under different strain rates and temperatures are shown in figure 2(b). It can be seen that the RS-value decreases with increasing temperature and approximately decreasing with increasing strain rate. Generally, higher RS-value corresponds to more violent DRX. The results in figure 2(b) indicate that DRX operates easier at lower deformation temperature and lower strain rate. Moreover, it is worth noting that, the RS-value at 500 °C & 10s⁻¹ is 0 indicating that the peak stress occurs at the strain of 0.91, namely WH effect predominant continuously.

3.2. Constitutive analysis

Equation (4) is the widely spread Arrhenius model which describe the constitutive relationship between strain rate (\( \dot{\varepsilon} \)), true stress (\( \sigma \)) and temperature (\( T \)) at a certain true strain. In the equation, \( Q \) and \( R \) are deformation activation energy (kJ mol⁻¹) and gas constant (8.31 kJ mol⁻¹ K⁻¹). \( A \), \( \alpha \), \( n \) are the material constants and \( \alpha = \beta/n' \), where \( \beta \) and \( n' \) are the material constants in the Arrhenius model for high stress level and low stress level in the equations (5) and (6) [29, 30], respectively.

![Figure 1. The flow curves of the Al-3.65Cu–0.98Li (wt%) alloy under different deformation conditions.](image-url)
By taking natural logarithm of the both side of equations (4)–(7) and linear fitting, the value of $\alpha$ is obtained. Moreover, the values of $n$ and $Q$ are obtained by the equations (7) and (8). After that, the value of $\ln A$ can be determined by linear fitting [7]. Meanwhile, the flow stress can be predicted by equation (9) after the determination of $\alpha$, $n$, $Q$ and $\ln A$ [31].

\[
\dot{\varepsilon} = A [\sinh(\alpha \varepsilon)]^n \exp(-Q/RT) \tag{4}
\]

\[
\dot{\varepsilon}_1 = A_1 \varepsilon^n \exp(-Q/RT) \tag{5}
\]

\[
\dot{\varepsilon}_2 = A_2 \sigma^n \exp(-Q/RT) \tag{6}
\]

\[
n = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln(\sinh(\alpha \varepsilon))} \bigg|_{\varepsilon} \tag{7}
\]

\[
Q = nR \frac{\partial \ln(\sinh(\alpha \varepsilon))}{\partial (1/T)} \bigg|_{\varepsilon} \tag{8}
\]

\[
\sigma = \frac{1}{\alpha} \left( \frac{\dot{\varepsilon} \exp(Q/RT)}{A} \right)^{\frac{1}{n}} + \left( \frac{\dot{\varepsilon}_1 \exp(Q/RT)}{A} \right)^{\frac{1}{2}} + 1 \right)^{\frac{1}{2}} \tag{9}
\]

Though this process, e.g. at the strain of 0.5, the values of $\alpha$, $n$, $Q$ and $\ln A$ are calculated to be 0.0147, 5.5777, 159.6197 $\text{kJ mol}^{-1}$ and 25.6217s$^{-1}$, respectively, the values of the four parameters at the strain of 0.05 $\sim$ 0.9 with an interval of 0.05 are calculated. And the relationship between the four parameters and strain were 7th ordered polynomial fitted as shown in figure 3. The polynomial functions between the parameters ($\alpha$, $n$, $Q$ and $\ln A$) and strain are as following:

\[
\alpha = f(\varepsilon) = 0.015 - 0.033 \varepsilon + 0.322 \varepsilon^2
\]

\[
-1.401 \varepsilon^3 + 3.320 \varepsilon^4 - 4.378 \varepsilon^5
\]

\[
+3.012 \varepsilon^6 - 0.843 \varepsilon^7
\]

\[
n = g(\varepsilon) = 7.88 - 41.31 \varepsilon + 335.76 \varepsilon^2
\]

\[
-1412.11 \varepsilon^3 + 3286.04 \varepsilon^4
\]

\[
-4278.88 \varepsilon^5 + 2917.9 \varepsilon^6 - 810.43 \varepsilon^7
\]

\[
Q = h(\varepsilon) = 215.0 - 490.1 \varepsilon + 3295.8 \varepsilon^2
\]

\[
-12644.3 \varepsilon^3 + 26112.9 \varepsilon^4 - 29683.0 \varepsilon^5
\]

\[
+17606.8 \varepsilon^6 - 4265.9 \varepsilon^7
\]

\[
\ln A = j(\varepsilon) = 35.06 - 80.14 \varepsilon + 536.04 \varepsilon^2
\]

\[
-2090.53 \varepsilon^3 + 4411.40 \varepsilon^4
\]

\[
-5142.86 \varepsilon^5 + 3141.87 \varepsilon^6 - 787.50 \varepsilon^7
\]

To involve the influence of strain, the strain compensated Arrhenius model is derived from equation (9) and it is denoted as:

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Figure 2. (a) Different stages of the flow curve, (b) the variation of RS-value under different strain rates and temperatures.
As a result, the comparison between predicted experimental stress is shown in Figure 4. The hollowed dots are the predicted value and the solid lines are the experimental. Most of the predicted values are close to the experimental. The relative error (R) and average absolute relative error (AARE) which are generally introduced as the evaluators in previous research [32, 33] are determined to be 0.9898 and 4.70%, respectively, revealing good prediction accuracy.

3.3. Processing map

3.3.1. Strain rate sensitivity index

The plastic deformation capability of an alloy is generally characterized by the strain rate sensitivity index (m) [10, 34]. The higher value of this index indicates better plasticity, even super plasticity. This m-value is generally calculated through the following equation at a certain strain [35–37].

\[
\sigma = \frac{1}{f(\dot{\varepsilon})} \left( \frac{\dot{\varepsilon} \exp (h(\dot{\varepsilon}) / RT)}{\exp (j(\dot{\varepsilon}))} \right)^{\frac{1}{f(\dot{\varepsilon})}} + \left( \frac{\dot{\varepsilon} \exp (h(\dot{\varepsilon}) / RT)}{\exp (j(\dot{\varepsilon}))} \right)^{\frac{2}{f(\dot{\varepsilon})}} + 1^{\frac{f(\dot{\varepsilon})}{2}}
\]

(11)

Figure 5 shows the response surface of m-value under different temperature and strain rate and partial corresponding inverse pole figure (IPF) maps of the EBSD results at the strain of 0.91. The m-value roughly increases with the increasing temperature and decreasing strain rate and it reaches a peak value of 0.26 at 500 °C & 0.01s⁻¹ and a valley value of −0.04 at 350 °C & 10s⁻¹, indicating the best and worst plasticity of this alloy under current condition, respectively. The IPF maps corresponding to the m-values of 0.07, 0.15 and 0.26 were presented in this figure. The thin white line and bold black line represent low angle grain boundary (LAGB, >2° and <15°) and high angle grain boundary (HAGB, >15°), respectively. It is denser of both LAGB and HAGB at the m-value of 0.07 comparing to the others, revealing stronger dynamic softening effect. At this condition, grain fragmentation is pronounced inside the squashed grains indicating DRX proceeded violently, moreover, massive LAGB indicating strong DRV operates, the comprehensive effect of the two prominent mechanisms on
Figure 4. The comparison between predicted and experimental flow stress.

Figure 5. The response surface of $m$-value under different temperature and strain rate and partial corresponding IPF maps of the EBSD results.
flow stress can also be verified by the RS-value which is approximately 19% at this condition. However, the deformation homogeneity at this condition is relative weak, which may lead to lower plasticity. At higher strain rate of 0.1s$^{-1}$ and the same temperature with a m-value of 0.15, few grain fragmentation is observed, simultaneously, LAGBs are much less but more uniform than these at 350 °C & 0.01s$^{-1}$. At 500 °C & 0.01s$^{-1}$ with an even higher m-value of 0.26, the grain size is more similar, indicating more uniform deformation.

3.3.2. Construction of processing map
A processing map consists of a power dissipation map which characterizes the PDE with contour map and an instability map which estimates the instability with an area in which the instability criterion is below zero. According to Prasad et al [38, 39], the PDE ($h$) and instability criterion ($\xi$) are denoted as follows:

$$ h = \frac{2m}{m+1} $$

$$ \xi(\dot{\varepsilon}) = \frac{\partial m}{\partial \ln \dot{\varepsilon}} + m^2 + m^3 < 0 $$

The processing map at the strain of 0.91 is obtained as shown in figure 6. The value on the contour line represents the PDE value and the shaded area is the instability region. The instability region concentrated in the area with high strain rate and low temperature, occupying approximately a window of 350 °C & 0.37 ~ 10s$^{-1}$. The rest area is recognized as safe area, obviously, with various PDE. Generally, higher PDE value corresponds to the DRV microstructure or the microstructure with partial or high recrystallization fraction [40]. For this alloy, the PDE value increases with rising temperature and reaches a peak of 0.39 at the temperature of 500 °C. As shown in figure 5, the microstructure evolution at this condition is uniform deformed and partially recrystallized. According to the distribution of PDE, the optimal area with a window of 440 °C & 0.01 ~ 0.368s$^{-1}$ can be obtained.

3.4. Microstructure analysis
Figure 7 shows the EBSD results under 450 °C with strain rates of 0.01s$^{-1}$, 0.1s$^{-1}$, 1s$^{-1}$, 10s$^{-1}$, the corresponding PDE values are approximately 0.25, 0.3, 0.24 and 0.16 respectively. Comparing the IPF maps under different conditions with each other, more LAGBs and fragment grains are observed at the strain rate of 0.01s$^{-1}$. With the increasing strain rate, the density of LAGB decreases steeply. At higher strain rate, some shear structures with 45° angle to compression direction are marked in red dashed rectangular, which could be the result of flow localization, of course, these were also recognized by the shaded area in processing map, namely the instability area. Figure 7(c) shows the misorientation distributions under the four conditions, the percentages of HAGB are presented and they are 15.45%, 15.55%, 14.52% and 12.26%, respectively. This has the same variation rule with PDE value. In another word, the PDE value, to some extent, can predict the percentage of HAGB. Figure 7(f) shows the dynamic recrystallization volume fractions under the four conditions, the strain rate of 0.01s$^{-1}$, DRXed grains takes near a half of the microstructure, however, this value falls down to 10.67% at the strain rate of 0.1s$^{-1}$. Instead, the percentage of substructure increases to 32.31% from approximately zero, indicating the main microstructure evolution mechanism changes from DRX to DRV with increasing strain.
rate. But, with further increasing of strain rate, the percentage of substructure decreases, and finally drops to 1.29% at the strain rate of 10 s$^{-1}$. In one word, for this alloy, DRX operates stronger only at sufficient low strain rate and its volume drops to a lower and stable state after strain rate exceeding 0.1 s$^{-1}$. Meanwhile, substructure forms to substitute the decreased volume fraction of DRXed grains, and this volume fraction decreases with further increasing strain rate.

Figure 8 shows the geometrical necessary dislocation (GND) maps under different conditions, which were calculated by MTEX, an open source and free toolbox based on MATLAB. Also, schematic diagrams of microstructures are given for all the GND maps. Figures 8(a)–(c) show the GND maps under 0.01 s$^{-1}$ with temperatures of 350 °C, 450 °C and 500. At 350 °C, it can be seen that, a fine grain band formed by massive DRXed grains and coarse grains coexist, accompanied with heterogeneously distribution of dislocation. Moreover, dislocation concentration line (dark red line in GND map) crossing grains can be observed at this condition, which results from the low annihilation speed of dislocation at lower temperature, accelerating dislocation pileups at already existed LAGB and providing driving force to the transformation to HAGB. At higher temperature of 450 °C, the DRXed grains are not as dense as them at lower temperature, but intensive dislocation cells are observed near grain boundaries. As shown in figure 8(b), high temperature promotes the motion and annihilation of dislocations, which decreases the dislocation resource for the formation of massive DRXed grains and dislocation concentration line. The dislocation motion is still hindered by the grain boundaries, which promotes the formation of intensive dislocation cells near these areas. In figure 8(c), fewer DRXed grains are observed on the grain boundaries, higher temperature facilitates homogeneous deformation in most grains. Pronouncedly dislocation cell structures are observed, which is a uniform DRV structure and has the tendency to operate uniform DRX.

Figures 8(d)–(f) show the GND maps under 450 °C with strain rates of 0.1 s$^{-1}$, 1 s$^{-1}$ and 10s$^{-1}$. At higher strain rate, the DRXed grain band gradually vanishes, instead, more intensive dislocation cells appear. Besides, the number of dislocation cell structure decreases with increasing strain rate, accompany with the high dense dislocation pileups extending from grain boundaries to interiors. The increasing strain rate accelerates the generation of dislocation and the motion of dislocation is impeded by grain boundary (which agrees with Li et al [41]), then dislocation pileup forms. Under higher temperature or lower strain rate, this process is weakened due to higher diffusion activation energy (offered by high temperature) and sufficient deformation time (offered by low strain rate) which promote the motion and annihilation of dislocations. Under the opposite conditions, dislocations were firstly hindered by grain boundary and dislocation pileups form, and thus the subsequent dislocation motions were hindered by the former dislocation pileups, finally causing the extending of high dense dislocation pileups to the interiors of grains.
4. Conclusions

This work investigated the flow behavior, constitutive modelling, processing map and microstructures of Al-3.65Cu–0.98Li (wt%) alloy by means of isothermal compression tests and microstructure analysis. The following conclusions were obtained:

1. The softening effect during hot deformation was strengthened by the decreasing strain rate and temperature, indicating violently DRX operation.

2. Strain compensated Arrhenius model is suitable to describe the flow behavior of this alloy. Because it can predict the flow stress with an R-value of 0.9898 and an AARE-value of 4.70%.

3. Based on the constructed processing map and verification of microstructure, the instable deformation window and optimal hot working window of this alloy was identified to be 350 °C & 0.37 ~ 10 s^-1 and 440 ~ 500 °C & 0.01 ~ 0.368 s^-1, respectively.

4. According to the GND analysis, the dislocations pileups are more intensive at lower temperature due to low motion and annihilation rate. On the contrary, the dislocation cell distributes more uniform at higher temperature with lower strain rate.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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