Flow Behaviour and Microstructure Evolution of 2196 Alloy during Isothermal Compression

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Abstract. The isothermal compression test of 2196 alloy was carried out by Gleeble-1500 isothermal simulator with deformation temperature from 430°C to 500°C and strain rate from 0.01\textsuperscript{s}\textsuperscript{-1} to 10\textsuperscript{s}\textsuperscript{-1}. The results show that the stress-strain curves are stable flow for the most deformation parameters. These stable flow exhibit a good ductility of 2196 alloy, also confirmed by no crack on the edge of the compressed samples. The constitutive equation is established using exponential relationship and the average activation energy \(Q\) is 189.2kJ/mol. Microstructure observation show that very few recrystallized grains at original grain boundaries and triple junctions for the all deformation parameters. It is clear that the dynamic recrystallization is difficult for the 2196 alloy suffered common thermal deformation, while the main microstructure evolution mechanism is dynamic recovery. At the same deformation temperature, the recrystallization fraction decreases with increased strain rate, while the proportion of subgrain increases. The recrystallization and dynamic recovery are two main factors for the microstructure characteristics.

1. Introduction

Al-Li alloy is one type of aluminum alloy with Li as the main element. It not only has the advantages of high specific strength and high specific rigidity comparing with conventional high-strength aluminum alloy, but also has good corrosion resistance and heat resistance [1]. Comparing with the conventional aluminum alloy, Al-Li alloy can reduce the structure mass by about 12\% and improve the rigidity by about 16\%. Adding 1\% (mass percent) Li to the aluminum alloy can reduce the density by 3\% and increase the rigidity by 6\% [2, 3]. Therefore, Al-Li alloy is an ideal material for aerospace structures in the 21st century [4].

The 2196 alloy, developed by Alcan Company, can replace 7075 alloy as the main structural material. It had been used for stringer, floor beams and seat rails in A380 aircraft. For a long time, precipitation hardening of Al–Cu–Li system always attract strong interest, which leads to large number of studies focus on the relationship between strength and aging regimes [5–7], and also hot deformation processing[8, 9]. Recently, microstructure evolution and mechanical properties of 2196 Al-Li alloy in hot extrusion process had been reported [10], where different deformed microstructures can be observed after hot extrusion with different temperatures and extrusion speeds.

The study on high temperature deformation behavior of Al-Li alloy is an important field. Mass of researches focus on the experimental test, establishing constitutive equation for simulation models, clarifying dynamic restoration mechanism, and obtaining processing mapping for choose reasonable process [11, 12]. Due to the tense relationship between microstructure and properties, researchers pay more attention to dynamic restoration mechanism. Dynamic recovery (DRV) and dynamic recrystallization (DRX) are co-responsible for the dynamic restoration during the isothermal compression process for the most Al-Li alloy. However, the proportion between the two behaviors is
different for the various alloys and their deformation parameters. For example, the large Al-Cu-Mn particles can stimulate nucleation of DRX in 2050 Al-Li alloy during hot compression [12].

In present study, isothermal compression test of 2196 alloy was carried out to study the stress-strain relationship and constitutive equation, and also to obtain the microstructure evolution characteristic. These data are all useful to optimize working process and improve the product quality.

2. Experimental Materials and Methods

The ingot of 2196 alloy was prepared by direct chill (DC) casting, and its chemical composition was shown in Table 1. The compression specimens are cut from the homogenized ingot and its size is Φ10mm×15mm. The isothermal compression was carried out on Gleeble-1500 isothermal simulator. The deformation temperatures are 430ºC, 460ºC, 480ºC and 500ºC. The strain rates are 0.01s⁻¹, 0.1s⁻¹, 1s⁻¹ and 10s⁻¹, which are chosen to very close to actual thermal deformation velocity of aluminum alloy extrusion. Prior to the test, the samples were heated to the targeted temperatures and then held for 3 minutes to ensure temperature homogeneity. During the compression, the instant temperature was measured by thermocouples welded to the center area of the sample surface. Nickel powder was daubed on the two end surfaces of the sample to reduce friction. The height reduction is 75%, corresponding to the true strain of 1.25. The compressed specimens were water cooled and then sectioned along the compression axis.

The specimens for optical metallographic were polished mechanically and etched in an acid solution. The optical micrographs (OM) were observed by ZEISS-AXIO. JEOL 7800F scanning electron microscope with electron back-scattering diffraction (EBSD) system was used to investigate the detailed substructure characteristics. The EBSD samples were obtained by mechanically grinding and electrochemical polishing.

Table 1. Chemical composition of 2196 alloy (wt/%)

| Cu  | Mg  | Li  | Ag  | Zr  | Ti  | Fe  | Si  | Al  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 2.7 | 0.5 | 1.8 | 0.45| 0.1 | ≤0.1| ≤0.1| ≤0.12| Bal.|

3. Results and Discussion

3.1. Original Microstructure

Figure 1 shows the optical microstructure and SEM image of the ingot after homogenization. In Figure 1a, near equiaxed grain with diameter of 200-400μm display a conventional DC casting microstructure. Although the most soluble coarse phases mainly Al2CuLi had been redissolved to α-Al matrix after homogenization, a small mount of residual Al2CuLi phase and insoluble Al7Cu2Fe phase less than 100μm still exist in matrix, shown Figure 1b. These unexpected phases participate in the following thermal deformation and are broken up to the smaller ones.

Figure 1. Optical microstructure (a) and SEM image showing residual coarse phases (b) for the homogenization treated ingot
3.2. True Stress-Strain Relationship

Figure 2 shows the true stress-strain curves of 2196 alloy at strain rates of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\), 1 s\(^{-1}\) and 10 s\(^{-1}\), and temperatures of 430\(^\circ\)C, 460\(^\circ\)C, 480\(^\circ\)C and 500\(^\circ\)C. It is obviously that the stress-strain curves are all stable flow only with slight wave, especially for the high temperatures. These stable flow curves exhibit a good ductility of 2196 alloy during the deformation conditions. This has been also confirmed by the compressed samples with no crack on the edge. In actually, our extrusion test and previous study\(^8\) had shown that the alloy is extruded easily under the general extrusion process. In this study, the stable flows for the strain rate of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) and 1 s\(^{-1}\), and slightly softening for the strain rate of 10 s\(^{-1}\), can be explained by the traditional microstructure evolution theory. Hot deformation at high temperature is a process of work hardening and dynamic softening. In the initial stage of deformation, dislocation density increases rapidly with the progress of isothermal compression, which shows that work hardening is stronger than dynamic softening. With the development of thermal deformation, the dynamic softening degree increases, which counteracts or even exceeds the work hardening process, so the stress tends to be stable or slightly decreased [13].

![Figure 2](image)

**Figure 2.** True stress-true strain curves of 2196 alloy under different deformation temperatures. (a) 430\(^\circ\)C; (b) 460\(^\circ\)C; (c) 480\(^\circ\)C; (d) 500\(^\circ\)C

It is should be noted that the flow stress increase at the strain rate of 10 s\(^{-1}\), which is obviously different from that of the lower strain rate of 0.01 s\(^{-1}\)-1 s\(^{-1}\). This may be explained by the temperature changes during the hot compression. Figure 3a shows the instant temperatures of the samples deformed at temperatures of 430\(^\circ\)C-500\(^\circ\)C and strain rate of 10 s\(^{-1}\). The temperatures tested from the thermoelectric couple gradually increase because of the deformation heating is not diffusion at the short time deformation. The increased instant temperature induces the deformation resistance instantly. Comparing with the deformation heating at the lower strain rates of 0.01 s\(^{-1}\)-1 s\(^{-1}\) in Figure 3b, the instant temperatures keep stable approximately, which is an essential factor for the stable flow stress.
Constitutive equation is very useful to establish material model for predict plastic strain and temperature during finite element numerical simulation. In this study, constitutive equation of 2196 alloy is established using exponential relationship. The relation of temperature and strain rate can be expressed by Z factor, that is, Zener-Hollomon parameter [14].

\[
Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) \tag{1}
\]

Where Z increases with the increase of strain rate and the decrease of deformation temperature, Q is the activation energy for hot deformation, reflecting the difficulty of hot deformation, and is also an important mechanical parameter for hot deformation of materials[15], T is the deformation temperature; \( \dot{\varepsilon} \) is the strain rate; R is the gas constant, 8.314J·mol\(^{-1}\)·K\(^{-1}\).

Under the condition of low stress level, the relationship between steady flow stress \( \sigma \) and strain rate \( \dot{\varepsilon} \) can be expressed by exponential equation [16]

\[
\dot{\varepsilon} = A_1 \sigma^{n_1} \tag{2}
\]

Combining equation (1) and (2), the Z is expressed as

\[
Z = A_1 \sigma^{n_1} \exp \left( \frac{Q}{RT} \right) \tag{3}
\]

Where \( A_1 \) and \( n_1 \) are constant independent of temperature. Combining equation (1) and (3), and taking partial differential of the natural logarithm on the two sides of formula (3):

\[
Q = R \left\{ \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma} \right\}_T \left[ \frac{\partial \ln \sigma}{\partial (1/T)} \right]_\varepsilon \tag{4}
\]

Where, the first item on the right represents the slope of \( \ln \dot{\varepsilon}-\ln \sigma \) relation curve and the second phase represents the slope of \( \ln \sigma-1/T \) relation curve.

For 2196 alloy in this study, the steady stress (when \( \varepsilon = 0.4 \)) is used to calculate \( \ln \sigma \). Corresponding to the strain rate and temperature, the relation curve of \( \ln \dot{\varepsilon}-\ln \sigma \) and \( \ln \sigma-1/T \) are plotted by linear regression method, shown in Figure 4. The linear correlation coefficients of Figure 4(a) and Figure 4(b) are greater than 0.97 and 0.99, respectively, which means the fitting is accurate by exponential equation expressing.

The deformation activation energy at various deformation temperatures can be obtained, and the average value \( Q \) is 189.2kJ/mol. The relation curve between \( \ln Z \) and \( \ln \sigma \) is drawn in Figure 5. The \( \ln Z \) and \( \ln \sigma \) are linear relationship, introducing that the high temperature flow stress behavior of 2196 alloy can be described by Z parameter.
Figure 4. Relationship of flow stress to strain rates and deformation temperatures:
(a) ln$\dot{\varepsilon}$-ln$\sigma$, (b) ln$\sigma$-1/T

Figure 5. lnZ-ln$\sigma$ relationship of 2196 alloy

Above all, the material parameters of $Q$, $n_1$ and $A$ are substituted into the equation (3), and the constitutive equation is as following:

$$\dot{\varepsilon} = 9.066 \times 10^6 \, \sigma^{5.884} \exp \left[ -189.2 \times 10^3 / (RT) \right]$$

This equation provides theoretical basis for hot working simulation, and has important practical significance to predict stress level under special deformation temperature and strain rate.

3.4. Microstructure Evolution
The thermoplastic deformation process is controlled by thermal activation [8]. During the deformation, the average kinetic energy of the alloy atom is higher, which results in larger amplitudes of vibration and promotes vacancy movement, dislocation formation, dislocation slip and climb. These defects and crystal structure changing are the main parts for studying the microstructure evolution.

Figure 6 shows the EBSD images of 2196 alloy deformed under different deformation conditions. The red line is small angle grain boundary ($\leq 15^\circ$), and the black line is the large angle grain boundary ($>15^\circ$). Black area, being coarse phases containing element Fe or Al2CuLi phase not redissolved during homogenization, is not resolved by JEOL 7800F scanning electron microscope.

It can be seen that the samples are transformed from equiaxed grains to elongated fiber after isothermal compression. The compressed microstructures are similar with that of extruded shown in literature [10]. Very few recrystallized grains (noted by white circles) are observed at original grain boundaries and triple junctions for the all deformation parameters. It is clear that the recrystallized grains are all similar for the present deformation parameters, which means that the dynamic
Recrystallization is difficult for the 2196 alloy suffered common thermal deformation, even during the extrusion with large plastic strain. So the main microstructure evolution mechanism for the alloy is dynamic recovery. This conclusion can be confirmed by the large number of sub-grain interior the original grains. There are a mass of small angle grain boundaries called sub-grain boundaries.

![Figure 6. EBSD images of 2196 alloy deformed under different deformation conditions](image)

(a) 430°C,0.01s⁻¹; (b)430°C,10s⁻¹; (c) 500°C,0.01s⁻¹; (d) 500°C,10s⁻¹

Table 2 shows the recrystallization fraction extracted from EBSD images of Figure 6. At the same temperature, the recrystallization fraction decreases with strain rate increasing, while the proportion of subgrain increases with strain rate increasing. For example, at the deformation temperature of 430°C, the recrystallization fraction decreases from 5.7% to 2.2%, while the proportion of subgrain increases from 74.7% to 81.2%. This means that no enough time for recrystallization at the strain rate of 10s⁻¹, and also no enough time for mass of low angle grain boundaries to recovery. This dynamic recovery is enhanced at the higher temperature of 500°C, with the recrystallization fraction from 5.8 to 3.2% and the proportion of subgrain from 69.7% to 71.0%. The higher temperature supplies a convenient condition for dynamic recovery. Generally, the recrystallization is not expected for the structural metal, especially for aluminum alloy. Because the recrystallization not only decrease the strength but also ductility, as well as fracture toughness, many researchers always make great efforts to alleviate recrystallization by different method. To choose a reasonable deformation temperature and strain rate, as well as their matching, is a basic way.

| Deformation conditions | 430°C  | 500°C  |
|------------------------|--------|--------|
|                        | 0.01s⁻¹| 10s⁻¹  | 0.01s⁻¹| 10s⁻¹  |
| Recrystallization fraction | 5.7%  | 2.2%  | 5.8%  | 3.2%  |
| Proportion of subgrain | 74.7% | 81.2% | 69.7% | 71.0% |

4. Conclusion
(1) The stress-strain curves are stable flow for the most deformation parameters. These stable flow exhibit a good ductility of 2196 alloy, also confirmed by no crack on the edge of the compressed samples.

(2) The constitutive equation is established using exponential relationship and the average activation energy Q is 189.2kJ/mol. The constitutive equation of flow stress was shown as follows:

$$\dot{\varepsilon} = 9.066 \times 10^6 \sigma^{5.884} \exp \left[-189.2 \times 10^3/(RT)\right]$$
Very few recrystallized grains are observed at original grain boundaries and triple junctions for all deformation parameters. It is clear that the dynamic recrystallization is difficult for the 2196 alloy suffered common thermal deformation, while the main microstructure evolution mechanism is dynamic recovery.

At the same deformation temperature, the recrystallization fraction decreases with increased strain rate, while the subgrain proportion increases.

5. References
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