Transition in Deformation Mechanism during High-Temperature Tensile Testing of Friction-Stir-Processed 5083 Aluminum Alloy

by

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The stress-strain relationship and microstructural evolution of a fine-grained 5083 aluminum alloy produced via friction-stir processing (FSP) during high-temperature tensile deformation were investigated. The FSP of the 5083 aluminum alloy resulted in the formation of a homogeneous fine-grained microstructure. Based on the stress-strain relationship, it was found that the 5083 aluminum alloy exhibited a large elongation, especially at a temperature above 693 K. The stress exponent and the activation energy for deformation, which were determined by the flow stress at a nominal strain of 0.03, were approximately 2.5 and 123 kJ/mol, respectively. These results suggest that grain boundary sliding accommodated by the solute drag motion of dislocations was the rate-controlling process in the early stages of deformation. The largest elongation of 350% occurred at 743 K and an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. In this case, the grain aspect ratio increased with increasing nominal strain, which indicated that equiaxed grains continuously elongated along the tensile axis during high-temperature deformation because of dislocation creep. The value of the stress exponent increased with increasing strain. From our experimental results, the dominant deformation mechanism was determined to change during the tensile test, and the contribution of dislocation creep to the high-temperature deformation increased as the deformation proceeded.

Key words:

High-temperature deformation, Al-Mg alloy, Grain-boundary sliding, Solute-drag creep, Friction-stir processing

1 Introduction

5083 aluminum alloy is a typical Al-Mg alloy that has been used for automotive body-sheet applications because it has excellent strength, corrosion resistance, and weldability. High-temperature forming of 5083 aluminum alloy into sheet metal parts has attracted considerable interest because conventional plastic forming at room temperature is limited by its lower formability. Thus, many studies have investigated the tensile deformation behavior at high temperature of Al-Mg alloys¹⁾⁻¹¹⁾. It is widely believed that the combination of an equiaxed, stable, fine-grained structure with a grain size of less than 10 μm and surrounded by high-angle grain boundaries (GBs) is the most important requirement for excellent elongation. According to the deformation-mechanism map developed by Ito for the Al-6 mol%Mg alloy at 673 K¹¹⁾, the deformation mechanism of an Al-6 mol%Mg alloy can change from solute drag to GB sliding with decreasing grain size. If the grain size is refined, high ductility can be expected because of the combination of

GB sliding and slip controlled by the solute-drag motion of dislocations.

In this study, the stress-strain relationship of a fine-grained 5083 aluminum alloy was investigated using high-temperature tensile testing over various temperatures and strain rates to demonstrate the high ductility achieved by combining GB sliding and dislocation slip controlled by the solute-drag motion of dislocations. Moreover, the transition in the deformation mechanism during high-temperature deformation of fine-grained 5083 aluminum alloy was investigated. A friction-stir processing (FSP), which was developed by Mishra¹²⁾, are known to reduce the grain size of metallic materials. In this study, FSP was used to obtain a fine-grained microstructure.

2 Experimental Procedure

An annealed 5083 aluminum alloy rolled sheet with dimensions of 6 mm (thickness) $\times$ 50 mm (width) $\times$ 300 mm (length) was used as the starting material for FSP. The

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composition of the alloy in mass% was Al-4.5Mg-0.68Mn-0.19Fe-0.13Si-0.11Cr-0.02Cu.

FSP was carried out using an SHH204-720 FSW machine (Hitachi Setsubi Engineering Co., Ltd.). Single-pass FSP was performed on the sheet along the rolling direction (RD) at a rotation rate of 1200 rpm (revolutions per minute) and a speed of $6.7 \text{ mm} \cdot \text{s}^{-1}$, followed by air cooling. A schematic drawing showing the orientation of the tensile test specimen relative to the track of the FSP is shown in Fig. 1. The welding tool was fabricated from a tool steel and comprised a concave shoulder 14 mm in diameter and an M6-threaded cylindrical pin 5.8 mm in length. The tool tilting angle was forward 3° , and the tool plunge depth was controlled to be 5.9 mm.

Specimens for tensile testing with gage dimensions of 6 mm (length) $\times$ 2 mm (width) $\times$ 2 mm (thickness) were carved out of the normal direction (ND)–transverse direction (TD) surface of the stir zone, as shown in Fig. 1. Next, tensile specimens were annealed at 473 K for 0.5 h to obtain a recovered microstructure. Tensile tests were conducted using a conventional testing machine (Instron Model 5586, Instron Corp.) at 643, 668, 693, 718 and 743 K with a constant crosshead speed corresponding to initial strain rates of 1.0×10^{-3} , 1.0×10^{-2} and $1.0 \times 10^{-1} \text{ s}^{-1}$. The tensile direction was parallel to the TD of the sheet. Moreover, to clarify the microstructural evolution and transition in deformation mechanism during the high-temperature deformation, the tensile specimens at 743 K with a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ were deformed to nominal strains of 0.48, 0.95, and 1.40. Prior to applying the load, the specimens were kept for 1200 s at the testing temperature to reach thermal equilibrium, which was measured using a thermocouple clamped close to the specimens. The tensile direction was parallel to the TD. Upon completion of the tests, the specimens were immediately cooled with a blower to preserve the microstructure.

The samples for microstructural observation were electropolished at 13 V for 180 s in a solution containing 10 vol.% HClO_4 + 20 vol.% $\text{C}_2\text{H}_4(\text{OH})_2$ + 70 vol.% $\text{C}_2\text{H}_5\text{OH}$ at 255 K. Microstructural observations and crystal orientation measurements were conducted on the ND–TD plane from the RD using a field-emission scanning electron microscope (FE-SEM; JEOL JSM-7001F, JEOL Ltd.) equipped with an electron backscattering diffraction (EBSD) detector. EBSD observations with a scanned area of $80 \text{ } \mu\text{m} \times 80 \text{ } \mu\text{m}$ were carried out using an accelerating voltage of 15 kV with a step size of 0.1 μm . The crystal orientation analyzes were conducted by HKL Channel 5 software (Oxford Instruments).

3 Results and discussion

3.1 Microstructure of FSP samples before and after annealing

Figure 2(a) shows the inverse-pole-figure (IPF) map and GB map constructed from the EBSD data for the stirred zone after FSP but before annealing. In the IPF map, the correspondence between the color and the crystallographic direction for each point is indicated in the standard stereographic triangle parallel to the RD of the FSP sheet. In the GB map, high-angle boundaries with a misorientation angle of $>15^\circ$ are shown as green lines, and low-angle boundaries between 2° and 15° are shown as red lines. Boundaries less than 2° were not taken into account because of the limited angular resolution of the EBSD system¹³. Moreover, noise-reduction treatment was applied in order to more clearly show the GBs. In these maps, horizontal and vertical directions are TD and ND, respectively. An equiaxed, fine-grained microstructure, surrounded by high-angle boundaries, formed homogeneously in the stirred zone. The mean grain sizes and grain aspect ratio (AR) are shown in Fig. 2. In this figure, d_{TD} and d_{ND} are the grain sizes determined from GB maps by the linear-intercept method in the TD and ND, respectively. The value of AR was defined as the ratio of d_{TD} to d_{ND} . The mean grain size and AR of FSPed 5083 aluminum alloy before annealing were about 4 μm and 1.1, respectively. (See Fig.2(a)) It was confirmed that FSP produced fine grains with an equiaxed homogeneous grain-size distribution throughout the processed region, and that FSP was an effective route to produce a fine-grained microstructure in a commercial 5083 aluminum alloy. It is widely believed that during FSP, the material experiences dynamic recrystallization, which is contributed to by the severe deformation from the intense stirring effect and a dynamic recovery and recrystallization process. It was observed that there were many low-angle boundaries present in the grains. This is in agreement with the occurrence of the dynamic recrystallization that takes places under continuous strain, coupled with the rapid recovery and migration of sub-grain/GBs during FSP¹⁴.

Figure 2(b) shows the IPF map and GB map for the stirred zone after FSP and annealing. There were some low-angle boundaries remaining after annealing. This indicates that this recovered microstructure composes of large subgrains. The mean grain size increased from 4 to 5 μm during annealing. The sample maintained a fine-grained equiaxed microstructure separated by high-angle boundaries after annealing.

3.2 Stress–strain relationship

Figure 3 shows the nominal stress–nominal strain curves from the tensile tests performed at 643, 668, 693, 718 and 743 K with initial strain rates of 1.0×10^{-3} , 1.0×10^{-2} and $1.0 \times 10^{-1} \text{ s}^{-1}$ after FSP and annealing. The yield stress and tensile stress increased with increasing strain rate and decreasing temperature. After reaching the maximum stress at

a nominal strain of around 0.05, the flow stress continuously decreased until fracture, and no steady-state flow occurred. The elongation-to-fracture increased with decreasing strain rate and increasing temperature and showed about 350% achieved at 743 K with $1.0 \times 10^{-3} \text{ s}^{-1}$. In this case, the stress-strain relationships indicated that the flow stress gradually decreased after the maximum stress exhibited superplastic-like deformation behavior. This indicates that GB sliding was the dominant deformation mechanism.

3.3 Deformation mechanism in the early stages of deformation

Generally, plastic deformation at high temperature is described using a constitutive equation ¹⁵:

$$\dot{\varepsilon} = A \frac{GDb}{kT} \left(\frac{\sigma}{G} \right)^n \left(\frac{b}{d} \right)^p \quad (1)$$

where $\dot{\varepsilon}$ is a strain rate, A is a dimensionless constant which incorporates microstructural features other than the grain size, G is the shear modulus, D is the diffusion coefficient, b is the Burgers vector, k is Boltzmann's constant, T is the absolute temperature, σ is the flow

Fig. 1 Schematic drawing showing the direction of friction-stir processing and the orientation of the tensile-test specimen relative to the track.

Fig. 2 IPF maps ((a-1) and (b-1)) and GB maps ((a-2) and (b-2)) obtained from EBSD measurements showing the microstructure of friction-stir-processed 5083 aluminum alloy before (a) and after (b) annealing.

stress, n is the stress exponent, d is the grain size, and p is the grain-size exponent. The shear modulus can be converted into the elastic modulus, E , using:

Fig. 3 Nominal stress–nominal strain curves at different strain-rates and different temperatures.

$$E = 2G(1 + \nu) \quad (2)$$

where ν is Poisson's ratio. The elastic modulus was calculated based on the temperature dependence of the elastic modulus of pure aluminum ¹⁶⁾:

$$E(\text{MPa}) = 8.039 \times 10^4 - 42.6 \times T \quad (3)$$

The mean grain size was slightly increased during annealing. However, in our study, it was assumed that the grain size was constant during annealing after FSP. The diffusion coefficient can be expressed using the following equation:

$$D = D_0 \exp\left(-\frac{Q}{RT}\right) \quad (4)$$

where D_0 is a frequency factor, R is the molar gas constant, and Q is the activation energy for deformation. Using Eq. (4), Eq. (1) can be rewritten as:

$$\dot{\varepsilon} = B \left(\frac{\sigma}{E}\right)^n \exp\left(-\frac{Q}{RT}\right), \quad (5)$$

where B is a constant. Therefore, the stress exponent can be calculated using:

$$n = \frac{\partial \log \dot{\varepsilon}}{\partial \log(\sigma/E)}. \quad (6)$$

Figure 4 shows a double-logarithmic plot of strain rate against normalized flow stress. The nominal stress at a nominal strain of 0.03 was used as the value of the flow stress because uniform deformation ended at a nominal strain of about 0.05, after which the flow stress gradually decreased. The stress exponent was evaluated directly from the plots in Fig. 4, and their values are also shown in the figure. The stress exponent gradually decreased with increasing

Fig. 4 Strain-rate dependencies of normalized flow stress to evaluate the temperature dependence of the stress exponent.

Fig. 5 The variation of normalized flow stress as a function of $1000/T$.

temperature, and exhibited 2.5 at 743 K. These results indicate that GB sliding is enhanced at higher temperatures. Generally, the dominant deformation mechanism in fine-grained materials at high temperature is GB sliding, which corresponds to a stress exponent of 2.5 ¹⁵⁾ and is in good agreement with the stress exponent obtained in the present study. This suggests that GB sliding contributed to the large ductile deformation at 743 K. Other researchers have reported that the deformed fine-grained structure exhibits superplastic characteristics during the early stages of deformation ¹⁷⁾⁻¹⁸⁾, and that conventional superplasticity in fine-grained Al-Mg alloys with grain sizes of the order of 10 μm is based on GB sliding as the primary deformation mechanism ¹⁹⁾⁻²⁰⁾. Our experimental results are in agreement with this previous research.

The deformation activation energy can be evaluated according to the following equation.

$$Q = nR \left. \frac{\partial \left(\ln \left(\frac{\sigma}{E} \right) \right)}{\partial \left(\frac{1000}{T} \right)} \right|_{\dot{\varepsilon}} \quad (7)$$

Figure 5 shows the Arrhenius plot for the normalized flow stress in the temperature range from 693 to 743 K at a constant strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ at a nominal strain of 0.03. The slope of the plots indicates $\partial \ln(\sigma/E) / \partial \ln(1000/T)$ in eq. (7). Using the value of the slope, n (2.5) and R , the value of deformation activation energy could be estimated. For the calculation of the slope, the normalized stress in the temperature range from 693 to 743 K was used because n value was found to be similar (2.5~3), as shown in Fig. 4. The estimated activation energy for deformation at a constant strain rate was 123 kJ/mol, which is close to that of Mg diffusion in an Al matrix (126 kJ/mol ²¹⁾), and much higher than that for dislocation pipe diffusion in Al (82 kJ/mol ²²⁾) and GB diffusion in Al (84 kJ/mol ²²⁾). It can be concluded that, in the initial stages of deformation, the dominant deformation mechanism giving rise to the superplastic-like deformation in the present specimens of friction-stir-processed 5083 aluminum alloy was GB sliding, which was rate-controlled by interdiffusion of Mg in Al, that is to say, the deformation is accommodated by solute-drag creep.

3.4 Deformation-mechanism transition during tensile testing

In order to clarify the transition in deformation mechanism during the high-temperature deformation, the microstructural evolution and the change of the stress component during the tensile test at 743 K were investigated. Figure 6 shows IPF maps and GB maps of the microstructure at nominal strains of 0.48, 0.95, and 1.40 at 743 K with a

strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. Figure 7 shows the value of the stress exponent evaluated at nominal strains of 0.48, 0.95, and 1.40 at 743 K. For comparison, the value of the stress exponent at a nominal strain of 0.03 is also plotted in Fig. 7. Figure 3 shows that the nominal strain of 0.03 corresponds to the strain hardening stage in the initial stages of deformation. Up to the maximum flow stress, the deformation along the gage section within the tensile specimen is uniform; however, after the maximum flow stress, the deformation will localize and necking will begin. In this study, the stress component after the maximum stress was evaluated without taking the localization into account.

At a nominal strain of 0.03, GB sliding was the dominant deformation mechanism, as shown in Fig. 4. At this strain, GB sliding can be accommodated by solute-drag creep because the GBs are able to emit and absorb dislocations. After the maximum stress, propagation of necking accompanied by grain elongation occurred. Equiaxed grains continuously elongated along the tensile axis during high-temperature deformation^{10), 23)}; therefore, the AR increased with increasing nominal strain. Accordingly, the value of the stress exponent increased with increasing strain. It is reported that the GB sliding is difficult to occur along the direction of the elongated grains²³⁾. The increase of AR means that dislocation creep would be the important deformation mechanism during the high temperature deformation and that the dominant deformation mechanism changed during the tensile test from the GB sliding to dislocation creep. It is widely considered that a slight necking occurs and propagates through the gauge section of the tensile specimen during high temperature deformation by GB sliding after the maximum stress of a nominal stress-nominal strain curve. The transition of deformation mechanism would be enhanced by necking. The low-angle boundaries found inside grains indicate that the dislocations generated during high-temperature deformation inside the grains and that were converted into a fine sub-grain structure by the recovery process. This fine-grained 5083 aluminum alloy produced by FSP exhibited microstructural stability during high-temperature deformation because abnormal grain growth did not occur. However, as shown in Fig. 6, the grains (d_{TD}) grew slightly to over 10 μm . We believe that the change in deformation mechanism may be due to this grain growth.

4 Conclusions

In this study, the stress-strain relationship and microstructural evolution of a fine-grained 5083 aluminum alloy produced by FSP were investigated. The results can be summarized as follows:

(1) EBSD results showed that the FSP of the 5083 aluminum alloy resulted in the formation of a homogeneous fine-grained

structure with an average grain size of about 4 μm .

(2) The stress-strain relationship indicated that the FSP 5083 aluminum alloy exhibited highly ductile behavior, especially above 693 K, with a maximum elongation of 350% at 743 K and an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$.

(3) The experimentally determined stress exponent and the activation energy for deformation were about 2.5 and 123 kJ/mol, respectively. These results suggest that GB sliding accommodated by solute-drag motion of dislocations was the

Fig. 6 IPF maps and GB maps obtained from EBSD measurements showing the microstructural evolution during deformation at 743 K with a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ at elongations of 0.48, 0.95, and 1.40. Loading was in the horizontal direction.

Fig. 7 Strain-rate dependencies of normalized flow stress to evaluate the transition of the stress exponent during deformation at 743 K.

rate-controlling process in the early stages of deformation.

(4) The grain AR increased with increasing nominal strain. Equiaxed grains continuously elongated along the tension axis during high-temperature deformation because of dislocation creep. The value of the stress exponent increased with increasing strain. The dominant deformation mechanism changed during the tensile test, and the contribution of dislocation creep to the high-temperature deformation increased as the deformation proceeded.

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