Superplastic Deformation Behavior in Mg–Al Alloy, AZ91, Grain Refined by Isothermal-rolling

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The isothermal rolling mill, which allows a large preform for materials having poor workability, has been developed. AZ91 alloy sheets made from Mg–9Al–1Zn tempered to a fine grain size by recrystallization have been prepared and tensioned at elevated temperatures. The maximum total elongation reached 170% and the m-value was found to be 0.5 in range of 1.0×10⁻⁴ s⁻¹ to 2.5×10⁻⁴ s⁻¹ except at the testing temperature of 573 K.
The activation energy required for high temperature deformation has been calculated to be about 124 kJ · mol⁻¹, which is almost equal to the self-diffusion energy of Mg for volume diffusion. Therefore, the superplastic deformation of the recrystallized AZ91 has been accompanied by a volume diffusion.

KEY WORDS: magnesium alloys; superplasticity; isothermal-rolling; high temperature deformation; activation energy; volume diffusion.

1. Introduction
The greatest fault of the Mg–Al alloys is that they are too brittle to work. So far, attempts to add various alloying elements have been performed to improve this brittleness, and many of these results have been reported.¹⁻⁴) There is recently a tendency to improve the mechanical properties by grain refinement based on a recrystallization technique. However, a major problem has developed as to how to produce a large preform below the recrystallization temperature in this technique. Because it is almost impossible to significantly deform Mg–Al alloys which have the hexagonal lattice structure.
As part of obtaining such a large preform, a powder metallurgy (PM)⁵) and an extrusion method called ECAE (Equal Channel Angular Extrusion)⁶) have recently developed superplasticity in Mg–Al alloys.
In this study, the isothermal-rolling mill, which allows such a large preform, has been developed. The recrystallized Mg–Al alloy sheets using this machine have then been tested at various temperatures and at various strain-rates below the recrystallization temperature. The total elongation and strain-rate sensitivity, the m-values, have been obtained and discussed.

2. Experimental Procedure
The AZ91 material, which has the lowest ductility of all the Mg–Al alloys, has been supplied in the form of ingots for this experiment. Its chemical composition is listed in Table 1. The AZ91 ingot was cut to fixed size of 70 mm length, 15 mm width and 10 mm thickness. These billets were heated at 573 K for 300 s in an electric furnace, and then isothermally rolled by 0.5 mm in every pass at 573 K roll surface temperature by using the isothermal-rolling mill shown in Fig. 1, in which the maximum temperature on the roll surface is 673 K and the maximum load is 196 MN. Finally, the rolling reductions in area were 90% with a 1mm thickness. These rolled specimens were further cut into a suitable size, and subsequently recrystallized at 648 K with the average grain size of about 20 µm as shown.

Table 1. Chemical composition of AZ91 alloy (mass%).

<table>
<thead>
<tr>
<th></th>
<th>Mg</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal.</td>
<td>9.0</td>
<td>0.8</td>
<td>0.1</td>
<td>0.1</td>
<td>0.06</td>
<td>0.06</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 1. Roll for isothermal rolling.
in optical microstructure of Fig. 2.

The tension tests were carried out at four different temperatures of 573 K, 598 K, 623 K and 648 K and at the five constant initial strain-rates of $1.0 \times 10^{-3}$ s$^{-1}$, $2.5 \times 10^{-4}$ s$^{-1}$, $1.0 \times 10^{-3}$ s$^{-1}$, $2.5 \times 10^{-3}$ s$^{-1}$ and $1.0 \times 10^{-2}$ s$^{-1}$ after reaching the setting temperatures and held for 10 min. A hydraulic machine with a vacuum chamber equipped with an induction heater was used for these tests at a heating rate of 20 K/min. The size of specimens for tension tests is shown in Fig. 3.

3. Experimental Results and Discussions

The tension test results to failure of the AZ91 sheets at high temperatures are shown in Fig. 4, plotting the strain-rate on the $X$-axis and the total elongation on the $Y$-axis. The values of the total elongation exceeded over 100% at the testing temperature over 623 K at strain-rate below $1.0 \times 10^{-2}$ s$^{-1}$, and the maximum total elongation reached 170% as shown in Fig. 5 on condition of testing temperature of 623 K and at strain-rate of $2.5 \times 10^{-3}$ s$^{-1}$. It seems that this maximum-value of total elongation is comparatively large in Mg–Al alloys which have a hexagonal lattice structure.

The results of the tension tests are shown in Fig. 6, plotting the strain-rate on the $X$-axis and the peak flow stress, which corresponds to a strain of 0.13, obtained from the stress vs. strain curves on the $Y$-axis. The $m$-values exceeded over 0.5 at strain-rates from $1.0 \times 10^{-3}$ s$^{-1}$ to $2.5 \times 10^{-4}$ s$^{-1}$ except at the testing temperature of 573 K. The total elongation and ultimate tensile strength at a normal temperature were 6% and 255.8 MPa in the neighborhood of $1.0 \times 10^{-3}$ s$^{-1}$, respectively.

The optical microstructures on several positions in Fig. 5 were shown in Fig. 7, where the gray parts indicated the Al$_2$Mg$_7$, called $\gamma$ phase, and the black parts indicated the cavities. The average grain size became larger during tension testing, and found to be about 30 $\mu$m after failure.

Generally, the ln $\sigma$ vs. ln $\dot{\varepsilon}$ curves present an “S” shape in superplasticity. Therefore, there are many cases where these curves are divided into three regions of I, II and III based.
on the magnitude of \( m \), and the region to produce the maximum \( m \)-value, that is, region II is defined as the hot regime. In addition to this regime, region II is seen and the maximum \( m \) exceeds 0.3 in Fig. 4. Next, it was decided to examine the mechanism in region II from an activation energy standpoint. A thermally activated relaxation process happens over the temperatures of the half melting point 0.5 Tmpt for many polycrystalline metals and alloys. This process means that a deformation mode changes from a lattice dislocation type to a local dislocation or diffusion type.

Assuming a thermally activated mechanism of hot deformation, the strain-rate \( \dot{e} \) at testing temperature \( T \) can be given by the following rate equation.

\[
\dot{e} = A \cdot f(\sigma) \cdot \exp(-Q/RT) \quad \text{(1)}
\]

Where, \( Q \) and \( R \) are the activation energy required for deformation and the gas constant, respectively, and \( A \) is a constant.

Here, \( f(\sigma) \) is determined by the following equation.

\[
f(\sigma) = (\sigma/K)(1/m) \quad \text{(2)}
\]

Where, \( \sigma \) is the peak flow stress and \( K \) is a constant.

If Eq. (2) is substituted into Eq. (1), the following equation is obtained.

\[
\ln \dot{e} = \ln A - (1/m) \ln K + (1/m) \ln \sigma - Q/RT \quad \text{(3)}
\]

Assuming that \( \ln \sigma \) is a constant, Eq. (3) is given as follows.

\[
\ln \dot{e} = \text{const.} - Q/RT \quad \text{(4)}
\]

Plotting the relation of \( \ln \dot{e} \) vs. \( 1/T \) by using Eq. (4) in Fig. 3, Fig. 8 is obtained and the activation energy \( Q \) is calculated to be 124 kJ·mol\(^{-1}\), in which the constant \( \sigma \) of 30 MPa is adopted.

The self-diffusion energies of Mg for both volume diffusion and grain boundary diffusion are summarized in Table 2.\(^7\) The activation energy of 124 kJ·mol\(^{-1}\) obtained from the high temperature tensile tests corresponds to the self-diffusion energy of Mg for volume diffusion. Consequently, hot deformation of the recrystallized AZ91 seems to be accommodated by the volume self-diffusion of Mg.

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**Fig. 7.** Optical microstructures after failure on several positions in Fig. 5.

**Fig. 8.** Activation energy required for high temperature deformation.
Mabuchi et al.\(^5\) indicated using the extruded PM AZ91 bar with grain sizes of 1.0–2.8 \(\mu m\) and IM AZ91 bar with grain sizes of 4.1–5.0 \(\mu m\) that the total elongation exceeded over 250% at the testing temperature of 573 K at strain-rate of 1.0 \(\times 10^{-2}\) s\(^{-1}\), and over 400% at the testing temperature of 523 K at strain-rate of 3.3 \(\times 10^{-4}\) s\(^{-1}\) for each materials, respectively, and attained the high \(m\)-values exceeded over 0.5 at wide strain-rate region for both materials.

Further they calculated that the values of activation energy required for superplastic flow for both materials were 121 kJ · mol\(^{-1}\), which was higher than the value for grain boundary diffusion, corresponding to the above data of 124 kJ · mol\(^{-1}\) obtained in this experiment.

Watanabe et al.\(^8\) indicated using the extruded AZ61 sheet material that a transition temperature of 585 K from a grain boundary diffusion mechanism at 523–573 K to a lattice diffusion mechanism at 598–673 K was observed in the normalized strain-rate vs. reciprocal temperature, and calculated that the activation energy was 90 kJ · mol\(^{-1}\) in the former region and 143 kJ · mol\(^{-1}\) in the later region respectively.

In this experiment, the activation energy of 124 kJ · mol\(^{-1}\) was calculated in the upper temperature region of the transition temperature, which is in the dominant lattice diffusion, using the 573 K isothermal-rolled AZ91 sheet material.

The microstructure by transmission electron microscopy (below, TEM) before tension test is shown in Fig. 9. A large number of serrations are seen in grain boundary after completely recrystallized. These serrations seem to prevent the fine grains from sliding or migrating in boundaries. As this result, though the \(m\)-values exceeded over 0.5 at strain-rates from 1.0 \(\times 10^{-4}\) s\(^{-1}\) to 2.5 \(\times 10^{-4}\) s\(^{-1}\), the total elongation of recrystallized AZ91 reached only 170%.

If an improvement of thermo-mechanical treatment technique inhibits these serrations from causing, the total elongation will become larger than 170% obtaining in the present experiment, because these serrations cause the stress concentrations and these concentrations cannot be relaxed by diffusional processes or dislocation movements.

### 4. Conclusions

The following conclusions were obtained as a result of examining the hot deformation of the Mg–Al alloy, AZ91 recrystallized sheets, using isothermal-rolling. The tension tests were carried out at four different temperatures and at five constant initial strain-rates.

1. The values of the total elongation exceeded over 100% at testing temperatures over 623 K with a strain-rate below 1.0 \(\times 10^{-2}\) s\(^{-1}\), and the maximum total elongation reached 170%.
2. The strain-rate sensitivity, \(m\)-values, exceeded over 0.5 with strain-rates from 1.0 \(\times 10^{-4}\) s\(^{-1}\) to 2.5 \(\times 10^{-4}\) s\(^{-1}\) except at a testing temperature of 573 K.
3. The activation energy, \(Q\), required for hot deformation was calculated to be 124 kJ · mol\(^{-1}\).
4. Hot deformation of the recrystallized AZ91 seemed to be accommodated by the volume self-diffusion of Mg.

### REFERENCES