Effect of Zr Addition on Dynamic Recrystallization during Hot Extrusion in Al Alloys

Hiroki Adachi¹, Kozo Osamura¹, Ken Kikuchi² and Jun Kusui²

¹Department of Materials Science and Engineering, Kyoto University, Kyoto 606-8501, Japan
²Technology Development Department Powder & Paste Division, Toyo Aluminium K. K., Osaka 529-1608, Japan

The presence of Al₃Zr precipitates promotes continuous dynamic recrystallization in Al-Zn-Mg alloys during hot extrusion, resulting in a fine-grained structure. The mechanism for this phenomenon was investigated by observing the change in microstructure under hot extrusion using high resolution EBSP. In the rear of the extrusion die mouth, grain boundary mobility is low since the flow stress is comparatively low and grain boundary migration is inhibited by Al₃Zr particles. Thus, in order to reduce the deformation-induced high dislocation density, continuous dynamic recrystallization occurs, which does not involve long-range grain boundary migration, resulting in a finer-grained structure. The flow stress, and hence, the mobility of the grain boundary increase near the die mouth. This promotes long-range grain boundary migration, which reduces the dislocation density. Continuous dynamic recrystallization is not observed under these conditions. Since Al₃Zr precipitates inhibit long-range grain boundary migration, increase of the Al₃Zr precipitate content expands the region where continuous dynamic recrystallization occurs towards the die mouth, resulting in a finer-grained structure.

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1. Introduction

There has been a great demand in recent years for the weight-decrease of various types of transportation equipment, which has necessitated the development of aluminum alloys with high specific strength. To this end, powder metallurgy (P/M), which solidifies powder produced by rapid solidification (RS) processing, is employed at present.¹⁻³ This RS-P/M method has the following advantages;¹,²,⁴ (1) Solute segregation can be remarkably reduced in comparison with the conventional casting method. (2) Since it is possible to compulsively dissolve additive atom, degree of freedom of composition selection in alloy design increases. (3) Precipitation of the non-equilibrium phase, such as amorphous phase and quasi-crystals, is expected.

Mesoalite is an alloy developed using RS-P/M by hot-extruding the RS powder produced by the atomization method.⁵⁻⁷ The composition of Meso10, a typical mesoalite alloy, is Al-9.5Zn-3Mg-1.5Cu-0.04Ag in mass%. The tensile strength and elongation of this alloy are 785 MPa and 8.7%, respectively.⁸ Extruding atomized powder containing 1.33 mass% Zr dissolved in the matrix (which exceeds the solid solubility limit of Zr in Meso10) was found to result in an increase in strength.⁹

Microstructural observation using high-resolution EBSP analysis revealed that this increase was due to the addition of Zr, which promoted continuous dynamic recrystallization (DRX) under hot extrusion and thus the formation of a large number of fine grains.⁸ It is believed to be the effect of Al₃Zr particles, which precipitate during the degassing treatment involving Zr addition. However, it is not clear why the presence of Al₃Zr particles promotes continuous DRX under hot extrusion. The present study aimed at revealing this phenomenon by high-resolution EBSP analysis of Meso10 with varying amounts of Zr addition, and determining how the Zr content influences fine grain formation during extrusion.

| Table 1 Chemical composition of specimens (mass%). |
|-----------------|----------------|----------------|-----------------|----------------|-----------------------|
|                 | Zn    | Mg    | Cu    | Ag   | Zr    | Al   |
| Meso10          | 8.76  | 2.93  | 1.62  | 0.04 | 0     | bal. |
| Meso10-0.4Zr    | 9.66  | 3.42  | 1.27  | 0.04 | 0.42  | bal. |
| Meso10-1.0Zr    | 9.61  | 3.01  | 1.39  | 0.04 | 1.03  | bal. |
| Meso10-1.3Zr    | 8.59  | 2.91  | 1.23  | 0.04 | 1.33  | bal. |

2. Experimental

The chemical composition of samples produced at TOYO ALUMINIUM K.K. is shown in Table 1. The cylindrical billet (diameter: 31.6 mm; length: 75 mm) for hot-extrusion was fabricated by cold isostatic pressing (392 MPa) powder produced by atomization. Atomized powder is under 100 mesh. Then, a degassing treatment was performed in flowing argon at 773 K for 3.6 ks. During this process, Al₃Zr particles precipitate in the Zr-containing alloy.⁹ Schema of the extrusion process is shown in Fig. 1. Hot-extruding the billet at 773 K, an extrusion ratio of 10 and an extrusion speed of 0.5 mm/s yields a cylindrical extrudate 10 mm in diameter. This extrudates were solution-treated at 763 K for 7.2 ks and quenched in water. They were then aged at 383 K for 108 ks. Mechanical properties of these specimens were characterized by tensile testing.

In order to investigate the microstructural change under extrusion, the extrusion run was interrupted once the billet was extruded to 55 mm. The billet, along with the extrusion die, was then immediately immersed in water, yielding samples that preserved the microstructure under extrusion. The orientation analysis of as-extruded and under-extruded samples was carried out using Electron Backscattering Pattern (EBSP). EBSP measurements were performed in a JEOL JSM-6500F scanning electron microscope (SEM), with software by TexSem Lab., Inc. EBSP measurements were
carried out at the 2 mm intervals at the center of underextruded samples, as is shown in Fig. 1(d). The scanning area and scanning step size were 3600 $\mu$m$^2$ and 200 nm, respectively. The texture change during extrusion was quantitatively analyzed using the orientation distribution function (ODF).

3. Experimental Results and Discussion

Table 2 shows the mechanical properties of extrudates that have been aged. Both the 0.2% proof stress and tensile strength increased monotonously with the amount of Zr addition. This is thought to be because the number of fine DRX grains increase with the addition of Zr.

Table 2 Mechanical properties of aged specimens obtained by tensile test and their Zr content dependence.

<table>
<thead>
<tr>
<th></th>
<th>0.2% proof stress, $\sigma_{0.2}$/MPa</th>
<th>Tensile Strength, $\sigma_{UTS}$/MPa</th>
<th>Elongation, (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Meso10</td>
<td>681</td>
<td>782</td>
<td>7.5</td>
</tr>
<tr>
<td>Meso10-0.4Zr</td>
<td>709</td>
<td>786</td>
<td>5.1</td>
</tr>
<tr>
<td>Meso10-1.0Zr</td>
<td>769</td>
<td>818</td>
<td>4.3</td>
</tr>
<tr>
<td>Meso10-1.3Zr</td>
<td>805</td>
<td>827</td>
<td>4.0</td>
</tr>
</tbody>
</table>

and at a density of 70,000/mm$^2$. The number of the DRX grains gradually increased with the decrease of the distance from the die mouth, up to 8 mm from the die mouth. Near the die mouth, it showed no increase and DRX did not occur. In Meso10-1.0Zr, the number of DRX grain increased with the decrease of the distance from the die mouth, up to 10 mm from the die mouth. The rate of increase in the number of fine grains was almost similar to that observed in the case of Meso10-1.33Zr. However, the evolution of DRX grains stopped closer to the die mouth than in Meso10-1.33Zr. In Meso10-0.4Zr and Meso10, the increase in the number of fine grains stopped at 14 and 18 mm rear from the die mouth, respectively. As the Zr content increased, the rate of increase
are reported as follows.\(^8,12,13\) Inhomogeneous local strains 
Formation mechanisms of fine grains by continuous DRX 
may not be observed because deformation has already 
hypotheses are considered. Let us first examine whether DRX 
occur in the vicinity of the die mouth. The following 
expanded, as a result of which the number of the fine grains 
in the number of DRX grains showed no significant change, 
however, the number of DRX grains stopped increasing 
closer and closer to the die mouth, and the DRX region 
expanded, as a result of which the number of the fine grains 
increased.

To clarify the reason why the DRX region expands with Zr 
addition, it is necessary to investigate why DRX does not 
occur in the vicinity of the die mouth. The following 
hypotheses are considered. Let us first examine whether DRX 
may not be observed because deformation has already 
completed by the time the die mouth is approached. 
Formation mechanisms of fine grains by continuous DRX 
are reported as follows.\(^8,12,13\) Inhomogeneous local strains 
are induced by grain boundary shearing near grain bounda-
ries of the initial coarse grain, and the local dislocation 
density increases. The dislocations are rearranged and a 
subboundary is formed. Then, misorientation of the sub-
boundary increases by the absorption of introduced disloca-
tions, and new DRX grains are formed along the grain 
boundary of the initial grains. Therefore, it is believed that 
new DRX grains do not form once deformation has 
completed, since no new dislocations are introduced into 
the initial grain. Since the initial equiaxial grain is elongated 
along the extrusion direction, and orients itself along ED // 
\(\langle 111 \rangle\) or \(\langle 100 \rangle\) as extrusion progresses, it is possible to 
observe the evolution of deformation by investigating the 
change of aspect ratio of initial grains and monitoring the 
change in orientation density, ED // \(\langle 111 \rangle\) and \(\langle 100 \rangle\). 
Orientation density is the degree of deviation of orientation 
from randomness when the intensity of random texture is 
defined to be 1. Aspect ratio of initial coarse grains was 
calculated by averaging the ratio of the minor axis to the 
major axis of the ellipse fit to the grains with a diameter 
larger than 5 \(\mu m\).

Figure 4 shows the change of aspect ratio of the coarse 
grains and orientation density, ED // \(\langle 111 \rangle\) and \(\langle 100 \rangle\), with 
the progress of extrusion in Meso10-1.33Zr. The aspect ratio 
of the coarse grains slowly increases and orientation density, 
ED // \(\langle 111 \rangle\) and \(\langle 100 \rangle\) is also almost constant from the 
extrusion mouth to 10 mm. This indicates that the plastic 
deformation quantity is small and the development of the 
texture is not remarkable. In this region, the continuous DRX 
is generated and the fine grains are formed, as is shown in 
Fig. 3. From 8 mm rear from the die mouth, the aspect ratio 
rapidly increases and the coarse grain is rapidly elongated in 
the extrusion direction. In this region, the plastic deformation 
quantity is large and the fiber texture, ED // \(\langle 111 \rangle + \langle 100 \rangle\) 
develops rapidly with the coarse grains becoming fibrous. 
In the vicinity of the die mouth, since the aspect ratio of coarse 
grains increases, the edge of the fibrous grains frequently 
comes out of the scanning area of EBSP measurement and the 
aspect ratio couldn’t be appropriately calculated. Therefore, 
though in the die mouth vicinity, the aspect ratio monoto-
nously does not increase, since the fiber texture continues to 
develop, large plastic deformation is occurred and actually, 
the aspect ratio is considered to increase. In Meso10-1.33Zr, 
though deformation occurs in the region of 2 to 8 mm from 
the die mouth, the formation of fine grain by continuous DRX 

![Figure 3](image1.png)

**Fig. 3** Change in the number per unit area of DRX grains in the center of the extrudate with the progress of extrusion.

![Figure 4](image2.png)

**Fig. 4** Change in average aspect ratio of coarse grains which diameter > 5 \(\mu m\) and \(\langle 100 \rangle\) and \(\langle 111 \rangle\) orientation density of ED in the center of the extrudate with the progress of extrusion in Meso10-1.33Zr.

Regardless of the amount of Zr addition, orientation density is about 2 times random 20 mm towards the rear of
the die mouth. It increases rapidly as the distance from the die mouth decreases and is saturated at about 20 times random. However, as the amount of Zr addition increases, the point where orientation density and the mobility of the grain boundary of coarse grains begin to increase approaches the die mouth. This point roughly corresponds to where the formation of fine grains by continuous DRX stops in which alloy. This indicates the following. The higher the amount of Zr added, the higher the number of Al$_3$Zr precipitate, the higher the force pinning grain boundary migration. 14) Grain boundary mobility does not increase in the vicinity of the die mouth, where the flow stress is high. As a result, the region where fine grains formed by continuous DRX expanded towards the die mouth, and fine grains were increased.

4. Conclusions

High-resolution EBSP was used to observe the micro-

structural change during hot extrusion in an attempt to find out why the number of fine grains formed during hot extrusion increased with the addition of Zr.

Though the formation of fine grains by the continuous DRX was not observed in the region of which the plastic deformation was large and the mobility of the grain boundary was high, i.e. in the vicinity of the die mouth, the continuous DRX was observed only in the region of which the plastic deformation was small and the mobility of the grain boundary was low. Since Al$_3$Zr precipitates pin grain boundary migration, a large flow stress is required for the grain boundary mobility to increase as the amount of Zr addition increases. Thus, since the region in which the grain boundary mobility is low expands towards the vicinity of the die mouth, the DRX region expands towards the die.

REFERENCES

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