Ultra-Fine Grain Development in an AZ31 Magnesium Alloy during Multi-Directional Forging under Decreasing Temperature Conditions*1

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Grain refinement of a magnesium alloy, AZ31, was studied in multi-directional forging (MDF) with decreasing temperature from 623 to 423 K. The MDF was carried out up to cumulative strains of around 5 with changing the loading direction during decreasing temperature from pass to pass. The structural changes are characterized by the development of many mutually crossing kink bands accompanied by MDF at low strains, followed by full development of very fine grains at high strains. The dynamic changes in grain size evolved can be expressed by two different power law functions of flow stress for the regions of flow stress above or below around 100 MPa. The MDF under decreasing temperature condition can accelerate the uniform development of much finer grains and the improvement in plastic workability, leading to the minimal grain size of 0.36 μm at a final processing temperature of 423 K. The mechanism of grain refinement is discussed in detail.

(Received January 26, 2005; Accepted May 6, 2005; Published July 15, 2005)

Keywords: magnesium alloy AZ31, multi-directional forging (MDF), continuous dynamic recrystallization, fine grain, hall-petch relation

1. Introduction

Magnesium (Mg) and its alloys are the lightest metallic structural materials and have the following several features, i.e. high specific strength, good electromagnetic interference shielding, good recyclability, and etc.1,2) Mg alloys are very attractive in application to automotive parts and casing of portable electrical devices in recent years, however, are categorized as hard plastic materials because of a limited ductility and formability due to the hexagonal closed-packed (HCP) crystal structure at room temperature. Then, structural Mg alloys have been manufactured less frequently by plastic working such as rolling and other forming processes compared with casting route.1,2) It is expected that several slip systems can be operated in addition to the basal slip system during warm and hot deformation, leading to increase in the plastic workability. It is also known2,3) that fine grains are developed in Mg alloys at relatively low strains during warm and hot working and result in much improvement of the plastic workability. Yang et al.4,5) investigated the grain refinement mechanism occurring in Mg alloy AZ31 during high temperature deformation and found that the formation mechanism of new grains is clearly different from conventional discontinuous dynamic recrystallization (dDRX). The number and the misorientation angle of kink bands evolved at low strains rapidly increase with further deformation, finally resulting in development of new fine-grains in high strain. They concluded that dynamic grain evolution in Mg alloy can be based on grain fragmentation taking place in original coarse grains and so controlled by deformation-induced continuous reactions assisted by dynamic recovery, i.e. continuous dynamic recrystallization (cDRX).

Kink bands are formed roughly perpendicular to the basal plane.5) In single pass compression, the basal plane of extruded Mg rod, which is parallel to the compression axis, is rotated and approaches perpendicular to the compression axis at high strain. As a result, fine grains cannot be fully generated throughout the material even in high strain.4,5) In the present study, optimum processes for fine grain development and improvement of the mechanical properties are studied in multi-directional forging (MDF) of Mg alloy with decreasing temperature from pass to pass. Fine-grained microstructure developed is investigated by optical and transmission electron microscopy (OM and TEM).

2. Experiment Procedure

A commercial AZ31 magnesium alloy was provided as a hot-extruded rod with the chemical composition as following: Al 2.86, Zn 0.75, Mn 0.68, Cu 0.001, Si 0.003, Fe 0.003 and balance Mg (all in mass%). The rectangular samples with a dimension of 31, 21 and 14 mm in each side (i.e. the axial ratio = 2.22:1.49:1) were machined from the rod parallel to the extrusion direction (see Fig. 1(a)). The samples were annealed for 7.2 Ks at 733 K and then furnace cooled, leading to the evolution of equiaxed grains with an average size of about 22 μm (see Fig. 3(a)).

Compression tests were carried out at a constant true strain rate of 3 × 10−3 s−1 by using a testing machine equipped with a quenching apparatus, which made it possible to quench a sample in water within 1.5 s after hot deformation was ceased.6) The sample was deformed by multi-directional forging (MDF) with changing the loading axis at an angle of 90° from pass to pass and with decreasing temperature from 623 to 423 K (Fig. 1). When a pass strain Δε is fixed at a constant, i.e. Δε = 0.8, the dimension ratio of the sample does not change in each pass and so the compression processing can be carried out to infinity.7) Deformed samples were cut along a plane parallel to the last compression axis. Microstructural observation was carried out by using OM and TEM under an accelerating voltage of 200 kV. The Vickers hardness was also measured at room temperature.

3. Results and Discussion

3.1 Deformation behavior

Typical true stress-cumulative strain (σ−ΣΔε) curves of
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Fig. 1 Schematic illustration of the thermo-mechanical processing method used in multi-directional forging (MDF) with continuous decreasing temperature in each pass. (a) The loading direction is changed in 90° with pass to pass (x → y → z → ...). A pass strain $\Delta \varepsilon = 0.8$. (b) The temperature decreases from 623 to 423 K. WQ indicates water quenching.

Mg alloy AZ31 during MDF are shown in Fig. 2. The $\sigma-\varepsilon$ relationship of single-pass compression for an as-annealed sample tested at 473 K is also shown by a broken line for comparison. The latter exhibits a sharp and higher stress peak of about 6.7 MPa after 1st-pass compression at 623 K, immediately followed by brittle fracture. On the other hand, the flow curves during early MDF show work softening following a peak stress, then followed by steady state flow at high strains in high temperature region. The amount of work softening after a peak stress decreases with dropping temperature, and at temperatures lower than 473 K steady state flow appears with no flow softening. It is interesting to note that the flow curves in single-pass and MDF tests at 473 K are clearly different from each other, i.e. the flow stress of around 110 MPa at $\Sigma \Delta \varepsilon \cong 3.2$ in MDF is about one-third of that in single-pass compression. Furthermore, the MDF processing was successful to deform the Mg alloy to $\Sigma \Delta \varepsilon = 4.8$ at 423 K which is below 0.5$T_m$ ($T_m$ is the melting point).

3.2 Optical microstructures

Figure 3 shows the microstructures of (a) as-annealed and (b) to (d) developed during subsequent MDF with decreasing temperature from 623 to 423 K. It can be clearly seen in Fig. 3 that the grain size decreases by straining with reducing temperature; the initial grain size of 22.3 $\mu$m decreases to about 6.7 $\mu$m after 1st-pass compression at 623 K (Fig. 3(b)), 3.8 $\mu$m after 2nd-pass compression at 523 K (Fig. 3(c)) and about 0.8 $\mu$m after 4th-pass compression at 473 K (Fig. 3(d)).

The flow curves in MDF at 623 K shows significant strain softening after a stress peak, followed by steady state flow (Fig. 2), where equiaxed fine-grains are homogeneously and almost fully developed in the whole area, as can be seen in Fig. 3(b). These results are almost similar to the previous data obtained by single-pass compression at 673 K.4,5) Yang et al.4,5) have found that initial coarse grains are fragmented by the boundaries of kink bands developed at early stages of deformation. The regions fragmented by kink bands are bounded by high angle boundaries in high strain, finally resulting in evolution of a new grain structure. They concluded that dynamic formation of new grains in Mg alloy can result from a series of strain-induced continuous reactions, that is to say continuous dynamic recrystallization (cDRX). Kink bands are commonly generated perpendicular against the basal plane of HCP lattice when the latter lies to parallel to the compression axis. During hot compression, the basal planes in the initial grains rotate gradually and approach near perpendicular to the compression direction. After that, the generation of kink bands as well as full development of new grains hardly takes place even in high strain.5) During MDF, in contrast, kink bands are developed in various directions and therefore crossed mutually due to change in the compression axis from pass to pass. This process should lead finally to homogeneous and full development of fine grains over the whole area.5)

The lower flow stresses appearing during MDF of 473 K (Fig. 2) can be resulted from such a drastic grain refinement taking place by repeated MDF with dropping temperature. During MDF at temperatures from 623 to 473 K, the average
grain size decreases from 22.3 to 0.8 \( \mu \text{m} \). In such fine-grained structures, grain boundary sliding can take place frequently and extensively even during low temperature deformation, leading to reduction of flow stress as well as improvement of ductility.

3.3 TEM microstructures

Figures 4(a) and (b) show typical TEM microstructures and the selected area diffraction (SAD) patterns for the samples deformed to \( \Sigma \Delta \epsilon = 4.0 \) at 453 K and \( \Sigma \Delta \epsilon = 4.8 \) at 423 K, respectively. Objective apertures of 6 and 2 \( \mu \text{m} \) in diameter were employed for SAD analysis in Figs. 4(a) and (b), respectively. The SAD patterns show almost uniform and fully continuous rings. These observations suggest that the microstructures are composed of polycrystalline ultra-fine grains surrounded by high angle boundaries. The average grain size in Figs. 4(a) and (b) was about 0.54 and 0.36 \( \mu \text{m} \), respectively. It was reported that the average grain size in AZ31 processed by Equal-Channel Angular Extrusion (ECAE) at 473 K and \( \Sigma \Delta \epsilon = 8.0 \) (i.e. after 8th pass ECAE) is about 1.1 \( \mu \text{m} \). In the present MDF processing, the average grain size of about 0.54 \( \mu \text{m} \) is developed at 453 K and \( \Sigma \Delta \epsilon = 4.0 \). The MDF processing may be a more effective method for grain refinement of Mg alloys during severe plastic deformation.

3.4 Relationship between flow stress and grain size

The relationship between the average grain size and the flow stress developed during MDF with dropping temperature as well as single-pass compression at high temperature is shown in Fig. 5. The results derived by means of OM and TEM are represented by open and closed marks, respectively. The symbols of triangle and circle indicate the data observed by MDF and single-pass compression at high temperature. Grain refinement cannot take place in an as-annealed Mg alloy at the temperatures lower than 473 K, because the alloy fractures in a brittle manner during single-pass compression (Fig. 2). During MDF with dropping temperature, on the other hand, dynamic grains which were evolved gradually decrease the size with repeating MDF of Mg alloy without cracking or fracture. At this moment, the minimal grain size of 0.36 \( \mu \text{m} \) is successfully attained by MDF to \( \Sigma \Delta \epsilon = 4.8 \) at 423 K. Figure 5 suggests that grain size may approach to 0.1 \( \mu \text{m} \) or less than it with further MDF at lower temperature. Figure 5 shows that grain size can be expressed by two power law functions of flow stress, i.e. \( \sigma = kD^{-N} \), in the region of flow stress above or below around 100 MPa. The grain size exponent is \( N \approx 0.9 \) in the region of \( \sigma < 100 \text{ MPa} \), where deformation is carried out at higher temperatures above 0.5\( T_m \). In contrast, \( N \) is about 0.2 in the region of \( \sigma > 100 \text{ MPa} \), where MDF is performed below around 0.5\( T_m \). The relationship between flow stress and dynamic grain size developed by MDF of pure polycrystalline Cu is also roughly similar to the results in Fig. 5; namely, \( N \) is 0.75 in the region of lower stresses below 200 MPa and \( N \approx 0.3 \) in that of higher stresses. It is interesting to note that a similar type of the relationship is held between \( \sigma \) and \( D \) irrespective of different recrystallization mechanisms operating in differ-
ent materials, *i.e.* discontinuous and continuous DRX. In pure polycrystalline Cu, dDRX and cDRX can operate in the regions of lower flow stresses and higher ones respectively.\(^ {12}\)

On the other hand, only cDRX can take place in HCP Mg alloy irrespective of the flow stress regions, as discussed above.

### 3.5 Room temperature hardness

The relationship between room-temperature hardness \(H_v\) and the average grain size \(D\) developed during MDF is represented in Fig. 6. It is seen clearly that \(H_v\) increases with decreasing average grain size and the relationship between \(H_v\) and \(D\) can be approximated by the following Hall-Petch eq. (1).

\[
H_v = H_0 + kD^{-1/2}
\]

The data for an as-annealed material (○) is removed for this analysis, because the other data are obtained from MDFed samples which have deformation-induced microstructures containing high density dislocations. Here, \(H_0 = 510\) MPa and \(k = 0.22\) MPa/m\(^{-1/2}\).

Koike *et al.*\(^ {13}\) have reported that the relationship between yield stress \(\sigma_y\) and grain size for various Mg alloys can be expressed by the form of \(\sigma_y = \sigma_0 + kD^{-1/2}\), where, \(\sigma_0\) is 90–210 MPa and \(k\) is 0.17 MPa/m\(^{-1/2}\). Using \(\sigma_y = 1/3H_v\), it follows that \(H_0 = 270\)–630 MPa and \(k = 0.17\) MPa/m\(^{-1/2}\). The present data of \(H_0\) and \(k\) in Fig. 6 are roughly similar to them within experimental scatter. It is suggested from Fig. 6 that ultra-fine grained Mg alloys, where \(D\) is reduced to around or less than 0.1 \(\mu m\) by MDF processing, possess high performance of twice higher room-temperature hardness or strength compared with conventional ones with \(D > 10 \mu m\).

### 4. Summary

Fine-grain evolution as well as improvement of room-temperature strength of Mg alloy, AZ31, was studied in
multi-directional forging (MDF) of a pass strain $\Delta \varepsilon = 0.8$ with decreasing temperature from 623 to 423 K. The main results are summarized as follows.

1. MDF of the Mg alloy with dropping temperature can be carried out up to high strain of around 5 even at temperatures less than $0.8 T_m$, although the as-annealed alloy fractures brittlely even at a strain of below 0.3 during single-pass compression at 473 K.

2. Evolution of fine-grained structure can be accelerated by MDF with changing the loading direction from pass to pass because kink bands are developed in various directions. The average grain size decreases with repeated MDF with dropping temperature, resulting in evolution of the minimal grain size of 0.36 $\mu$m at $\Sigma \Delta \varepsilon = 4.8$ at 423 K.

3. The relationship between dynamic grain size and flow stress during MDF can be expressed by two power law functions of flow stress with different slopes in the regions of flow stress above or below 100 MPa. The relationship can be expressed by $N = 0.9$ in $\sigma > 100$ MPa and $N = 0.2$ in $\sigma < 100$ MPa.

4. The relationship between room-temperature hardness ($H_v$) and dynamic grain size ($D$) can be approximated by a Hall–Petch relation, $H_v = H_0 + KD^{1/2}$. Here, $H_0 = 510$ MPa, and $K = 0.22$ MPa/m$^{1/2}$.

Acknowledgments

The authors are intended to the following bodies for the financial supports: Ministry of Education, Science and Culture (Grant-in-Aid for Scientific Research (c) (No. 15560623 and No. 16560627)) and the Light Metals Educational Foundation, Japan.

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7. The dimensions of the sample in three perpendicular direction are named as $L_x$, $L_y$, and $L_z$, respectively. When a pass strain of 0.8 is applied in $x$ direction, the following relationships are held.

\[
\varepsilon_x = \ln \frac{L_y}{L_x} = -0.8
\]

\[
\varepsilon_y = \varepsilon_z = -\frac{1}{2} \varepsilon_x = 0.4
\]

If $L_z$ is 1, $L_x$ and $L_y$ should be 1.49 and 2.22 respectively. When the loading direction is changed at an angle of 90° and $\Delta \varepsilon$ = 0.8 is applied in each pass, the dimension ratio of the sample is always constant, i.e., 1:1.49:2.22, irrespective of each MDF pass.