Isochronal Annealing Behavior of Magnesium Alloy AZ31 after Hot Deformation*

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Annealing behaviors of hot-deformed magnesium alloy AZ31 were studied at temperatures of 373 to 673 K by optical and SEM/EBSD metallographic observation. The hot-deformed alloy had fine-grained structures developed by grain fragmentation due to continuous dynamic recrystallization (cDRX). Temperature dependence of the average grain size (D) is categorized into three temperature regions, *i.e.* (a) an incubation period for grain growth at < 450 K, (b) rapid grain coarsening at 473–523 K, and (c) normal grain growth at $T > 550$ K. The number of fine grains per unit area, however, is reduced remarkably even in region (a). These lead to grain coarsening taking place continuously in the whole temperature regions. In contrast, the deformation texture scarcely changes even after full annealing at high temperatures. It is concluded that the annealing processes operating in hot-deformed magnesium alloy with cDRXed grain structures can be mainly controlled by grain coarsening accompanied with no texture change, that is continuous static recrystallization (cSRX).

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1. Introduction

Magnesium (Mg) is one of the lightest metals in practical use and is of growing interest as a next-generation lightweight material because of its high specific strength and excellent technological properties.¹⁾ Most Mg alloy products have been produced by casting route, while recently request for the wrought products has been increased with development of forming process technology. Mg alloys show generally low cold-workability because of a few slip systems in the hexagonal close-packed (HCP) structure. One of possible methods breaking through the low ductility is to develop fine-grained structures in Mg alloys. Grain refinement could improve not only the plastic workability, but also the strength at ambient temperature.²⁾ Recently, severe plastic deformation (SPD) by using equal channel angular extrusion (ECAE),^{3,4)} multi-directional forging (MDF),⁵⁾ etc., has been carried out on the Mg alloys to develop a fine-grained structure. The authors have studied thermo-mechanical processes for development of ultra-fine grained Mg alloys. They developed fine-grained structures with the grain size of submicrons by using MDF under decreasing temperature conditions and succeeded much improvement of the plastic workability and also the mechanical properties due to grain refinement.⁵⁾

The texture control will also be important for increase in the ductility of deformed Mg alloys,⁶⁾ because, for example, the basal plane becomes perpendicular to the forging direction in high strain during hot deformation.⁷⁾ Further improvement of the ductility of deformed Mg alloys will be carried through the texture control due to annealing treatment following plastic deformation. There have been a few reports on the annealing behaviors of hot-deformed Mg alloys. The annealing characteristics and the texture changes in a widely used Mg alloy, *e.g.* AZ31, have been almost unknown, as far

as the authors know.⁸⁾

The present work aimed to investigate the annealing characteristic of a Mg alloy AZ31 after hot deformation. The structural changes taking place during hot deformation and subsequent isochronal annealing were systematically studied at various reheating temperatures. Changes in the grain structure and the texture developed during and after hot deformation are analyzed in detail, and the mechanisms of annealing processes also are discussed.

2. Experimental Procedure

A commercially produced Mg alloy AZ31 was provided as a hot-extruded rod with a diameter of 19 mm. The chemical composition was as follows; Al 2.68, Zn 0.75, Mn 0.68, Cu 0.001, Si 0.003, Fe 0.003, and balance Mg (all in mass%). Cylindrical samples of 8 mm in diameter and 12 mm in height were machined from the rod parallel to the extrusion direction. Then 6 evenly spaced annular grooves of 0.12 mm depth were machined into the end faces of cylinders in order to promote the retention of a graphite lubricant (DAG 154). The samples were annealed at 733 K for 7.2 ks and then furnace cooled, leading to the evolution of equiaxed grains with an average size of about 22 μm .

Compression tests were carried out at constant true strain rates on a testing machine equipped with a water quenching apparatus,⁷⁾ which made it possible to quench the samples within 1.5 s after deformation was ceased. The samples were deformed up to strains of 1.2 at 573 K and at two true strain rates of 3×10^{-3} and $3 \times 10^{-1} \text{ s}^{-1}$, followed by quenching in water. They were cut to plates with 2 to 3 mm thickness parallel to the compression axis, and then annealed for 1 ks in air using a muffle furnace at temperatures ranging from 373 to 673 K. Each plate was mechanically and electrolytically polished and then etched in a solution of 6% picric acid and 94% methanol. As-deformed and the subsequent annealed microstructures were examined by using optical microscopy (OM) and orientation imaging microscopy (OIM). The

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Fig. 1 Effect of strain rate on true stress–true strain curves for AZ31 alloy at 573 K.

distributions of grain size were analyzed by using an image analysis program of “AnalySIS” (Soft Imaging System Co.). Crystallographical orientation and texture changes were examined by SEM/OIM. The Vickers hardness was measured on each plate at room temperature under a condition of 3 N.

3. Results and Discussion

3.1 Hot deformation behaviors

Figure 1 shows typical true stress–true strain curves for the AZ31 alloy compressed at 573 K and at strain rates of 3×10^{-3} and $3 \times 10^{-1} \text{ s}^{-1}$. It is seen in Fig. 1 that the flow stresses as well as the shape of the flow curves are sensitively affected by strain rate. The flow curve at $3 \times 10^{-3} \text{ s}^{-1}$ shows a smooth stress peak following gradual strain hardening in relatively low strain, followed by strain softening and steady-state flow in high strain. The flow shape at a high strain rate of $3 \times 10^{-1} \text{ s}^{-1}$, in contrast, indicates a sharper stress peak following rapid strain hardening and then rapid strain softening and steady-state flow in high strain. These suggest that mechanisms of structural change operating during deformation depend sensitively on strain rate.

3.2 Structural changes during hot deformation

It is well known that new grain structures are developed in relatively low strain during warm and hot deformation of Mg alloys.^{2,7)} Changes in the volume fraction and the average size of new grains (V_{rex} and D_{rex}) with deformation at 573 K are shown in Fig. 2. New grains start to develop at around the stress peak and V_{rex} rises rapidly during work softening and then approaches a saturation value in high strain. In contrast, D_{rex} in the new grained regions decreases slightly during work softening and approaches a saturation value. It is considered that D_{rex} developed at low strains is roughly similar to that in high strain.⁷⁾ With increasing strain rate, the strain at which V_{rex} approaches a constant increases and conversely D_{rex} in high strain decreases from 4 to $2.7 \mu\text{m}$.

Fig. 2 Effects of strain and strain rate on (a) the volume fraction (V_{rex}) and (b) the average size of new grains (D_{rex}) for AZ31 alloy deformed at 573 K.

Fig. 3 Effect of strain rate on changes in room-temperature hardness with deformation at 573 K for AZ31 alloy.

Figure 3 shows strain dependence of the room temperature hardness (H_v) for the hot-deformed Mg alloy. H_v increases monotonously with strain and approaches to a saturation in high strain irrespective of strain hardening and softening in the flow curves (Fig. 1). The increasing rate of H_v and the saturation value in high strain are higher with increasing strain rate. The H_v – ϵ relationships in Fig. 3 may correspond to those between the reciprocal D_{rex} and strain in Fig. 2. Namely, H_v increases with reduction of the average grain size

Fig. 4 Microstructures developed in AZ31 alloy (a) deformed to $\varepsilon = 1.2$ at 573 K with a strain rate of $3 \times 10^{-1} \text{ s}^{-1}$, followed by annealing for 10^3 s at (b) 473 K, (c) 523 K and (d) 673 K. CA indicates the compression axis.

D due to increasing the volume fraction of new grains, and approaches to a saturation value in high strain, where D_{rex} is almost constant. This may suggest that the room temperature hardness of hot-deformed Mg alloy can be controlled mainly by grain refinement rather than increasing dislocation densities introduced by deformation. These results will be discussed in detail in section 3.6.

3.3 Structural changes during isochronal annealing

Figure 4 shows a series of typical optical microstructures evolved before and after annealing at various temperatures for the AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K and at $3 \times 10^{-1} \text{ s}^{-1}$. The authors^{7,9,10} previously discussed that new grains evolved in Mg alloy during hot deformation can result from a kind of strain-induced continuous recrystallization, but not from conventional discontinuous recrystallization. The process of new grain formation in hot-deformed Mg alloy is described briefly as follows: (1) Kink bands are developed frequently in grain interiors at low strains and the misorientation and the number of the boundaries of kink bands rise rapidly with deformation because HCP structure have a limited number of slip systems. (2) Their intersections take place with deformation, leading to fragmentation of original grains. The average size of the fragmented domain is almost the same as that of new grains. (3) It is concluded, therefore, that the new grain formation can result from grain fragmentation by development of kink band and so be controlled by deformation-induced continuous reactions assisted by dynamic recovery. This is generally called as *in-situ dynamic recrystallization* or *continuous dynamic recrystallization (cDRX)*. It should be noted that new grains

were not fully developed throughout the material even in high strain, as can be seen in Fig. 4(a) and discussed in elsewhere.⁷⁾

Figures 4(b) to (d) show that such strain induced new grains coarsen homogeneously with increase in reheating temperature. After annealing at temperatures below 473 K, some rather coarse grains still remain and are clearly distinguishable from surrounding fine grains [Fig. 4(b)]. After annealing at $T \geq 503 \text{ K}$ [Figs. 4(c) and (d)], it is difficult to distinguish the coarse grains from the surrounding ones which coarsen homogeneously, finally leading to a fully development of an almost equiaxed grain structure.

Figure 5 shows changes in the average grain size (D) in the whole area with annealing temperature for the AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K and at strain rates of 3×10^{-3} and $3 \times 10^{-1} \text{ s}^{-1}$. D was measured by using a linear line intercept method. D is hardly changed during annealing at low temperature $< 473 \text{ K}$. D increases rapidly at 473–523 K and then increases slowly with rising temperature. Such a grain size change looks like that occurring in conventional deformed cubic metals during annealing.¹¹⁾ The temperature range from 300 to 450 K may correspond to an incubation region for recrystallization, when a period of annealing is 1 ks, that from 473 to 523 K a recrystallization region and the high temperature side above around 550 K a region for normal grain growth, respectively. The temperature, at which rapid grain growth takes place, falls a little with increasing strain rate. This result also is similar to that for annealing of deformed cubic metals.¹¹⁾

Fig. 5 Effect of strain rate on changes in average grain size in the whole area, D , with annealing temperature for AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K.

3.4 Quantitative analysis of microstructures and texture

Typical grain size distributions before and after annealing at 373 to 673 K are shown in Fig. 6 for the samples deformed to $\varepsilon = 1.2$ at 573 K and at $3 \times 10^{-1} \text{ s}^{-1}$. The distribution at 373 K is almost similar to that for as-deformed state and shows a bimodal shape with two peaks at around 2 and 20 μm . The fine grains correspond to those newly developed by hot deformation and the coarser ones to the remained original grains [see Fig. 4(a)]. Upon annealing at $T > 473 \text{ K}$, the relative frequency of fine grains rapidly decreases and the range of coarse grain sizes gradually increases with increase in temperature. With further annealing at $T > 523 \text{ K}$, the grain size distribution is similar to a log-normal type with a single peak at around 20 μm [see Figs. 6(e) and (f)].

Figure 7 shows changes in the number of fine grains per unit area, N , less than 10 μm in diameter with annealing temperature. It is remarkable to note that N decreases clearly even at low temperatures of $T < 450 \text{ K}$. This is in contrast with the result of the average grain size in Fig. 5, *i.e.* there is no remarkable grain coarsening in this temperature region. It is concluded from Fig. 7 that grain growth starts to take place mainly in the fine-grained regions simultaneously just after annealing even at low temperature [Figs. 6(a) to (c)]. Further annealing in the temperature region between 473 and 523 K leads to a rapid grain coarsening accompanied by a rapid decrease in N . Then the grain size distribution changes from a double peak to a single peak type, as shown in Figs. 6(c) and (d). During this process, some coarse grains containing high density dislocations [see Fig. 8(a)] may be consumed by recovered growing grains in the surroundings. During annealing at $T > 523 \text{ K}$, the average grain size gradually increases accompanied with a little decrease in N , suggesting the occurrence of normal grain growth.

Typical OIM micrographs with the inverse pole figures for compression direction are shown in Fig. 8. Figures 8(a), (b) and (c) correspond to the microstructures in Figs. 4(a), (b) and (d). Disorientation (θ) $\theta > 2^\circ$, $\theta > 4^\circ$ are delineated by thin white and black lines, while high-angle boundaries

Fig. 6 Changes in grain size distribution with annealing temperatures for AZ31 alloy deformed to $\varepsilon = 1.2$ at $3 \times 10^{-1} \text{ s}^{-1}$ and at 573 K.

Fig. 7 Effect of strain rate on the relationship between the number of fine grains with less than 10 μm in diameter per unit area, N , and annealing temperature for AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K.

including kink bands⁷⁾ with misorientations more than 15° are by bold black lines. Different colors for each grain correspond to different crystallographic orientations. It is clearly seen in Fig. 8 that most of the grains developed have a strong texture near (0001) compared with those near (10 $\bar{1}$ 0) or (11 $\bar{2}$ 0). It is known⁷⁾ that extruded rod of Mg alloys has a strong texture in which the basal plane of the HCP lattice lies parallel to the extrusion direction (and so the compression direction in the present case). The basal plane rotates

Fig. 8 OIM micrographs and inverse pole figures of AZ31 alloy (a) deformed to $\varepsilon = 1.2$ at 573 K with at $3 \times 10^{-1} \text{ s}^{-1}$, followed by annealing for 10^3 s at (b) 473 K and (c) 673 K. Thin white lines correspond to boundaries of misorientation $> 2^\circ$, thin black line $> 4^\circ$ and thick black lines $> 15^\circ$, respectively. Colors correspond to crystallographic orientations indicated in the inverse pole figure.

gradually from around 0° to near 90° to the compression axis with deformation. It is remarkable to note in Fig. 8 that such a deformation texture with (0001) perpendicular to the compression axis exists stably even after full annealing at high temperatures. It is concluded that strain-induced fine grains evolved in Mg alloys can grow remarkably and, in contrast, the deformation texture is scarcely changed with annealing.

3.5 Static restoration mechanisms operating in cDRXed Mg alloys

The present experimental results described above, can be summarized as the followings.

- (1) The grain size distribution shows a single peak type during annealing at $T > 500 \text{ K}$ where rapid grain coarsening takes place [Figs. 6(d) to (f)].
- (2) Grain coarsening takes place continuously during annealing even at low temperatures (Figs. 5 and 7).
- (3) The deformation texture is scarcely changed during grain coarsening (Fig. 8).

Fig. 9 Effect of strain rate on changes in room-temperature hardness with annealing temperature for AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K.

Such annealing behaviors are in contrast with those for conventional deformed cubic metals, in which discontinuous recrystallization takes place accompanied with nucleation of new grains and their long range grain boundary migration. It is concluded, therefore, that annealing processes operating in hot-deformed Mg alloy is controlled mainly by grain coarsening accompanied with no texture change, that is *continuous static recrystallization (cSRX)*. It is interesting to note that dynamic evolution of new grains in Mg alloys results from continuous DRX taking place during hot deformation, as described above, and continuous static recrystallization can subsequently operate in cDRXed grain structure during annealing.

It is suggested by Fig. 8 that the texture of wrought Mg alloy may be hardly controlled by using conventional methods, *i.e.* plastic working followed by annealing. It is also seen in Fig. 8(a) that dislocation boundaries with low angle misorientations are developed in remained coarse grains, and the cumulative misorientations at near the original grain boundaries are within a few degrees, *i.e.* these is almost no lattice orientation gradient evolved.⁷⁾ This is in contrast with the cases of hot-deformed grains and conventional DRXed grains in cubic metals. Namely, some high orientation and strain gradients are always formed near the grain boundaries, leading to nucleation with various orientations followed by long range grain boundary migration.^{11–14)} Thus, deformation process accompanied with various strain paths, such as multi-directional forging (MDF),⁵⁾ may be useful for texture modification of Mg alloy.

3.6 Effects of hot deformation and annealing on room-temperature hardness

Figure 9 shows changes in room-temperature hardness (H_v) with annealing temperature for the AZ31 alloy deformed to $\varepsilon = 1.2$ at 573 K and at strain rates of 3×10^{-3} and $3 \times 10^{-1} \text{ s}^{-1}$. H_v begins to decrease gradually at low temperatures, then rapidly at 473–523 K, and finally approaches a constant value in high temperature above

Fig. 10 Effect of strain rate on the relationships between room-temperature hardness and average grain size for AZ31 alloy (a) as-hot worked to $\epsilon = 1.2$ at 573 K and at various strain rates (solid marks) and (b) followed by annealing for 10^3 s at various temperatures (open marks). ($\square$) shows the data for a full-annealed initial sample.

523 K. Such H_v - T relationships are roughly similar to the N - T ones in Fig. 7 compared with the data of D in Fig. 5. The values of H_v and N at $3 \times 10^{-1} \text{ s}^{-1}$ are always larger than those at $3 \times 10^{-3} \text{ s}^{-1}$ except the temperature range from 473 to 523 K. Effect of strain rate on the changes in H_v at 473–523 K is different from those for N or D in hot-deformed structure. This will be discussed in the following.

The room temperature hardness of hot-deformed Mg alloy should be influenced not only by deformation-induced dislocation substructures, but also new fine grains developed. The relationships between H_v and D developed before and after full annealing are summarized in Fig. 10 according to the Hall-Petch equation, *i.e.* $H_v = H_0 + kD^{-1/2}$, where H_0 and k are experimental constants. It is seen clearly in Fig. 10 that H_v - D relationships for as-deformed and full-annealed states can be approximated by a different Hall-Petch equation. The relationship is also dependent sensitively on strain rate. It is considered that the difference in H_v for a same grain size in as-deformed and full annealing state can result from reduction in dislocation densities as well as fine grains during annealing. A Hall-Petch slope (k) at a lower strain rate should be smaller compared with that at high strain rate, because dynamic recovery takes place more frequently during deformation.

It is concluded from the above examination, therefore, that a main factor controlling the value of H_v for hot-deformed and subsequent full-annealed Mg alloy can result from grain refinement or coarsening due to the Hall-Petch relationship, but a little from changes in dislocation densities due to dynamic or static recovery. This is in contrast with that for

conventional strain-hardened cubic metals,¹¹⁾ *i.e.* changes in H_v during hot deformation and subsequent annealing can result mainly from increase or decrease in dislocation densities in substructures.

4. Conclusions

Isochronal annealing behaviors of hot-deformed Mg alloy AZ31 were studied at temperatures from 373 to 673 K. The main results obtained are summarized as follows.

- (1) Temperature dependence of the average grain size (D) is categorized into three temperature regions, *i.e.* (a) an incubation period of grain growth at < 450 K, (b) rapid grain coarsening at 473–523 K and (c) normal grain growth at $T > 550$ K. The number of fine grains per unit area, however, is reduced remarkably even in region (a). It is concluded, therefore, that grain growth takes place continuously during annealing in the whole range from low to high temperature.
- (2) A bimodal distribution of grain size in as-deformed state changes to a single peak type after rapid grain coarsening taking place in medium to high temperature. The relative frequency of fine grains decreases rapidly and that of coarse grains increases gradually with annealing.
- (3) The deformation texture with the basal plane (0001) perpendicular to the compression axis can exist stably at high temperature and so scarcely change even after full annealing.
- (4) It is concluded from the results (1) to (3) that the annealing processes operating in hot-deformed Mg alloy with fine grains, developed by continuous dynamic recrystallization (cDRX), are controlled mainly by grain growth accompanied with no texture change, that is continuous static recrystallization (cSRX).
- (5) The relationships between room-temperature hardness and average grain size for as-deformed and full-annealed states can be approximated by different Hall-Petch relationships, which also depend sensitively on strain rate during hot deformation.
- (6) The main reason for the changes in the room temperature hardness during hot deformation and subsequent annealing can result from grain refinement or coarsening, and a little from change in the dislocation density due to dynamic or static recovery.

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