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Special Issue on Severe Plastic Deformation for Production of Ultrafine Structures and Unusual Mechanical Properties: Investigating Role of High-Density Lattice Defects

Mechanical Properties of Magnesium Alloy AZ31 after Severe Plastic Deformation*1

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Grain refinement of magnesium alloy AZ31 was studied in multidirectional forging (MDF) under decreasing temperature conditions. MDF was carried out up to large cumulative strains of 5.6 with changing the loading direction during decrease in temperature from pass to pass. MDF can accelerate the uniform development of very fine- grained structures and an increase of the plastic workability at low temperatures. New grain structures with the minimal grain size of 0.23 µm can be developed by continuous dynamic recrystallization at a final processing temperature of 403 K. As a result, the multidirectional- forged alloy showed excellent higher strength as well as moderate ductility at room temperature, and also a superplastic elongation of over 300% at 423 K. The mechanisms of strain-induced and fine-grained structure development and of the excellent plastic deformation are discussed in detail. [doi:10.2320/matertrans.ME200705]

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Keywords: *severe plastic deformation, multidirectional forging, magnesium alloy, workability, strain-induced grain refinement, continuous dynamic recrystallization, Hall-Petch relation*

1. Introduction

Magnesium (Mg) alloys are the lightest metallic structural materials and have the following several features, such as high specific strength, good electromagnetic interference shielding, and *etc.*[1] They exhibit a limit ductility due to the hcp lattice and are categorized as hard plastic materials. The Mg structural materials have been manufactured less frequently by plastic working such as rolling and other forming process than by casting route.[1,2] It is expected, however, that several slip systems can operate on basal and non-basal planes during warm and hot deformation, leading to an increase of the plastic workability. It is also known[2,3] that fine grains are developed in Mg alloys at relatively low strains during warm and hot working. The plastic workability of such fine-grained Mg alloys can be much improved accompanied by superplasticity due to operation of grain boundary sliding.[3]

The aims of present work were to study the optimum processes for grain refinement of Mg alloy and to improve the mechanical properties at ambient temperature. Fine-grained structures were developed in a Mg alloy AZ31 by using multidirectional forging (MDF) under decreasing temperature conditions from pass to pass.[4,5] The process of grain refinement taking place during MDF and the mechanical properties of the products were investigated in detail. The mechanisms of grain refinement and plastic deformation of the fine-grained Mg alloy are analyzed and discussed.

2. Experiment Procedure

A commercial Mg alloy AZ31 was provided as a hot-extruded rod with the following chemical composition: 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 mm in length, 21 mm in width and 14 mm in thickness (the axis ratio of 2.22 : 1.49 : 1) were machined from the rod parallel to the extrusion direction. The samples were annealed at 733 K for 7.2 ks and then furnace cooled, leading to the evolution of almost equiaxed grains with a diameter of about 22 µm.

MDF were carried out in compression at a constant true strain rate of $3 \times 10^{-3}\ s^{-1}$ in vacuum by using a computer-aided testing machine equipped with a quenching apparatus. The later made a sample possible to quench in water within 1.5 s after hot deformation was ceased.[4] The samples were deformed by repeated MDF with changing the loading axis at an angle of 90° with a pass strain ($\Delta\varepsilon$) of 0.8 (see Fig. 1(a)). The sample dimension did not change during MDF when $\Delta\varepsilon$ was kept at constant.[5] MDF was carried out with decreasing temperature from 623 K to 403 K, or at a constant temperature of 623 K, and then quenched in water in each pass. Deformed samples were cut along planes parallel to the last compression axis, and microstructural observations were carried out by using optical microscopy (OM) and transmission electron microscopy (TEM) under an accelerating voltage of 200 kV.

Tensile specimens with a gauge dimension of 6 mm in length, 3 mm in width, and 0.7 mm in thickness were machined parallel to the final compression axis (CA) of multidirectional-forged (MDFed) samples. The tensile axis (TA) was perpendicular to the CA (Fig (b)). Tensile tests were conducted in vacuum by using an Instron-type testing machine which was equipped with a hydrogen gas quenching apparatus.[6] Tensile tests were carried out at temperatures of 298 K and 423 K and at initial strain rates ranging from $8.3 \times 10^{-6}\ s^{-1}$ to $8.3 \times 10^{-3}\ s^{-1}$. The Vickers hardness tests were also performed at room temperature on the MDFed samples.

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(a)

(b)

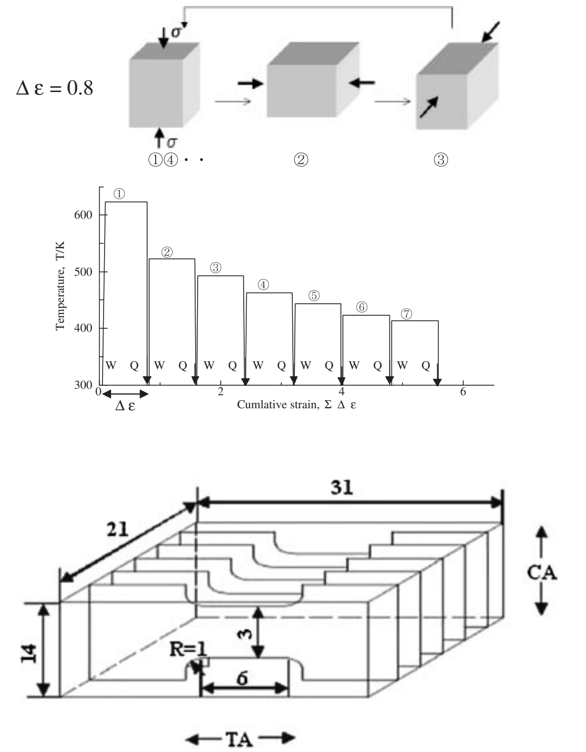

Fig. 1 (a) Schematic illustration of multidirectional forging (MDF) with a pass strain ($\Delta\varepsilon$) of 0.8 under decreasing temperature conditions. WQ indicates water quenching. (b) Tensile specimens machined from MDFed Mg alloy. The tensile axis (TA) was perpendicular to the final compression axis (CA).

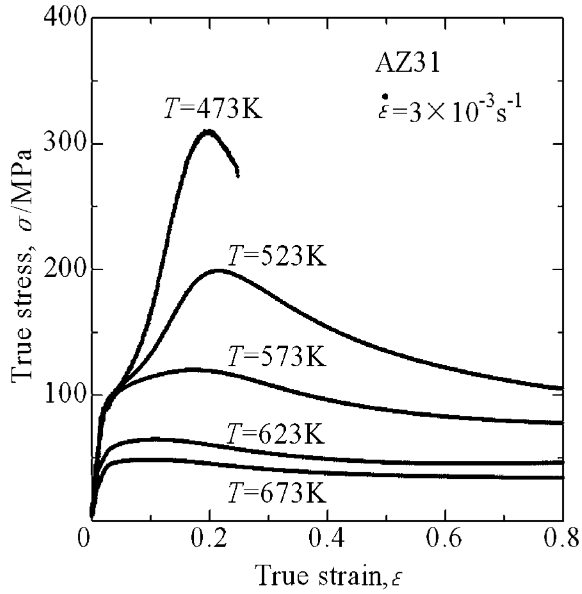

Fig. 2 Temperature effect on true stress-true strain curves of Mg alloy AZ31 during single pass compression at a strain rate of $3 \times 10^{-3}\,s^{-1}$.

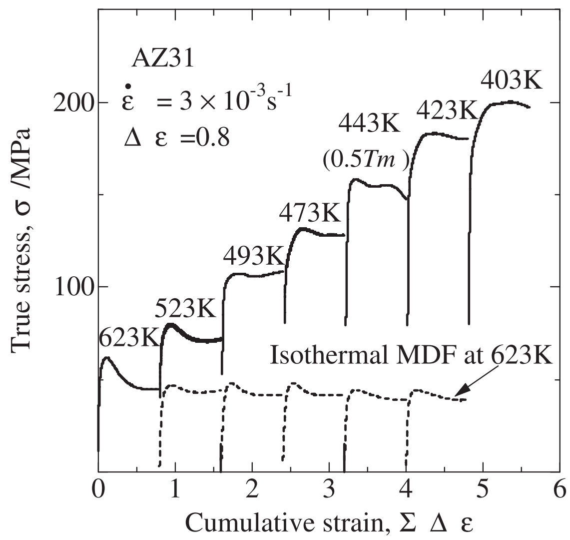

Fig. 3 True stress-true strain curves of Mg alloy AZ31 during isothermal MDF at 623 K (broken line) and MDF under decreasing temperature conditions from 623 K to 403 K (solid line).

3. Results

3.1 Deformation behaviors

The annealed Mg alloy AZ31 was deformed in compression at a true strain rate of $3 \times 10^{-3}\,s^{-1}$ and at temperatures ranging from 473 K to 673 K. A series of the true stress-true strain (σ–ε) curves is shown in Fig. 2. At high temperatures above 573 K, the σ–ε curves show work softening following a smooth stress peak at low strain, then followed by steady-state like flow at high strains. Such flow behaviors are generally similar to those of conventional dynamic recrystallization (DRX) behavior appearing in cubic metals.[7] At low temperatures below 523 K, in contrast, the σ–ε curve shows rapid work hardening in low strain and a sharp stress peak at around $\varepsilon = 0.2$, and then work softening followed by steady state flow at high strains. The σ–ε curve at 473 K exhibits a sharp and higher stress peak of above 300 MPa at around $\varepsilon = 0.2$, immediately followed by brittle fracture.

Typical true stress-cumulative strain (σ–$\Sigma\Delta\varepsilon$) curves during isothermal MDF at 623 K and during repeated MDF under decreasing temperature conditions from 623 to 403 K are shown in Fig. 3. The first flow curve at 623 K shows a stress peak followed by work softening and subsequently steady- state like flow at high strains. The amounts of the peak stress and work softening after the peak decrease with repeated isothermal MDF at 623 K (broken line). A steady state flow appears in high cumulative strain and the flow stresses of around 40 MPa do not change during MDF.

On the other hand, the σ–$\Sigma\Delta\varepsilon$ curves during MDF with decreasing temperature conditions are depicted by solid line in Fig. 3. They show work softening following a peak stress and then followed by steady-state flow during early MDF at higher temperatures. With dropping the processing temperature, the values of flow stress increase accompanying with decrease in the amount of work softening after the stress peak. At temperatures below 493 K, steady-state flow appears without a peak stress as well as flow softening. It is interesting to note that the the σ–ε curve at 473 K in Fig. 2 and that appearing at around $\Sigma\Delta\varepsilon = 3$ during MDF at 473 K (Fig. 3) is clearly different from each other. Namely, the flow stress peak is 310 MPa at $\varepsilon = 0.2$ and then brittle fracture occures in Fig. 2, and, in contrast, that appearing during MDF at 473 K ia about 128 MPa which is about one-third of the former. Furthermore, the MDF processing was successful to deform the Mg alloy to cumulative large strains up to

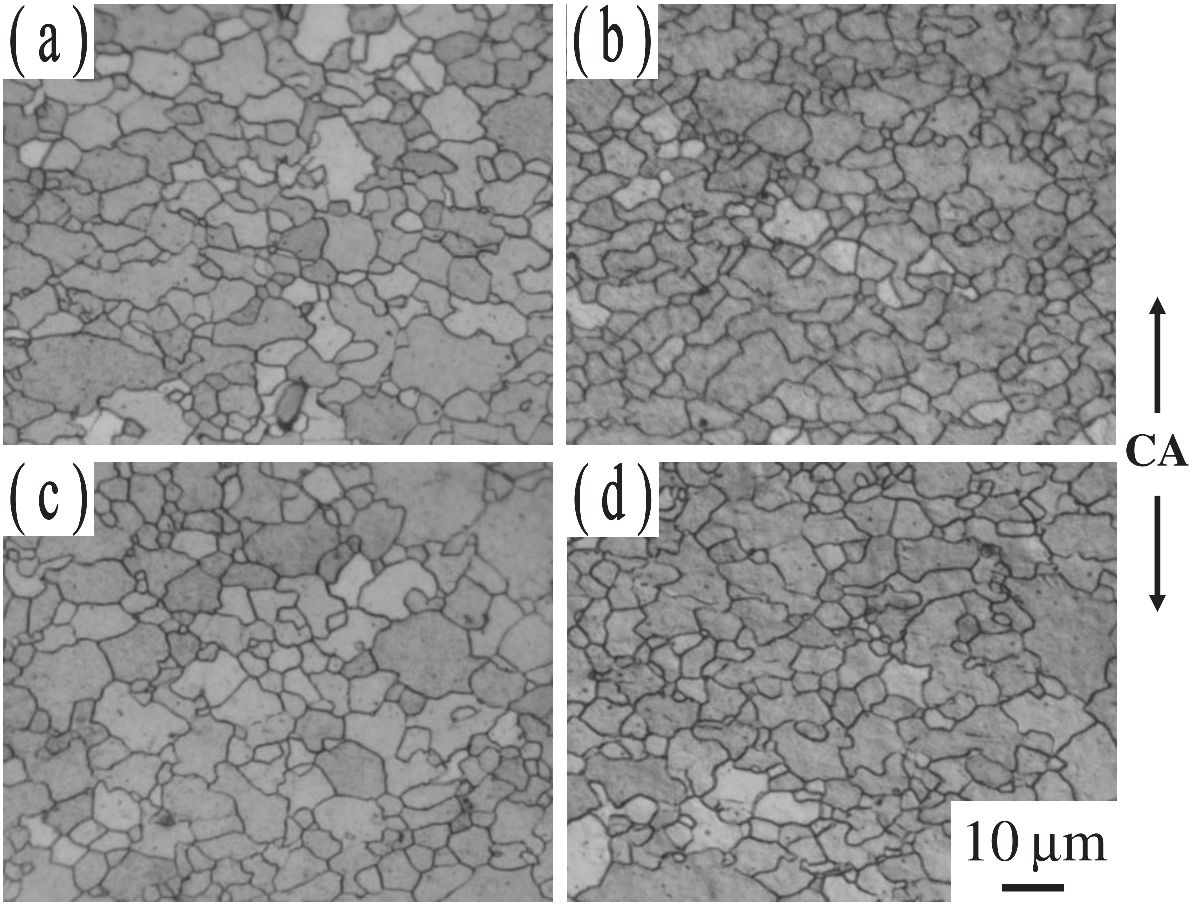

Fig. 4 Optical microstructures evolved in Mg alloy AZ31 during isothermal MDF at 623 K. CA indicates compression axis. (a) $\Sigma\varepsilon = 0.8$, (b) $\Sigma\varepsilon = 1.6$, (c) $\Sigma\varepsilon = 2.4$ and (d) $\Sigma\varepsilon = 4.8$.

$\Sigma\Delta\varepsilon = 5.6$ at 403 K which is below than $0.5T_m$ (T_m is the melting point).

3.2 Microstructural changes during MDF

Figure 4 shows the OM microstructures developed during isothermal MDF at 623 K. The initial grain size of 22.3 μm decreases to about 6.7 μm after 1st-pass compression, but this grain size does not change with further MDF to high strain of $\Sigma\Delta\varepsilon = 4.8$. On the other hand, Fig. 5 shows the OM microstructures developed during repeated MDF with decreasing temperature from 623 to 443 K. It can be clearly seen in Fig. 5 that the grain size decreases rapidly by straining with reducing temperature; i.e. the initial grain size of 22.3 μm decreases to about 6.7 μm after 1st-pass compression at 623 K (Fig. 5(b)), 3.8 μm after 2nd-pass at 523 K (Fig. 5(c)), 1.3 μm after 3rd-pass at 493 K (Fig. 5(d)), and 0.8 μm after 4th-pass at 473 K (Fig. 5(e)). The grain size developed by the 5th-pass compression at 443 K is further decreased, but difficult to be measured by using OM (Fig. 5(f)).

Figure 6 shows a typical TEM microstructure and the selected area diffraction (SAD) pattern developed in the sample deformed to $\Sigma\Delta\varepsilon = 5.6$ at 403 K. Many diffraction points with a streak in the SAD pattern suggest that rather large internal stresses can be evolved in this strain-induced grain structure.[8] The SAD pattern in Fig. 6 shows almost uniform and fully continuous rings, suggesting that this microstructure is composed of polycrystalline ultra-fine grains surrounded by high angle boundaries. The average grain size of 0.23 μm is fully developed by the 7th-pass compression at 403 K.

Changes in the average grain size evolved in the Mg alloy AZ31 during isothermal MDF at 623 K and during MDF under decreasing temperature conditions were shown by a broken line and a solid line in Fig. 7, respectively. The average grain size developed during isothermal MDF is almost constant within the experimental scatters and about 6.7 μm at strains up to $\Sigma\Delta\varepsilon = 4.8$. On the other hand, the dynamic grain size evolved during MDF with dropping temperature conditions decreases drastically with repeated MDF. It is concluded in Fig. 7 that the MDF processing under dropping temperature conditions may be a most effective method for grain refinement of Mg alloys.

3.3 Tensile deformation at warm temperature

Tensile tests were carried out at 423 K by using the fine-grained Mg alloy AZ31 processed by MDF under decreasing temperature conditions. Figure 8 shows grain size dependence of the true stress-nominal strain (σ-ε_n) curves tested at an initial strain rate of $8.3 \times 10^{-4}\,s^{-1}$. The σ-ε_n curve of the 22.3 μm sample shows the highest stress peak at a strain of 30%, followed by gradual work softening and fractured at a total strain of about 60%. With decreasing the grain size, flow stresses rapidly decrease accompanying with remarkable

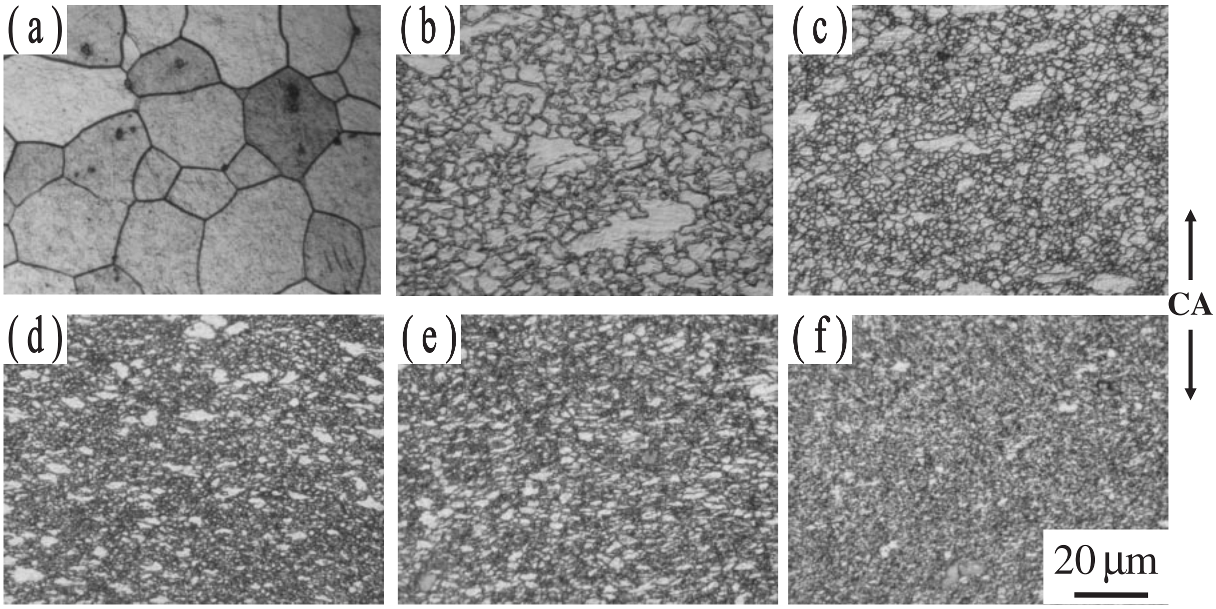

Fig. 5 Optical microstructures evolved in Mg alloy AZ31 during MDF under decreasing temperature conditions. (a) $\varepsilon = 0$, (b) T = 623 K, $\Sigma\Delta\varepsilon = 0.8$, (c) T = 523 K, $\Sigma\Delta\varepsilon = 1.6$, (d) T = 493 K, $\Sigma\Delta\varepsilon = 2.4$, (e) T = 473 K, $\Sigma\Delta\varepsilon = 3.2$, and (f) T = 443 K, $\Sigma\Delta\varepsilon = 4.0$.

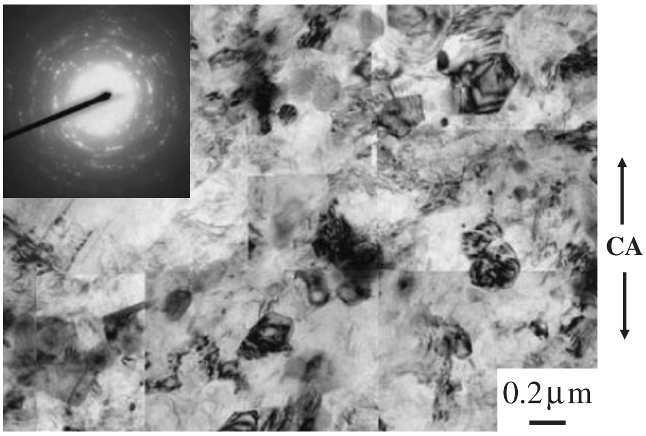

Fig. 6 Typical TEM microstructure and the diffraction pattern of Mg alloy AZ31 deformed up to $\Sigma\Delta\varepsilon = 5.6$ by MDF at T = 403 K.

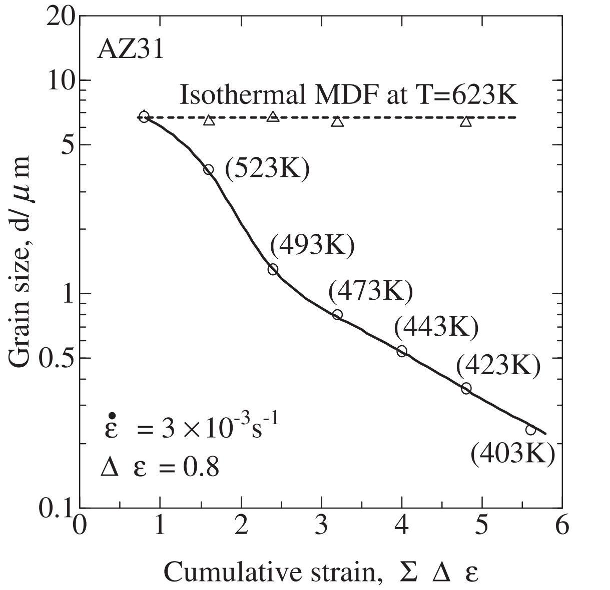

Fig. 7 Grain size changes in Mg alloy AZ31 with isothermal MDF at 623 K (broken line) and with MDF under decreasing temperature conditions from 623 K to 403 K (solid line).

increase in total elongation. The σ-ε_n curve of the 0.36 μm ultra-fine grained Mg alloy shows a smooth stress peak at around 80% strain following gradual work hardening, and then small work softening, leading to a total elongation to fracture of about 185%.

Typical σ-ε_n curves, tested at 423 K under various strain rates from $8.3 \times 10^{-6}\,s^{-1}$ to $8.3 \times 10^{-3}\,s^{-1}$ for the 22.3 μm and the 0.36 μm Mg alloy, were depicted by broken and solid lines in Fig. 9. Total elongations to fracture for the 22.3 μm samples change scarcely with strain rate, and are almost below 60%. On the other hand, the average flow stresses for the ultra-fine grained samples are far below than those for the 22.3 μm samples and the total elongations to fracture increase remarkably with decreasing strain rate. The total elongation to fracture at $8.3 \times 10^{-6}\,s^{-1}$ attains to over 300%. It is concluded from Fig. 9 that grain refinement taking place during MDF process under dropping temperature conditions can result in remarkable improvement of the ductility of Mg alloy. The present ultra-fine grained Mg alloy exhibited a maximum total elongation of more than 370% at 393 K.[9)]

3.4 Tensile deformation and hardness at room temperature

Tensile tests were carried out at 293 K and at 8.3×10^{-3} s^{-1} by using the ultra-fine grained Mg samples processed by MDF under dropping temperature conditions. Figure 10 shows grain size dependence of the true stress-nominal strain (σ-ε_n) curves measured in tension at room temperature. The σ-ε_n curve of the 22.3 μm sample shows a monotonous work hardening following yielding and then a stress peak just

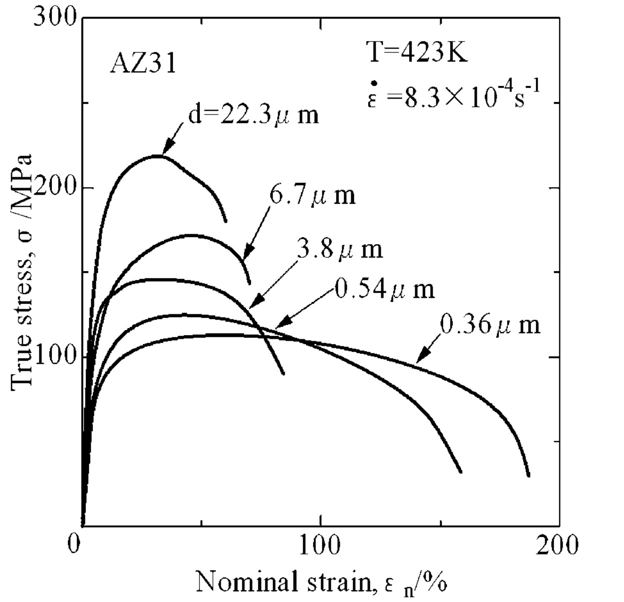

Fig. 8 Effect of grain size on true stress- nominal strain (σ-ε_n) curves at 473 K and at $8.3 \times 10^{-4}\,s^{-1}$ for Mg alloy AZ31 processed by MDF under dropping temperature conditions.

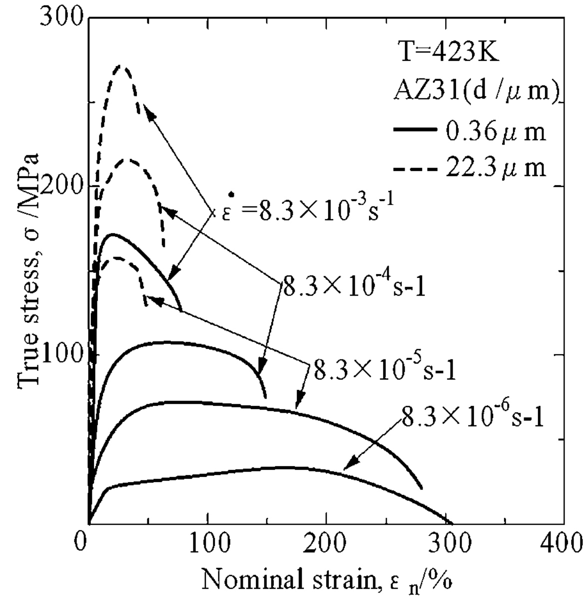

Fig. 9 Strain rate dependence of true stress-nominal strain (σ-ε_n) curves at 423 K for Mg alloy AZ31 with the grain sizes of 0.36 µm and 22.3 µm.

before fracture at a total elongation of about 35%. It is remarkable to note in Fig. 10 that the yield and peak stresses just before fracture increase remarkably accompanying with rather large magnitude of work hardening and moderate total elongation with decreasing grain size. The σ-ε_n curve of the 0.36 µm sample shows the highest yield stress over 400 MPa and the peak stress of 526 MPa at a total elongation of 13%. This yield stress is almost 5.3 times larger than that for the as-annealed sample.

Figure 11 shows the relationships between yield flow stress at a strain of 0.2% (σ_y) or room-temperature hardness (H_v) and the average grain size (d) developed during MDF. The results of H_v and σ_y are represented by open and closed circles, respectively. It can been seen clearly in Fig. 11 that the relationship between H_v or σ_y and d can be approximated by the following Hall-Petch eqs. (1) and (2) with almost similar slopes of 0.23 and 0.21.

$$H_v = 500 + 0.23d^{-1/2} \quad (1)$$

$$\sigma_y = 80 + 0.21d^{-1/2} \quad (2)$$

Here the data for the as-annealed samples ((○) and (●)) are not taken into account because they do not contain deformation- induced high-density dislocations. The maximum values of H_v and σ_y for the ultrafine grained sample are over about 2 and 4 times larger than those for the annealed one, respectively. It is concluded that excellent improvement of the mechanical properties of Mg alloy can be attained by the MDF processing under decreasing temperature conditions.

4. Discussion

4.1 Grain refinement in Mg alloy during MDF

It is well known[2,3] that fine grains are developed in Mg alloys at relatively low strains during warm and hot working. The authors[10,11] investigated grain refinement mechanisms occurring in the Mg alloy AZ31 during high temperature deformation and discussed that the mechanism of new grain formation is clearly different from conventional discontinuous dynamic recrystallization (dDRX). The dDRX involves the nucleation of new, dislocation free grains followed by subsequent long-distance growth through migration of high angle grain boundaries.[7] During hot deformation of Mg alloy, in contrast, kink bands are frequently evolved in original grain interiors at low strains. The number and misorientation angle of kink bands rapidly increase with further deformation, finally resulting in development of new fine-grained structures at strains above 0.5. They concluded that dynamic grain evolution taking place in Mg alloy can be based on grain fragmentation due to formation of kink bands in original grain interiors and so controlled by deformation-induced continuous reactions assisted by dynamic recovery, i.e. in-situ or continuous dynamic recrystallization (cDRX).[10] During steady state flow in each pass strain (i.e. $\Delta\varepsilon = 0.8$), equiaxed new grains are homogeneously developed in a whole volume, as can be seen in Figs. 4 to 6.

Kink bands are formed roughly perpendicular to the basal plane of the Mg alloy.[11] In single pass compression, the basal plane of the extruded Mg rod, parallel to the compression axis, is rotated and approaches perpendicular to the compression axis at high strains. After that, the generation of kink bands as well as full development of new grains hardly take place even in high strain above 0.5.[10,11] During MDF, in contrast, kink bands are developed in various directions and so crossed mutually due to change of compression axis from pass to pass. This process should finally lead to a homogeneous and full development of fine grained structure in a whole volume.[5] The grain size developed in high strain does not change during isothermal MDF at 623 K (Figs. 4 and 7). However, it decreases rapidly with decrease in temperature under the present MDF conditions (Figs. 5 to 7), because the density of kink band developed may be controlled by

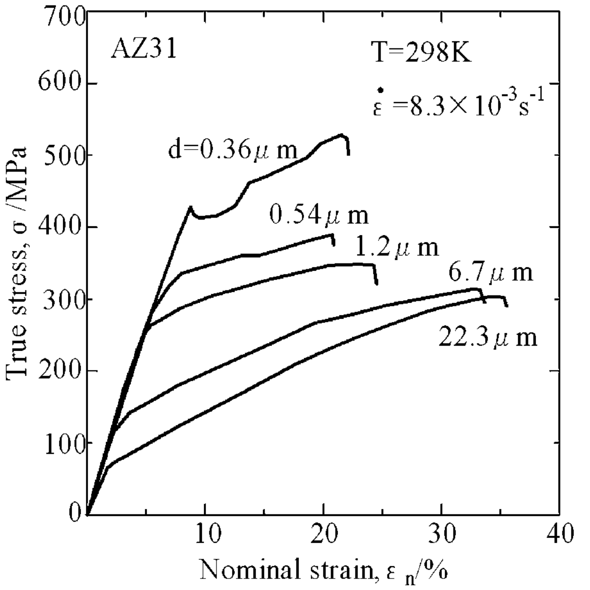

Fig. 10 Effect of grain size on true stress-nominal strain (σ-ε_n) curves at 298 K and at 8.3×10^{-3} s^{-1} for Mg alloy AZ31 processed by MDF with dropping temperature conditions.

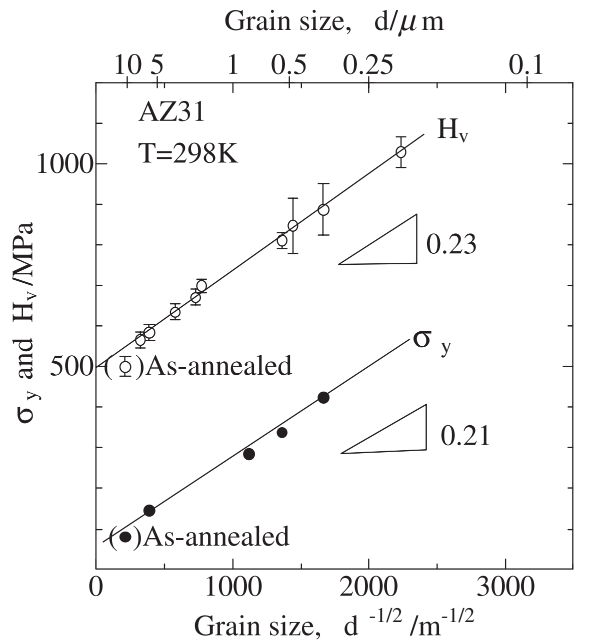

Fig. 11 Relationships between yield stress (σ_y) or hardness (H_v) at 298 K and grain size developed by MDF of Mg alloy AZ31.

difficulty of slip deformation and also of compatibility requirement of neighboring grains at lower temperature.

The flow stress appearing at around $\Sigma\Delta\varepsilon = 3$ during MDF at 473 K is about one-third of that for the as-annealed sample (Figs. 2 and 3). This may be resulted from such a drastic grain refinement taking place by repeated MDF under dropping temperature conditions. During MDF at temperatures from 623 K to 473 K, the average grain size decreases from 22.3 μm to 0.8 μm at $\Sigma\Delta\varepsilon = 3$. In such fine-grained structures, grain boundary sliding can take place frequently even during low temperature deformation,[9] leading to a reduction of flow stress as well as improvement of ductility (Figs. 8 and 9).

4.2 Mechanical properties of ultrafine grained Mg alloy

Figures 8 and 9 suggest that superplastic deformation can take place at 423 K in the ultrafine grained Mg alloy processed by MDF under dropping temperature conditions. The authors investigated the superplastic behaviors of the Mg alloy, the results of which are described in detail elsewhere.[9] Here the main results obtained are summarized briefly. During tensile deformation of a 0.36 μm ultrafine grained Mg alloy at 393 to 473 K, the initial grain size increases or decreases with lower or higher strain rate deformation, respectively. This can result in relatively large stress exponents of above 5. The strong deformation texture, i.e. the basal plane roughly parallel to the tensile axis (see Fig. 1(b)), exists stably during tensile deformation. It is concluded that superplasticity of strain-induced fine-grained Mg alloy can be controlled mainly by grain boundary sliding, while grain rotation hardly takes place. Such superplastic characteristics should be examined in more detail in near future.

On the other hand, mechanical properties of the ultrafine grained Mg alloy are excellent for not only strength, but also ductility at ambient temperature (Fig. 10). It has been reported in Ref. 2, 12–14 that such excellent mechanical properties can be attained in various ultra-fine grained materials processed by severe plastic deformation (SPD). Recently, Tsuji *et al.*[15] investigated the grain size dependence of room temperature strength as well as ductility of an ultra-fine grained aluminum (Al) alloy 1100 and an interstitial free (IF) steel processed by the accumulative roll-bonding (ARB) method. Figure 12 shows the relationships between grain size and uniform elongation of Al alloy and IF steel processed by ARB and also of the present Mg alloy processed by MDF. It can be seen in Fig. 12 that grain size dependence of the uniform elongation is almost the same in the Al and IF steel irrespective of the different crystal lattice. It is remarkable to note that the uniform elongation of these metals suddenly drops to a few per cent at the grain sizes below 1 μm. In contrast, the uniform elongation of the Mg alloy with hcp lattice decreases gradually with decreasing grain size, but is always more than 13% even in the ultrafine grained region below 1 μm. Tsuji *et al.* discussed that the dropping of uniform elongation at grain sizes below 1 μm may be resulted from small or even negative work-hardening rate after yielding appearing in the ultrafine grained materials, that is plastic instability.

By the way, Koike *et al.*[16] studied dislocation microstructures developed by tensile deformation at room- temperature for a polycrystalline Mg alloy AZ31. They observed that non-basal **a** dislocations active only near grain boundaries, while basal **a** dislocations are dominant in grain interiors for a 50 μm coarse-grained sample. In contrast, in a 7 μm fine grained Mg alloy non-basal **a** dislocation segments are around 40% of the total dislocation density even in the grain interiors. If the data obtained by Koike *et al.* can be extrapolated to a range of submicron grain size, non-basal **a** dislocations can be more activated to develop in grain interiors by compatibility stress concentration.[16] This can

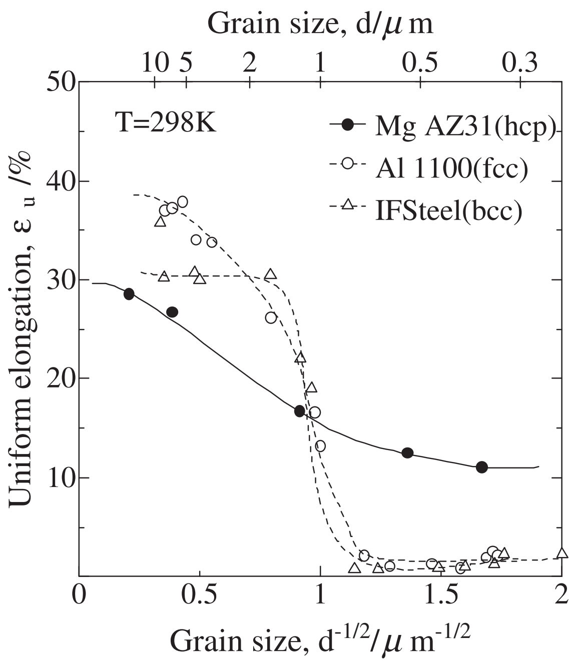

Fig. 12 Grain size dependence of uniform elongation measured in tensile test at room temperature for Mg alloy AZ31 processed by MDF, and for Al alloy 1100 and IF steel processed by severe plastic deformation. The data for cubic metals are reported by Tsuji *et al.*[15)]

activate various kinds of slip systems operating in non-basal and basal planes, and then result in relatively large work-hardening as well as moderate uniform elongation appearing in the ultra-fine grained Mg alloy, as can be seen in Fig. 10.

5. Summary

Grain refinement as well as improvement of the mechanical properties of magnesium alloy AZ31 was studied in multidirectional forging (MDF) under decreasing temperature conditions. The main results obtained are summarized as follows.

(1) MDF under dropping temperature conditions can accelerate the evolution of finer-grained structures in Mg alloy. The minimal grain size of 0.23 μm was developed at $\Sigma\varepsilon = 5.6$ and at 403 K.

(2) Total elongation of ultra-fine grained Mg processed by MDF is above 300% at 423 K and at $8.3 \times 10^{-6}\,\mathrm{s}^{-1}$, suggesting superplasticity taking place in the Mg alloy.

(3) The ultrafine grained Mg alloy shows always a moderate uniform elongation more than 13% at room temperature even in the range of ultrafine grain size below 1 μm.

(4) The relationship between room-temperature hardness (H_v) or yield stress (σ_y) and strain-induced grain size (d) can be approximated by the following Hall-Petch equations in the range of all grain size introduced by MDF, i.e. $H_v = 500 + 0.23d^{-1/2}$ and $\sigma_y = 80 + 0.21d^{-1/2}$.

Acknowledgments

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