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ULTRAFINE GRAIN EVOLUTION IN Mg ALLOYS, AZ31, AZ61, AZ91 BY MULTI DIRECTIONAL FORGING

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Abstract: AZ31, AZ61, and AZ91 Mg alloys having different initial grain sizes were multi directionally forged (MDFed) under decreasing temperature condition from 673K to 473K to a cumulative strain $\Sigma\Delta\varepsilon$ of 3.2. A pass strain of $\Delta\varepsilon$ = 0.8 and a strain rate of 3 x 10^{-3} s^{-1} were employed. In all the alloys, the average grain size decreased rapidly with cumulative strain during MDF. Twinning and kinking dominantly contributed to grain fragmentation. After straining to $\Sigma\Delta\varepsilon$ = 3.2 (i.e., after 4 passes of MDF), equi-axed ultrafine grains (UFGs) with an average size around 1 μm were uniformly evolved. The room-temperature hardness was gradually raised with decreasing grain size. The increment in hardness looked more significant with increasing content of Al in the alloys, even while the obtained grain size was coarser. This would be induced by coarser and higher density of $Mg_{17}Al_{12}$ precipitates in addition to high higher Al content. The mechanisms of UFG evolution during MDF and the mechanical properties of UFGed alloys are analyzed and discussed.

1. INTRODUCTION

A numerous number of researches on severe plastic deformation (SPD) have been carried out to obtain ultrafine grained (UFGed) metallic materials [1-5], because it is assumed that UFGed structure induces notable improvement of mechanical properties of strength and formability. Various methods for SPD processes have been developed and actually applied also to Mg alloys [2-5]. The poor ductility due to limited number of slip systems in Mg alloys, the most of SPDs of Mg alloys are carried out at elevated temperature. From such researches, it is being revealed that UFGed Mg alloys possess quite large ductility as well as high strength. Kai et al. have processed AZ90Mg alloy by high pressure torsion and reported superplasticity of about 800% at 423K [2]. Xing et al. have reported that the UFGed AZ31Mg alloy produced by multi directional forging (MDF) exhibits superior balance of strength and ductility; ultimate tensile strength of 530 MPa, ductility about 13% at room temperature and superplasticity at 423K [3]. A similar tendency was observed also in the MDFed AZ61Mg alloy [4]. Such large ductility of the UFGed Mg alloys is opposite property observed in the other cubic metallic materials with UFGed structures. Xing et al.[3] and Miura et al.[4] have attributed the large ductility of the UFGed Mg alloys to extensive occurrence of grain boundary sliding (GBS). Actually, it is reported that GBS takes place even at ambient temperature in Mg [6]. Because the easier occurrence of GBS causes superplasticity at relatively low temperature, it can be called as *low temperature superplasticity* [3].

Although the mechanical properties of Mg alloys are being modified by means of grain refinement and alloying, further improvement especially strengthening is highly desirable. Miura et al. have

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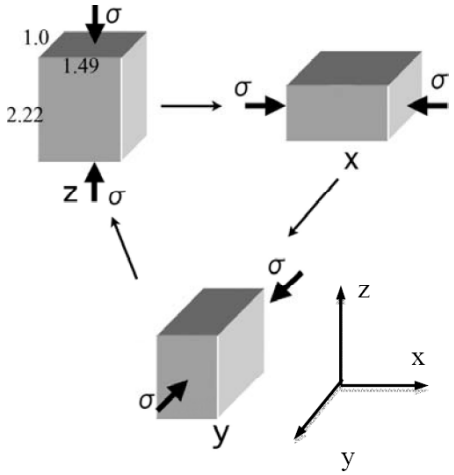

Fig. 1. A schematic representation of multi directional forging when a pass strain of $\Delta\varepsilon = 0.8$ is employed. Forging axis is changed by 90 degrees at each pass $z \rightarrow x \rightarrow y \rightarrow z$... Numbers indicate the aspect ratio of the sample.

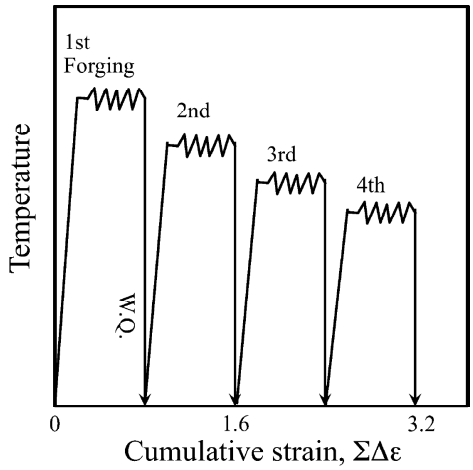

Fig. 2. Schematic representation of the present thermomechanical process by multi directionally forging (MDF) under decreasing temperature condition. The forging temperature was gradually decreased pass by pass. MDF with $\Delta\varepsilon = 0.8$ was carried to a cumulative strain of $\Sigma\Delta\varepsilon = 3.2$ at maximum. W.Q. indicates water quench.

suggested from the experimental results of MDF of AZ61Mg alloy [7] that the alloy would possess comparably higher potentials of easier evolution of UFGs by MDF and higher strength at room temperature than AZ31Mg one. This implies that higher content of Al in the AZ series of Mg alloys may cause further improvement of mechanical properties. This is the motivation of the present study. For this purpose, the Mg alloys, such as AZ31, AZ61 and AZ91, are MDFed and the tendency of UFG evolution was examined. Furthermore, improvement of the mechanical property of the MDFed Mg alloys was also studied.

2. EXPERIMENTAL

AZ31, AZ61 and AZ91 Mg alloys with initial grain sizes of 22, 57, and 155 mm respectively, all of which were obtained by annealing at 733K for 2 h of the commercial extruded rods, were spark cut to rectangular shaped samples with dimensions of 31 x 21 x 14 mm^3. The different initial grain sizes were due to largely different initial microstructure of the supplied commercial extruded rod alloys. Even while finer grain size is desirable for MDF because of more rapid evolution of UFGed structure with decreasing initial grain size [8], they were subsequently MDFed. MDF was carried out on an Instron-type mechanical testing machine at a true strain rate of $3 \times 10^{-3}\ s^{-1}$ in vacuum. A pass strain of $\Delta\varepsilon = 0.8$ was employed. During MDF, forging temperature was gradually decreased pass by pass from 673K to 473K. The procedure of MDF and the thermomechanical process employed in the present MDF under decreasing temperature condition are schematically drawn in Figs. 1 and 2. The forging temperature profile during MDF must be drastically changed depending on the alloys due to the largely different initial grain size and distribution of β phase, i.e., $Mg_{17}Al_{12}$. The appropriate MDF temperature profile depending on alloy was determined by several forging tests at various temperatures at each pass. The determination process of the MDF temperature is precisely described elsewhere [4]. The forging temperature was finally determined as 623K → 523K → 493K → 473K for AZ31Mg, 623K → 573K → 523K → 503K for AZ61Mg and 673K → 623K → 573K → 523K for AZ91Mg alloys, respectively. While the aspect ratio of the sample is theoretically unchanged during MDF, the sample had to be reshaped by mechanical polishing because of shape change especially after the1st and 2nd passes of MDF. The shape change taking place at such early passes of MDF should be due to the strong texture developed in the extruded rods. MDF was carried out to cumulative strain of $\Sigma\Delta\varepsilon = 3.2$ at maximum, i.e., after 4 passes. The sample could be uniformly forged without any cracking even at 473K at minimum. Such obvious improvement of plasticity at such relatively low temperature was induced by evolution of UFGs, as will be shown later. After MDF, the evolved microstructure was observed on the plane parallel to the final forging axis using optical microscopy (OM) and orientation imaging

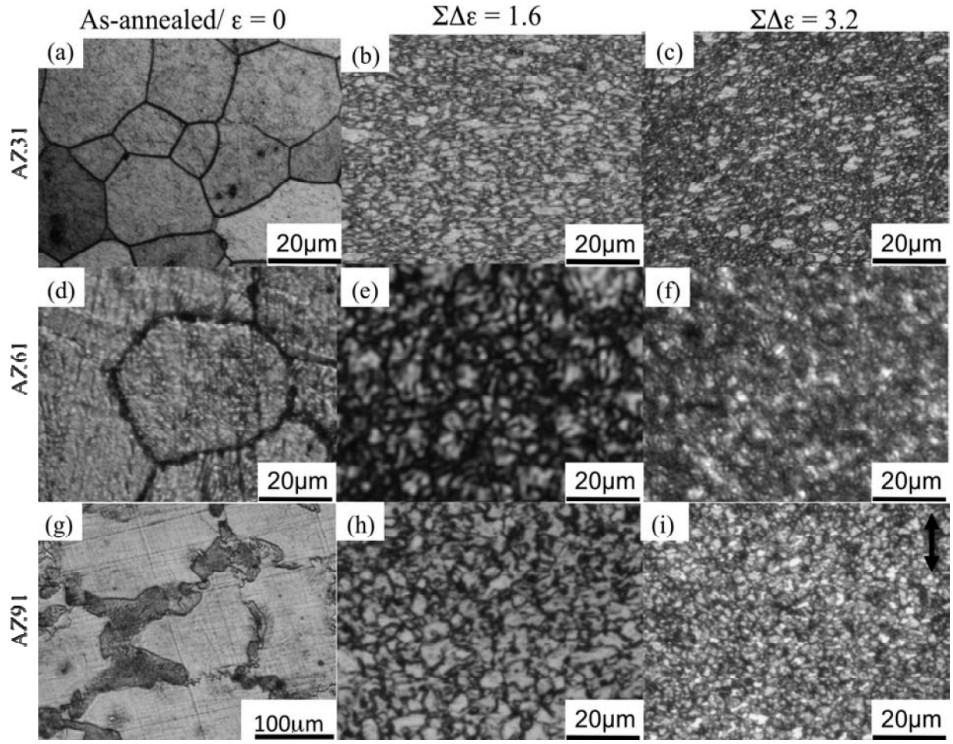

Fig. 3. Evolved microstructures in AZ31, AZ61, and AZ91Mg alloys during MDF under decreasing temperature condition to cumulative strain of ΣΔε = 3.2 at maximum. An arrow marks indicates the final forging axis.

microscopy (OIM). The Vickers hardness of the MDFed alloys was also investigated.

3. RESULTS AND DISCUSSION

3.1. Microstructural evolution during MDF

Fig. 3 exhibits the typical microstructures evolved during MDF of all the alloys. The volume fraction of the phase/ precipitates $Mg_{17}Al_{12}$ in the as-annealed initial microstructure tends to increase with increasing content of aluminum, while it can be hardly observed in the AZ31Mg alloy. Only by 2 passes of MDF, i.e., at ΣΔε = 1.6, coarse initial grains were drastically fragmented to have much finer and equiaxed homogeneous microstructure. It is matter of natural because each of the MDF temperature was determined to have such microstructure after preliminary experiments at various temperatures, as already mentioned above. By further MDF up to ΣΔε = 3.2, quite fine grains were almost uniformly evolved in the whole areas independent of alloys. The average grain size of 0.8 μm, 1.2 μm and 2.6 μm was achieved for the AZ31, AZ61, and AZ91 Mg alloys respectively. The b phase was also fragmented to much finer size during MDF, and therefore, it cannot be identified at ΣΔε = 3.2 anymore. This would contribute to the uniform distribution of fine precipitates especially in AZ91Mg alloy, which can induce strengthening, as shown later. It is interesting to see in Fig. 3, however, the grain size could not be the same even after severe plastic deformation up to ΣΔε = 3.2.

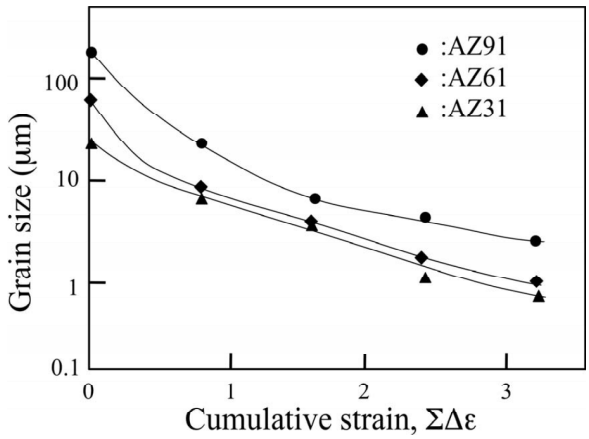

Fig. 4. Change in the average grain size of AZ31, AZ61, and AZ91Mg alloys during MDF under decreasing temperature condition to cumulative strain of ΣΔε = 3.2 at maximum.

The grain size change during MDF is summarized in Fig. 4. It is evident the grain size decreases rapidly at the early stage of MDF and, then, gradually at higher strains. This result suggests that different mechanisms of grain refinement would be operative and, still more, further finer grain size can be easily

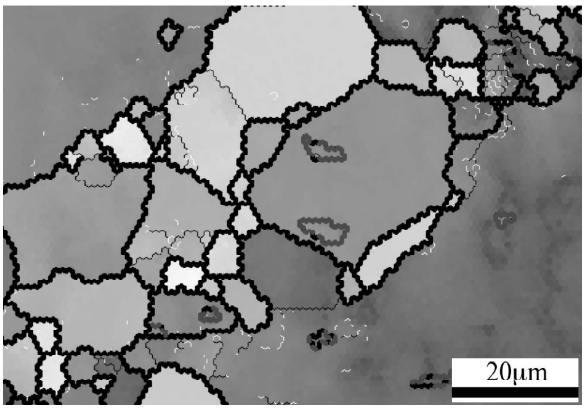

Fig. 5. Microstructure evolved in AZ61Mg alloy at $\Sigma\Delta\varepsilon$ = 0.3 and at 623K. Thin and bold black lines indicate low angle 3 ≤ θ 15 and high angle 15 ≤ θ grain boundaries. The thin white and bold brown lines are sub boundaries with θ < 3 and twin boundaries, respectively.

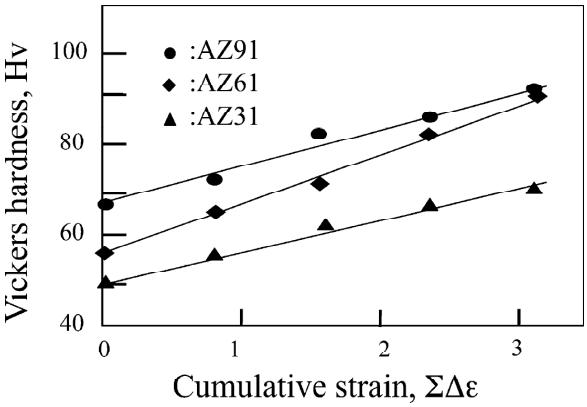

Fig. 6. Room-temperature hardness change of AZ31, AZ61, and AZ91Mg alloys during MDF to cumulative strain of $\Sigma\Delta\varepsilon$ = 3.2 at maximum under decreasing temperature condition. The hardness at ε = 0 is that of the as-annealed sample.

achieved by prolonged MDF. In fact, Xing et al. succeeded to obtain an average grain size of about 0.23 μm in the AZ31Mg alloy at $\Sigma\Delta\varepsilon$ = 5.6 [3]. It is interesting to note in Fig. 4 that the change in the grain size becomes more gradual with decreasing grain size. Therefore, large difference of grain size became smaller at higher strain region. The ratio of grain refinement ($\Delta d/d$) is 0.036, 0.02, and 0.02 in the Mg alloys AZ31, AZ61, and AZ91, respectively. Rather large grain refinement occurring in AZ61and AZ91 may be resulted from larger fractions of β precipitates in these alloys.

Belyakov et al. have experimentally shown that grain fragmentation during MDF is more proceeded when initial grain size is smaller [8]. This would be presumable due to the effective role of grain boundaries on grain refinement. In addition, it is known that deformation microstructure also contributes to grain fragmentation in case of Mg alloys. Among of them, deformation twinning and kinking are the most important mechanisms. The former contributes dominantly to grain fragmentation when grain size is coarse, but the latter when finer [9]. Twinning hardly takes place when grain size becomes smaller than 10 mm [10]. Of course, dislocations can also affect much to the grain refinement especially after severe plastic deformation to large strain region. These mechanisms are all strongly concerned with those of continuous dynamic recrystallization (cDRX). The final grain size achieved by the mechanisms of DRX is a function of temperature at a fixed strain rate condition [11]. The grain size, therefore, obtained should be larger when forged at higher temperature. The smaller difference in the grain size with increasing cumulative strain must be affected by some factors; i) recovery and coarsening of UFGs during MDF, ii) impediment of grain coarsening by precipitates, and iii) probability of twinning and kinking. The latter two factors would become more important in the alloys with higher content of aluminum, and therefore, the difference in the average grain size became relatively smaller with increasing cumulative strain. Actually, It is reported that UFGed AZ31Mg alloy possesses nature of quite low thermal stability, while it drastically improved in AZ61Mg alloy [7]. Still more, that twinning appears more frequently when stacking fault energy is reduced [12].

The evolved microstructure during MDF was observed using OIM and a typical map for AZ61 deformed to $\Sigma\Delta\varepsilon$ = 0.3 and at 623K, i. e., before the end of first pass of MDF, is exhibited in Fig. 5. New grains appeared preferentially along the initial grain boundary. This feature is similar to those of the other cubic metallic materials, while the mechanisms are quite different. What more important in Fig. 5 is that twinning and kinking appeared frequently and contributed to the grain fragmentation. From the analysis of the OIM map, it is revealed that kink bands were parallel either to basal or prismatic slip plane. It is notable to see in Fig. 5 that twins are formed mainly in the relatively coarser grains, and kinking in finer grains or near to grain boundaries. This evidently indicates the change in the dominant mechanisms of grain refinement during MDF. That is, twinning is most important mechanism when grain size is coarse, however, it changes to kinking with decreasing grain size. Of course, cDRX, which

is controlled by dislocation pile up and recovery to form low to medium grain boundaries, plays important role in all the process. The observed rather rapid decrease in the grain size at an early stage of MDF, therefore, should be much induced by twinning.

3.2. Hardness change during MDF

Room-temperature hardness change was investigated and the results for the present Mg alloys are summarized in Fig. 6. It can be seen in Fig. 6 that the hardness increased almost proportionally to cumulative strain irrespective of the alloys. When compared the hardness depending on alloy, it becomes higher with increasing content of aluminum, even while the grain size becomes larger and MDF temperature higher. The higher MDF temperature induces lower dislocation density due to easier recovery, and therefore, lower hardness. It is concluded, hence, the highest hardness in the AZ91Mg alloy must be because of the higher volume fraction of precipitates $Mg_{17}Al_{12}$. H_v = 90 was achieved at $\Sigma\Delta\varepsilon$ = 3.2 i.e., after 4 passes of MDF of AZ91Mg alloy. Because the hardness is seen to increase almost proportional to cumulative strain, hardness must be easily raised more with increasing pass number of MDF, therefore, with decreasing grain size.

It is interesting in Fig. 6 that the slope of hardness increment in AZ61Mg is largest, which is closely related with the tendency of the rapid grain refinement (see Fig. 4). Miura et al. suggested that more rapid grain refinement in AZ61Mg alloy due to easier occurrence of twins [7]. Because of the easier occurrence of twins with increasing content of aluminum, the AZ61 and AZ91Mg alloys should possess higher potential of more rapid grain fragmentation. The grain refinement and strengthening in the AZ61 and AZ91Mg alloys should be, therefore, drastically improved when the coarse initial microstructure before MDF is modified.

4. CONCLUSION

The AZ31, 61, 91Mg alloys were multi directional forging (MDF) under decreasing temperature condition up to cumulative strain of $\Sigma\Delta\varepsilon$ = 3.2 at maximum. The grain size decreased with increasing pass number of MDF and ultrafine grained (UFGed) structure with average grain size of about 1 μm could be obtained. The achieved microstructure at $\Sigma\Delta\varepsilon$ = 3.2 was strongly affected by the initial grain size. It is estimated that, therefore, finer initial grain size causes more rapid evolution of UFGed microstructure. The higher content of aluminum in the AZ series of Mg alloy induced higher volume fraction of precipitates, which affected much on strengthening. The coarse β phase observed in the initial microstructure was fragmented to quite finer sizes during MDF.

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