The Fine-Grained Structure in Magnesium Alloy Containing Long-Period Stacking Order Phase

Tatsuya Morikawa¹, Kenji Kaneko¹, Kenji Higashida¹, Daisuke Kinoshita¹, Masanori Takenaka¹,² and Yoshihito Kawamura²

¹Department of Materials Science and Engineering, Kyushu University, Fukuoka 819-0395, Japan
²Department of Materials Science, Kumamoto University, Kumamoto 860-8555, Japan

Microstructural characteristics of warm-extruded magnesium alloy (Mg₉₇Zn₁Y₂) containing long-period stacking order (LPSO) phase has been investigated using SEM and TEM as the first step to understand the effect of warm-extrusion on its mechanical property. Particular attention has been paid on the microstructural change in the hcp matrix caused by warm extrusion.

The microstructure developed by the warm extrusion at 623 K consists of elongated grains with fine-lamellae of LPSO phase and fine-grained matrix of hcp phase. The grain size of the hcp matrix observed on the cross section perpendicular to the extruding direction was about 1 µm, indicating that remarkable grain refinement was occurred during the extrusion since the grain size of as-cast alloy was around 0.5 mm. Those fine grains in the extruded alloy included abundant stacking faults, and HAADF-STEM observation revealed that the stacking faults were enriched by Zn and Y. In addition, grain boundaries were also enriched by those solute elements, which must contribute to stabilizing such fine-grained structure.

Keywords: grain boundary, fine-grained structure, transmission electron microscopy

1. Introduction

Magnesium alloys are one of the key materials to respond the urgent requirement for designing highly efficient system to solve the environmental problem. Particularly, Mg alloys containing Zn and rare earth elements such as Y or Gd have attracted much attention because not only of their characteristic microstructure but also of their unique performance in mechanical property.¹ The most essential characteristic in microstructures of these alloys is a long-period stacking ordered (LPSO) phase formed in the hcp matrix. The atomistic structure of the LPSO phase has been examined by Abe et al. using novel analyzing methods such as HAADF-STEM.²⁻⁴ However, there still remain problems on the relation between their mechanical performance and characteristic microstructures. One of the most interesting issues to be discussed here is the effect of warm-extrusion on the mechanical property. Yoshimoto et al. reported that usual warm extrusion (extruding ratio:10, 623 K) caused a marked enhancement of its strength without losing ductility which is necessary for practical plastic working.¹⁻⁵ In general, strengthening due to the mechanism such as solution hardening or precipitation hardening decreases the ductility of materials. However, such tradeoff relation between the strength and ductility does not appear in the present extruded Mg alloy. The only method for combining strength with ductility is grain-refinement although its fundamental mechanism has not been clarified yet.⁶⁻¹⁰

In the present study, as the first step to understand the mechanism increasing the strength without loss of ductility in the warm-extruded Mg₉₇Zn₁Y₂, the microstructure has been examined, where particular attention has been paid not only on the grain-refinement due to the extrusion but also the distribution of solute atoms Zn and Y in the 2H matrix.

2. Experimental

As-cast Mg₉₇Zn₁Y₂ alloy was extruded at 623 K. The extrusion ratio was 10 (equivalent strain of 2.3). Both microstructures of as-cast and extruded specimens were observed by scanning electron microscopy (SEM), where sample surfaces were polished by lapping papers, and cleaned by ethylene glycol containing 1% nitric acid. Microstructures in extruded alloys were observed also by transmission electron microscopy (TEM). For preparing TEM samples, extruded rods were sliced into disks with 1 mm thickness, and rectangular specimens were cut out from the disks. Those specimens were mechanically polished and thinned to foils by ion milling. The foil normal is parallel to the extruded direction. Orientation image mapping (OIM) was performed using electron back scattering diffraction (EBSD), where sample preparation was made using a section polisher.

3. Results and Discussion

Figures 1(a) and 1(b) show SEM images obtained from the as-cast and the extruded specimen, respectively. Figure 1(b) is obtained from the cross-sectional plane perpendicular to the extruding direction. White areas dispersed in both figures are considered to be the LPSO phase, according to the literature 4) and 5). The distribution of LPSO phase in the extruded alloy in Fig. 1(b) becomes finer than those of the as-cast alloy in Fig. 1(a). In the observation of the cross section parallel to the extruding direction, LPSO phase was observed to be elongated along the extruding direction. This indicates that the warm extrusion causes a fine dispersion of elongated LPSO phase in the 2H matrix. However, even after warm extrusion as observed in Fig. 1(b), the size and spacing of LPSO phase are still more than 10 µm, indicating this alloy should be regarded as a kind of composite material consisting of two phases of LPSO and 2H matrix.

*¹Present address: KOBE STEEL LTD.
*²Graduate Student, Kyushu University
Figure 2(a) shows an orientation image mapping (OIM) obtained from the cross-sectional plane perpendicular to the extruding direction by using electron back-scattering diffraction (EBSD). The each color in this figure designates the crystallographic orientation with respect to the sample normal which is parallel to the extruding direction according to the color key of the unit triangle (Fig. 2(b)). In Fig. 2(a), the areas of black or irregular aggregation of dots with various colors correspond to the regions where the OIM system could not analyze their diffraction patterns, which indicates that the areas should consists of LPSO phase. Areas of regular OIM mapping of 2H phase are also seen in Fig. 2(a). There appear not only fine-grains with the grain size of 1–3 μm but also relatively large grain with the size of 10μm. Small grains with the grain size within a few micrometers might be formed by dynamic recrystallization during extrusion. On the other hand, relatively large grains observed in the left-bottom in the figure are considered to be non-recrystallized grains, since the orientation with respect to the sample normal indicates nearly parallel to {1010} which corresponds to one of the preferential orientations induced by extrusion in hcp crystal. In addition, a gradation in blue color is also seen in the grain, which also suggests that the relatively large grain oriented to {1010} is as-deformed grain.

More detailed observation using TEM was conducted to examine the effect of the warm extrusion on the micro-structural change in the 2H matrix. Figure 3(a) shows a TEM bright-field image obtained from a matrix area where a fine-grained structure is formed. The diffraction pattern obtained from the fine-grained area is shown in the top right of the figure. There appears a large scattering of orientation, indicating the formation of high angle boundaries. Figures 3(b) and 3(c) show dark-field images of the same area as the bright field image of Fig. 3(a). They are obtained from the selected spots indicated by white arrows labeled (b) and (c) in the diffraction pattern. Bright areas in Figs. 3(b) and 3(c) correspond to a part of fine grains, demonstrating that the grain size in the matrix becomes about 1μm. Considering that the initial grain size of the as-cast alloy was around 500 μm, it is understood that remarkable grain-refinement occurred during the warm extrusion.

Figure 4(a) shows a bright field image of fine grains, and the enlargement of the area indicated by white rectangle is shown in Fig. 4(b). It is to be noted in these figures that lamellar bands are seen along the basal plane. However, characteristic extra spots due to LPSO phase was not confirmed in the diffraction patterns obtained from this area.
In order to examine the details of the lamellar bands, high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) was conducted.

Figure 5 shows a HAADF-STEM image obtained from the matrix including grain boundaries. Significant Z-contrasts appear not only as horizontal white bands but also along the curved grain boundary lying in the left of the figure. The white bands seem to be extending from the grain boundary. The bright Z-contrasts indicate the enrichment of heavy atoms, Zn or Y in the present study. Considering that such solute atoms have a tendency to segregate into stacking faults, the horizontal white bands indicate stacking faults enriched by Zn or Y atoms. In addition, it is to be noted that such solute segregation occurs also on the grain boundary, which must inhibit the migration of grain boundaries to stabilize the fine-grained structure formed during the warm extrusion. In order to make more detailed analysis on the distribution of Zn and Y in the matrix, energy dispersive X-ray spectroscopy (EDS) was employed.

Figure 6(a) shows a bright-field image obtained from the matrix including a grain boundary and stacking faults. In this figure, a grain boundary is lying from upper-left to bottom-right, and the bottom left side of the grain boundary is seen as a dark area because of the difference of diffraction condition of the two grains. Inside the grain observed to be a bright area, there appear fine lamellar bands almost perpendicular to the grain boundary. Figs. 6(b) and (c) show EDS elemental distribution images of Zn and Y, respectively. Comparing the both EDS images of 6(b) and (c) with the bright field image 6(a), the areas enriched by Zn and Y atoms appear almost the
same areas of the fine lamellar bands in Fig. 6(a). The lamellar bands are considered to be stacking faults enriched by Zn and Y. Besides, it is to be noted that the areas around the grain boundary have high intensity due to Zn atoms. These results show that Zn and Y are segregated not only to stacking faults but also to grain boundaries, which is consistent with the result obtained by HAADF-STEM in Fig. 5 although Fig. 6(b) shows that Zn atoms tend to be preferentially distributed at grain boundaries.

Further characteristic microstructure to be noted was observed in the matrix. Figure 7 shows a HAADF-STEM image obtained from a fine grain in the matrix. Abundant stacking faults enriched by Zn and Y are seen as bright bands. In addition to the stacking faults, solute atom segregation is also observed along the white arrows. They are considered to be so-called kink bands where lattice bending of basal plane abruptly occurs. Although the bending angle at kink bands is less than 10 degrees, they can act as small angle tilt boundaries, so that kink band formation might contribute to grain subdivision during the warm extrusion.11)

4. Summary

Microstructures in Mg\textsubscript{97}Zn\textsubscript{1}Y\textsubscript{2} alloy extruded at 623 K were observed by SEM and TEM. The extruded alloy consists of the dispersed block of LPSO phase and matrix. OIM obtained by SEM-EBSP and dark-field mode of TEM revealed that the matrix includes fine grains with the grain size around 1–3 µm and high angle grain boundaries, suggesting that dynamic recrystallization during extrusion must contribute to the formation of fine-grained structure in the matrix. HAADF-STEM images and EDS elemental distribution images exhibited that Zn or Y were enriched at grain boundaries and stacking faults inside the grains in the matrix, which would increase the thermal stability of the fine-grained structure.

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REFERENCES