Grain refinement of magnesium alloy sheets by ARB using high-speed rolling mill

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Abstract. Applications of ARB to magnesium alloys were limited due to low deformability. The authors recently found that the rollability of the alloys is significantly improved in high-speed rolling. It is supposed that the severe plastic deformation of magnesium alloy sheets is feasible if rolling in ARB processes is conducted at high speed. In this study, AZ31B and ZK60A sheets are processed by ARB up to five cycles at 423K with a speed of 1000m/min. Vickers hardness increases with increasing number of ARB cycles, while the tensile strength shows the maximum after the second cycle. The grain size is reduced significantly at the first cycle and decreases gradually from the second cycle. The mean grain sizes after five cycles are 1.6µm for AZ31B and 1.8µm for ZK60A. It is concluded that ARB using high-speed rolling is effective for grain refinement of magnesium alloys.

1. Introduction

Magnesium alloys are lightweight structural materials, however wrought products are not used so widely as cast products. Although strengthening of magnesium alloys is demanded, cold/warm working processes have not been applied widely because of the low ductility. This is due to the lack of slip systems below 500K. Rolled sheets are used for laptop PCs, automobiles, etc. The sheets are mostly manufactured by multi-pass hot rolling in industries. Applicable reduction in thickness in cold/warm rolling is limited by edge-cracking and fracture.

Accumulative roll bonding (ARB) is the severe plastic deformation process applicable to sheets [1]. Ultra-grain refinement by ARB process was reported for most metals and alloys. However, applications of ARB to magnesium alloys are limited due to the low deformability. Some researchers conducted hot (>500K) ARB where deformability is sufficient but strain hardening is negligible [2-5]. The authors applied cold ARB to Mg-Li alloy sheet which shows higher deformability due to dual-phase (hcp + bcc) microstructure [6]. Formation of ultrafine grains by cold ARB was reported. However, effects of normal ARB (ARB below recrystallization temperature) to practical magnesium alloys have not been investigated.

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The authors recently found that high-speed rolling is very effective to improve the deformability of magnesium alloys in rolling [7-9]. For instance, AZ31 sheets can be reduced 60% without fracture in one-pass cold rolling at 2000m/min. Major reason for the improvement is the temperature rise during the rolling. In the high-speed heavy rolling, large plastic work and frictional work convert to heat while the heat is less transferred to cold rolls due to short contacting time. Accordingly, the sheet temperature increases so that non-basal slip or dynamic recrystallization takes place [9,10].

It is supposed that the SPD of magnesium alloy sheets is feasible if the rolling operation in ARB process is conducted at high speed. In this study, AZ31B and ZK60A sheets are processed by ARB at 423K with a speed of 1000m/min. AZ31 is the most popular magnesium alloy, while ZK60 is a high-strength wrought alloy.

2. Experiment
Prior to ARB, AZ31B (Mg-3%Al-1%Zn-0.4%Mn) sheets were annealed after rolling and ZK60A (Mg-5.4%Mn-0.56%Zr) sheets were solution-treated after extrusion. The both sheets were 2.5mm thick, 30mm wide and 300mm long. A high-speed rolling mill of which roll diameter was 530mm was used. One sheet was held for 900s in an electric furnace at 423K. The thickness of the sheet was reduced 50% by one pass rolling operation at 1000m/min under unlubricated conditions. Bonding of two sheets was not performed at the first cycle. After passing the roll gap, the sheet was immediately quenched into water shower. At the second cycle, the 1.25mm thick sheet was cut into two halves in length. The sheet surfaces were degreased but not scratch brushed. The sheets were stacked and processed under the same ARB conditions as shown in Figure 1. ARB was repeated up to five cycles (equivalent strain=4.0) for both AZ31B and ZK60A sheets.

![Figure 1. Illustration of Accumulative Roll-Bonding (ARB) Process conducted in this study.](image)

The microstructures of the initial materials and the ARB processed specimens were observed with an optical microscope. The OM observation was conducted on the plane perpendicular to the transverse direction (TD plane, or longitudinal section). The mean intercept length was measured as the grain size. After chemical polishing, the pole figures on the mid plane were measured by the Schulz reflection method with Cu-Kα X-ray radiation. The intensity was normalized by that of randomly oriented magnesium sample. Mechanical properties of the sheets were measured by tensile test at room temperature on an Instron-type machine. The tensile-test specimens were machined so that the tensile axis was parallel to the rolling direction. The gauge length was 10 mm and its width was 5 mm. The crosshead speed was 0.5 mm/min, so that the initial strain rate was $8.3 \times 10^{-4}$ s$^{-1}$. 
3. Results and Discussion

After the first ARB cycle, both AZ31B and ZK60A alloy sheets showed small edge cracks. As the propagation of edge cracks is relatively slow, the width was not trimmed much after each cycle. The both alloys were subjected to five cycles without fracture. However it was not possible to take sufficient tensile specimen from the ZK60A sheets after five cycles.

Figure 2. Optical micrographs of AZ31B sheets as a function of number of ARB cycles.

Figure 3. Optical micrographs of ZK60A sheets as a function of number of ARB cycles.

Change in microstructure of AZ31B is shown in Figure 2. In the figure, ‘0-cycle’ means the initial microstructure. The initial sheet is covered with equiaxed grains of which mean grain size is 7.5 µm. The grain size is significantly reduced to 2.1 µm at the first cycle may be due to dynamic recrystallization under high Z parameter [9,10]. Grain size does not change much after the second cycle. Microstructural evolution of ZK60A is shown in Figure 3. The initial microstructure of ZK60A is not homogeneous containing equiaxed and elongated grains due to the previous extrusion process. With increasing number of ARB cycles, the fraction of fine equiaxed grains increases, the
microstructure becomes more homogeneous and the mean grain size decreases gradually. Changes in the mean grain size are compared in Figure 4. The grain size is significantly reduced at the first cycle and decreases gradually in general from the second cycle. Difference between the two alloys is not so large. Enlarged micrographs of the 5-cycle processed sheets are shown in Figure 5. The mean grain sizes are 1.6µm for AZ31B and 1.8µm for ZK60A.

Changes in (0002) pole figures are shown in Figure 6. All the ARB-processed sheets have basal texture. After the first ARB cycle, AZ31B shows the basal texture with two peaks on RD. It means that c axis is tilted 15-20 deg. back and forth to the rolling direction. The angle between the two peaks θ is shown on the figure. Basal texture with double peaks is a typical rolling texture of magnesium alloys. ZK60A accompanies two other peaks on the TD at the early stage. The two peaks are retained components of the extrusion texture (0002) ⊥ ED. With increasing number of ARB cycles, θ decreases and the basal texture turns into single-peak basal texture. This is due to the sheet-stacking in ARB process [11]. It is known that single-peak basal texture is a shear texture or surface texture component of magnesium alloys [12] so that the texture forms beneath the surfaces. As the two sheets are roll-bonded together in ARB process, the surface locates at the center of the thickness at the next cycle. Therefore, the texture on the midplane changes from the rolling texture to the shear texture with increasing number of ARB cycles.

Changes in tensile strength and elongation are shown in Figure 7. The tensile strength increases at the first cycle. The increase of ZK60A is higher than that of AZ31B. After the second cycle, the elongations of the both alloys is low and the tensile strength decreases gradually with increasing the number of ARB cycles. AZ31B and ZK60A show similar trend, though the tensile strength of ZK60A is approximately 30MPa higher than that of AZ31B. It seems that the change in tensile strength does

![Figure 4. Mean grain size as a function of number of ARB cycles.](image)

![Figure 5. Optical micrographs of the sheets after 5 ARB cycles.](image)
not agree with that in grain size. Change in Vickers hardness is shown in Figure 8. The hardness increases until the second cycle and does not change much after the second cycle. These trends correspond with that in the grain size shown in Figure 4. The hardness increases with decreasing grain size. It is notable that the impression formed by the indentation on longitudinal section is not square but diamond (rhombus) shape elongated in the thickness direction. This is due to the plastic anisotropy on longitudinal section. In order to investigate the anisotropy, two diagonal lengths of the impression were measured separately. HV\text{ND} is the hardness calculated with the diagonal length in the thickness direction only, while HV\text{RD} is the hardness with the diagonal length in the rolling direction. They are also shown on Figure 8. HV\text{RD} is always higher than HV\text{ND} due to the evolution of basal texture as expected. The flow stress of magnesium alloys is asymmetric and tensile flow stress along $a$-axis is higher than that in $c$-axis [13]. As $c$-axis is aligned in the thickness direction (ND) in the textured sheet, the impression is longer in the direction. The anisotropy generally increases with number of ARB cycles. It means that textured magnesium sheets are easier to be compressed in the thickness direction than in the rolling direction.

In most metals and alloys, tensile strength shows good correlation with Vickers hardness [14]. However, it is found that the hardness and the strength do not show similar trend with an increase in the number of ARB cycles. The fact implies that unmatured fracture occurs during tensile test of the ARB-processed sheets. A possible explanation for the fracture is the evolution of single-peak basal texture. Number of slip systems is insufficient for magnesium alloys so that more shear banding and twinning and fracture is expected at room temperature, in particular in well-textured sheets.

4. Conclusion
AZ31B and ZK60A sheets are processed up to five ARB cycles (strain of 4.0) at 423K by ARB with a speed of 1000m/min. Vickers hardness increases with the number of ARB cycles, while the tensile strength shows the maximum after the second cycle. The grain size is reduced significantly at the first cycle and decreases gradually from the second cycle. The mean grain sizes after five cycles are 1.6µm
for AZ31B and 1.8 µm for ZK60A. It is concluded that ARB process is effective for grain refinement and hardening of magnesium alloys.

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Figure 7. Tensile properties as a function of number of ARB cycles.

Figure 8. Vickers hardness as a function of number of ARB cycles (dotted lines show Vickers hardness calculated from one diagonal length of the impression).