Improvement of Mechanical Properties of Magnesium Alloy ZK60 by Asymmetric Reduction Rolling

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Abstract The improvement of mechanical properties of ZK60 processed by asymmetric reduction rolling (ARR) was investigated in this paper. The grain refinement and basal texture intensity decrease were attributed to the introduction of shear stress produced by ARR process. Compared to conventional symmetrical rolled (SR) ZK60 alloys, ARRed ZK60 exhibited finer, more homogeneous grains and higher mechanical properties. The intensity of basal texture of ARRed ZK60 after annealing was lower than that of SRed ZK60 after annealing. ZK60 sheet with good combination of strength and ductility could be obtained by ARR process. The yield strength (YS) and ultimate tensile strength (UTS) of the ARRed ZK60 sheet were increased 150% and 91.3%, compared to those of SRed ZK60 sheet, from 80 to 200 MPa and from 140 to 264 MPa, respectively. Simultaneously, the elongation to failure increased by 68.75% in the ARR sheet (27%) when compared to that of the SR sheet (16%).

KEY WORDS: ZK60; ARR; Microstructure; Texture; Mechanical properties

1 Introduction

Magnesium alloy is an attractive lightweight structural metal due to high specific strength and specific stiffness, good damping capacity, excellent machinability and good

castability [1–5]. However, their wide applications are restricted due to their limited strength, ductility and formability. The weak ductility and formability of magnesium alloys at room temperature and intermediate are related to their hexagonal close-packed (hcp) crystal structure, which have limited operative slip systems [6–8]. Thus, improving the ductility and formability is one of the major subjects in magnesium alloys research. ZK60 alloy is one of the most typical high strength wrought magnesium alloys. The ZK60 alloy treated by optimum T6 treatment, i.e., solution treatment at 375 °C for 3 h and subsequent artificial aging treatment at 175 °C for 10 h, had good combination of high strength and good ductility [9]. The conventional thermal–mechanical processes such as rolling and extrusion were effective to improve mechanical properties with the sacrificing ductility of ZK60 [10, 11]. In order to obtain the high mechanical properties and good ductility simultaneously, some novel thermal–mechanical processes were proposed, such as combination of repeated upsetting and forward extrusion [12], integrated extrusion and equal channel angular pressing [13] and combination

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of twinning deformation and aging treatment [14]. Even though the novel thermal–mechanical processes above were effective to enhance the strength and ductility of ZK60, the cost was too high and the productivity was quite low compared to the conventional rolling and extrusion. Thus, how to get the high strength and good ductility with low cost and high productivity would be a great challenge to researcher. The ARR process with a minor change of the conventional rolling machine had a good effect on improvement of mechanical properties and ductility for AZ31 alloy [15]. Due to the absence of any detailed observation on ZK60 processed by ARR, the improvement of mechanical properties of ZK60 was investigated in this study. It is expected that ARR exhibits good effect on mechanical properties and ductility improvement of ZK60.

2 ARR Process and Stress Analysis

The schematic of the ARR process is shown in Fig. 1. The ARR rolling machine was designed on the basis of SR rolling machine. Therefore, the ARR and SR processes have the same roll diameters and rotation speed in this study. The support plate in front of the bite entrance becomes a unique difference between ARR and SR rolling machine. The introduction of support plate is expected to make the upper and lower part of sheet deform asymmetrically. The aim of adding the support plate to SR machine is to introduce the shear stress during rolling. In order to confirm the introduction of shear stress, the ARR process was stimulated by commercial finite element (FE) software: DEFORM-2D. In the FE stimulation, the geometric models have the identical dimensions and rotation speed of

SR machine, i.e., rotation speed, diameter of rolls and roll barrel are 42 rpm/min, 200 and 350 mm, respectively. The other necessary parameters for FE stimulation are shown in Table 1. The details of FE stimulation of ARR were elaborated in Ref. [15].

The different reductions (25, 50 and 65%) were selected for FE stimulation to investigate the deformation behaviors of the rolled ZK60 sheet. The distributions of the effective stress of ARRed and SRRed ZK60 sheets are shown in Fig. 2. In SR process, the distributions of the effective stress are always symmetrical with respect to the middle layer of the sheet as shown in Fig. 2d–f, while in ARR process, distributions of the effective stress are asymmetric, and the extent of asymmetry increases with the increase in reduction. It can be noted, there is only one stress concentration band, just like a shear band, emerged around the rolling entrance. The asymmetry of effective stress becomes more significant with the increase in rolling reduction. This result is consistent with the effective strain distribution of the sheet at a 25% reduction as shown in Fig. 3.

3 Experimental Procedures

The starting material sheet with the thickness of 10 mm was supported by Shanxi United Magnesium Company with nominal chemical composition Mg-5.5Zn-0.5Zr (wt%) and Mg balanced (ZK60). The as-received ZK60 sheet was the as-cast alloy with minor forging for making good sheet contour. The as-received ZK60 sheet was homogenized at 400 °C for 20 h. The homogenized ZK60 sheet was heated to 450 °C for 10 min prior to rolling. The

Fig. 1 Schematic of the ARR process **a** initial stage; **b** stable rolling stage

ZK60 sheets were subjected to total two passes (with 25% reduction for each pass) SR and ARR. The annealing between two rolling passes was performed at 450 °C for

5 min. The thickness of ZK60 sheet was reduced from 10 to 5.6 mm, and the total reduction was 44%.

All optical microstructures (OM) were observed at room temperature in the transverse direction (TD) of rolled ZK60 sheets. The grain sizes were determined by using the line-intercept method. Electron backscattered diffraction (EBSD) measurements were taken at the midplane in the thickness of the rolled sheets, and samples preparation consisted of mechanical grinding, mechanical polishing, electropolishing and cleaning [16]. The EBSD measurements were taken on a Zeiss Supra 55 FEG-SEM at 20 kV, with a working distance of 20 mm, a tilt angle of 70° and a scanning step of 0.4 μm . The specimens for tensile tests had a gauge length of 15 mm, width and thickness both of 5.6 mm, respectively. And tensile tests were carried out parallel to the rolling direction at room temperature with an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$.

Table 1 Parameters of ARR FE simulation

Parameters	Unit	Value
Friction coefficient		
Roll sheet, μ_1		0.3
Support plate sheet, μ_2		0.1
Thermal properties		
Thermal conductivity	$\text{N}(\text{s } ^\circ\text{C})^{-1}$	159
Thermal expansion		
Heat capacity	$\text{N}(\text{MM}^2 \text{ } ^\circ\text{C})^{-1}$	1.7675
Interface heat transfer coefficient	$\text{N}(\text{sMM}^2 \text{ } ^\circ\text{C})^{-1}$	4.5
Thermal expansion		2.2×10^{-5}
Convection coefficient	$\text{N}(\text{sMM} \text{ } ^\circ\text{C})^{-1}$	0.2

Fig. 2 Effective stress distributions of the rolling sheets with different reductions obtained from FE simulation: **a** 25%, **b** 50%, **c** 65% of the ARR sheet; **d** 25%, **e** 50%, **f** 65% of the SR sheet

Fig. 3 Effective strain distributions of the rolling sheets with 25% reductions obtained from FE simulation: **a** the ARR sheet; **b** the SR sheet

4 Results and Discussion

4.1 Microstructure Evolution

The microstructure of the as-received and homogenized ZK60 sheet at 400 °C for 20 h is shown in Fig. 4. It is composed of coarse grains (with average grain size of $\sim 300\ \mu\text{m}$) and large number of twins within nearly all the coarse grains, as Fig. 4a demonstrated. Twinning was considered as the main deformation mode in the process of minor forging for the as-cast ZK60 with very coarse grains. The characteristics of the as-homogenized ZK60 were coarse grains and fine static recrystallization (SRX) grains indicated by arrows A, B and C. The twins in the as-received ZK60 were substituted by DRXed grains. The coarse grains in Fig. 4a became smaller after homogenization as shown in Fig. 4b, which indicated that the coarse grains in the as-received ZK60 were divided by twins or partly be swallowed by DRXed grain growth.

The microstructure of the as-homogenized ZK60 sheet processed by SR is shown in Fig. 5. The microstructural characteristics of the ZK60 alloy sheet processed by the first-pass SR were the formation of twins, intersection of twins, elongation of the grains and dynamic recrystallization (DRX) grains, which were indicated by arrows A, B, C and red ellipses as shown in Fig. 5a. The twins within

coarse grains disappeared and substituted by fine DRXed grains after annealing which were indicated by arrows D and E in Fig. 5b. The amount of coarse grains and twins in ZK60 alloy processed by the second-pass SR decreased dramatically compared to that processed by the first-pass SR. The microstructure of ZK60 alloy processed by the second-pass SR was inhomogeneous after annealing. The grain size of DRXed grains in annealed ZK60 after two passes SR was in the range of 5–15 μm . However, the coarse grains had the grains size of $\sim 100\ \mu\text{m}$. Those coarse grains inherited from the as-received ZK60 because their size was big and closed to the one of the as-received ZK60. The percentage of DRXed grains was $\sim 70\%$ in two passes SRed ZK60 in the condition of annealing. The other 30% grains are deformed grains inherited from the as-received ZK60.

The microstructures of the as-homogenized ZK60 sheet processed by ARR are shown in Fig. 6. It is obvious that the major deformation mode of ZK60 during the first-pass ARR is twinning, and the number of twins seems more than that of the first-pass SRed ZK60. It is strange that many black areas indicated by red ellipses could not be observed by OM. In order to check the details of the black areas, scanning electron microscope (SEM) was employed. The high-magnification image of black area is shown in Fig. 7. These areas were characterized as fine grains (not fully

Fig. 4 Optical micrographs of ZK60 **a** the as-received ZK60; **b** the as-homogenized ZK60

Fig. 5 Optical microstructures of ZK60 subjected to repeated SR and annealing at 450 °C: **a** the first SRed; **b** the first SRed and annealed; **c** the second SRed; **d** the second SRed and annealed

Fig. 6 Optical microstructures of ZK60 subjected to repeated ARR and annealing at 450 °C: **a** the first ARRed; **b** the first AARed and annealed; **c** the second ARRed; **d** the second ARRed and annealed

Fig. 7 Details of black area in the first-pass ARRed ZK60 sheets

DRXed grains with grain size of 2–10 μm). Fine grains and limited coarse grains were obtained after annealing as shown in Fig. 6b. The disappearance of coarse grains in ZK60 sheet processed by the first ARR after annealing was attributed to the consumption of fine and not fully DRXed grains growth in black areas and twins. After two passes ARR and annealing, the ZK60 sheet exhibited much finer and homogeneous grains compared to those processed by SR. The average grains size (AGS) of ZK60 after two passes ARR and annealing was $\sim 15 \mu\text{m}$. The percentage of DRXed grains was $\sim 85\%$ in two passes ARRed ZK60 in the condition of annealing. The other 15% grains are deformed grains inherited from the as-received ZK60.

The grains with basal pole parallel to ND were very stable during SR process compared to grains with other orientations, because those grains are not favorable for dislocation basal slip. Thus, the coarse grains in SRed ZK60 were considered as basal grains [17, 18]. The compression stress could employ on the rolling sheet during the process of SR and ARR. The shear stress would be supported when support plate was introduced to SR process according to the result of FE simulation [15]. Due to the combined action of compression and shear stress, the basal grains with hard orientation which were not favorable for dislocation slip in the process of SR would become the site where dislocation slips easily in the process of ARR. The density of dislocation in ARRed ZK60 was higher than those of SRed ZK60, so finer DRXed grains could be observed in ARRed ZK60 after annealing, which is proved in Fig. 8b, d. Due to the introduction of shear stress in the process of ARR, the stability of basal grains decreases and the coarse basal grains which should remain in SR process were consumed in ARR process. Thus, the microstructure of ARRed ZK60 after annealing was more homogeneous than that of SRed ZK60 after annealing.

Fig. 8 {0002} pole figure of annealed ZK60 sheets process by the second pass of **a** SR, **b** ARR

4.2 Texture Analysis

Figure 8 shows the {0002} pole figures of 44% total rolled samples in annealed condition. The major texture components of SRed ZK60 could be expressed as ND//{0001}, and the maximum intensity of basal texture is about 9.6, which is shown in Fig. 8a. The ARRed ZK60 exhibits basal textures with 10° – 15° tilting away from ND as shown in Fig. 8b. The intensity of basal texture of ARRed ZK60 was weaker than that of SRed ZK60.

4.3 Mechanical Properties

The nominal stress–strain curves of ZK60 after two passes SR and ARR in annealed condition are shown in Fig. 9. The ARRed ZK60 sheet in annealed condition exhibits higher mechanical properties than that of SRed AZ91 sheet in annealed condition. The yield strength (YS) and ultimate tensile strength (UTS) of the ARRed ZK60 sheet were increased 120 and 91.5%, from 100 to 220 MPa and from 190 to 364 MPa, respectively. Simultaneously, the elongation to failure increased by 68.75% in the ARR sheet (27%) when compared to that of the SR sheet (16%).

Fig. 9 Stress–strain relations of ZK60 sheets processed by SR and ARR in annealed condition

Table 2 Comparisons of the room temperature properties of ZK60 alloy using different processings

No.	YTS (MPa)	UTS (MPa)	Elongation (%)	Strain rate (s ⁻¹)	Processing method	References
1	250	350	6	2×10^{-3}	Twin roll cast + rolling at 350 °C	[19]
2	199	306	24	6×10^{-4}	Hot rolling at 400 °C	[20]
3	278	342	18	Not support	Twin roll cast + rolling at 300 °C	[9]
4	200	360	25	5×10^{-4}	12P ECAP at 300 °C	[21]
5	396	440	9	5×10^{-4}	12p ECAP at 300 °C + cold rolling	[21]
6	150	310	30	3.3×10^{-3}	4P ECAP at 250 °C	[22]
7	215	289	37	1×10^{-3}	4P CEC at 230 °C	[23]
8	225	285	25	1×10^{-3}	5P RU at 250 °C	[12]
9	135	285	34	1×10^{-3}	3P RU at 250 °C + sheet extrusion at 220 °C	[12]
10	310	351	17	1×10^{-3}	DE + ECAP, 2p °C at 350 °C	[13]
11	269	315	12	1×10^{-3}	DE at 380 °C	[24]
12	266	175	32	1×10^{-3}	4p ECAP Bc at 240 °C + 4p Bc at 180 °C	[24]
13	80	140	16	1×10^{-3}	SR at 450 °C	Present work
14	200	264	27	1×10^{-3}	ARR at 450 °C	Present work

The comparisons of room properties of ZK60 using different processings are listed in Table 2. It is clearly shown that the YTS and UTS of ARRed ZK60 were comparable to those of ZK60 processed by other processing methods. The elongation of ARRed ZK60 was higher than most of elongation of ZK60 processed by other processing methods. The characteristics of processing methods listed in Table 2 were complex, high cost, low productivity and limitation of product size, such as ECAP, CEC and RU. Overall considered the productivity, cost and product size, the ARR process had potential of getting good combination of strength and ductility and application in industrial production.

Grains size and texture are the two key factors influencing the mechanical properties. According to Hall–Petch relation [25], it is easy to understand that the YS of ARRed ZK60 is higher than that of SRed ZK60.

The basal dislocations in the grains with soft orientation could be activated more easily compared to the basal dislocations in hard orientation at room temperature [26]. The Mg alloys with high intensity of basal texture means the ratio of the grains with basal texture was higher than that of Mg alloys with lower intensity of basal texture. From the intensity of basal texture point of view, the SRed ZK60 could have better mechanical properties than ARRed ZK60. However, the tensile test exhibits opposite results.

In a word, the fine grains and low intensity of basal texture are two competitive mechanisms affecting the mechanical properties. According to the results of the mechanical properties, the grain size is more effective to enhance the mechanical properties than the intensity of basal texture in this study.

Furthermore, the increase in elongation of ARRed ZK60 sheets could attribute to weak intensity of basal texture, homogeneous of the fine grains. Dislocation in Mg alloy with weak intensity of basal texture could slip easily at room temperature. Fine grains mean that the proportion of the grain boundary is big and the deformation energy absorption is sufficient. Moreover, homogeneous grains could make deformation uniform and avoid the appearance of excessive concentration of local stress.

5 Conclusions

Based on the results obtained and observations made in the present investigation, the conclusions can be summarized as follows:

- (1) The ARR process had higher efficiency to refine grains than the SR process. The ARRed ZK60 in annealed condition exhibited finer and more homogeneous grains compared to the SRed ZK60 in annealed condition.
- (2) The major texture components of SRed ZK60 could be expressed as ND//{0001}, and the maximum intensity of basal texture is about 9.6. The ARR process could decrease the intensity of basal texture and make the basal textures tilt 10°–15° away from ND due to the introduction of shear stress. The intensity of basal texture of ARRed ZK60 was weaker than that of SRed ZK60.
- (3) ARR process is an effective way to obtain good combination of high strength and ductility of ZK60 without loss of productivity and cost increase. The

yield strength (YS) and ultimate tensile strength (UTS) of the ARRed ZK60 sheet were increased 150 and 91.3% compared to the SRed ZK60 sheet, from 80 to 200 MPa and from 140 to 264 MPa, respectively. Simultaneously, the elongation to failure increased by 68.75% in the ARR sheet (27%) when compared to that of the SR sheet (16%).

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