Dynamic Recrystallization and Grain Growth in a ZK60 Magnesium Alloy Sheet Produced by Isothermal Rolling

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Abstract. A ZK60 magnesium alloy was subjected to isothermal rolling (IR) at 275 and 300°C. This processing resulted in grain refinement through dynamic recrystallization (DRX) at both temperatures. The recrystallized volume fractions of 82 and 95% and average sizes of fine grains of 2.5 and 3.7 μm were achieved after IR at 275 and 300°C, respectively. It was shown that the ultrafine-grained structure produced by DRX at 300°C exhibited higher stability under following static and dynamic annealing than that produced at 275°C. This fact was attributed with the formation of a less constrained DRX structure at higher temperature of IR. As a result, the sheet produced from the ZK60 alloy at 300°C showed superior superplastic properties. Conversely, it was not feasible to enhance the superplastic properties in the ultrafine-grained alloy produced at 275°C because significant grain growth occurred during further processing of the as-rolled alloy.

Introduction

Magnesium alloy sheet products have a great potential for aircraft, automotive and electronic industries due to low density and high specific strength [1]. However, the fabrication of Mg sheets is a difficult process consisting of numerous sequential steps of cold or warm rolling passes with a small reduction and intermediate recrystallization annealings. The number of passes can attain 40, and only a low alloy Mg as an AZ31 (Mg-3%Al-0.8%Zn in wt.pct.) with moderate strength is a suitable candidate for sheet fabrication. This is why sheets from Mg alloys have found only limited application, and more than 90 pct. of Mg articles are produced by die-casting process [1,2] and hot working from high strength AZ91 (Mg-9%Al-0.8%Zn in wt.pct.) and ZK60 (Mg-6%Zn-0.65%Zr in wt.pct.) alloys, respectively.

An important current objective is therefore to apply more effective procedures for the fabrication of sheets from high alloy Mg. It is also apparent, that only superplastic forming with high strain rates will be suitable for the fabrication of complex parts from Mg alloy sheets. Since the use of this technology for the manufacturing of high-volume automotive components strongly depends on the production forming time, which has to be ~1 min for each part, so the superplastic strain rate should be $\sim 10^{-2}$ s^{-1} [3].

Recent results have demonstrated that Mg alloys processed by rolling could be deformed superplastically after going through dynamic recrystallization (DRX) which resulted in a fine-grained structure [4,5]. It is effective to reduce the working temperature for obtaining ultrafine-grained structure in order to improve superplasticity. Unfortunately, Mg alloys are not capable of extensive grain refinement by conventional rolling. In addition, fine-grained Mg alloys tend to significant grain coarsening due to rapid grain growth at elevated temperature. Alternatively, intense plastic straining (IPS) was suggested as a more effective process for grain refinement of Mg alloys to produce high plastic properties [6-10]. One can expect that superplastic Mg sheets may be successfully produced after pretreatment deformation process leading to extensive grain refinement through DRX in optimal working conditions. To date, however, very limited data are available for developing ultrafine-grained Mg alloy sheets; hence their microstructure under various working conditions and their superplastic properties are not well understood. The objective of this study is to demonstrate an effective IPS - isothermal rolling (IR) processing route for producing ultrafine-grained microstructure and superior superplastic properties in sheets of the ZK60 magnesium alloy.

Material and Experimental Procedure

A commercial ZK60 alloy with a chemical composition of Mg-5.8%Zn-0.65%Zr (in weight %) was manufactured by direct chill casting and, then, homogenized at 450°C for 24 hours. Next, the ZK60 alloy was extruded with total strain of about 0.8 at temperatures ranging from 390 to 330°C and finally was cut into cylinders with 90 mm in diameter and 180 mm in length. These cylinders were compressed at 350°C with a strain of 1.4. Blocks with 8.4 mm thickness and 45 mm width were machined from the compressed billet. These blocks were heated at either 275°C or 300°C for 1.8×10^{3} s and then rolled with reductions ranging from 20 to 50% per pass up to the final thickness of ~2.5 mm. The total reduction was ~70%. The rolling direction (RD) was parallel to the prior compression axis. A 6-high-mill with isothermal internal rollers 65 mm in diameter and 250 mm in length was used. The internal rollers were heated to 275 or 300°C, respectively.

Tensile samples with a 6 mm gauge length and 3 mm gauge width were machined directly from these sheets; the stress axis was parallel to RD. Tensile tests were performed at temperatures ranging from 225 to 300°C and strain rates ranging from 1.4×10^{-4} to 5.6×10^{-2} s^{-1}. An Instron universal testing machine (Model 1185) equipped with a three-zone split furnace was used. Temperature accuracy was within ±2°C. The values of the strain rate sensitivity ($m= \partial (\lg\sigma)/ \partial(\lg\dot{\varepsilon})$) were determined by strain-rate-jump tests [11]. Details of the techniques for structural characterization were described in a previous paper [12].

Results and Discussion

Effect of Rolling Temperature on Dynamic Recrystallization. It was shown that a high alloy Mg such as ZK60 could be very easily rolled with excellent sheet quality due to initial thermomechanical processing involving extrusion and compression. The sheets were produced only for 3-4 passes with reduction from 8.4 to 0.35-2.5 μm in thickness without cracking at 275 and 300°C. This attractive rolling becomes possible because of grain refinement attained through the pretreatment deformation process. IPS by conventional extrusion and compression being a very simple technique leads to the formation of about 75% recrystallized structure with average grain size of 4.9 μm providing enhanced workability of the ZK60 alloy under the IR. Although grain refinement is needed for successful rolling of ZK60 alloy, it is important to know the final structure of the sheets.

Fig. 1 shows the microstructures of the specimens after IR at 275 and 300°C. In the rolled specimens the volume fractions of fine grains increase to 82 and 95% and the grain sizes decrease to 2.5 and 3.7 μm at temperatures of 275 and 300°C, respectively, indicating that ultrafine-grained structures are developed through DRX during rolling. It is clearly seen that DRX leads to grain refinement; reduction in the grain size is more essential at a lower temperature of 275°C. In the all rolled specimens the ultrafine-grained microstructures appear very homogeneous due to isothermal conditions of the rolling. TEM micrographs show that many particles are formed at the boundaries and within interiors of grains. It is also apparent that there is a difference between rolled structures. Recrystallized structure formed at higher rolling temperature is less constrained. In this condition, the dislocations are less visible within the grains, grains exhibit a reasonably equiaxed grain shape and the grain boundaries are more straightened. This difference in recrystallized structure may affect microstructure and properties during subsequent processing of the rolled alloy. Therefore, the advantage of smaller grain size produced by DRX at lower rolling temperature should be further investigated with the aim of determining microstructural stability and superplastic response of the sheet ZK60 alloy.

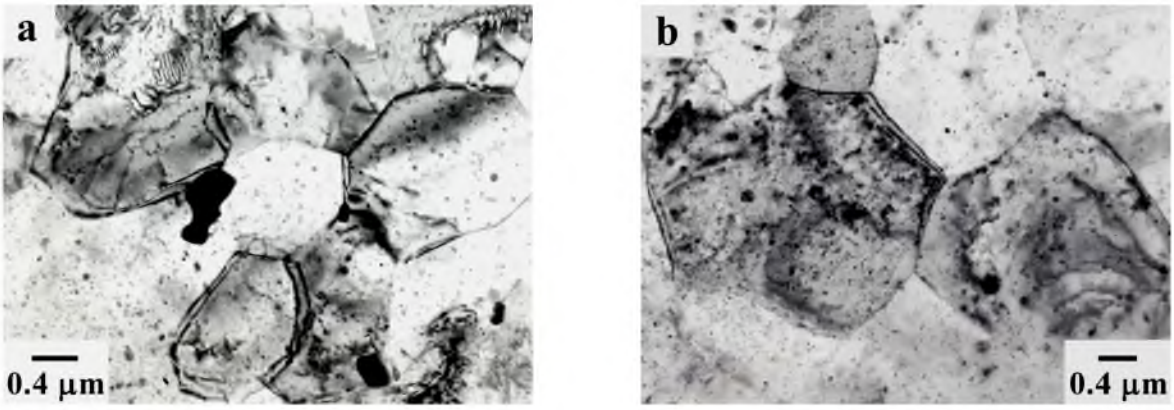

Fig. 1. TEM micrographs of samples after isothermal rolling at (a) 275 and (b) 300°C.

Tensile Testing of the Sheet ZK60 Alloy. Fig. 2 shows the typical true stress-true strain (σ-ε) curves for the rolled ZK60 alloy at an initial strain rate of 5.6×10^{-4} s^{-1} and temperatures of 250 and 275°C. Only the curves for these conditions are shown since optimum superplastic elongation was found near these temperatures and strain rate. It is apparent from Fig. 2 that the specimens rolled at 275°C exhibit much higher stresses than the specimens rolled at 300°C. Extensive strain hardening takes place initially in the specimens rolled at 275°C. After reaching a maximum, the flow stress extensively decreases until fracture. A shift of the peak stress to a higher strain and a reduction in the strain-hardening rate takes place in the specimens rolled at 300°C. The flow stress decreases with strain gradually after the peak stress; extensive softening was observed just before failure.

Tensile data are summarized in Table 1. It is seen that the material rolled at 300°C demonstrates excellent superplastic behavior at 250 and 275°C. The maximum strain rate sensitivity value of 0.54 and total elongation of 1170% were achieved at 250°C after rolling at 300°C. It is important to note that the ZK60 alloy rolled at 300°C exhibited a superplastic elongation of 417% at 275°C and 1.4×10^{-2} s^{-1} that corresponds to the attainment of high strain rate superplasticity. Decreasing the rolling temperature to 275°C results in a reduction in superplastic properties. After rolling at 275°C the strain rate sensitivity value of 0.42 and a moderate elongation of 409% were achieved at 250°C and 5.6×10^{-4} s^{-1}. It is apparent that the smaller grain size produced at lower temperature does not lead to a direct improvement of superplasticity and careful structure examination is needed for understanding the relationship between formed microstructure and the resulting material properties.

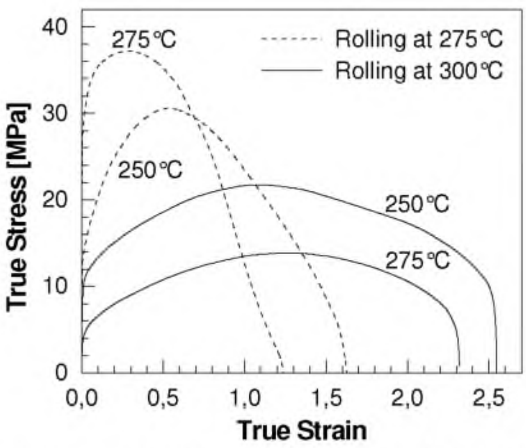

Fig. 2. True stress-true strain curves at a strain rate of 5.6×10^{-4} s^{-1}.

Table 1. The superplastic properties of the ZK60 alloy rolled at 275 and 300°C and pulled to failure at a strain rate of 5.6×10^{-4} s^{-1}.

	d [μm]	T [°C]	m	δ [%]
Rolling at 275°C	2.5	250	0.42	409
		275	0.23	244
Rolling at 300°C	3.7	250	0.54	1170
		275	0.51	918

For this purpose microstructures of the rolled specimens were carefully examined after annealing in the grip sections of specimens tensioned at 250 and 275°C. Fig. 3 depicts the results in terms of grain size distribution. The charts show that rolling at 300°C gives a significantly more stable ultrafine-grained structure than rolling at 275°C. The microstructural transformations after annealing were minimal in the specimens rolled at 300°C. Ultrafine grains were homogeneously distributed, as depicted in Fig. 4b and the average grain size was 3.7 and 3.9 μm after annealing at 250 and 275°C, respectively (Table 2). In contrast, microstructures in the specimens rolled at 275°C appear to be very unstable. Significant grain growth occurs within the specimens during annealing (Fig. 4a). The grain size distribution charts exhibit two maximum corresponding to the average grain sizes of 4 and 27.6 μm when annealing at 250°C and 5.7 and 32.6 μm when annealing at 275°C (Fig. 3, Table 2). The percentage of fine grains after annealing at 250 and 275°C was about 79 and 58%, respectively.

The microstructural evolution under dynamic conditions was examined in the gauge sections of specimens tensioned at the temperatures of 250 and 275°C at a strain rate of 5.6×10^{-4} s^{-1} (Table 2). It is apparent there is grain growth in the gauge section at 250 and 275°C. However, the fine grain size is only slightly increased in the specimens rolled at 300°C. The grain aspect ratio (AR), defined as the ratio of the grain dimension in the longitudinal direction to that in the transverse direction, increases from ~1.54 to ~1.85 with increasing temperature from 250 to 275°C. It is known [11,13] that an AR of 1.5 is attributed to the highest contribution of grain boundary sliding (GBS) to total deformation. The ultrafine-grained structure is generally retained in the specimens rolled at 275°C and tested at 250°C. However, the material does not demonstrate high elongation in contrast to that rolled at 300°C. Increasing the testing temperature results in the rapid grain growth in the specimens rolled at 275°C and this is the reason for a significant reduction in elongation in these specimens.

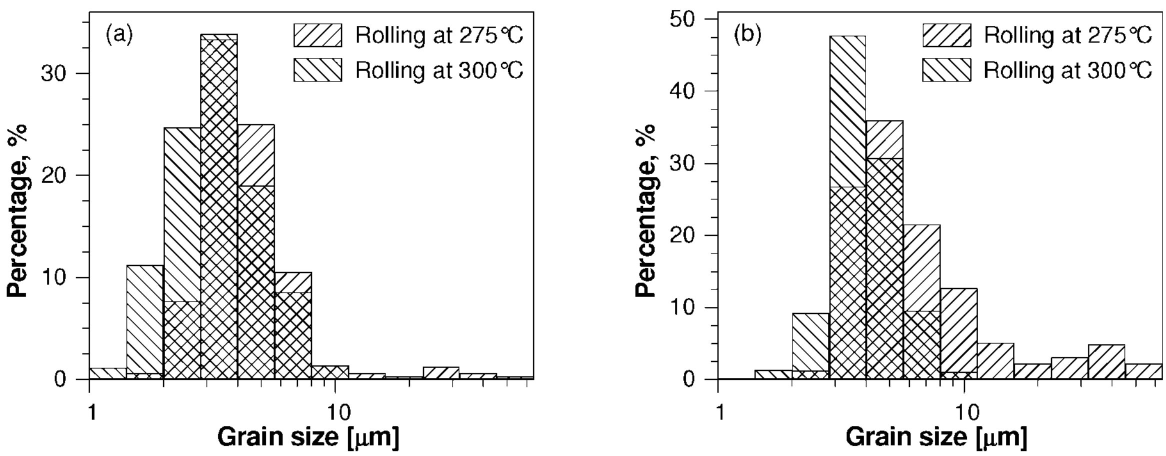

Fig. 3. Grain size distribution charts for specimens after rolling and static annealing at (a) 250°C and (b) 275°C.

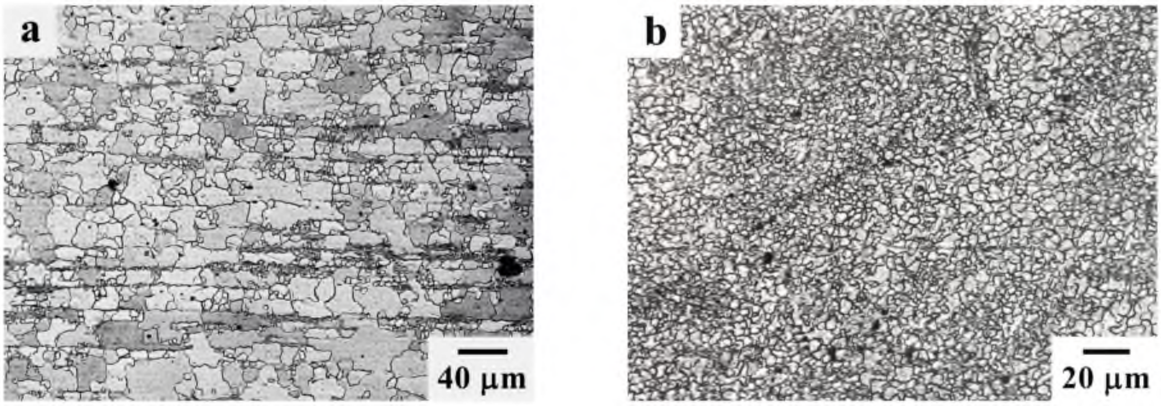

Fig. 4. Optical micrographs of samples annealed at 275°C after rolling at (a) 275 and (b) 300°C.

Table 2. Average grain sizes and volume fractions of the grains after static annealing (L_s, V_s), and superplastic deformation (L_d, V_d) and grain aspect ratio (AR) of the samples rolled at 275 and 300°C and pulled to failure at a strain rate of 5.6×10^{-4} s^{-1} and temperatures of 250 and 275°C. The elongation to failure in pct is also indicated.

	Rolling at 275°C				Rolling at 300°C	
T [°C]	250 409%		275 244%		250 1170%	275 918%
L_s [μm]	4	27.6	5.7	32.6	3.7	3.9
V_s [%]	79	21	58	42	-	-
L_d [μm]*	5.8/4.2	45.4/24	6.82/4	54/19.7	5.4/3.5	7.2/3.9
AR	1.38	1.89	1.71	2.76	1.54	1.85
V_d [%]	93	7	75	25	-	-

*Numerator and denominator are grain sizes measured in the longitudinal and transverse directions, respectively.

Rolling Process for Grain Refinement. The results show that despite the smaller grain size attained by IR at 275°C grain refinement at this lower temperature is less effective for the achievement of high superplastic properties in the sheet ZK60 alloy. It is evident that the features of ultrafine-grained structure affect the superplastic behavior of the rolled alloy. It is also apparent from a comparison of TEM micrographs that the recrystallized structure formed at 275°C is more constrained than that formed at 300°C. The enhanced dynamic recovery and DRX during IR at 300°C lead to more equilibrium and stable DRX structure and therefore provide more favorable conditions for following development of GBS. As a result, the specimens subjected to IR at 300°C demonstrate significantly higher elongations than those rolled at 275°C. In contrast, the recrystallized structure produced at 275°C is unstable at elevated temperatures. It is seen from a comparison of Figs 1a and 5 that static annealing after IR at 275°C converts the structure of the rolled specimens into the less constrained state: the grain boundaries are more regular after annealing, the dislocations are less visible within the grains, the number of fine particles is reduced within the coarsened grains. The latter is probably the reason for grain growth and a significant reduction in elongation in the specimens rolled at 275°C. It can be concluded that the highest superplastic ductility is attained in the specimens rolled at 300°C due to an optimal combination of two different factors [11,13]:

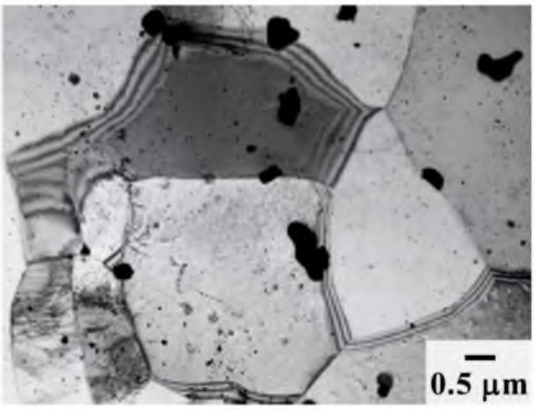

Fig. 5. TEM micrograph of a sample after rolling at 275°C and annealing at 275°C for 1.8 ks.

(1) Strain hardening (Fig. 2) associated with a more equilibrium state of the recrystallized structure.

(2) Strain rate hardening (m=0.5 (Table 1)) associated with significant structure stability under superplastic conditions (Table 2).

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