journal of materials processing technology 197 (2008) 116–121

journal homepage: www.elsevier.com/locate/jmatprotec

Effect of adding Sc and Zr on grain refinement and ductility of AZ31 magnesium alloy

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ARTICLE INFO

Article history:
Received 28 September 2006
Received in revised form
10 January 2007
Accepted 2 June 2007

Keywords:
Magnesium alloys
Scandium
Zirconium
Grain refinement
Rolling
ECAE

ABSTRACT

Grain size in the microstructure dominates the ductility of Mg–Al–Zn magnesium alloy. The strengthening and grain refinement of aluminum alloys by Sc have been extensively studied recently. As is well known, Zr refines grains in magnesium alloys. This work presents a novel approach for enhancing the grain refinement of magnesium alloy by adding Sc and Zr. Rolling and equal channel angular extrusion (ECAE) are performed to refine the grains. Experimental results indicate that adding 0.03–0.06 wt% of Sc and 0.05–0.15 wt% of Zr reduced the mean grain size of AZ31 to 2.47 μm. The ductility of AZ31–0.15Zr–0.06Sc modified alloy can be increased to 324% at 300 °C. Results of this study demonstrate that both Sc and Zr are important in controlling grain size and ductility.

1. Introduction

The hcp structure limits the industrial applications of magnesium alloys owing to low ductility at a low forming temperature. Numerous efforts in the recent decade have attempted to enhance the ductility of magnesium alloys using grain refinement technology by deformation or adding alloying elements. Some studies have demonstrated the feasibility of enhancing both ductility and mechanical properties by grain refinement (Bussiba et al., 2001; Chang et al., 2003; Jin et al., 2005; Miyahara et al., 2005). As is well known, Zr plays an important role in the refinement of grains of aluminum alloys (Lee et al., 2000; StJohn et al., 2005). Adding Sc supports the effect of Zr to reduce further the grain size in Al–Mg, Al–Sc, and 2XXX and 5XXX aluminum alloys (Yin et al., 2000; Ocenasek and Slamova, 2001; Fuller et al., 2002; Yu et al., 2004). Although previous literature provides invaluable information about the grain refinement of aluminum alloys, the individual effects of Zr and Sc on the magnesium alloys remains poorly understand. Magnesium alloy that has a uniformly fine grain structure may have good formability even at a relatively low forming temperature, as well as favorable mechanical properties at room temperature. Also, many researchers have demonstrated that ECAE effectively reduce the grain size and rearranges the basal planes of magnesium alloys (Jin et al., 2005; Miyahara et al., 2005). The processed material has the potential to exhibit high-strain-rate superplasticity.

In this work, Sc is coupled with Zr as a grain-refining agent in AZ31 magnesium alloy. Casting, hot rolling and ECAE processes of these AZ31–Zr–Sc series alloys are performed to evaluate how these minor additives affect the grain refinement and ductility.

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doi:10.1016/j.jmatprotec.2007.06.021

Table 1 – Sc and Zr contents (wt%)

Alloy no.	Zr	Sc
A	0.15	0.03
B	0.15	0.06
C	0.05	0.03
D	0.05	0.06

2. Experimental procedures

Magnesium alloys were prepared using commercial AZ31 and master alloys Al–10Zr and Al–2Sc. The Zr contents were selected as to 0.05 and 0.15%, and the Sc contents were selected as 0.03 and 0.06%. Table 1 presents the compositions of these additives. Casting was performed at 700 °C under a protective gas that contained 0.03% SF_6 with KCl and $MgCl_2$ added. Following casting, the ingots were machined to 150 mm × 100 mm × 25 mm and homogenized at 400 °C for 15 h.

The specimens were rolled using a 2-Hi roller at a rolling temperature of 400 °C. Casting ingots with a thickness of 25 mm were rolled in multi-passes with reduction ratio of 30% in each pass. The final thickness of the rolling test specimens was 3 mm. Some specimens were rolled to 12 mm to be used in ECAE tests. The rolled materials were then finished by annealing at 300 °C for 30 min.

The alloy adopted in the ECAE test was AZ31–0.15Zr–0.06Sc. AZ31 was also used to compare the effects of the alloying elements. Rolled materials were machined into ECAE specimens with dimensions of 12 mm × 12 mm × 70 mm. The ECAE test was performed using a die fabricated by tool steel and the internal angle between two channels was 90°. The extrusion speed of ECAE tests was 10 mm/s. The specimen was rotated through 90° about the longitudinal axis and reloaded into the channel from the other end between consecutive passes. The number of extrusion passes was two and the extrusion temperature was 225 °C. Following extrusion, the materials were annealed at 190 °C for 1 h.

After processing, the specimens were sectioned in the rolling direction or the ECAE direction to determine the grain size and the microstructure using optical microscopy. The linear intercept approach was adopted to determine the mean grain size. SEM was adopted to determine the morphology and the distribution of the grains following the deformation. TEM was adopted to identify further detailed characteristics of the microstructures. Tensile test of the rolled specimens was performed to measure the ductility of the grain-refined alloys. Testing temperature was set from room temperature to 300 °C and the strain rate was $10^{-3}\ s^{-1}$.

3. Results and discussion

Fig. 1 shows the microstructure of AZ31–Zr–Sc alloys, which is identical to typical as cast AZ31. The grains are extremely large, between 50 and 200 μm. The microstructures comprise mostly an Mg-rich phase, precipitates of $Al_{12}Mg_{17}$ and some randomly dispersed Mn particles that can be easily identified. Fig. 2 presents microstructures of rolled AZ31–Zr–Sc alloys and AZ31. The strong effect of mechanical deformation on grain refinement is evident. The grain size of rolled AZ31–Zr–Sc alloys was dramatically reduced to 3.0–4.5 μm (Table 2). Although rolling effectively reduced the grain size of AZ31, the grain size of rolled AZ31 was still around 20 μm, as determined by dynamic recovery and recrystallization during the hot working (Myshlyaev et al., 2002). As temperature increases and strain is present during rolling, the dislocation climbs and non-basal slips in the system increase

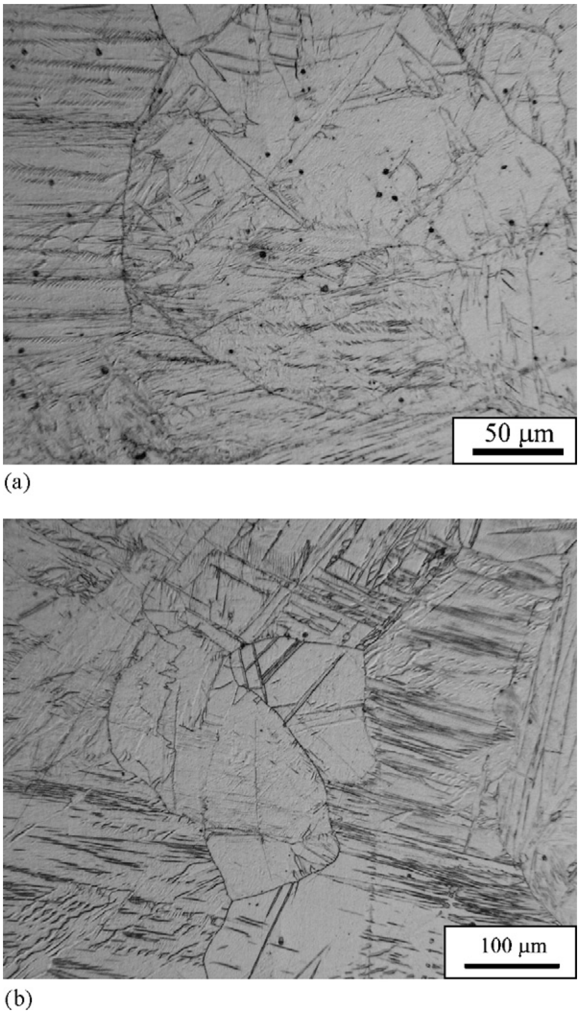

Fig. 1 – Microstructure of as-cast: (a) AZ31–0.05Zr–0.06Sc and (b) AZ31.

Table 2 – Grain size of rolled specimens (μm)

Alloy no.	Positions that measured			Average
	1	2	3	
A	3.0	3.3	3.7	3.35
B	3.6	3.9	1.7	3.74
C	4.2	4.0	4.5	4.24
D	3.2	2.8	3.0	3.00
Average grain size of rolled specimens				3.58

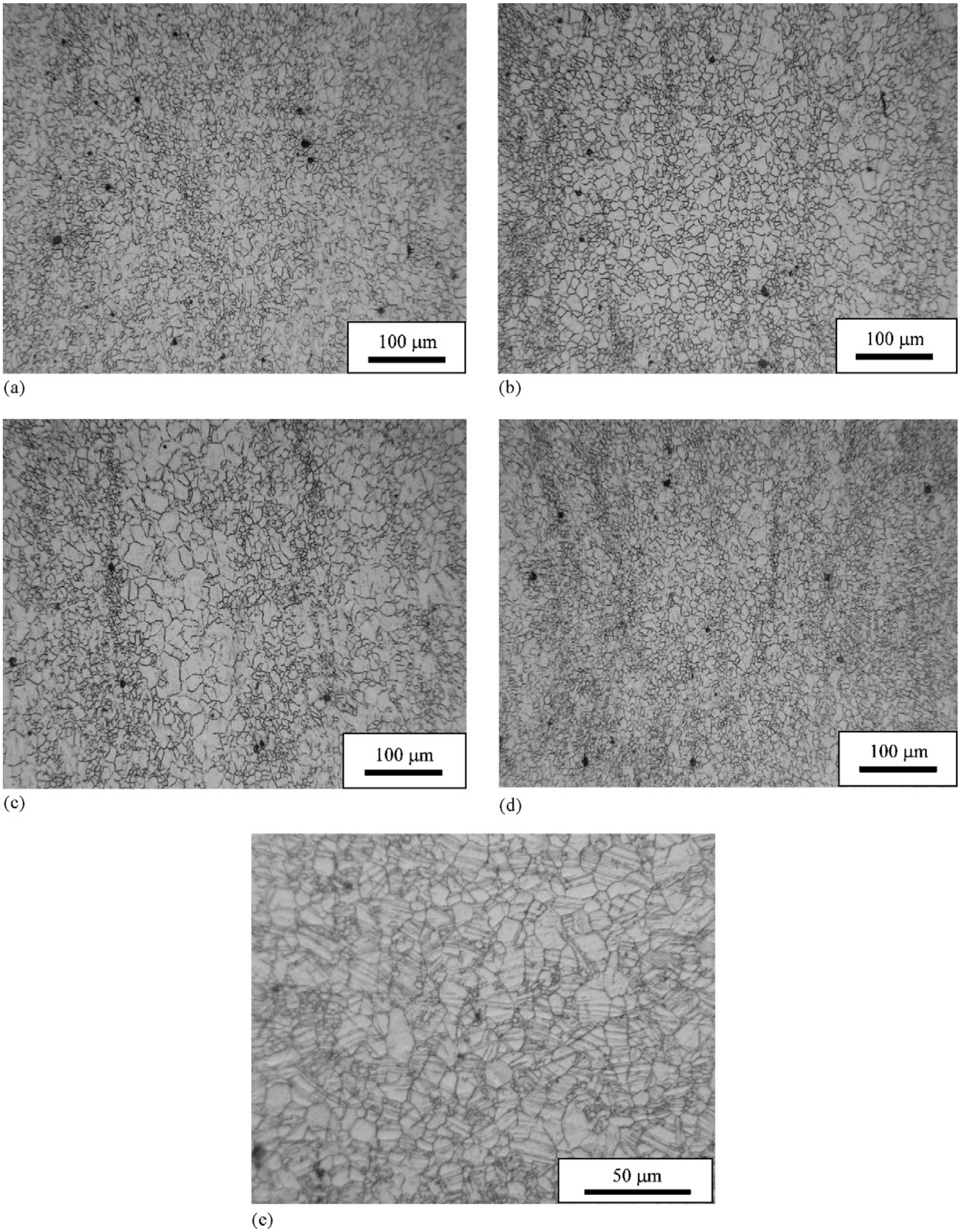

Fig. 2 – Microstructures of as-rolled: (a) AZ31–0.05Zr–0.03Sc; (b) AZ31–0.05Zr–0.06Sc; (c) AZ31–0.15Zr–0.03Sc; (d) AZ31–0.15Zr–0.06Sc; (e) AZ31.

(Fig. 3). When the degree of misorientation is sufficiently high, dynamic recrystallization nucleates in the twinning region and grains start to be refined. Many deformation twins were still present in the AZ31 grains (Fig. 2(e)). In AZ31–Zr–Sc alloys, the effect of adding Zr and Sc on grain refinement is to form nanoprecipitates to retard grain boundary migration. Zr particles (Qian et al., 2002) with a size of 1 μm are dispersed in the Mg grains (Fig. 4) and Sc reacted with Mn to form fine rod-like Mn–Sc compound (Fig. 5) (von Bush et al., 1999; Smola et al., 2002). The precipitates of Mn–Sc are very fine (only 20 nm long)

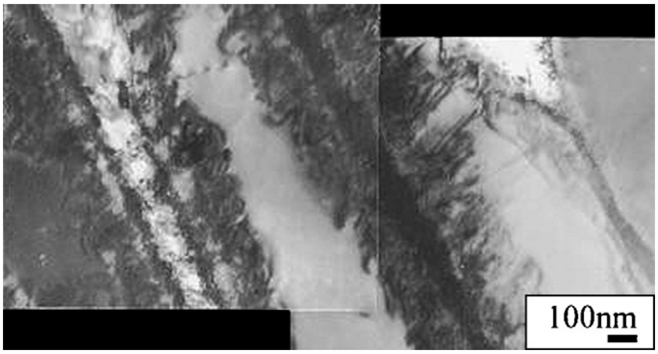

Fig. 3 – As-rolled AZ31–Zr–Sc with many dislocation pile-up at the grain boundaries.

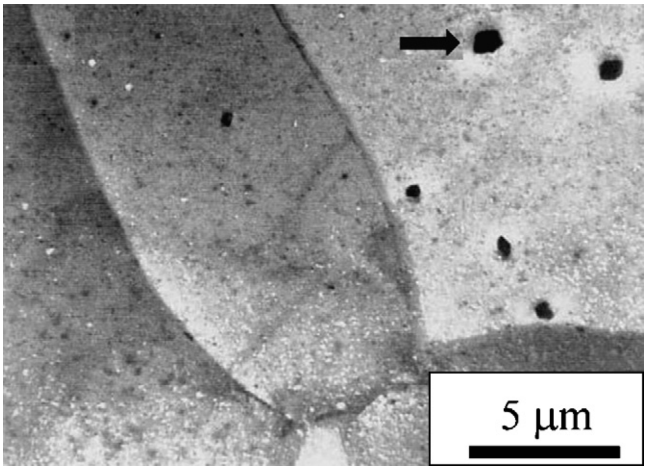

Fig. 4 – Finely dispersed Zr particles in rolled AZ31–0.05Zr–0.03Sc that had been annealed at 300 °C.

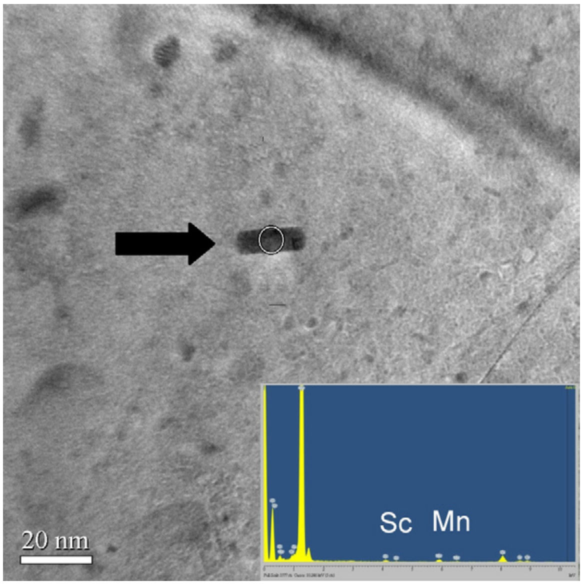

Fig. 5 – Nano-scale precipitates of Mn–Sc.

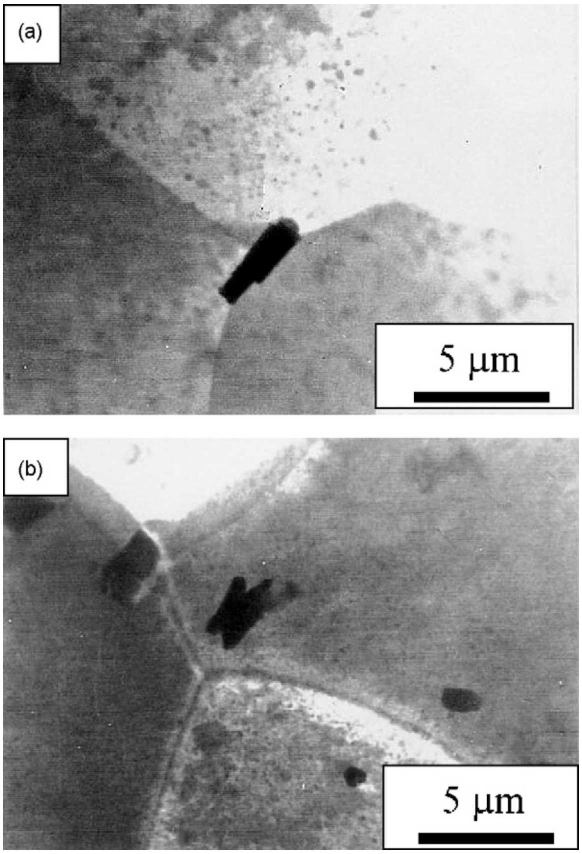

Fig. 6 – Grain boundaries of AZ31–0.05Zr–0.03Sc were pinned by Zr and fine Mn–Sc particles.

and are commonly pinched at the grain boundaries (Fig. 6). Those fine particles, large or small, may reduce or even arrest the motion of the grain boundaries. These fine particles provide more nucleation sites during the transformation from twinning to dynamic recrystallization, and these particles also restrict the growth of recrystallized grains during hot working.

Notably, some indistinct lamella structures were observed parallel to the rolling direction. Fine recrystallized grains (smaller than 1 µm) were separated by large grains (grain size of 10 µm). The spaces between these flows of smaller grains were about 100 µm (Fig. 2(c)). This finding closely resembles that of Barnett et al. (Barnett et al., 2005), who found lamella structures in rolled AZ31 structures. According to their results, when AZ31 were rolled to a give total strain, as the number of rolling passes increased, the lamella became more obvious, because the number of recrystallized grains increased. Since the alloys herein were rolled by multi-pass deformation, the lamella structures of recrystallized grains may be produced by localized stress concentration and the generation of heat by deformation.

The microstructures of AZ31–0.15Zr–0.06Sc following ECAE did not differ markedly from those of the rolled specimens. The mean grain size was slightly refined after ECAE. The only difference was that the grain size distribution of alloys after ECAE was more uniform than that of the rolled materials, and no lamella structure was found in ECAE specimen. The mean grain size was 2.47 µm for AZ31–0.15Zr–0.06Sc and 3.17 µm

Table 3 – Grain size of ECAE specimens (μm)

Alloy	Positions that measured			Average
	1	2	3	
AZ31–0.15Zr–0.06Sc	2.9	2.0	2.5	2.47
AZ31	3.0	3.3	3.2	3.17

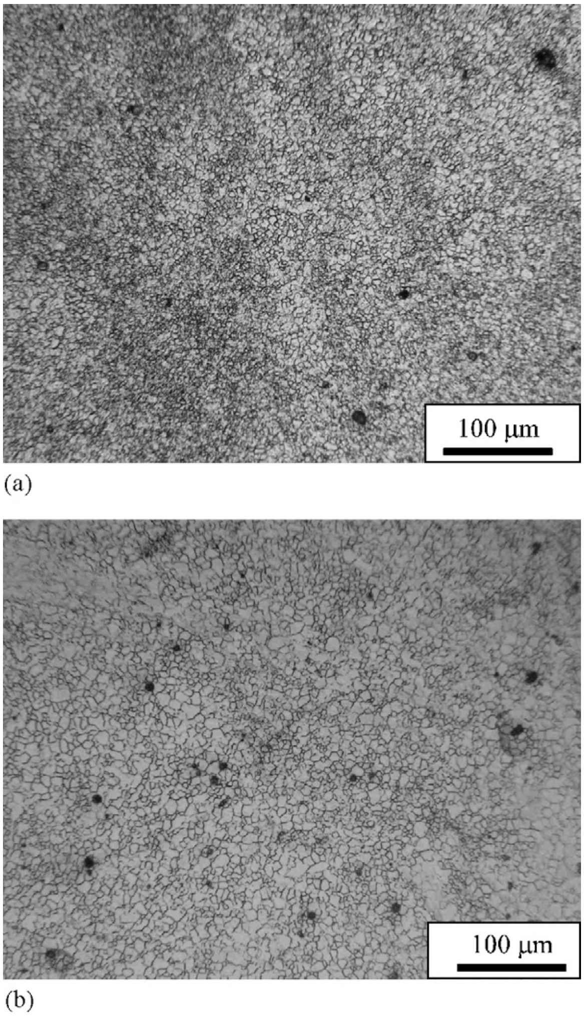

Fig. 7 – Microstructure following ECAE process: (a) AZ31–0.15Zr–0.06Sc and (b) AZ31.

for AZ31 (Table 3). The grain size of AZ31–0.15Zr–0.06Sc was slightly smaller than the grain size of AZ31 (Fig. 7), indicating that the grains may be directly refined by rolling the casting ingot with added Sc and Zr. No fine grain microstructure needed to be produced by extrusion or ECAE process.

The ductility of AZ31–0.15Zr–0.06Sc that was deformed by rolling or ECAE was measured by performing tensile tests, at temperatures of 23, 200, 250 and 300 °C. The rolled materials were annealed at 300 °C for 30 min and the materials that had undergone ECAE were then annealed at 190 °C for 1 h. Fig. 8 presents the test results. The elongation of rolled materials was increased from 8 to 226% and that of the materials that had been processed by ECAE was increased from 10 to 324% as the testing temperature increased. The ductility of materials that had undergone ECAE exceeded that of the rolled specimens. The microstructures of AZ31–0.15Zr–0.06Sc at the cross section in the tensile direction were studied by optical microscopy, as presented in Fig. 9. The deformed microstructures contained deformation twins at a low-test temperature,

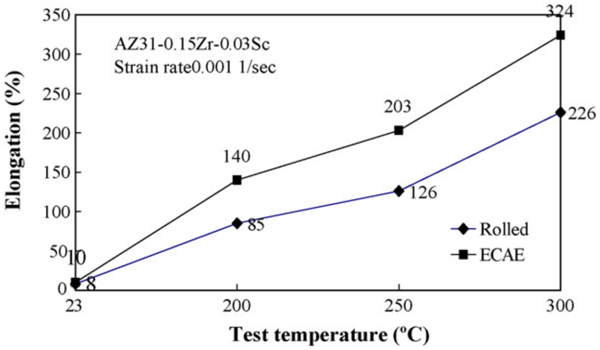

Fig. 8 – Ductility of AZ31–0.15Zr–0.03Sc alloy after rolling and ECAE.

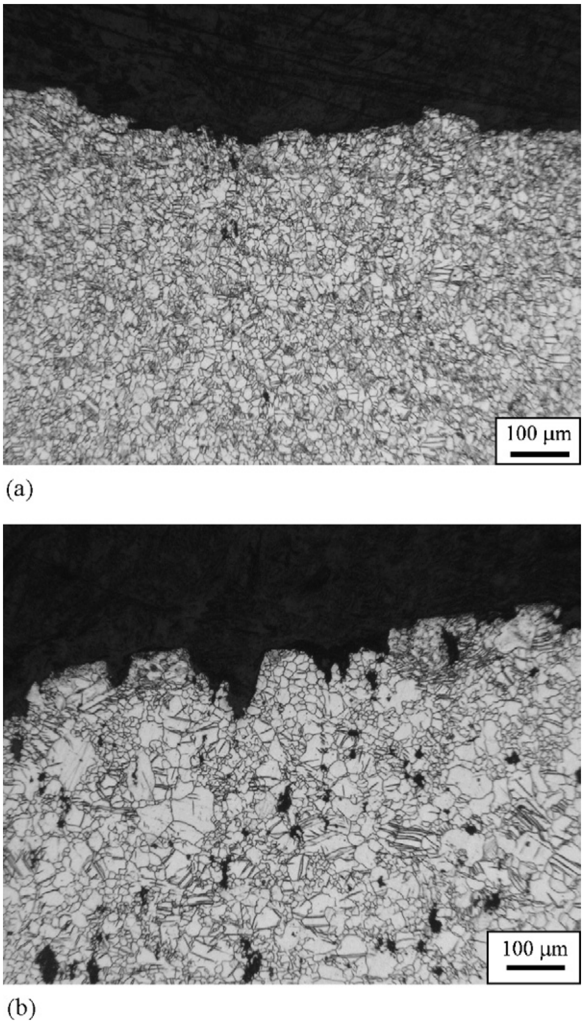

Fig. 9 – Tensile fracture sections of AZ31–0.15Zr–0.06Sc tested at: (a) 23 °C and (b) 300 °C.

and the grains remained equiaxed and uniformly distributed. The fracture surface was very smooth (Fig. 9(a)), indicating that dislocation pile-up or climb dominated the deformation. As the test temperature increased, the number of deformation twins decreased, and some large grains were present. Many intergranular cavities near the fracture surface were observed at 270 °C, and the fracture surface began to form a serrated surface (Fig. 9(b)). These cavities were all formed at the boundaries among large grains and aligned in the tensile direction. These cavities are associated with the Nabarro–Herring phenomenon, in which the self-diffusion of grains causes plastic flow from boundaries under tensile stress to boundaries under compressive stress. The deformation close to the fracture surface was caused mainly by grain boundary sliding (grain boundary diffusion). The presence of large grains indicated that the grain growth was not restricted by those particles that had been formed by Sc and Zr, and that cavities formed and the specimens began to fail. This finding demonstrated that the distribution and thermal stability of these fine particles had to be improved to increase the deformation temperature. This finding also represents the most important obstacle to be solved to producing materials with good formability. Future works will focus on the amount of additives and the processing conditions that promote dispersion and thermal stability at high deformation temperature.

4. Summary

This work proposed a novel approach for enhancing the grain refinement of magnesium alloy by adding Sc and Zr. The effects of Sc and Zr are summarized as follows.

1. Adding a slight amount of Sc and Zr effectively reduced the grain size of AZ31 after rolling and ECAE.
2. Sc and Zr enhanced the transformation of deformation twins to equiaxed grains during hot rolling.
3. The precipitate particles of Sc and Zr effectively pinned the grain boundaries during deformation at elevated temperature.
4. The refined AZ31–Zr–Sc alloys contained equiaxed fine grains and thus had a greater ductility than conventional AZ31 alloy.

Acknowledgements

The authors are grateful to C.S. Chen, PhD and Y.C. Fann for helping with experiments. We would also like to thank A.K. Li, PhD and C.C. Yang, PhD of Industrial Technology Research Institute for their helpful suggestions.

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