Materials and Design 31 (2010) 4043–4049

Contents lists available at ScienceDirect

Materials and Design

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

Effects of trace Er addition on the microstructure and mechanical properties of Mg–Zn–Zr alloy

Jing Zhang [a,b,*], Qi Ma [a], Fusheng Pan [a,b]

[a] *College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China*
[b] *National Engineering Research Center for Magnesium Alloys, Chongqing 400044, China*

ARTICLE INFO

Article history:
Received 1 March 2010
Accepted 5 May 2010
Available online 10 May 2010

Keywords:
A. Non-ferrous metals and alloys
E. Mechanical properties
F. Microstructure

ABSTRACT

The effects of trace Er addition on the microstructure in Mg–9Zn–0.6Zr alloy during casting, homogenization, pre-heating, and hot extrusion processes were examined. The mechanical properties of alloys with and without Er were compared. The results showed that Er exhibited a lower solubility in solid magnesium and formed thermally stable Er- and Zn-bearing compounds. The Er-bearing alloy exhibited a considerably improved deformability, as well as a fine and uniform microstructure. Moreover, dynamic precipitation of fine $MgZn_2$ particles with a modified spherical morphology occurred during hot extrusion, resulting in a tensile yield strength of 313 MPa and a high elongation to failure value of 22%. Further aging of the Er-bearing alloy led to an increment of another 30 MPa in yield strength. In addition, Er markedly increased the thermal stability of the alloy structure.

1. Introduction

As the lightest structural metal materials, magnesium alloys have attracted significant interest in the last decade for weight reduction in aerospace, automobile, and tool applications. However, the use of magnesium alloys is still very limited due to their inadequate strength, numerous difficulties associated with the processing technology, and high cost of either expensive compositional elements used or special processing routes involved. Therefore, the wide application of magnesium alloys depends heavily on the development of low-cost and high performance new wrought alloys. Due to the intrinsic nature of magnesium alloys, such as hexagonal close packed (hcp) lattice structure having limited number of independent slip system, no allotropic transformation along with temperature variation, the improvement of strength and ductility of magnesium therefore have to depend on the combination of various strengthening mechanism.

Strengthening via grain size control is particular effective to magnesium alloys because the Hall–Petch coefficient of magnesium alloys is notably high [1,2]. Hot deformation accompanied by dynamic recrystallization has been shown to be a promising route for refining the microstructure [3,4]. However, commercial wrought magnesium alloys suffer from high tendency of grain growth at high temperature due to lack of effective solutions to stabilize the microstructures [5,6]. This will induce significant loss of strength and ductility during secondary warm or hot deformation processing of semi-products. In addition, further strengthening via aging hardening through heat treatment such as T4 or T6 can hardly be realized [7].

Mg–Zn system alloys have more pronounced response to age hardening compared to other magnesium alloys. As widely used commercial high-strength wrought alloy, ZK60 (nominal composition Mg–6.0Zn–0.6Zr (wt.%)) alloy has been investigated in detail. The precipitation sequence from a supersaturated solid solution (SSSS) has been reported to be as follows [8,9]:

$$\begin{aligned}\text{SSSS} &\rightarrow \text{GP zones} \rightarrow \beta'_1(\text{MgZn}_2) \rightarrow \beta'_2(\text{MgZn}_2)\\ &\rightarrow \beta(\text{MgZn or Mg}_2\text{Zn}_3)\end{aligned}$$

Investigations have shown that the peak aged strengthening is associated with the rod-shaped transition β'_1 phase, forming with their growth axis parallel to the $[0\,0\,0\,1]_{Mg}$ direction. The precipitation hardening effect, however, is still far below that observed in aging hardenable aluminium alloys, due to the coarse distribution of the rod-shaped $MgZn_2$ precipitates.

Trace element addition is one of the most effective ways to enhance precipitation hardening effects [10,11]. Among them, rare earth elements (RE) have been received particular attention. Rare earth (RE) additions, such as Y, Nd, Yb, Gd, Ce, were known to produce second-phase particles combining with Zn and matrix in Mg [12–18]. These phases normally have higher thermal stability and cannot be dissolved during conventional thermo-mechanical treatments. It is reported that these particles can be utilised to affect

---

* Corresponding author at: College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China. Tel.: +86 23 65111167; fax: +86 23 65102821.
E-mail address: jingzhang@cqu.edu.cn (J. Zhang).

0261-3069/$ - see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.matdes.2010.05.008

recrystallization and grain size [7]. Dispersion strengthening effects by these RE and Zn-bearing intermetallics were also reported [19,20]. However, consumption of Zn atoms by forming these Zn-bearing particles can cause decrease of the solid solubility of Zn in Mg matrix, resulting in reduction of the solution and age hardening strengthening effects brought by zinc [17].

In this study, a higher Zn element content, compared with that in ZK60 magnesium alloys, was used in order to compensate for the solute loses in Mg matrix. A rare earth element Er, which has been received less attention than other RE, was added as a trace additive. Er has a large solubility in Mg. According to Mg–Er phase diagram [21], the maximum solubility of Er in Mg at the eutectic temperature (570 °C) is as high as 6.9 at.%. The microstructure evolution, including the formation and transformation of precipitates in Mg–9Zn–Zr alloys with and without trace element of Er during casting, homogenization, pre-heating, and hot extrusion were compared and correlated with the mechanical properties. The strengthening mechanism is also discussed.

2. Experimental procedures

Commercial high-purity Mg (>99.9%) and Zn (>99.95%), and master alloys Mg–31% Zr and Mg–30% Er were used to prepare the experimental alloys to the compositions of Mg–9Zn–0.6Zr and Mg–9Zn–0.6Zr–0.5Er (wt.%). All the materials added were melted in a steel crucible inside an electrical resistance furnace. The Mg–Er master alloy was added after all the other metal/alloys were completely melted and mixed. After being held at 750 °C for 30 min, the melt was cooled down to around 720 °C, and cast into bar ingots of 85 mm in diameter by semi-continuous casting with $SO_2 + CO_2$ gas mixture protection and water cooling. The ingots were then homogenized at 315 °C for 20 h and cooled in a furnace.

Before the ingots were extruded, both the ingots and the extrusion die were heated to 400 °C for 85 min. To investigate the effect of the pre-heating treatment on the microstructure, small samples for microstructure observation were also heat treated with the same heating regime and then quenched in water at about 70 °C to retain the high temperature microstructures. Extrusion was conducted under a controlled constant force by a XJ-500 Horizontal Extrusion Machine made in China. The extrusion ratio was 25. After extrusion, the alloys were cooled in open air. The extruded samples were further aged at 200 °C for 10 h. Microstructure stability of the alloys was evaluated by a solution heat treatment at 400 °C for 1.5 h.

Cylindrical samples of 60 mm in gauge length and 10 mm in diameter were machined from the as-extruded and as-aged bars for tensile tests at room temperature. Tensile properties were determined from a complete stress–strain curve. 0.2% yield strength (YS), ultimate tensile strength (UTS) and elongation to failure (Elongation) were obtained based on the average of three tests.

Microstructures were observed by optical microscopy, as well as scanning electron microscopy (SEM) using a TESCAN VEGA2 scanning electron microscope equipped with an INCA Energy 350 energy dispersive X-ray spectrometer (EDX). Phase constitutions were determined by a Rigaku D/max 2500PC X-ray diffractometer with the use of Cu Kα radiation. Grain size distribution was statically analysed using a Transcend Tciimage image software. In this analysis, 8–10 grains in each of 10 viewing fields for each sample were randomly selected and measured. For each grain, the values for long axis and short axis were separately recorded, which were

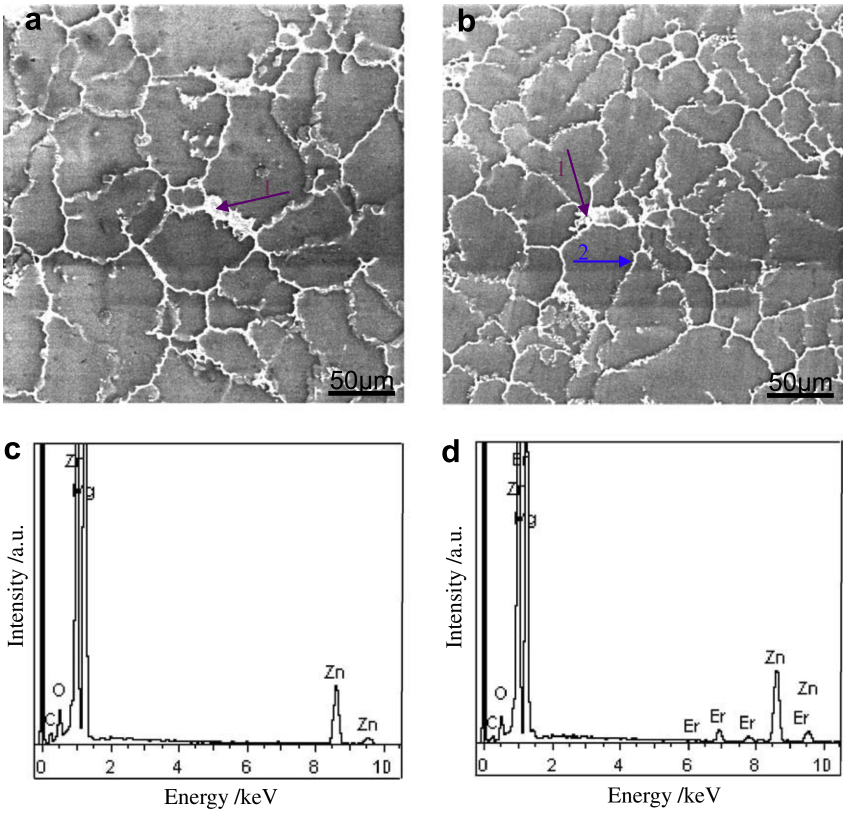

Fig. 1. SEM images of the as-cast microstructures of (a) Mg–9Zn–0.6Zr and (b) Mg–9Zn–0.6Zr–0.5Er alloys and corresponding EDX point analysis spectrum for (c) particle 1 and (d) particle 2 in (b).