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Effect of Ce addition on the microstructure and tensile properties of extruded Mg–Zn–Zr alloys

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ABSTRACT

The microstructure and tensile properties of extruded Mg–Zn–Zr–Ce alloys with different Ce contents were investigated. Ce addition to ternary Mg–Zn–Zr alloy resulted in significant grain refinement as well as a change in the type of second phase particles from MgZn to $Ce(Mg_{1-x}Zn_x)_{11}$, which has a C-centered orthorhombic structure with lattice parameters of a=0.999 nm, b=1.146 nm, and c=0.976 nm. However, fine $MgZn_2$ precipitates were found to exist in the extruded alloys regardless of the level of Ce content. The yield and ultimate strengths of the extruded alloys were significantly increased by Ce addition although the presence of coarse $Ce(Mg_{1-x}Zn_x)_{11}$ particles has an adverse effect on ductility.

1. Introduction

The development of high strength and low cost Mg alloy extrusions is considered essential to support the growing need for lightweight components in the automotive industry [1]. Unfortunately, however, there are few Mg alloy extrusions that fulfill the property and cost requirements of the automobile industry. In most cases, this is due to the fact that high strength Mg alloys, such as Mg–8Al–0.5Zn (AZ80) and Mg–6Zn–0.5Zr (ZK60), exhibit low extrusion speeds in the range of 0.5–2.5 m/min, which limits the production efficiency of Mg extrusion products [1]. Such alloys are susceptible to hot-cracking at high extrusion speed; this cracking results from the low incipient melting temperature of the second-phase particles that exist in the microstructure. One of the methods to produce sound extrusions with high strength Mg alloys at fast speed is to modify alloy compositions toward having more thermally stable second-phase particles. Promising alloying elements for this purpose include the rare-earth (RE) elements, especially Ce, which has been extensively utilized to enhance the creep resistance of Mg alloys at high temperature [2–4]. Recently, Yu et al. reported that the extrusion speed of the ZK60 alloy can be increased to as high as 10 m/min by the addition of 1 wt% Ce [5]. Although a few studies on the microstructure and mechanical properties of extruded ZK60 alloys containing Ce have been reported thus far [5–7], detailed microstructural characterization for these alloys is still regarded as incomplete. In the present study, the microstructure of ZK60 alloys with varying levels of Ce content from 0.5 to 1.5 wt% was characterized to elucidate its effect on the tensile properties of the extruded alloys.

2. Experimental procedure

Alloys with nominal compositions (wt%) of Mg–6Zn–0.5Zr, Mg–6Zn–0.5Zr–0.5Ce (ZKE600), Mg–6Zn–0.5Zr–1.0Ce (ZKE601), and Mg–6Zn–0.5Zr–1.5Ce (ZKE602) were prepared by induction melting using graphite crucibles under an inert atmosphere with a CO_2 and SF_6 mixture. The analyzed compositions of the alloys are Mg–5.65 wt% Zn–0.52 wt% Zr for ZK60, Mg–5.44 wt% Zn–0.61 wt% Zr–0.36 wt% Ce for ZKE600, Mg–5.67 wt% Zn–0.53 wt% Zr–0.77 wt % Ce for ZKE601, and Mg–5.71 wt% Zn–0.58 wt% Zr–1.40 wt% Ce for ZKE602. To cast a billet, each alloy was stabilized for 10 min in a molten state at 700 °C and then poured into a steel mold preheated to 200 °C. After casting, the alloys were homogenized at 440 °C for 4 h and then water-quenched to induce a supersaturated solid solution. The dimensions of each billet were 80 mm in diameter and 150 mm in length. Indirect extrusion experiments were implemented at an initial billet temperature of 250 °C, a ram speed of 0.65 mm s^{-1}, and an extrusion ratio of 50. Microstructural examinations were conducted on the midsections parallel to the extrusion direction (ED) using a Nikon EPIPHOT 200 optical microscope, a Quanta 200 field-emission scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS), and a JEM-2100F Cs-corrected transmission electron

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microscope (TEM) equipped with EDS. Grain size was measured by using a linear intercept method; $d=1.74\times L$, where d is the grain size and L is the linear intercept size. The sizes and area fractions of grains were averaged with values measured from 16 optical micrographs for every alloy. TEM samples were fabricated using a focused ion beam (FIB) technique. X-ray diffraction (XRD) analyses were conducted with a Rigaku D/MAX-2500/PC in the back reflection mode with Cu-K_α radiation. Tensile tests at room temperature were performed at an initial strain rate of $1\times 10^{-3}\,s^{-1}$ using round tensile specimens with a gage length of 25 mm and a gage diameter of 6 mm.

3. Results and discussion

Fig. 1 shows the optical micrographs of the extruded alloys. These micrographs reveal the microstructures, which consist of fine equiaxed α-Mg grains formed by dynamic recrystallization (DRX) and relatively coarse grains elongated along the ED, indicating that DRX is not complete for some grains after extrusion. The area fractions of the incompletely recrystallized grains appear to increase as the Ce content increases. The values for these area fractions are 2.6%, 4.9%, 6.1%, and 6.8% on average for the ZK60, ZKE600, ZKE601, and ZKE602 alloys, respectively. However, the sizes of the equiaxed DRX grains become smaller as the Ce content increases and the average grain sizes, considering both the fine equiaxed and coarse elongated grains, are 2.2, 1.9, 1.2, and 1.1 μm for the ZK60, ZKE600, ZKE601, and ZKE602 alloys, respectively. The grain sizes of the extruded alloys are much smaller than those of the homogenized alloys prior to extrusion, which are in the range of 80–120 μm.

Fig. 2 shows SEM micrographs of the extruded alloys; these images clearly reveal the presence of coarse second-phase particles in the α-Mg matrix. Particles appearing aligned along the ED in the ZK60 alloy were characterized as MgZn containing about 3.6 at% Zr, as has been similarly reported elsewhere [8]. However, a different type of globular particle with sizes of several tens micrometers can be seen in the ZKE alloys. As has been previously reported, these particles are already present in the homogenized ZKE alloys prior to extrusion [5,6]. The area fractions of these particles increase from 1.2% to 4.1% as the Ce content increases from 0.5 to 1.5 wt%. EDS analysis reveals that the particles present in the three ZKE alloys have similar compositions of around 52.0% Mg–39.6% Zn–8.4% Ce (at%) regardless of the level of Ce content. As can be seen in Fig. 3, XRD analysis indicates that the MgZn phase in the ZK60 alloy is replaced by another phase (tentatively denoted as CeMgZn) due to Ce addition. The XRD results also reveal that $MgZn_2$ precipitates, typically observable in wrought ZK60 alloy [5,8], are present in the ZKE alloys as well. This suggests that Ce addition does not alter the type of fine precipitates, which form via dynamic precipitation during extrusion.

In order to more clearly identify the CeMgZn phase in the ZKE alloys, TEM analysis was performed with the ZKE602 alloy containing 1.5 wt% Ce. Fig. 4 shows a TEM sample fabricated by FIB; this sample has a CeMgZn phase with an α-Mg matrix. Analyses of electron diffraction patterns obtained from the CeMgZn phase, shown in Fig. 4be, indicate that the phase coincides well with $Ce(Mg_{1-x}Zn_x)_{11}$, which has a C-centered orthorhombic structure with lattice parameters of around $a=0.999$ nm, $b=1.146$ nm, and

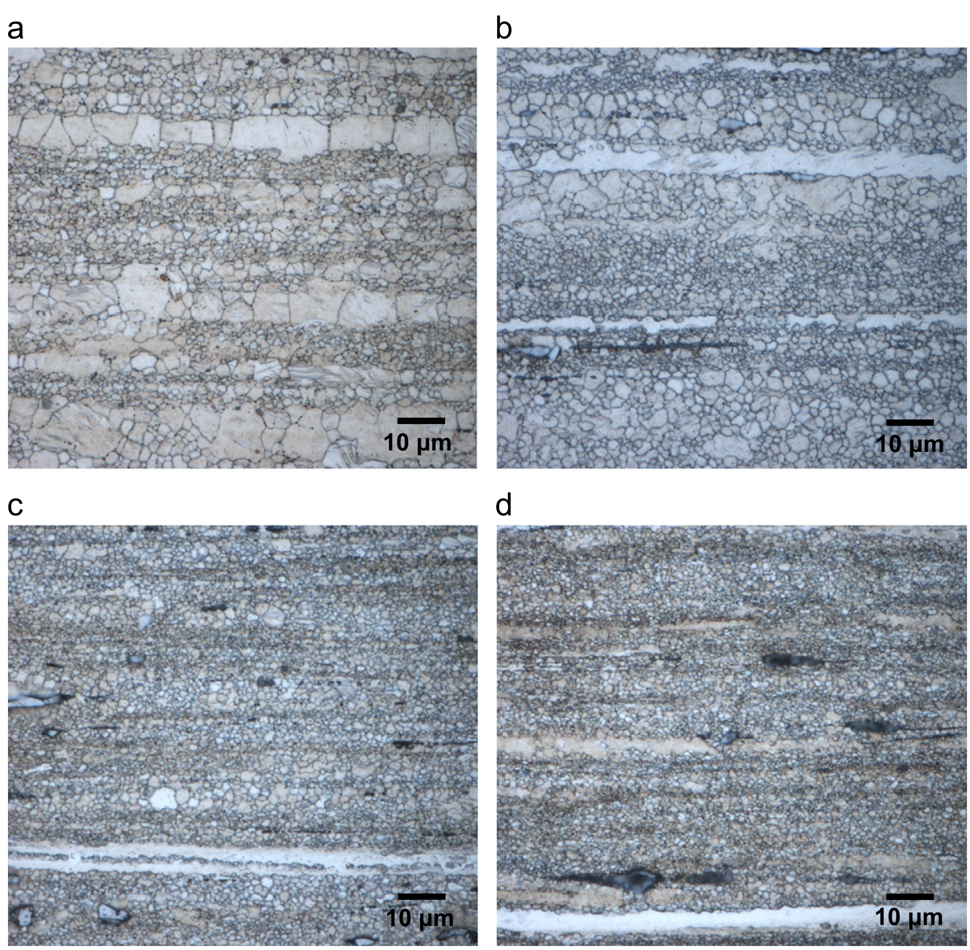

Fig. 1. Optical micrographs of extruded Mg–Zn–Zr–Ce alloys: (a) ZK60, (b) ZKE600, (c) ZKE601, and (d) ZKE602. Extrusion direction is horizontal.