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Influence of Ce addition and homogenization temperature on microstructural evolution and mechanical properties of extruded Mg-Sn-Al-Zn alloy

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The effects of Ce addition and homogenization temperature (HT) on the microstructure and tensile properties of extruded Mg-7Sn-1Al-1Zn (TAZ711) alloy were investigated. It was found that Ce addition resulted in the formation of a thermally stable lath-type Ce_3Sn_5 phase and that a decrease in HT caused an undissolved spherical Mg_2Sn phase to remain in the homogenized billets. With an increase in the amount of the Ce_3Sn_5 and Mg_2Sn particles, the number of fine Mg_2Sn precipitates formed dynamically during extrusion decreased gradually owing to a reduction in the amount of Sn dissolved in the matrix, which, in turn, led to an increase in the size of dynamically recrystallized grains because of a weakened grain boundary pinning effect. The tensile strength of the extruded alloys decreased with increasing amount of added Ce and decreasing HT, mainly because of the reduced number of precipitates and enlarged grain size. The yield strength and ultimate tensile strength of the alloy were inversely proportional to the area fraction of the Ce_3Sn_5 and Mg_2Sn particles present in the homogenized billets, whereas the strain-hardening ability of the alloy was directly proportional to the area fraction.

1. Introduction

In recent years, extruded Mg alloys have been in the spotlight in the transportation industry given the superiority of their mechanical properties to those of cast Mg alloys, which results in an apparent weight loss effect, as well as the simplicity of their fabrication process in comparison to rolling. However, further improvements in the strength of extruded Mg alloys are required in order to make them more competitive with Al alloys and steel. The mechanical properties of extruded Mg alloys are predominantly determined by microstructural evolution during extrusion, in which process dynamic recrystallization (DRX) and/or dynamic precipitation (DP) are generated. That is, the nucleation and growth of new grains and/or the formation of fine precipitates occurs vigorously under hot extrusion conditions because both strain and heat energies are imposed simultaneously to the material. This drastic variation in microstructure during extrusion is strongly dependent on the process variables. Mg extrusion products are generally fabricated by the following processes: casting, homogenization treatment, and hot extrusion. Here, extrusion parameters such as temperature, ram speed, and extrusion ratio significantly affect the DRX and/or DP behavior during the extrusion process, which, in turn, results in a variation in the mechanical properties of the extruded alloys [1–4]. For example, extrusion at high temperatures, fast ram speeds, and/or large extrusion ratios promotes the growth of newly formed dynamically recrystallized (DRXed) grains owing to the high grain boundary mobility. These extrusion conditions also reduce the number of dynamically formed precipitates because of the increased solubility of alloying elements on account of the high deformation temperature, which eventually results in a decrease in the strength of the extruded alloys. This result has been widely reported for various extruded Mg alloys such as Mg-7.95Sn-0.95Al-0.95Zn (wt%) [2], Mg-8Al-0.5Zn (wt%) [3], and Mg-1Zn-2Y (at%) [4].

Homogenization treatment can also play an important role in DRX and/or DP behavior during extrusion, thus affecting the resultant properties after extrusion because the microstructural state of a billet before extrusion changes considerably with the homogenization conditions [5–8]. The purpose of homogenization treatment after casting is to increase the hot workability of an alloy and extend the range of process parameters applicable during extrusion. Homogenization treatment is also used to dissolve undesirable large second phases formed during solidification and to generate a supersaturated α-Mg matrix, which enables the formation of fine precipitates during extrusion. If homogenization

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treatment is conducted at insufficient temperatures and/or for insufficient times for full dissolution of dissolvable second phases, then some of these second phases will remain along the grain/dendrite boundaries and within the grains. Particles present in a homogenized billet can have a beneficial effect on the nucleation of new DRXed grains [5,9,10] and on the suppression of their growth [11,12] during extrusion, thereby improving the strength of the extruded alloy on account of the increased DRX fraction and refined grain size. However, the presence of undissolved particles in a billet can prevent the formation of precipitates during extrusion, which, in turn, would decrease the strength of the extruded Mg alloys. In our previous study [5], the effect of homogenization treatment on DRX and DP behavior during extrusion was investigated in a Mg-7Sn-1Al-1Zn (wt%) (TAZ711) alloy, which has recently attracted considerable interest owing to its excellent combination of high extrudability and strength [13,14]. It was found that extrusion using a billet subjected to insufficient homogenization treatment reduces in the total number of dynamic precipitates formed during extrusion and consequently decreases the strength of the extruded alloy [5]. This indicates that the homogenization conditions are an important factor affecting the microstructure and mechanical properties of extruded Mg alloys.

It is known that the addition of the rare earth element Ce can have a great effect on the mechanical properties of extruded Mg alloys [15–18]. For example, the addition of a small amount of Ce (0.2 wt%) to pure Mg resulted in a significant increase in the tensile ductility (from 9.1% to 31%) of the extruded alloy [15]. It was also reported that the addition of Ce ($<$ 1.0 wt%) to a Mg-6Al-1Zn (wt%) alloy resulted in the formation of thermally stable Al_4Ce particles in a homogenized billet, which consequently improved the tensile properties of the extruded alloy owing to grain refinement by the fine Al_4Ce particles [16]. Furthermore, Mg-Zn-Ce particles formed by the addition of Ce (0.5–1.0 wt%) to a Mg-6Zn-0.5Zr (wt%) alloy increased the area fraction of the DRXed grains and the tensile strength of the extruded alloy by accelerating the DRX behavior during extrusion [17]. In addition, it was recently demonstrated that the microstructure of extruded TAZ711 alloy is also considerably influenced by Ce addition; with increasing Ce content, the DRX fraction increases but the number of precipitates decreased, because of the formation of Ce_3Sn_5 particles [18]. However, no in-depth studies have been conducted on the variation in the DRX and/or DP behavior during extrusion and the microstructure and mechanical properties of Ce-containing TAZ711 alloys with regard to homogenization treatment conditions. This study therefore investigated the effects of the addition of Ce and the homogenization temperature (HT) on the microstructural evolution in a TAZ711 alloy, and we determined the relationship between the microstructural characteristics and the tensile properties of the extruded alloys.

2. Experimental procedure

The nominal composition of the studied alloys was Mg-7Sn-1Al-1Zn-*x*Ce, hereafter referred to as TAZ711-*x*Ce (where $x=0$, 0.2, and 0.5 wt%). Cast billets of the TAZ711-xCe alloys were prepared by first melting the alloys in an electric resistance furnace at 800 °C under an inert atmosphere composed of a mixture of CO_2 and SF_6, then holding the molten metal at 730 °C for 15 min to stabilize it, and finally pouring it into a steel mold preheated to 210 °C. The chemical compositions of the cast billets were subsequently confirmed by inductively coupled plasma spectrometry (ICP; Thermo Xseries II) as being Mg-6.96Sn-0.98Al-1.04Zn, Mg-6.87Sn-1.06Al-1.07Zn-0.20Ce, and Mg-6.79Sn-1.04Al-1.04Zn-0.49Ce (wt%); these compositions were very close to their respective nominal values. Each alloy was heat treated for 24 h at temperatures of 400 °C, 450 °C, and 500 °C; the homogenized billets are hereafter denoted as 400HT, 450HT, and 500HT, respectively. For extrusion, the homogenized billets (Ø50 mm × 200 mm) were preheated at 250 °C for 1 h in a resistance furnace alongside dies with an angle of 90°. Indirect extrusion was then performed at an initial billet temperature of 250 °C, a ram speed of 1 mm/s, and an extrusion ratio of 20. The capacity of the employed indirect extrusion machine was 500 t, and no lubricant was utilized owing to the absence of friction between the billet and the walls of the container. A schematic diagram of this indirect extrusion process can be found elsewhere [19].

The microstructure of the homogenized billets and extruded bars was analyzed by optical microscopy (OM), field-emission scanning electron microscopy (FE-SEM), and energy-dispersive X-ray spectroscopy (EDS). The area fraction of the second-phase particles in each homogenized billet was measured using feature image processing software (INCA) and automated SEM image scans performed over an area of 1 mm^2. The average size of the DRXed grains of the extruded samples was determined by measuring the linear intercept size in five FE-SEM micrographs. The tensile properties of the extruded samples were measured at room temperature (RT) by using an Instron 4206 universal testing machine with a crosshead speed of 1.5 mm/min. Tensile tests were performed three times using dog-bone-shaped specimens (gage section: Ø6 mm × 25 mm), and the average values of these measurements were used in this study.

3. Results and discussion

The SEM micrographs of the homogenized billets reveal that the TAZ711 billets contain an undissolved Mg_2Sn second phase (Fig. 1a–c). The TAZ711-500HT billet has very few small particles (area fraction: ~0.1%), but the 450HT and 400HT billets contain some large (~1–10 μm) particles. In addition, a relatively large number of clusters of fine particles (~0.5–0.8 μm) are distributed along the dendritic cell boundaries in the 400HT billet (Fig. 1c); this is because in this alloy composition, the temperature of 400 °C was insufficient to completely eliminate the dendritic structure of the cast alloy owing to its low thermal energy, which corresponds to the kinetic energy of the atoms and molecules [5]. In contrast, all the TAZ711-0.2Ce and TAZ711-0.5Ce alloys contain lath-type undissolved particles along the grain boundaries and inside the grains (Fig. 1d–i); from EDS analysis, these particles are revealed to be as a thermally stable Ce_3Sn_5 phase [18]. The Ce-containing billets homogenized at 500 °C essentially contain only the lath-type Ce_3Sn_5 phase because the Mg_2Sn phase is almost fully dissolved by high-temperature heat treatment (Fig. 1d and g). In contrast, those homogenized at 450 °C contain both lath-type Ce_3Sn_5 particles and a small number of spherical Mg_2Sn particles (Fig. 1e and g), and the billets homogenized at 400 °C additionally contain clusters of fine Mg_2Sn particles (Fig. 1f and i). It should be noted that the Ce-containing billets (TAZ711-0.2Ce and TAZ711-0.5Ce) exhibit a combined second-phase distribution of Mg_2Sn particles, which are present in the corresponding Ce-free billets (TAZ711), and Ce_3Sn_5 particles formed by the addition of Ce. From the variation in the total area fraction of the undissolved second-phase particles shown in Fig. 2, it can be clearly seen that the amount of particles increases with increasing Ce content because of the formation of the Ce_3Sn_5 phase. Furthermore, this amount increases significantly as HT decreases (from 0.1% to 4.5% for TAZ711, from 1.9% to 5.1% for TAZ711-0.2Ce, and from 2.5% to 6.0% for TAZ711-0.5Ce). In all the alloys, the degree of increase is more pronounced when the temperature decreases from 500 °C to