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The effect of double extrusion on the microstructure and mechanical properties of Mg–Zn–Ca alloy

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ABSTRACT

The Mg–4.50Zn–1.13Ca (wt%) alloy was subjected to double extrusion, and the microstructure and mechanical properties of the extruded alloys were investigated. Double extrusion refined the microstructure significantly, resulting in the enhancement of mechanical properties of the double-extruded alloys compared with the single-extruded alloy. The yield strength of the single-extruded alloy was 173 MPa, while the value was increased to 320 MPa and 370 MPa after subsequent second extrusion at 300 °C and 250 °C, respectively. The second extrusion ratio had little effect on the microstructure and mechanical properties of the double-extruded alloys, while grain size was decreased and strength was increased with the decrease of double extrusion temperature. The double-extruded Mg–Zn–Ca alloy exhibited superior elevated temperature mechanical properties, which was attributed to the fine stable $Ca_2Mg_6Zn_3$ intermetallic compound particles dispersed in the alloy.

1. Introduction

There has been a growing demand for high strength magnesium alloys in transportation industries to reduce the weight of vehicles [1]. Microstructure refinement is one of the most effective ways to improve the mechanical properties of Mg alloys [2,3]. Magnesium alloys with fine-grained microstructure exhibited high strength and ductility at room temperature [4].

Thermo-mechanical processes refined microstructure of Mg alloys through dynamic recrystallization during hot deformation [5,6]. The grain size of dynamic recrystallization of the wrought alloys was affected by initial grain size [7] and intermetallic compounds [8]. Calcium was one of the most effective elements to refine the microstructure of Mg alloys [9,10]. Ca and Mg combined with Zn formed the stable compound $Ca_2Mg_6Zn_3$ [11]. Therefore, the wrought Mg–Zn–Ca alloys had the potential to produce fine-grained microstructure and attracted many researchers' attention. Ultrafine-grained microstructure of Mg–5.12Zn–0.32Ca (wt%) alloy with an average grain size of 0.7 μm was obtained by subjecting the extruded alloy to equal channel angular pressing (ECAP) [12]. The Mg–4.7 wt% Zn–0.5 wt% Ca alloy exhibited ultimate tensile stress of 329 MPa and elongation to failure of 15.8% after extrusion at 250 °C with extrusion ratio of 18 [13] due to the grain refinement and the dispersed precipitates. High ductility with elongation to failure of 34% was obtained in Mg–4.0 wt% Zn–0.5 wt% Ca alloy extruded at 320 °C with area reduction of 16 [14]. Such superior ductility was mainly due to the uniform microstructure and fine spherical second phase in the matrix [14].

Compared with single extrusion, multi-extrusion was more effective to refine the microstructure and improve the mechanical properties of metals [15]. The yield strength of Mg–2.70Nd–0.20Zn–0.41Zr (wt%) alloy was increased from 163 MPa to 275 MPa after second extrusion, due to much finer and more homogenous microstructures [16]. Two-pass extruded Mg–8Li–2Zn alloy exhibited excellent superplasticity with a maximum elongation of 758% at 563 K because the grain refinement promoted grain-boundary sliding (GBS) [17].

The researches on deformation of Mg–Zn–Ca alloys were mainly focused on single-step processing [13,14,18], multi-step plastic deformation of the Mg–Zn–Ca alloys was seldom investigated. In this study, Mg–Zn–Ca alloy was subjected to two-step extrusion in order to refine the microstructure and improve the mechanical properties. The effect of double extrusion parameters on the microstructure and tensile properties of the alloys was investigated.

2. Experimental procedures

Mg–4.50Zn–1.13Ca (wt%) ingot with diameter of 350 mm and length of 1730 mm was fabricated by semi-continuous casting,

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Table 1
The processing parameters of second extrusion.

Alloy	Temperature (°C)	Ratio	Ram speed (mm/s)
300R16+350R8	350	8	10
300R16+350R12	350	12	10
300R16+350R16	350	16	10
300R16+300R12	300	12	10
300R16+250R12	250	12	10

which has been described elsewhere [19]. The ingot was firstly extruded into a 60 mm diameter rod through a three-hole mold with a diameter of 420 mm at 300 °C with an extrusion ratio of 16:1 and an extrusion rate of 6 mm/min. Then the 60 mm diameter rod was further extruded with the parameters shown in Table 1 in order to investigate the effect of extrusion parameters on the microstructures and mechanical properties of the alloy.

Tensile tests using specimens with a gauge length of 15 mm and thickness of 2 mm were conducted at room temperature using an Instron 5569 universal test machine with a crosshead speed of 1 mm/min. The elevated tensile tests were conducted on Instron 5500R tensile machine at the temperature range of 100–300 °C with a strain rate of $1.67 \times 10^{-3}\ s^{-1}$.

The specimens for microstructural observation were cut along the extrusion direction. Samples for optical microscopy (OM) and scanning electron microscope (SEM) observation were etched in a solution of acetic picral (10 ml water+6 g picric acid+5 ml acetic acid+100 ml ethanol) after mechanical polishing. TEM specimens were ion-milled to perforation at an ion accelerating voltage of 3 kV. Microstructure analyses were conducted using Tecnai G^2 F30. The average DRXed grain sizes were measured using Image-Pro Plus 5.0 software, and at least 500 grains were recorded. Crystallographic texture of the extruded samples was examined by Bruker X-ray diffractometer.

3. Results

3.1. Microstructures

Fig. 1 shows the optical microstructures of the single-extruded and double-extruded alloys. The alloy after first extrusion consisted of equiaxed grains with an average grain size of about 12 μm and stringers of the second phase particles oriented along extrusion direction (Fig. 1a). The microstructures of the double-extruded alloy were significantly refined compared with the single-extruded alloy, as shown in Fig. 1. The average grain sizes for the single and double-extruded alloys are listed in Table 2. The average grain size of the alloy decreased with decreasing second-extrusion temperature. After second-extrusion at 250 °C, the DRX grain was too fine to be resolved in the optical microscope (Fig. 1f), and small amounts of unrecrystallized regions could be observed in the alloy, as shown in Fig. 1f. While the grain sizes of alloy extruded at 350 °C with different extrusion ratios (350R8, 350R12 and 350R16) had no obvious differences.

Fig. 2 shows SEM microstructures of the single and double-extruded alloys. It can be clearly seen that the second phase was broken up and dispersed along the extrusion direction after first extrusion, as shown in Fig. 2a. After second extrusion, the distance between the stringers of the second phases became narrow, and the second phases in the stringers became finer, exhibiting a more homogenous distribution of the second phases, as shown in Fig. 2b–f. The grain size of the alloy after second extrusion at 250 °C was estimated to be 1 μm, as shown in Fig. 2f.

Fig. 3 shows the typical bright field TEM images of single and double-extruded alloys. It is clearly seen that the grain size was refined after second extrusion and the grain size decreased with decreasing second-extrusion temperature. The fragmented second phase particles along extrusion direction were observed in the single-extruded alloy (Fig. 3a). And the similar stringers were also found in double-extruded alloys. Small amount of spherical particles were observed to precipitate in the single-extruded alloy, which were identified to be $Ca_2Mg_6Zn_3$ by selected electron diffraction patterns, as shown in Fig. 3a. A few precipitates were observed in the grains and at the grain boundaries of the alloys second-extruded at 350 °C and 300 °C, respectively (indicated by arrows in Fig. 3b and c). And no intragranular precipitates were detected when the second-extrusion temperature decreased to 250 °C (Fig. 3d).

3.2. Textures

Fig. 4 shows the (0001) pole figures for the single and double-extruded Mg–Zn–Ca alloys. The pole figures suggested that basal poles of the single and double-extruded alloys were perpendicular to the extrusion direction, which was a typical texture of extruded Mg alloys [20]. The double-extruded alloys exhibited weaker basal texture compared with that of the single-extruded alloy. And the intensities of basal texture for the double extruded alloys were similar except the alloy second-extruded at 250 °C, which contained some unrecrystallized regions.

3.3. Mechanical properties

Fig. 5 shows the typical stress–strain curves of the single and double-extruded Mg–Zn–Ca alloys. The ultimate tensile strength (UTS), tensile yield strength (TYS) and elongation to failure of the alloy were summarized in Table 2. The effect of extrusion ratios and temperatures on the mechanical properties is presented in Fig. 6a and b, respectively. The single-extruded alloy exhibited ultimate tensile strength and yield strength of 173 MPa and 251 MPa, respectively. After second extrusion, the alloys showed a pronounced improvement of tensile strength (Fig. 5 and Table 2). The yield strengths were increased from 173 MPa for the single-extruded alloy to approximately 230 MPa, 320 MPa and 370 MPa after second extrusion at 350 °C, 300 °C and 250 °C, respectively. The second-extrusion ratio had little effect on the mechanical properties, as shown in Fig. 6a. But the second-extrusion temperatures had a great influence on the mechanical properties of the double extruded alloys (Fig. 6b). With increasing extrusion temperature, the tensile strength decreased while the elongation to failure increased obviously. It is noted that the obvious yield phenomenon was observed in the double-extruded alloy except the alloy second extruded at 250 °C, as shown in Fig. 5.

Fig. 7 shows the ultimate tensile strength of the single-extruded alloy and the alloys second-extruded at 300 °C and 250 °C. The single-extruded Mg–Zn–Ca alloy exhibited much higher strength than the as-extruded [21] and as-rolled AZ31 alloy [22] above 100 °C, and the decrease of strength was much slower than that of commercial AZ31 alloy. The ultimate tensile strength of the double-extruded alloy was even higher than that of the as-extruded Mg–Al–Ca–Mn alloy [23] when tensile-tested at 150 °C. The double-extruded alloys exhibited the ultimate tensile strength above 220 MPa at 200 °C. The strength of the double-extruded Mg–Zn–Ca alloy was much higher than that of the single-extruded alloy when tensile-tested below 300 °C.