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Effect of Temperature on Grain Refinement of Mg-3Al-1Zn Alloy Processed by Equal Channel Angular Pressing

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In this work the effect of temperature on grain refinement of Mg-3Al-1Zn alloy (AZ31B) processed by equal channel angular pressing using route A is described. The deformation sequences consisted of equal channel angular pressing passes at 200 °C followed by passes at 150 °C. Nonhomogeneous grain size distribution promotes shear band formation at 150 °C. Shear bands with microcracks inside were analyzed by electron backscatter diffraction technique.

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1. Introduction

Due to their low specific weight, magnesium and its alloys are prospective materials for various industrial applications [1]. Although wrought magnesium alloys are only less used because of their poor workability at room temperature, they possess better mechanical properties compared to cast alloys [1]. One of the basic requirements to achieve these properties is homogeneous grain size. To produce materials with homogeneous grain size distribution specific deformation techniques are required. One of them is equal channel angular pressing (ECAP) [2]. However, during ECAP processing magnesium alloys are usually deformed at temperatures higher than 200°C where more slip systems are active [3]. Grain refinement at higher temperature is thus limited by recrystallization and grain growth. In order to obtain satisfactory grain refinement and hence to improve strength and ductility, ECAP should be realized at lower temperature.

Recently the pure magnesium was processed by ECAP at decreasing temperatures down to room temperature without using backpressure [4]. In another study the AZ31 magnesium alloy has been ECAPed using backpressure down to $125 \,^{\circ}$ C [5]. In case of pure magnesium ECAPed at room temperature there was no mention about shear band formation. In the latter case the authors used no more than two ECAP passes at individual temperature and final microstructure consisted of shear bands.

The aim of this article is to perform multitemperature ECAP of AZ31 magnesium alloy down to 150 °C and characterize the resulting microstructures by means of light microscopy and electron backscatter diffraction (EBSD).

2. Experimental material

The material used in this investigation was 13 mm thick rolled plate of magnesium alloy AZ31B (nominal composition Mg-3wt%Al-1wt%Zn) produced by AMTS, Israel. The initial microstructure of wrought AZ31B alloy has the texture typical of hot-rolled magnesium alloy with basal planes oriented perpendicular to the normal direction (ND) of the plate. Average grain size of 20 μ m was determined by EBSD technique. Billets with dimensions $10 \times 10 \times 70 \text{ mm}^3$ were machined from the rolled plate, with their long sides parallel to the rolling direction (RD). The billets were oriented with the rolling plane (RD-TD) perpendicular to the exit channel of the ECAP die (Fig. 1a). The ECAP die with angles $\Phi = 90^{\circ}$ and $\Psi = 45^{\circ}$ was used. The billet lubricated by molybdenum disulphide with graphite powder was inserted into the entrance channel of the preheated die, left there for 3 min and then extruded. The temperature of the ECAP die was monitored by the thermocouple placed inside the die near the plane of intersection of the two channels. The pressing speed for all passes was 5 mm/min.



Fig. 1. Scheme of ECAP die together with reference axes for initial rolled specimen (a) and specimen after ECAP (b).

AZ31B alloy was processed by ECAP using route A (no rotation between passes). Two different multitemperature ECAP cycles were performed: (1) two passes

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at 200 °C were followed by two passes at 150 °C, (2) four passes at 200 °C were followed by one pass at 150 °C. The samples for light microscopy were mechanically ground to 2400 grid using SiC paper, polished with diamond suspension through 3 μ m and 1 μ m, and with a colloidal silica solution. Etching of the samples for light microscopy was performed in acetic picral solution. EBSD technique incorporated in a dual-beam microscope FEI Quanta 3D FEG (working voltage 10 kV) was used to analyze the shear bands containing microcracks. The samples for EBSD analysis were prepared similarly as for light microscopy, after polishing with colloidal silica solution the ion beam polishing was performed on a Gatan-PECS. EBSD maps were measured at a step size of 0.15 μ m. The analysis of the EBSD data was performed with TSL 5.3 OIM analysis software. All microstructural observations were done on the plane defined by the extrusion direction (ED) and the normal direction (ND) (Fig. 1b).

3. Results and discussion

3.1. Light microscopy observation of microstructures

Light microscopy observation of the AZ31B alloy after first multitemperature ECAP cycle (2 × 200 °C + 2 × 150 °C) is shown in Fig. 2. After two passes at 200 °C nonhomogeneous grain size was observed with average grain size diameter of 3.1 μ m and large elongated grains in the range of 10–20 μ m (Fig. 2a). Subsequent two ECAP passes at 150 °C lead to the formation of shear bands in the regions close to the billet surface (Fig. 2b).



Fig. 2. Light microscopical observation of microstructure of the AZ31B alloy after ECAP using route A: (a) 2×200 °C, (b) 2×200 °C + 2×150 °C in the middle part of the billet, (c) 2×200 °C + 2×150 °C in the surface region.



Fig. 3. Light microscopical observation of microstructures of the AZ31B alloy after ECAP using route A: (a) 4×200 °C, (b) 4×200 °C + 1×150 °C in the middle part of the billet, (c) 4×200 °C + 1×150 °C in the surface region.

Shear bands were commonly observed after severe plastic deformation of AZ31 alloy [6, 7]. The nonhomogeneous grain size distribution obtained after two passes at 200 °C promotes formation of the shear bands during ECAP at 150 °C. Moreover, microcracks occurred in some shear bands close to the upper surface of the billet (Fig. 2c). The central part of the billet after multitemperature ECAP is characterized by relatively homogeneous fine grain size distribution ($\approx 1 \ \mu$ m) where sporadically isolated larger (5 μ m) grains were present (Fig. 2b). The average grain size in this area determined by EBSD was 1.6 μ m.

Figure 3 shows the light optical microstructure after the second multitemperature ECAP cycle $(4 \times 200 \,^{\circ}\text{C} +$ 1×150 °C). A homogeneous grain size distribution was observed after four passes at 200°C with average grain size diameter of 3.5 μm (Fig. 3a). Consequently, the microstructure after subsequent ECAP pass at 150 °C consisted of mixture of larger grains (5 μ m) and smaller $(1.6 \ \mu m)$ grains. The shear band formation was significantly reduced. But still shear bands with microcrack were observed in the surface region where large elongated grains were present (Fig. 3c). The larger grains in the surface region might result from the higher temperature close to the die wall. Also relatively slow extrusion rate and consequently longer time in the die at higher temperature support grain growth. In order to obtain additional information about shear band formation during ECAP of AZ31 alloy at 150°C an EBSD analysis of the resulting microstructure was performed.

3.2. EBSD study of the shear band microstructure

Inverse pole figure (IPF) map taken from a billet after two passes at 200 °C followed by two passes at 150 °C is shown in Fig. 4. Black lines and white lines represent high-angle grain boundaries (misorientation higher than 10°) and low-angle grain boundaries (misorientation lower than 10°), respectively. Microcrack situated inside the shear band was observed (Fig. 4A). Shear bands are known as places where the plastic deformation is strongly localized [8]. An accompanying effect is extensive grain size refinement inside the shear band, which in this case leads to an average grain size diameter of 1 μ m. Due to the strong localization of deformation to the shear band the large elongated grains below the shear band are not refined. These large grains were not refined during the ECAP passes at 200 °C. The following two passes at 150 °C were not sufficient to refine those large grains, but rather supported the nonhomogeneous distribution of deformation flow and shear band formation. Large grains marked as B and C are curved to the ED direction by deformation flow localized to the shear band. In this case large grain acts as an obstacle for localized deformation of the shear band which consequently lead to microcrack initiation inside the shear band.



Fig. 4. Inverse pole figure map after multitemperature ECAP $(2 \times 200 \,^{\circ}\text{C} + 2 \times 150 \,^{\circ}\text{C})$. (A) Microcrack inside the shear band. (B, C) elongated grains curved by deformation localized to the shear band.

Figure 5a shows (0002) pole figures of grains surrounding the microcrack and of grains far away from the microcrack (Fig. 5b). The texture of fine grains around the microcrack (Fig. 5a) does not correspond to the texture of all other grains in the billet (Fig. 5b). This is caused by the free surface around microcrack, where grains behave differently than inside the bulk. It can also be proposed that the microcrack appeared at the beginning of the ECAP pass and the grains did not rotate to the final position as shown in Fig. 5b. The elongated grain D(Fig. 4) is not curved because it was away from the localized deformation flow. The crack propagates through the grains and is parallel to basal planes of grains situated around the crack (Fig. 4). This implies that due to extensive localization of deformation to the grains inside the shear band the slip occurred mainly on basal planes and the stress locally exceeded the strength of the material. As a result the microcrack inside the shear band was observed.



Fig. 5. (0002) pole figure of grains surrounding microcrack (a) and grains far away from the microcrack (b).

Large elongated grains contain a relatively high fraction of low-angle grain boundaries with misorientation up to 10°. Some of them lie close to the high-angle grain boundaries (Fig. 4, arrow 1) and some of them pass across the grain (Fig. 4, arrow 2). These low-angle grain boundaries gradually change their misorientation and finally become high-angle grain boundaries. This is known as continuous dynamic recrystallization [8]. On the other hand, recrystallized grains (arrow 3 and 4 in Fig. 4) with different orientation than deformed grain imply that also discontinuous dynamic recrystallization occurred during ECAP at 150°C.

4. Conclusions

The effect of temperature on grain refinement of magnesium alloy AZ31 processed by ECAP using route A

down to 150 °C was presented. Two ECAP passes at 200 °C are not sufficient to produce homogeneous grain size distribution. This consequently support shear band formation during the subsequent ECAP at 150 °C. Increasing the number of ECAP passes at 200 °C to four significantly homogenizes the spatial grain size distribution. As a result the shear banding after subsequent ECAP at 150 °C is significantly suppressed. An EBSD analysis revealed that the prevailing mechanism of grain refinement was continuous dynamic recrystallization, but the signs of discontinuous dynamic recrystallization were also observed after ECAP at 150 °C. Microcrack propagation is parallel to basal planes of grains surrounding the microcrack.

Acknowledgments

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