DYNAMIC RECRYSTALLIZATION DURING ECAP OF MAGNESIUM SINGLE CRYSTAL

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Abstract

In the present paper, magnesium single crystals were processed by a single pass via equal channel angular pressing (ECAP) at 230°C in order to examine the dynamic recrystallization behaviour. Microstructure and microtexture were investigated by scanning electron microscope equipped with electron backscattered diffraction camera. The deformed microstructure was heterogeneous, composed of deformed (but unrecrystallized) and recrystallized zones. Formation of differently shaped and oriented dislocation walls is discussed as well as the character of high angle grain boundaries occurring in recrystallized zones.

Keywords: Magnesium, single crystal, ECAP, recrystallization

1 Introduction

Equal channel angular pressing (ECAP) provides a technique for producing microstructures with grain sizes in the submicrometer or even nanometer range in bulk materials [1]. This procedure has been successfully applied to aluminium alloys but is more complicated in hcp metals such as magnesium due to usually low forming capability [2]. Several research groups have achieved some promising results concerning improvement of mechanical properties of magnesium alloys [1,3]. However, the information available on mechanisms of grain refinement during ECAP of hcp metals is rather limited.

In majority of studies, ECAP processing was carried out at elevated temperatures. Thus, the grain refinement is achieved by dynamic recrystallization (DRX). According to the nature of the process, two modes of DRX can be generally distinguished, discontinuous DRX (DDRX) and continuous DRX (CDRX). DDRX is a classic nucleation of strain-free grains and subsequent growth at the expense of deformed regions. The CDRX is a recovery process characterized by formation of low angle boundaries (LABs) via dislocation accumulation and the gradual increase of their misorientation resulting in the formation of high angle boundaries (HABs). It was observed that magnesium and its alloys undergo CDRX rather than DDRX during deformation at moderate temperatures (~200-250°C) [4,5]. Several processes occurring during CDRX of magnesium were described in which the role of original grain boundaries [6] or twins [7] on the recrystallization process is mainly emphasized. However, exact mechanism of CDRX remains unclear.

Most of the investigations have been focused on the grain refinement of polycrystals, where the formation of new grains depends on presence and amount of original grain boundaries. There is a question how do the new grain boundaries form when there is a boundary-free structure at the beginning of deformation. Analysis of deformation structures of magnesium single crystal after one ECAP pass enables to investigate the occurrence of new grains directly from the deformation-free structures.
This paper aims to investigate the formation of new grain boundaries via CDRX from boundary-free structure during ECAP deformation of magnesium single crystal.

2 EXPERIMENTAL

The single crystal (20 mm in diameter and 60 mm in length) was grown by Bridgman method from 99.99% magnesium in graphite crucible under argon atmosphere with the pulling rate of 10 mm/h. Laue backscatter reflection technique and electron backscatter diffraction (EBSD) method were used to determine its crystallographic orientation. The ECAP sample with dimensions of 10 mm × 10 mm × 35 mm was cut from the single crystal using electro-spark cutting machine.

The ECAP die and the corresponding orientation of the single crystal are depicted in Fig. 1. The ECAP processing was performed using a die having internal angles \( \Phi = 90^\circ \) and \( \Psi = 45^\circ \). The single crystal was held for 60 min in the die preheated to 230°C and then processed in a single pass with the speed of 5 mm/min followed by water cooling. Molybdenum disulphide and graphite powder were used as lubricants.

After ECAP, the microstructural characterizations were performed using field emission scanning electron microscope (Ultra 55 Zeiss Ultra, working voltage of 20 kV) equipped with a high-speed EBSD camera (EDAX/TSL). The samples were mechanically ground using SiC papers followed by polishing with water-based diamond suspension (particle size up to 1 µm). Final preparation was performed using mechanical-chemical polishing in a solution of colloidal silica (Struers OPS) followed by etching in solution of 30% HNO₃.

![Figure 1 Schematic illustration of the ECAP die and initial orientation of the single crystal. Initial orientation is shown in (0002) and (10\( \bar{1} \)0) pole figures. Dark gray rectangle shows the observation area. Coordinate system (ID – insert direction, ED – extrusion direction and TD – transverse direction) is also included.](image)

3 RESULTS AND DISCUSSION

3.1 Microstructure

Inverse pole figure (IPF) map and (0002), (10\( \bar{1} \)0) pole figures of the single crystal after single ECAP pass are shown in Fig. 2. The map covers an area \( \sim 1.8 \times 4 \, \text{mm}^2 \). The microstructure consists of large deformed (i.e. unrecrystallized) areas and recrystallized regions. Unrecrystallized regions have orientation of basal poles close to A positions in (0002) pole figure (pink-violet-yellow colours in EBSD map) or close to B position in (0002) pole figure (blue colour in EBSD map). Unrecrystallized areas are characterized by smooth transition in colour contrast which indicates gradual tilting of the matrix due to successive arrangement of dislocations during ECAP. In segments with basal poles in A positions, twins can be distinguished and are
visible in magnified image. According to the EBSD measurement, twins are {10\(\overline{1}\)2} twins, as the HABs between the matrix and the twins have misorientation of 86°\((11\ \overline{2}\ 0)\) ± 5° [8].

The recrystallized grains are situated in “bands” and are characterized by blue-green colours in the IPF map. These colours correspond to orientation of basal poles close to B position in (0002) pole figure.

The (0002) pole figure in Fig. 2 shows that the density of basal poles is higher in the position A than at the edges (close to B position). This is because the fragments in A position are less recrystallized than fragments in B position, where the basal poles are more scattered. The difference between the amount of recrystallization may lie in the difference in activity of the slip systems in these segments, especially with the possibility B segment to be deformed via the (a) basal slip which is not possible in segments with A orientation [9].

**Figure 2** IPF map of the ECAPed single crystal together with the (0002) and (10\(\overline{1}\)0) direct pole figures from the same area as the IPF map covers. Pole figures are coloured according to the legend of densities of [0001] and {10\(\overline{1}\)0} poles. Symbols A and B denote orientations found in deformed single crystals. Magnified image shows presence of {10\(\overline{1}\)2} twins.

3.2 Formation of LABs in unrecrystallized regions

Two stages of CDRX occurred during the ECAP process. First, dislocation walls i.e. LABs, are formed. Second, some of these LABs are transformed into HABs and new CDRXed grains are formed. In this paper, these two stages are considered separately. The regions where almost no evidence of new DRXed grains with HABs is apparent are called unrecrystallized regions. The regions where new grains with HABs are found, are recrystallized regions. Unrecrystallized regions represent areas suitable for analysis of formation of very first subrains and LABs before the structure is fully recrystallized.

The IPF map from unrecrystallized area is shown in Fig. 3 a. HABs are depicted in black and the LABs in white colour. The map comprises differently oriented LABs which are shown by black arrows. There are
LABs lying more or less parallel to shear direction (at 45° to the ED direction) and other LABs which are perpendicular on them. LABs lying parallel to shear direction transform in some parts to HABs. Their appearance can be explained as a consequence of intensive shear stress. When the strain reaches a certain value, LABs are formed to release the accumulated strain and can give origin to the shear bands. The orientation of basal planes is favourable for basal slip and thus the deformation is prone to concentrate in the bands [10].

These LABs can act as preferential sites for appearance of new recrystallized grains with HABs. Newly appeared grains are shown in Fig. 3a by yellow arrows. Some of the new grains with HABs are small and located individually at the LABs. They can appear e.g. as a consequence of bulging of these LABs. Other new grains are quite big (more than ~20 \(\mu\)m) and seem to appear via connection of LABs marked by black arrows, i.e. are formed between the bands.

![Figure 3](image)

Figure 3: IPF maps of (a) unrecrystallized region and (b) recrystallized region. (b) is accompanied by misorientation angle distribution and the misorientation axis distributions for 30° boundaries.

### 3.3 Recrystallized regions

Figure 3b displays the IPF maps of the chosen area containing new recrystallized grains with the HABs. The map is accompanied with misorientation angle distribution. The two local maxima are visible in the distribution. The first local maximum belongs to the LABs and the second one to the HABs with the misorientations around 30°. Misorientation axis distributions for 30° boundaries is also displayed in Fig. 3b showing that these boundaries have misorientation axis close to \(\langle 0001\rangle\). The boundaries with the misorientations around 30° are frequently observed in deformed magnesium and its alloys (e.g. [6]). Ostapovets et al. [11] attributed the occurrence of the 30° peak to highly coincident grain boundaries (i.e. characterized by low values of the reciprocal density of the coincidence site lattice, \(\Sigma\)) with \(\langle 0001\rangle\) misorientation axis formed in recrystallized areas. The occurrence of this 30° local peak is frequent even after more ECAP passes [4]. These low-\(\Sigma\) boundaries may introduce local minimum energy configurations. Thus, the 30° HABs are stable and do not have a need to increase the misorientation to higher angles.

Within the DRXed grains, the LABs are present and are depicted by red arrows in both Figs. 3a,b. These LABs are straight, transverse the DRXed grains interiors in arbitrarily and thus divide the DRXed grains into smaller grains. EBSD analysis showed that the LABs are perpendicular to \(\langle 0001\rangle\) basal planes of grains. The observation of this kind of LABs is quite frequent [6,10]. With progressing deformation they can incorporate more dislocations resulting in separation of two parts of the DRXed grain.
4 CONCLUSIONS

Oriented magnesium single crystal was processed by single ECAP pass at the temperature of 230°C in order to investigate the mechanism of formation of new grains from the boundary-free structure. The deformed microstructure was heterogeneous, composed of deformed (but unrecrystallized) and recrystallized zones. (10\(\bar{1}2\)) twinning was observed in unrecrystallized zones. CDRX started with formation of differently oriented dislocation walls, which served as preferential sites for formation of new grains with HABs. In recrystallized areas, HABs with local maximum at the misorientation of 30° were observed. Long LABs formed perpendicular to the (0001) slip plane were frequently observed within the recrystallized grains causing the fragmentation into smaller grains.

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