Microstructural evolution of the AlMgMnZr alloy during severe plastic deformation

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Abstract. The effect of severe plastic deformation (SPD) on microstructure of Al-5.4%Mg-0.52%Mn-0.1%Zr alloy was studied. The methods of equal channel angular pressing (ECAP) and cold rolling were used to obtain fine-grained microstructure. The evolution of average misorientation angle and portion of HAB during SPD was determined. The lattice dislocation density was evaluated from EBSD data from the lattice curvature using the via Frank's equation.

Keywords – Aluminum alloys, Severe plastic deformation, Microstructure

1. Introduction
The development of various regimes of severe plastic deformation to obtain fine-grained microstructure in aluminum alloys has recently attracted more attention [1-2]. The formation of a fine-grained structure in these alloys is a convenient way to achieve a new level of physical and mechanical properties. Dynamic recrystallization is an important mechanism which make it possible to control the microstructure and mechanical properties of various materials [3-4]. The most significant advantage of dynamic recrystallization is that the necessary microstructure can be formed directly during deformation [3-5].

The process of dynamic recrystallization depends significantly on the temperature, strain and strain rate. In particular, large elongation ratios frequently used in the pressing technology, which lead to significant values of the strain. However, after reaching a certain critical degree of strain, it ceases to play the role of a control parameter, and then, the final properties of the alloy are determined by the temperature and strain rate [6]. Thus, the aim of the present study was to determine the evolution of the microstructure of the AlMgMnZr alloy various regimes of severe plastic deformation (SPD).

2. Material and methods
The studied Al-5.43%Mg-0.52%Mn-0.1%Zr wt.%. alloy was produced by continuous casting. The obtained billets were homogenized at 360°C for 6 h. Then the following treatments were used:
(a) To eliminate the disadvantages of the cast structure, an intermediate deformation was carried out: the rolling at room temperature with a total reduction of 70% and subsequent annealing at 400°C for 2 h followed by cooling in air. This material condition is denoted hereafter as coarse-grained (CG).
(b) The bars with cross section of 20×20 mm² and length of 100 mm were subjected to equal channel angular pressing (ECAP) via route BC with a 90° route after each pass. The ECAP processing
was performed at 300°C to the true strain of ~12. This material condition is termed as fine-grained (FG).

(c) The samples of studied alloy after treatment (b) were subjected to addition cold-rolling at room temperature with a total reduction of 80%. This material condition is denoted as severely strained (SS).

The microstructural investigations and elemental analysis were performed by Quanta 600FEG scanning electron microscope (SEM) equipped with an electron back scatter diffraction (EBSD) pattern analyzer incorporating an orientation imaging microscopy (OIM) system. The transmission electron microscopy (TEM) observations were carried out in the bright field using JEOL JEM-2100 microscope equipped with an INCA energy dispersive x-ray spectrometer.

Due to the experimental error of the EBSD method, all low-angle boundaries with a misorientation of less than 2° were excluded from consideration using the grain expansion parameter in the TSL software. The misorientation of 15 ° was used as a criterion for distinguishing low- and high-angle boundaries (LAB and HAB, respectively).

3. Results and discussion

Representative microstructures of the studied AlMgMnZr alloy in various states is shown in figure 1.

![Figure 1. OIM maps of studied alloy in CG (a); FG (b) and SS (c) material states.](image)

The main structural parameters are listed in Table 1. In the studied alloy recrystallization annealing at a temperature of 400°C, leading to the formation of the microstructure with heterogeneous grain size distribution (figures 1 (a) and 2 (a ’)). Large and relatively equiaxed grains have an average size of 22.0 μm, and small grains located along the boundaries of large grains have an average size of 6.5
μm (figure 1 (a)). Uniformly distributed $\text{Al}_6\text{Mn}$ particles with incoherent interfaces and average size of 35 nm were found inside the grains [7]. The specific fraction of HAB estimated using EBSD analysis is found to be $\sim$ 93% and the average misorientation angle is 33 ° (figure 2 (a)). The dislocation density in the CG state is $\sim 10^{13}$ m$^{-2}$.

**Table 1.** Microstructure; parameters of studied aluminum alloy in different material states.

| State | CG | FG | SS |
|-------|----|----|----|
| Grain size, μm | 22 | 1.5 | 1.0 |
| Fraction HAB’s, % | 93 | 62 | 43 |
| Average Misorientation Angle, ° | 33 | 27 | 21 |
| Size of $\text{Al}_6\text{Mn}$ particles, nm | 35 | 25 | 25 |

ECAP processing, leading to the refinement of recrystallized grains. The average grain size in this state is about 1.5 μm which is 15 times smaller as compared to the CG state (figures 1 (b) and 2 (b')). The fraction of recrystallized grains is 90%. The mean size of $\text{Al}_6\text{Mn}$ particles located inside the grains did not change remarkably during ECAP (table 1). The average misorientation angle and the portion of HAB in this state are decreased to 27 ° and 62% (figure 2 (b)), respectively.

The EBSD analysis of studied alloy in the SS material condition showed that the grains tends to acquire an elongated shape (figures 1 (c) and 2 (c')), the average grain size is $\sim$ 1 μm. According to transmission electron microscopy, the average size of $\text{Al}_6\text{Mn}$ particles was not affected by addition cold rolling (table 1). The average misorientation angle and the portion of HAB decreased to 21 ° and 43% (figure 2 (c)), respectively. Note that a gradual decrease in the average misorientation angle in the studied alloy is associated with a gradual increase in the fraction of LAB under deformation.

The lattice dislocation density was evaluated from EBSD data from the lattice curvature using the kernel average misorientation option [8] via Frank's equation:

$$\theta = 2\sin \frac{\theta}{2} = \frac{Nb}{h}$$

where $\theta$ is the misorientation angle created by a dislocation wall of height $h$ consisting of $N$ dislocations, and $b$ is the Burgers vector. In EBSD analysis, distance $h$ corresponds to the scanning interval. The dislocation density $\rho$ is then given by the ratio of the dislocation number per surface area, Eq. (2):

$$\rho = \frac{N}{S}$$

where the surface area of the hexagon $S$ (scanning geometry) is $S = \sqrt{3}h^2/2$. It follows that the dislocation density can be estimated from the following relationship:

$$\rho = \frac{2\theta}{\sqrt{3}hb}$$

The values used for dislocation density calculations are given in table 2.
Figure 2. Misorientation angles (a, b, c) and grain size distributions (a’, b’, c’) of studied alloy in CG state (a, a’); FG state (b, b’) and SS state (c, c’).

Table 2. The mean parameters for eqs. (1-3).

| State | h, μm | Θ_{KAM} | b, nm | ρ, m⁻² |
|-------|-------|---------|-------|--------|
| FG    | 0.2   | 0.72830 | 0.286 | 1.4×10^{14} |
| SS    | 0.05  | 1.09021 | 0.286 | 8.8×10^{14} |

It is seen that calculated value of dislocation density in FG (7.7±3.2)×10^{15}m⁻² and SS (2.8±3.0)×10^{15}m⁻² material conditions is in good agreement with that measured using TEM images.
4. Summary and conclusions
The main conclusions derived based on the present study could be summarized as follows:

1) The average misorientation angle and portion of HAB are gradually decreased during grain refinement of studied aluminum alloy.

2) The mean size of Al₆Mn particles didn’t change remarkably under severe plastic deformation and subsequent annealing.

3) The severe plastic deformation leading to significant increase of dislocation density from ~10^{13} m^{-2} in CG state to 8.8×10^{14} m^{-2} in SS state.

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