Materials Science & Engineering A 706 (2017) 319–329

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Microstructure and texture of pure magnesium after room-temperature lateral extrusion

O.V. Antonova[a,*], A.Yu. Volkov[a], D.A. Komkova[a], B.D. Antonov[b]

[a] Institute of Metal Physics, Ural Branch, Russian Academy of Sciences, 18S. Kovalevskaya St., Yekaterinburg 620108, Russia
[b] Institute of High Temperature Electrochemical, Ural Branch, Russian Academy of Sciences, 20 Akademicheskaya St., Yekaterinburg 620219, Russia

ARTICLE INFO

Keywords:
Magnesium
Microstructure
Texture
Plastic deformation
X-ray diffraction
Electron microscopy

ABSTRACT

Three specimens of pure magnesium with different columnar structure orientations have been subjected to severe plastic deformation (SPD) by lateral extrusion (LE) at room temperature. As a result of the deformation ($\varepsilon \sim 3.9$), 1-mm thick plates were obtained. It is shown that the deformation led to a substantial size reduction of the initial structure by more than two orders of magnitude. The plates have a bimodal structure where large grains of several microns are surrounded with smaller ones of 0.1–0.5 µm. It is expected that the main mechanism of grain size reduction is related to twinning processes along with slip and dynamic recrystallization. All the plates show basis texture of varying degrees of sharpness. Under tensile tests the plasticity of the three plates varies between 8% and 16%.

1. Introduction

Magnesium and its alloys are the lightest metallic structural materials. They are also of growing interest as a next-generation material because of the excellent mechanical properties [1]. To date, the application of these materials is limited due to low workability at room temperature. Since magnesium and its alloys are metals with hexagonal close packed crystal structure, their active slip systems are basal planes $(0\,0\,0\,1)<1\,1\,\bar{2}\,0>$, prisms $\{1\,\bar{1}\,0\,0\}<1\,1\,\bar{2}\,0>$ and pyramids $\{1\,0\,\bar{1}\,1\}<1\,1\,\bar{2}\,0>$ [2]. The critical resolved shear stresses (CRSS) are considerably higher for the non-basal modes than for basal slip and decrease as temperature increases. In accordance with the available reference data, the difference of CRSS values for single crystals is nearly twofold, when in case of large-grain polycrystals of magnesium and its alloys a non-basal CRSS is about 1.1–2.5 times of that for basal slip [3–7]. Thus, while basal slip is expected to dominate at room temperature, non-basal modes would become more important when temperature increases. It is the lack of slip systems at low temperatures that accounts for the fact that articles of magnesium alloys are normally made at the temperature of 200–400 °C. One of the possibilities to improve the formability is to refine the structure. In recent years, severe plastic deformation (SPD) has offered the possibility to make grain sizes significantly smaller than those produced using conventional thermomechanical processing. Equal channel angular extrusion (ECAE) is one of such severe plastic deformation processes that lead to microstructural refinement in bulk materials [8,9]. Among the great number of studies dedicated to the issues of deformation behavior during SPD of magnesium and its alloys, we would like to focus on [10–15], which offer detailed studies of microstructures, texture evolution and properties of magnesium and its alloys subjected to ECAE. Yoshinaga et al. [4] reported that after two ECAE passes at 200 °C, the average grain size in Mg-0.9%Al alloy was about 17 µm. Suwas et al. [11] showed that deformation of magnesium by multi-pass ECAE through routes A, B and C at 250 °C leads to the formation of a non-uniform grain size in the range of 6–8 µm. Note that it was possible to cold roll ECAE processed material through route B up to 80% reduction without any problem. In [12], ECAE samples were cut out from a hot-rolled magnesium sheet so as to ensure their orientation appropriated for basal slip during deformation. It was possible to impart large plastic deformation by reducing the temperature gradually from 250 °C to room temperature through 8 passes. As a result of such processing, the average grain size was ~ 250 nm. In conclusions, the authors point out the main role of continuous or discontinuous dynamic recrystallization depending on temperature modes of each pass. During ECAE, the recrystallization mechanism has been found to be orientation-dependent. Texture analysis of ECAE-processed samples indicates that the deformation mechanism leading to an ultrafine grain size is slip-dominated.

Therefore, resolving the problem of magnesium plasticity increase is a critical task. This study presents the results of investigation of microstructure, texture and mechanical properties of polycrystalline magnesium after SPD at room temperature.

* Corresponding author.
E-mail address: olgaanton4@gmail.com (O.V. Antonova).

http://dx.doi.org/10.1016/j.msea.2017.08.080
Received 22 May 2017; Received in revised form 18 August 2017; Accepted 21 August 2017
Available online 24 August 2017

2. Materials and methods

An ingot of commercially pure magnesium (99.98%) was used as the initial material. The large-grain ingot had columnar structure. The average grain diameter was 2.4 mm and height was 7.7 mm. Three cylindrical specimens of 30 mm in diameter and 50 mm in length were cut out from the central section of the ingot. Specimen 1 was cut along the longitudinal axis of grains. Specimen 2 was cut perpendicularly to the first one. Specimen 3 was cut at 45° with relation to the first two. These specimens were subjected to SPD by lateral extrusion (LE) in specially designed work tool at room temperature. The deformation method is schematically shown in Fig. 1a. The method was described in detail in the previous paper [13]. As result, thin plates were obtained. The width of plates was 30 mm, the thickness was 1 mm. The plates showed no visible trace of cracks, except for a little crackling along the edges. Fig. 1b shows some options for load application to the three specimens. In case of specimen 1, it was close to the growth direction of columnar structure, and load was 1.2 GPa. For specimens 2 and 3, where the original grain orientations had been changed significantly, loads were 0.9 GPa and 0.7 GPa, respectively. The press power was determined by a dynamometer. The load was calculated as the power applied divided by the cross sectional area. Deformation degree was $\varepsilon \sim 3.9$.

Methods of X-ray diffraction (XRD; DMAX 2200 RIGACU diffractometer), electron backscatter diffraction analysis (EBSD; QUANTA 200 FEI electronic microscope) and transmission electronic microscopy (TEM; JEM 200 Cx electronic microscope) were used to characterize microstructure and texture.

X-ray structure analysis of all the specimens was carried out under identical conditions in monochromatic CuKα radiation at a rate of 4°/min. Specimens for EBSD were subjected to mechanical polishing with abrasive paper and electro-polishing in 92% methyl alcohol, 5% nitric acid, 1% acetic acid and 2% glycerol reagent at 10 V. Foils for TEM were produced by means of electrolytic polishing, in a solution containing 20% of nitric acid and 80% of methyl alcohol at 10 V. All the specimens were prepared at room temperature.

Mechanical properties of magnesium after SPD were assessed using two methods: 1) based on microhardness of the specimens measured by means of the PMT-3 instrument; 2) based on the results of tensile tests. The tensile tests were carried out at room temperature using an Instron test machine. Test samples were cut out of 1-mm thick plates along the direction of flowing out. The length of the gauge part of the samples was 50 mm, the width was 10 mm. The strain rate was selected based on the work [13] and equal to 0.2 mm/min ($7 \times 10^{-5}\,s^{-1}$).

3. Results

3.1. X-ray diffraction analysis

Fig. 2 shows diffraction patterns of specimens 1, 2 and 3 before and after SPD. All the three specimens were differently oriented in relation to the direction of load application. In the first specimen (Fig. 2a, line 1), when the compression direction was approximately along the axis of structure of cast magnesium, there was a very intensive line (0 0 0 2). It can primarily indicate the basal texture. Fig. 2a (line 2) shows the diffraction pattern of specimen 2. A set of lines typical for materials with hexagonal close-packed lattice can be observed, but their intensity ratio is different from that in the reference X-ray pattern of polycrystalline magnesium in balanced condition. A strong line $\{1\ 0\ \bar{1}\ 1\}$ typical of polycrystalline magnesium was observed in Fig. 2a (line 3) for specimen 3.

The X-ray diffraction patterns of specimens deformed to $\varepsilon \sim 3.9$ are shown in Fig. 2b. It can be seen that there are significant changes in line intensity. Note that regarding specimen 1 after SPD (Fig. 2b, line 1), apart from the strong line (0 0 0 2), mention should be made of weak lines that were not observed in the initial states. As regards the other two specimens (Fig. 2b, lines 2 and 3), the intensities of all the observed orientations are redistributed towards increase of the line (0 0 0 2). Therefore, one can assume that, as a result of the above deformation pattern, basal textures with different sharpness levels have been formed in the magnesium plates. During deformation, the maximum resistance against formation of these textures was observed in specimen 3 (Fig. 2b, line 3), where plenty of differently oriented grains still remained in the material even after severe deformation.

By calculating the Schmidt factor f for all orientations observed in the initial specimens we have assessed possible activation of slip systems. As expected, for specimen 1 with the preferred grain orientation of (0 0 0 1) in its initial state, the deformation is the result of slipping in the non-basic plane only. A. Chapuis and J.D. Driver are reported in [16] that maximum Mg CRSS value at room temperature in pyramid planes is 100–150 MPa, and in basal planes – below 10 MPa. High values of load (1.2 GPa) at all-round compression that were applied to deform our specimen could have been sufficient to activate non-basal slip systems.

In accordance with X-ray diffraction patterns (Fig. 2), the other two specimens in the initial state had grain orientations that were favorable for basal slip development (Table 1). This is proven by much lower values of the specific load required for deformation of specimens 2 and 3: 0.9 and 0.7 GPa, respectively. Moreover, such values of the specific load may well be conducive to the activation of non-basal slip in favorably oriented grains, for which a high Schmidt factor exists, for

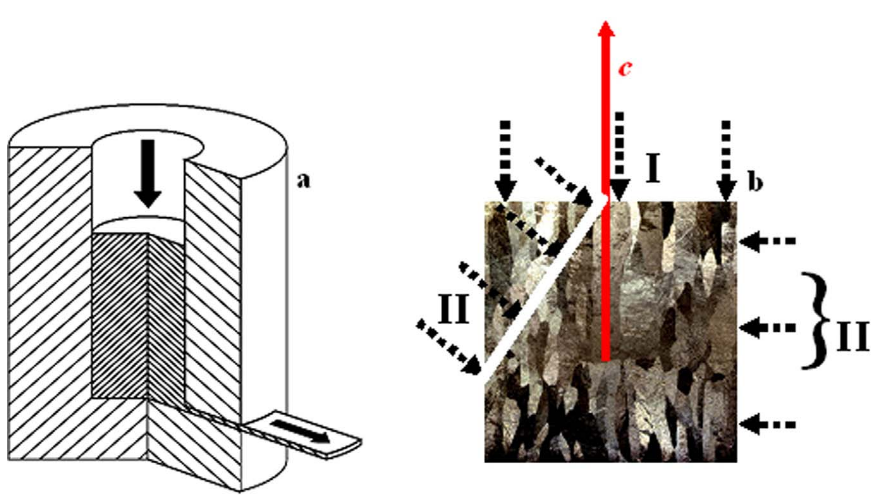

Fig. 1. The scheme of LE (a) and the scheme of load application (b).