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Evolution of microstructure and microtexture during the hot deformation of Mg–3% Al

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Abstract

The orientation relationships associated with different mechanisms of new grain formation during the hot deformation of a Mg–3.4% Al–0.33% Mn alloy were investigated using electron back-scattered diffraction (EBSD) techniques. Compression tests were carried out at 350 °C with a strain rate of 0.001 s^{-1} on samples machined from extruded tubes. Three types of microstructural features were produced at this temperature: (i) microbands (MBs); (ii) bulged regions; (iii) new grains formed by continuous dynamic recrystallization (cDRX). The formation of the MBs is attributed to the collection of basal dislocations in the MB boundaries. Both the bulges as well as the new cDRX grains are formed as a result of dislocation-based processes that produce *c*-axis rotations toward the loading axis (i.e. away from the radial direction–transverse direction (RD–TD) plane). Once nuclei have formed, however, the new grains have their *c*-axes located fairly close to the RD–TD plane. In this way dynamic recrystallization leads to the retention of the main characteristics of the initial RD–TD texture.

Keywords: Magnesium; Dynamic recrystallization; Dislocation boundaries; Orientation imaging microscopy; Misorientation relationships

1. Introduction

In many metals dynamic recrystallization (DRX) leads to the randomization of initial textures and so can be of practical interest with regard to subsequent forming [1]. According to the literature, the mechanisms involved in the DRX of magnesium are fairly complex and the nucleation mechanisms are considered to be affected by the deformation conditions [2,3]. Above the critical value of the Zener–Hollomon parameter for the activation of twinning ($\sim 7 \times 10^{12}$ s^{-1}) [4] the interaction between slip dislocations and twin boundaries leads to the development of highly distorted regions and the eventual formation of high angle boundaries. This mechanism has been referred to as low temperature dynamic recrystallization (ltDRX) [3,5,6]. As the Zener–Hollomon parameter of deformation is decreased, more and more cross-slip occurs [7–9] and dislocation rearrangement leads to the formation of numerous low angle boundaries (LABs) [3,10,11]. Subsequent continuous rotation of the new subgrains results in a progressive increase in the misorientation [3,12]. This process is generally referred to as continuous DRX or cDRX. Finally, when the activation energy of plastic flow approaches that for volume self-diffusion ($\sim$135 kJ mol^{-1} [13]), the controlling deformation mechanism becomes the climb of basal dislocations [3,4]. The larger strains attained in this way induce strain localizations at grain boundaries that can promote DRX nucleation either by bulging (one of the mechanisms of discontinuous DRX or dDRX) or by subgrain formation within slip bands [14–17].

Regardless of the mechanism (ltDRX, cDRX or dDRX), recrystallization is not usually accompanied by sharp changes in the crystallographic texture. Yi et al. [18] have reported, for example, that the orientation distribution function (ODF) intensities were similar in their initial material and in samples deformed into the DRX

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regime. (Nevertheless, they did observe a slight shift in the location of the main texture component.) Other investigators [10,19] have reported that most of the newly recrystallized grains have orientations similar to those of the matrix grains, but with slightly weaker intensities. Such recrystallization appears to promote 30° ⟨0 0 0 1⟩ rotations that preserve the basal texture or at least delay its decomposition [20,21].

These investigations were mostly performed on highly deformed samples, so that it was not possible to separate the influence of nucleation as opposed to growth. The present study was therefore undertaken with the aim of characterizing the formation of the LABs and new grains that developed in the high temperature regime. The rotations and mechanisms involved during the DRX of Mg will be outlined together with their impact on the flow stress and crystallographic texture.

2. Experimental

2.1. Uniaxial compression

The present investigation was carried out on a wrought magnesium alloy (AM30) developed at the GM Global Research Center in Warren, MI [22]. The tubes from which the samples were made were extruded using porthole dies and produced by Timminco Metals in Aurora, CO; they had nominal outer diameters of 70 mm and wall thicknesses of 4 mm. The samples were machined out of the tube walls with their axes aligned along the extrusion direction (ED); the specimens had heights of 4.5 mm and diameters of 3 mm so that the height/diameter ratio was 1.5, following ASTM standards.

Uniaxial compression tests were performed along the ED using a model 510 MTS servohydraulic mechanical testing machine. The specimens were heated to 350 °C and then held for 5 min prior to compression. Straining was carried out to true strains of 0.05–0.2 at a strain rate of 0.001 s^{-1}. A rapid cooling system was employed to preserve the as deformed microstructure by cooling each sample to ambient temperature within 2 s of deformation with the aid of an ejection lever.

2.2. Texture and microstructure characterization

The initial macrotexture was measured using a Siemens D500 diffractometer equipped with a goniometer and using MoK_α radiation. Corrections for peak defocusing and background intensity were made using experimentally determined defocusing curves established on random powder samples. The measured incomplete pole figures were obtained from the (0 0 0 2), (10–10) and (10–11) Bragg peaks. The data were then analysed using the arbitrary defined cell (ADC) method [23] with the TexTools 3.2 software to calculate the orientation distribution functions and complete pole figures. For each pole figure standard θ–2θ scans were run to obtain the exact positions of the peaks. Prior to the X-ray diffraction measurements the specimens were polished to a mirror finish and then etched with a solution containing 4.2 g picric acid, 10 ml acetic acid, 10 ml water and 70 ml ethanol.

The microtexture was examined with the aid of a scanning electron microscope equipped with an EBSD detector. Specimens were mounted in cold curing resin, polished and subsequently etched in a solution composed of 10 ml nitric acid, 30 ml acetic acid, 40 ml water and 120 ml ethanol. Automated electron back-scattered diffraction measurements were performed with a field emission type microscope. An acceleration voltage of 20 keV, together with a working distance of 15 mm and a sample tilt angle of 70° were selected to maximize back-scattered electron diffraction. Additionally, a step size 0.3 μm was employed to provide a minimum ratio of 1:3 between the step size and the size of the smallest feature of interest.

2.3. Misorientations and Rodrigues–Frank space

In order to shed some light on the mechanisms associated with the different microstructural changes described below, the rotations involved were measured carefully. For this purpose, the orientation matrices identifying two crystallites A and B in the specimen coordinate system are labelled $\mathbf{g}_A$ and $\mathbf{g}_B$, respectively. Then, the misorientation matrix $\mathbf{M}_{AB}$ relating these crystallites, where crystallite A is arbitrarily chosen to be the reference system, is given by [24]:

$$\mathbf{M}_{AB} = \mathbf{g}_B \mathbf{g}_A^{-1} \tag{1}$$

This matrix defines a rotation that converts the coordinate system of the reference crystallite into that of the other crystallite. The angle–axis pair associated with $\mathbf{M}_{AB}$ was then defined as:

$$\omega = \arccos(1/2[\mathrm{trace}(\mathbf{M}_{AB}) - 1]) \tag{2}$$

$$[d_1, d_2, d_3] = [m_{23} - m_{32},\ m_{31} - m_{13},\ m_{12} - m_{21}] \tag{3}$$

Here ω is the misorientation angle between crystallites A and B, d_{ij} ($i, j = 1, 2, 3$) are the axial components of the rotation axis d and m_{ij} ($i, j = 1, 2, 3$) are the elements of $\mathbf{M}_{AB}$. Note that the minimum angle–axis pair representation is used here; it is obtained by taking the crystal symmetry into account [24]. Although identification of the misorientation axes associated with low angle boundaries of less than 5° is subject to considerable error, this problem does not effect higher angle misorientations [25,26]. For this reason the present work deals only with misorientations of 5° and more, in which case the errors involved in determining the rotation axis are reduced.

The angle–axis pair can be represented in three dimensions by combining the unit vector **d** and the rotation angle ω using the Rodrigues formula [27] given by:

$$R = \tan\frac{\omega}{2}[d_1, d_2, d_3] \tag{4}$$

Each misorientation can then be described by the three components R_1, R_2 and R_3 of the Rodrigues–Frank (RF) vector. When the minimum angle–axis pair representation