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Texture enhancement during grain growth of magnesium alloy AZ31B

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Abstract—The microstructure and texture evolution during annealing of rolled Mg alloy AZ31B, at temperatures ranging from 260 to 450 °C, is characterized, and a grain growth exponent of $n = 5$, indicating inhibition of grain growth, is observed. Broadening of the normalized grain size distributions, which indicates abnormal grain growth, was observed at all temperatures investigated. It is shown, using a Zener-type analysis for pinning of grain boundaries by particles, that impurity-based particles are responsible for grain growth inhibition and abnormal grain growth. The strong basal texture which develops during rolling of the Mg alloy, resulting in an initial peak intensity in the (0002) pole figure of nine multiples of a random distribution (MRD), increases to ~15 MRD during annealing at 400 and 450 °C. Moreover, a specific texture component $\{0001\}\langle 11\bar{2}0\rangle$ is observed in the orientation distribution, which increases from 10 to 23 MRD at 400 °C. It is hypothesized that the anisotropic grain boundary properties (i.e. low angle boundaries have low energy and mobility) are responsible for the texture strengthening. Additionally, electron backscattered diffraction reveals the recrystallized microstructure to contain a significant number of boundaries with ~30° misorientation about the $\langle 0001\rangle$ direction, and this boundary type persists throughout most annealing treatments explored.

Keywords: Grain size distribution; Grain boundary; Orientation distribution; Abnormal grain growth; Anomalous grain growth

1. Introduction

Rolled magnesium alloy sheets typically exhibit tension–compression yield strength asymmetry due to the presence of a strong basal texture, with the *c*-axis of the crystal parallel to the through-thickness direction of the sheet. This contributes to poor cold formability and hinders broader application of these lightweight materials. For some applications, thermomechanical processing is followed by various annealing schedules in order to obtain a recrystallized microstructure, to alter the texture or to relieve residual stresses. In the case of Mg alloys, it has been found that the basal texture is retained after recovery and recrystallization annealing [1]. In this way, hexagonal close-packed (hcp) Mg alloys are more similar to many body-centered cubic alloys than they are to face-centered cubic (fcc) alloys, since the latter frequently undergo strong crystallographic texture evolution during recrystallization [2]. Even though a considerable amount of research has been devoted to developing an understanding of the recrystallization behavior of Mg alloys (e.g. [3]), little work has been done to explore the effect of grain growth annealing on microstructure and texture evolution.

This paper documents the kinetics of grain size evolution during annealing, including the abnormal grain growth phenomenon that has been observed in this alloy [4] and the texture evolution during grain growth. Subsequently, the possibility that these two phenomena (anomalous grain growth and texture strengthening) are correlated is explored. Finally, the role of grain boundary character distribution (GBCD) on the mechanism of texture evolution is considered. AZ31B was chosen for this study to assist in the practical application of the findings. In such a commercial alloy, complicating factors threaten to obscure the desired information (for example, the solute elements and second-phase particles will be shown to play an important role in determining grain growth behavior). However, it has proven possible to draw conclusions from the observations.

2. Experimental methods

2.1. Material, heat treatment and metallography

The material investigated was part of a larger study focused on the effect of shear rolling on the microstructure and properties of magnesium alloys. Alloy AZ31B sheet of thickness 1.52 mm was obtained from Magnesium Elektron North America and shear rolled at Oak Ridge National Laboratory, with both the rolls and the metal held at 200 °C. The details of the processing are described elsewhere [5]. One conclusion of that broader study was that the range of shear-rolling conditions explored induces

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quantitative changes in the resulting basal texture of the sheet, such as a slight asymmetric tilting of the basal poles from the sheet normal direction (ND) toward the rolling direction (RD) and broadening of the basal peak toward the transverse direction (TD), which induces some effect on the anisotropy of properties between the RD and TD [5]. However, the qualitative appearance of the textures is largely unchanged. As such, the fact that the material was shear (rather than conventionally) rolled is not viewed as an important aspect in what follows.

Samples were then heat-treated at temperatures ranging from 260 to 450 °C for durations ranging from 1 to 604,800 s (7 days) in order to produce samples of varying grain size (5–50 μm). The short duration anneals ($t < 960$ s (16 min)) were performed in a KNO_3 salt bath. For all other annealing durations, an air furnace was used. All the samples were quenched in room-temperature water after annealing. For optical and scanning electron microscopy (SEM) based metallographic examination, the samples were ground to the sheet mid-plane, cold mounted in epoxy and mechanically ground up to 1200 grit SiC paper, followed by 3 and 1 μm oil-based diamond paste and finally 0.06 μm colloidal silica polishing. The specimens were etched in an acetal–picral solution (4.2 g of picric acid, 10 ml of acetic acid, 70 ml of ethanol and 10 ml of water) for 5–10 s.

The grain size is measured from optical micrographs using the lineal intercept method in MATLAB toolbox Linecut, following the ASTM Standard E112-96 [6]. The measured grain sizes were plotted as histograms using Origin software, with a bin size of 1 μm, and then fitted with the log normal distribution to obtain the arithmetic mean grain size. The data were then normalized by this mean, plotted as histograms with a bin size 0.2 μm and then fitted with a Gaussian distribution to obtain the breadth of the normalized distribution. During normal grain growth, the breadth of this distribution remains invariant [7]. Any evolution of this breadth is used as an indicator of abnormal grain growth [8,9].

2.2. Texture measurements

The texture was measured on the as-received sheet and after all annealing heat treatments. The measurements were made on the sheet mid-plane, to obtain the bulk texture and avoid any surface effects, such as a near-surface texture gradient, which has been observed previously [10]. The measurements were made using a Panalytical X'pert Pro multi-purpose diffractometer with Cu K_α radiation operating at 40 kV and 45 mA, in Schultz reflection method [11] to minimize defocusing at higher χ tilts. An incomplete ($\chi = 0$–80°) basal (0002) pole figure was obtained for all annealing conditions. An experimental defocusing curve was obtained from a randomly textured Ti powder sample. Background subtraction, defocusing correction and normalization were carried out using MATLAB toolbox MTeX. The raw data was smoothed using a Gaussian filter of 5° [12] in order to get rid of spurious intensity spikes. For selected annealing conditions, the $(10\bar{1}0)$, (0002), and $(10\bar{1}1)$ incomplete pole figures were collected and the texture data were analyzed using MTeX to generate complete orientation distributions (ODFs) and full pole figures. In MTeX, the reference frame was set as X‖a, Y‖b* and Z‖c, such that the RD is aligned with the $\langle 11\bar{2}0\rangle$ of the crystal for the Euler angles (0°, 0°, 0°).

2.3. Second-phase particle characterization

To evaluate the evolution of the second-phase particles during annealing, the volume fraction and the particle size were measured using a JEOL 6700 scanning electron microscope in secondary electron (SE) and backscattered electron (BSE) mode. The composition was obtained from energy-dispersive spectroscopy (EDS), using the Oxford-Pgt Spirit EDS software. The particle size was measured using a lineal intercept technique from the SEM–BSE images. The volume fraction was obtained based on the stereological concept [13] that, in a randomly obtained planar section, the area fraction occupied by the particles is equal to the volume fraction of the particles. In order to obtain the area fraction, multiple (6–8) appropriately thresholded SEM images were obtained using analysis software, ImageJ.

2.4. Orientation imaging maps

After carrying out standard metallographic preparation as described above, the samples chosen for electron backscattered diffraction (EBSD) were cleaned with a plasma etching and coating system to remove the deformation layer and facilitate indexing. EBSD was performed within a FEI Quanta 200FEG scanning electron microscope equipped with an EDAX/TSL system with a Hikari EBSD detector. The objective of these experiments was to determine the character of the grain boundaries present in the system and to obtain the grain boundary misorientation distribution. The electron microscope was operated at 20 kV, with a working distance of 15 mm at a magnification of 500×. The step size and the spot size during EBSD data collection were 0.5 μm and 5.5 (corresponding to a beam current of 11 nA), respectively. The confidence index of the diffraction patterns was between 0.5 and 0.7, indicating that the quality of pattern identification was 72–87% for the samples examined.

3. Results

3.1. Characterization of as-rolled AZ31B sheet

The microstructure of the as-received sheet material is relatively fine grained and has few deformation twins (Fig. 1a). The grain size distribution is observed to be log normal (Fig. 1b), with a mean lineal intercept grain size of 5.3 μm, and the width of the initial normalized grain size distribution is 0.55 (Fig. 1c). The fine grains and low twin density are evidence of dynamic recrystallization during hot rolling of the sheet. In addition, a few fine second-phase particles and some larger (∼2 μm) particles, revealed by SEM–EDS to be Al–Mn compounds, are present in the optical image. The $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$ recalculated full pole figures obtained from the orientation distribution of the as-received sheet are shown in Fig. 1d. From these pole figures, it is seen that the (0002) basal poles are nearly aligned with the sheet normal direction. The maximum intensity is ∼9 multiples of a random distribution in the (0002) pole figure, with a slight spread towards the rolling direction, in accordance with previous observations of shear-rolled Mg alloy sheets [14]. Notably, the $(10\bar{1}0)$ pole figure exhibits near-radial symmetry, but complete orientation distribution analysis (shown below) revealed that there is a slight preferred orientation.