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Orientation dependent recrystallization mechanism during static annealing of pure magnesium

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ABSTRACT

In the present study, orientation dependent recrystallization mechanism in pure magnesium is discussed in light of the experimental results. Commercially pure magnesium was subjected to cold rolling of 90% reduction in thickness followed by annealing at 200 °C for different soaking times. Nucleation of no newly oriented grains could be observed during annealing. However, the rate of very low angle grain boundary (VLAGB) movement was found to be the rate controlling step for the formation of recrystallized grains. The formation of sub-grains from the parent grain was observed during annealing of the samples irrespective of the orientation of the grains. However, the rate of sub-grain formation was found to be fastest in the grains of orientations $> 40°$ from the normal direction (ND) of the sample. It was further observed that the growth rate of orientations/grains was decreased with increasing their deviation from ND of the sample. A dominant basal texture was observed in the samples and the maximum weakening of basal texture was observed during 300 s of annealing time.

1. Introduction

Poor formability at room temperature deformation of magnesium (Mg) and its alloys restricts their use in different structural applications such as automobiles and aerospace [1–3]. This is attributed to an inadequate number of slip systems in Mg and its alloys [4,5]. Attempts have been made in the past to improve the formability of Mg through different thermo-mechanical processing, such as extrusion, asymmetric rolling, equal channel angular processing, etc. [6–16]. At room temperature the critical resolved shear stress (CRSS) for basal slip is lower than that of non-basal slip, such as the prismatic and pyramidal slip systems [17–19]. The basal slip system alone does not provide the required independent slip systems to satisfy the Von Mises criterion of five independent slip systems for homogeneous deformation. This means that extension twinning is required to accommodate deformation at room temperature. With increasing temperature, the CRSS value quickly decreases for non-basal slip systems and activation of higher order slip systems is observed [17–19]. Variation of the processing conditions and addition of alloying elements are also expected to regulate the texture of Mg for improving its formability. For example, addition of rare earth elements and Ca (calcium) to Mg weakens the basal texture after recrystallization and increase the ductility as well as formability of the Mg alloys [15,16,17,20–26].

The weakening of the recrystallization texture is related to the factors like particle simulated nucleation (PSN), shear band induced nucleation (SBIN) and deformation twin induced nucleation (DTIN) [26–38]. Further, it has been reported that the recrystallization texture does not dependent on the orientation of the nuclei only but on the growth of the specific orientation also. The preferential growth of the recrystallized grains with their c-axis parallel to ND has been reported in pure Mg [39] and AZ31 sheet [40,41]. Most of the above workers have attributed the weakening of texture to its nucleation phenomenon during recrystallization in Mg alloys. Recently, nucleation of no new orientations/grains was reported by R. K. Sabat and S.K. Sahoo [42] during static recrystallization of pure Mg. Nucleation of no new orientations at the twin-twin intersection was also reported by Sabat et al. [43] through ‘ex situ’ EBSD (electron backscattered diffraction) investigation. The authors [42] have also been reported that the rotation of sub-grains through 10–30° with respect to the c-axis of the parent grain during annealing. However, the number of grains studied by the authors was statistically low and there was no clear information about the rate of formation of sub-grains in the parent grains of different orientations and their effect on the bulk texture during annealing [42]. Hence, an experiment was designed to study the effect of initial orientation on the rate of formation of sub-grains during annealing of the cold rolled pure Mg at 200 °C for varying annealing times. The detailed

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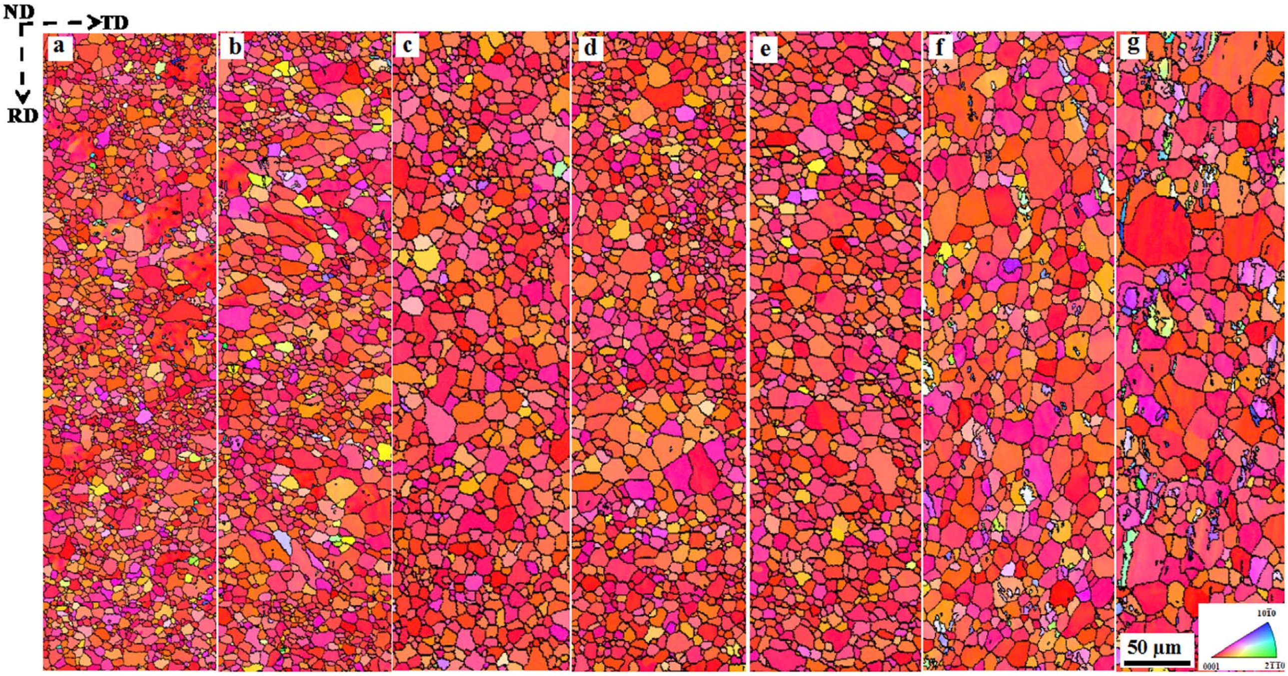

Fig. 1. IPF maps of the samples after annealing for different annealing times: (a) 10 s, (b) 30 s, (c) 120 c, (d) 300 s, (e) 600 s, (f) 1800 s and (g) 3600 s.

microstructural analysis (including texture) has been carried out and reported in the present study.

2. Experimental

2.1. Material and Sample Preparation

Commercially pure Mg, obtained from General Motors, USA, was subjected to cold rolling of 90% reduction in thickness in a laboratory rolling mill. Such an unusually high reduction percentage in cold rolling of Mg was achieved through MSCR (multistep cross rolling) [44]. The rolled samples were then subjected to annealing at 200 °C for 10 s, 30 s, 120 s, 300 s, 600 s, 1800 s and 3600 s of soaking times respectively. Annealing was carried out in a tubular furnace under inert (Argon) atmosphere. The desired temperature of 200 °C was attained with a heating rate of 5 °C per sec. The samples were kept inside the furnace at 200 °C for different soaking time followed by air cooling. The rolled and annealed samples were metallographically polished followed by electro-polishing for subsequent textural and microstructural characterizations. Standard methods were adopted for metallographic polishing whereas electro-polishing was carried out using an electrolyte containing mixture of ethanol to ortho-phosphoric acid by 3:5 ratio (by volume) at 0 °C. Electro-polishing was carried out at 3 V for 30 s and subsequently at 1.5 V for 2 min.

2.2. X-Ray Diffraction (XRD)

XRD was carried out in a Bruker D8-Discover system using CuK$_\alpha$ radiation. Six pole figures, (0002), $(10\bar{1}0)$, $(10\bar{1}1)$, $(10\bar{1}2)$, $(10\bar{1}3)$and $(11\bar{2}0)$, were measured on the ND plane containing the RD-TD (rolling direction-transverse direction). Orientation distribution functions (ODFs) were estimated using the MTEX software [45].

2.3. Electron Backscattered Diffraction (EBSD)

EBSD was carried out on a FEI-Quanta 200-HV SEM (scanning electron microscope). Data acquisition and analyses were performed by using the TSL-OIM™ version 6.0 software. Beam and video conditions were kept identical between the scans and a step size of 0.2 μm was used. The average confidence index (CI) was kept > 0.5 for all the scans. However, the EBSD analyses were performed only for points with CI > 0.1.

3. Results and Discussions

3.1. Microstructure Evolution

Fig. 1 shows the inverse pole figure (IPF) maps of the Mg samples at different annealing conditions. It may be noted that the EBSD study was infeasible for the cold rolled sample before annealing because the diffraction patterns were too indistinct. Hence, the EBSD has been carried out only for the annealed samples. The short term annealed samples i.e.

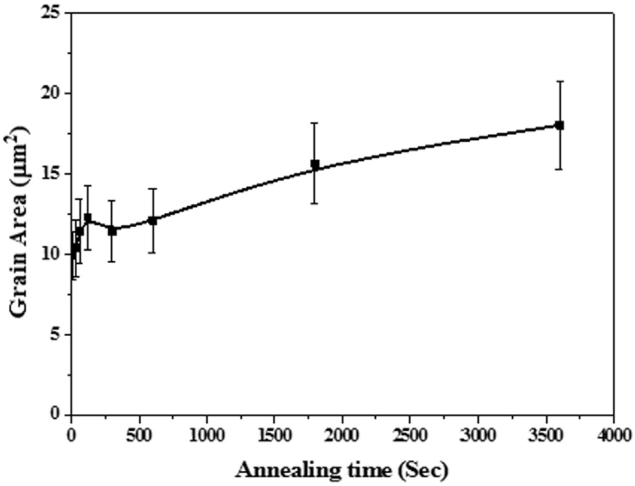

Fig. 2. Average grain size of the samples as a function of annealing time.