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Growth mechanism of extension twin variants during annealing of pure magnesium: An 'ex situ' electron backscattered diffraction investigation

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ABSTRACT

Pure magnesium was subjected to plastic deformation through CSM (continuous stiffness measurement) indentation followed by annealing at 200 °C for 30 min. Nucleation of no new grains was observed neither at the twin–twin intersections nor at the multiple twin variants of a grain after annealing. Significant growth of off-basal twin orientation compared to basal twin orientation was observed in the sample after annealing and is attributed to the partial coherent nature of twin boundary in the later case. Further, growth of twins was independent of the strain distribution between parent and twinned grains.

1. Introduction

Twinning plays an important role during plastic deformation of magnesium (Mg) and its alloys [1,2]. This has often been attributed to limited number of slip systems available in these materials at room temperature deformation. Two types of deformation twins are mainly observed: extension twinning, $\{10\bar{1}2\}$ [3] and contraction twinning, $\{10\bar{1}1\}$ or $\{10\bar{1}3\}$ [4]. Formation of extension twin requires a low value of critical resolved shear stress (CRSS) whereas contraction twin requires a high value of CRSS [5]. Contraction twins generally form in the localized regions and serve as effective sites for nucleation [6–8]. However, strain accumulation inside the extension twins is low and hence becomes resistant to nucleation during annealing [6]. The coherent structure of extension twin boundaries makes the boundary stable during annealing. Recently Nie and Xin [9,10] proved that annealing treatment leads to segregation of solute atoms at twin boundaries in Mg alloy and reduce the thermal activation further. Stability of extension twin boundaries is also reported by Levinson et al. [11] and Li et al. [6] in AZ31 sheet. However, no work related to nucleation and growth of specific extension twin variant during static recrystallization is reported in the literature. Keeping this in mind, the nucleation and growth of extension twin variants during static recrystallization have been investigated in the present study.

2. Experimental Details

Commercially pure magnesium, obtained from General Motors, USA, was subjected to hot rolling at 250 °C to impart 90% reduction in thickness followed by annealing at 400 °C for 1 h. The annealed sample was then metallographic polished. A scratch of 1 mm length was made on the polished surface using a CSM (Continuous Stiffness Measurement) indentation machine at a speed of 6 mm/min with constant load of 10 N. From this point onwards the scratched sample is reported as deformed sample in this report. The deformed sample was then annealed at 200 °C in a tubular furnace for 30 min of soaking time. Electron back scattered diffraction (EBSD) was carried out on the sample before and after annealing. It may be noted that EBSD was performed on the same region of the sample before and after annealing. This process is known as 'ex situ' annealing in contrast to in situ annealing [12]. Only deformed sample was electro-polished before EBSD measurement as EBSD could be performed after annealing of the deformed sample without electro-polishing. Electro-polishing was carried out using an electrolyte containing the mixture of ortho-phosphoric acid to ethanol (of 3:5) at 3 V for 30 s and 1.5 V for 2–3 min.

EBSD was carried out on a FEI-Quanta 200-HV SEM (scanning electron microscope). Data acquisition and analyses were performed using the TSL-OIM version 6.0 software. Beam and video conditions were kept identical between the scans and a step size of 0.5 μm was used during the scans. The black region in the inverse pole figure (IPF) map was identified as non-indexed region with confidence index (CI) < 0.1. These regions are attributed to the highly deformed region of the material. Grain size was measured with the TSL software as the circle-equivalent

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diameter based on scan area for each grain. The misorientation in a grain is represented as grain orientation spread (GOS) and kernel average misorientation. GOS is defined as the misorientation between all measurement points of a grain and the grain average orientation. Similarly, KAM is the local misorientation between neighbouring points in a grain and is defined as the average misorientation of a point with all of its neighbours with the proviso that misorientations exceeding some tolerance value (maximum misorientations) are excluded from the averaging calculation [13,14]. Extension twin was estimated as rotation of the c-axis of parent grain by 86° about <$11\bar{2}0$> direction. The nature of twin boundary was measured by plotting the boundary trace normal at the parent – twin interface. The common plane between the parent and twin grain was measured from the EBSD map using the TSL software. The pole figure for the common plane was plotted for both the twin and parent grains, and boundary trace normal was then superimposed in the pole figure. If the boundary trace normal passes through the parent and twin orientation in the pole figure, it is known as coherent boundary or else it is incoherent boundary [15].

3. Results and Discussion

Fig. 1 shows the IPF maps and corresponding (0002) & ($10\bar{1}0$) pole figures of the sample before and after annealing at 200 °C for 30 min of soaking time. The initial hot rolled and annealed microstructure along with its (0002) pole figure is also presented in Fig. 1. The initial sample had a strong basal texture with strain free grains of ~30 μm average grain size. The extension twins of {$10\bar{1}2$} type were observed in the sample (Fig. 1a and b). The texture in the form of (0002) and ($10\bar{1}0$) pole figures for deformed and annealed samples are shown in Fig. 1c and Fig. 1d respectively. Apart from basal texture components, the presence of off-basal texture components may be observed in both the samples. However, no significant change in texture components was observed in the sample after annealing. An increase in maximum texture intensity after annealing of the sample may be observed which indicates that there might be the growth of the initial grains/orientations. Hence, a detailed microstructural analysis has been carried out at different locations of the sample.

The microstructure of the deformed and annealed sample was partitioned mainly into near basal grains (marked as '1', '2' and '3' in Fig. 1), near prismatic grains (marked as '4' and '5' in Fig. 1) depending on the orientation of c-axes with respect to normal direction (ND) of the sample. The magnified view of basal grains superimposed with high angle grain boundaries of the sample before and after annealing is shown in Fig. 2. The orientation relationship between the parent grain (marked as filled circle) and twinned grain (marked as filled rectangle) is shown in Fig. 2e where the orientations are represented in a (0002) pole figure. It was noticed that nearly 86° orientation relationship exists between the parent grain and the twinned grain. Further, significant growth of these extension twins was observed in the basal grains (grains numbered as 1, 2 & 3 in Fig. 2) after annealing. The orientation of these grown twins was already present in the deformed sample before annealing, as shown by hcp (hexagonal close packed) unit cells in Fig. 2a and c. The growth of deformation twinning was attributed to the incoherent nature of extension twin boundary [15] which was identified for the grain marked as '3' and is shown in Fig. 2f. The dashed black arrow mark represents the boundary trace normal, red and blue circle corresponds to the orientations of the parent and twinned grains in

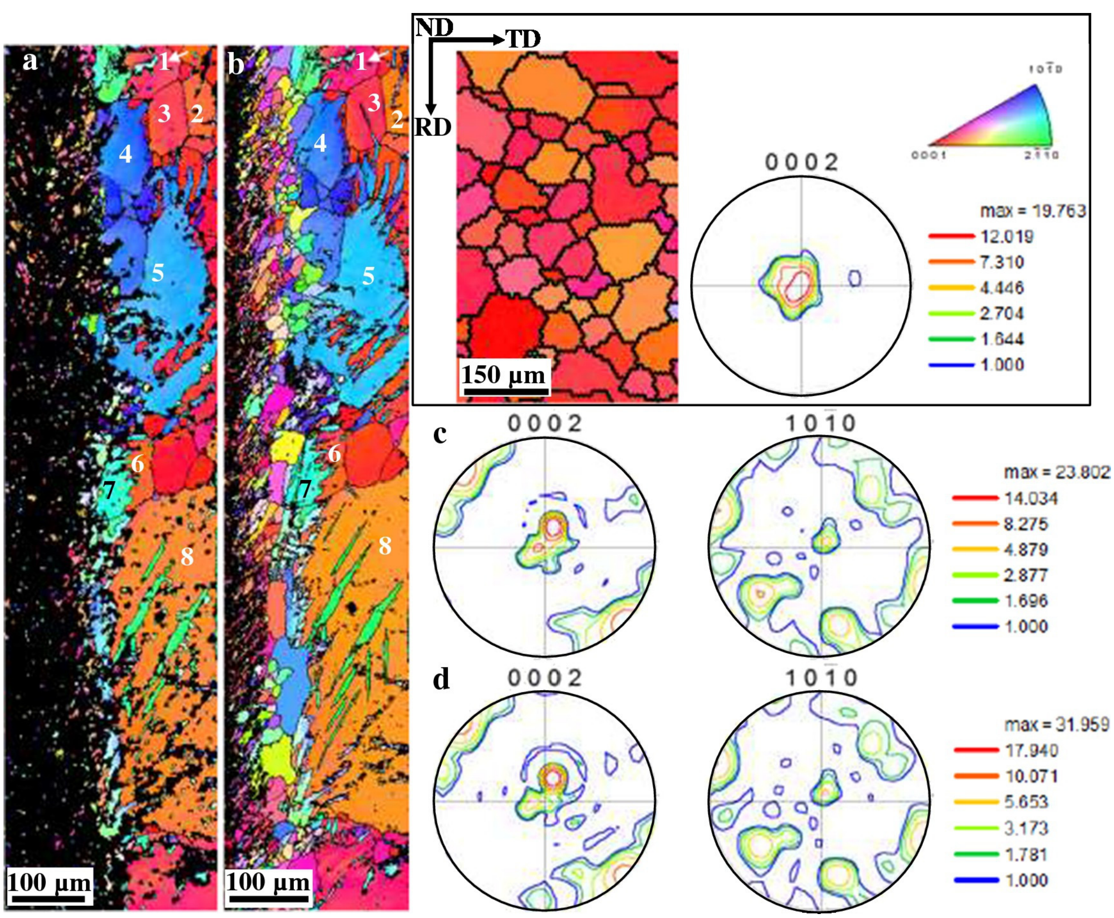

Fig. 1. IPF maps and texture of the sample before and after annealing: (a, c) deformed sample adjacent to the scratch before annealing; (b, d) same sample after annealing. Rectangle box: IPF map and texture of the initial hot rolled and annealed sample. The grains marked as '1–8' are further presented in subsequent figures.