In Situ Electron Backscatter Diffraction Analysis for Microstructure Evolution and Deformation Models of Mg–Ce Alloy During Uniaxial Loading

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Abstract

Previous studies showed that significant increases in elongation in Mg–Ce alloys due to the Ce addition and the solute drag effect by Ce addition were ascribed to the non-basal dislocation slip activating and the texture altering. The microstructure evolution and deformation models of extruded Mg-0.5 wt%Ce alloy rods under uniaxial tension have been studied using in situ electron backscatter diffraction. The basal and non-basal slips were characterized by using slip line trace analysis. The results provide evidence for that pyramidal slip activated during deformation, besides basal slip and extension twinning, which contributes to the texture weakening and ductility increasing in Mg-0.5 wt%Ce alloy.

Keywords Texture · Non-basal slip · Twinning · In situ electron backscattered diffraction (EBSD) · Trace analysis

1 Introduction

Magnesium alloys have much less formability than steel or aluminum alloys at room temperature due to their hexagonal (hcp) crystal structure with limited slip systems. There are increasing attempts to enhance the workability and ductility of magnesium alloys by grain refinement, alloying and texture modification [1–5] to overcome the inherent limitation. The texture of Mg alloy could be randomized by Y, Nd, Gd and Ce additions [3, 5–10] as reported, and the elongation of Mg–Ce extruded rods at room temperature could be drastically improved to 32%, which was ascribed to that the Ce addition altering the texture and therefore basal slips is more easily operated [3]. Texture modification had actually contributed to the ductility improvement in Mg alloys due to the high Schmid factors of basal $\langle a \rangle$ slip [11]; however, twinning and $\langle c+a \rangle$ slip, which can provide the strain along [0001] axis, were considered to be necessary deformation model for the compatibility of Mg alloys [12]. While

the critical resolved shear stress (CRSS) for $\langle c+a \rangle$ slip in magnesium is about 2.5 times higher than that for $1/3\langle 11-20 \rangle$ basal slip [13], the $\langle c+a \rangle$ slip should be more difficult activated during deformation in pure Mg or AZ31 alloy at room temperature. Chino et al. reported that the unique compression behavior of Mg-0.2%Ce was ascribed to non-basal slips [14], and more discussion had been focused on the solute drag effect with rare earth addition in Mg alloy and then its effect on the activation of non-basal dislocation slip [15]. However, the deformation models of Mg–Ce binary alloy are still unclear, and it is interesting to characterize the deformation models during deformation, and the results could help to understand the reason for the texture evolution and ductility improvement in Mg–Ce alloy.

The present work aims to study the deformation models of Mg-0.5 wt%Ce alloy at uniaxial tension loading using in situ electron backscattered diffraction (EBSD). The twinning, the basal $\langle a \rangle$ dislocation or $\langle c+a \rangle$ non-basal slips will be characterized, and the correlation between initial microstructure and deformation models is discussed.

2 Experimental

The material studied in the current work is Mg-0.5 wt%Ce magnesium alloy rods (marked as Mg–Ce alloy) prepared by hot extrusion. The microstructure evolution of the sample

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during tensile test was observed by in situ EBSD. A micro-test system provided by Deben UK Limited with a load cell of 5 kN capacity, installed inside a LEOTM 1450 scanning electron microscope (SEM) fitted with a TSLTM EBSD camera, was used for the experiments. Figure 1 shows the schematic view of the in situ tensile test and the stress–strain curve during test. The tensile direction (TD) is parallel to the extrusion direction (ED) of the sample. Polishing was done before in situ tensile test on the observation planes of tension samples using a sequence of ethanol-based diamond suspensions of 6, 3 and 1 μm , respectively. This was followed by fine polishing using colloidal silica suspension (OPS) and a final 2–4 s etching using a solution of 5% HNO_3 , 15% acetic acid, 20% H_2O and 60% ethanol before SEM and EBSD observations. The sample was tensional at room temperature at the displacement rate of 0.1 mm/s, corresponding to a strain rate of 0.0005 s^{-1} , and the polished surface of the sample was tilted by 70° to face the EBSD detector. The tensile test was carried out in steps. EBSD patterns from the same area of the sample surface were collected by stopping loading at extension intervals of 0.1 mm and holding the sample under load for the duration of the EBSD data collection, typically lasting 30 min. The loading was stopped at the strain of 23% due to the weakening Kikuchi pattern with the increasing stain.

After the data collection in EBSD, dates with confidential index (CI) value >0.3 have been taken into account for data analysis by OIM software, and the inverse pole figure (IPF) map, band contrast map and the (0001), (10-10), (1-210) and (11-2-2) pole figures of same area in the sample were processed. To permit identification of active slip systems, the slip trace inclination to the tensile direction was measured from SEM micrographs after tensile strain 23%, and the crystallographic orientation was determined by EBSD

Fig. 1 Stress–strain curve of Mg–Ce alloy during in situ tensile loading

mapping. Twinning in grains 1–5 and dislocation slip in grains A–K were examined totally (see Fig. 2 for the initial microstructures). The twinning boundaries are marked red, green or blue depending on the rotation of the lattice across the twin boundary. Extension twin boundaries ($86^\circ < 1-210 > \pm 5^\circ$), contraction twin boundaries ($56^\circ < 1-210 > \pm 5^\circ$) and double twin boundaries ($38^\circ < 1-210 > \pm 5^\circ$) are outlined in red, green and blue, respectively.

3 Results and Discussion

Figure 1 shows the stress–strain curves of Mg–Ce alloy under in situ (interrupted) tensile test conditions. The applied stresses at strain of 3%, 6%, 15%, 20% and 23% dropped down where the loading had been stopped for data collection in EBSD. The strain hardening behavior in the sample is similar as that for Mg–Ce alloy [2], suggesting that the grain structure evolution obtained from the in situ EBSD could be representative of the microstructure evolution during uninterrupted testing.

Figure 2 shows the IPF maps of Mg–Ce alloy at strains of 0, 3%, 6%, 15%, 20% and 23%, and Fig. 3 shows the Kikuchi band contrast maps with characterized twins at strains of 0, 6% and 15%. {10-12} twinning was identified in grain 1 to grain 5 at strain of 6% according to the IPF map (Fig. 2b) and the contrast map (Fig. 3b), where {10-12} twin boundaries were outlined by red line. Those twins will grow with increasing strain. Few {10-11}–{10-12} double twins and {10-11} contraction twins were also observed on the sample at strain of 3%, 6% and 15% (Fig. 3), but double twins and contraction twins always appear thin and long [16], and then, the relative frequencies of the twins were not able to be determined. Besides, the increasing misorientation with increasing strain in grains in the IPF maps suggests that dislocation slips were operated in most grains. The deformation resulted in the rough smooth and then low EBSD indexing rates [17]. More black areas ($\text{CI} < 0.3$) appear in Fig. 2 with increasing tensile strain.

Figure 4 shows the SEM micrographs of the Mg–Ce alloy at strain of 23%, and the slip trace in grains A–K was measured from the SEM images. Figure 4 also shows the crystallographic orientations of grains A–K which were determined by EBSD mapping as shown in Fig. 2a, and the ideal trace of the intersection of the basal, prismatic and pyramidal planes was inspected in the pole figure for each grain, and after comparing the ideal trace with the slip trace on the grain surface, the possible slip systems were determined and Fig. 4 only provides the pole figures of the slip plane for the grain A to grain K. For example, only {10-11} pole figure of grain C was provided here because the dislocation slip occurred on its (10-11) plane. The results suggested that the basal slip was activated in grains B, D and E and the dislocation slip

Fig. 2 IPF maps of Mg–Ce alloy at different tensile strains of **a** 0, **b** 3%, **c** 6%, **d** 15%, **e** 20%, **f** 23%. The tensile direction (TD) is parallel to the extrusion axis (ED). The colors in the IPF maps represent

the orientations of the normal direction of the grains according to the color key. And those grains and boundaries in which its CI value is lower than 0.3 in the data collection were shown as black

Fig. 3 Kikuchi band contrast maps of Mg–Ce alloy after strain of **a** 0, **b** 6%, **c** 15%. Color key for image quality map: the {10-12} extension twin boundaries ($86^\circ\langle 1-210\rangle\pm 5^\circ$) are outlined in red, the {10-

11} contraction twin boundaries ($56^\circ\langle 1-210\rangle\pm 5^\circ$) outlined as green and the {10-11}–{10-12} double twins boundaries ($38^\circ\langle 1-210\rangle\pm 5^\circ$) outlined as blue

on (10-11) plane was activated in grains C, G and H, and the dislocation slip on (11-22) plane was activated in grains A, J and I. In grain K, no slip trace was observed and the deformation occurred on the grain boundaries supported by the voids on the grain boundaries between grain K and its neighbor grains. The SEM micrographs also show that the surface was roughened after tension deformation. Except for the slip trace in the grain interior, most displacement of the surface occurred along the original grain boundaries.

{10-12} extension twinning, {10-11} contraction and {10-11}–{10-12} double twinning are the most possible twinning models in the deformation of magnesium alloy, and at the tension loading, contraction and double twinning are preferred for lot of commercial magnesium alloy, such as AZ31 [18], because there is contraction along the *c*-axis when typical ring basal texture exists in the AZ31 alloy [19]. At this loading condition, few contraction and double twinning were observed in the Mg-0.5%Ce alloy according to the Kikuchi band map in Fig. 3b. And the volume fraction of twinning in this sample is quite low, and most of them are {10-12} extension twinning. There are two possible reasons responsible for the low twinning volume fraction. Firstly, the main texture of the Mg–Ce alloy showed that the *c*-axis has near 65° – 75° with the ED (Fig. 4a), and few grains have their *c*-axis under contraction, and the CRSS for contraction twinning is much higher than that for extension twinning [20]; therefore, dislocation slip and few extension twinning are suppressed under this loading condition. Secondly, the

contraction and double twinning always were observed at higher strain deformation due to the higher CRSS, but it was difficult to index the twinning boundaries by EBSD mapping with increasing strain. However, we still can confirm that there was low volume fraction of contraction and double twins in this Mg–Ce alloy compared to other alloys with ring basal texture because more slip systems were activated at this loading condition.

Slip on basal $\{0001\}\langle 11-20\rangle$ and $\{10-12\}\langle -1011\rangle$ extension twinning were considered as dominant deformation models in the deformation of magnesium at room temperature. As mentioned before, the increasing ductility in Mg–Ce alloy was ascribed to high Schmid factor value on basal slip [3]. In this observed area of Mg–Ce alloy, slips on basal, pyramidal {10-11} and {11-22} planes were determined together (Fig. 4) and the fraction of each slip is close to each other, but no {10-10} slip was observed in the observed grains. Those non-basal slips accommodate straining in the *c*-direction and improve the deformation compatibility of the Mg–Ce alloy. Prismatic $\{10-10\}\langle 11-20\rangle$ and pyramidal $\{10-11\}\langle 11-20\rangle$ and also $\langle c+a\rangle$ slip $\{11-22\}\langle -1-123\rangle$ had been thought as an accommodation mechanism [21] for magnesium alloy at room and elevate temperatures, but the fraction of non-basal slip by total strain is quite low in most wrought alloy. Figure 4 shows the equivalent fraction of non-basal slip compared to basal slip in those observed grains A–K of Mg–Ce alloy, which suggests that Ce addition somehow changes the activation

Fig. 4 Slip traces and accompanying pole figures illustrating the method used to identify the most likely active deformation mode after strain of 23%. The line on the pole figures corresponds to the trace of the slip plane (arrowed pole) closest to the observed slip trace

of deformation modes on the pyramidal planes in Mg. And recently, study on Mg–Y alloy also reported a high occurrence of $\langle c+a \rangle$ slip and increased activation of compression/secondary twins [7, 15]. But the actual reason for this higher activation remains unclear. In our previous work, the segregation of Gd near dislocation core could be ascribed to the obstacle of dislocation slip on basal system and then

promoted the activation of non-basal slip [22], and in the study of Mg–Li single crystal deformation at room temperature, evidence for $\langle c+a \rangle$ slip was also found [23] which ascribed to c -axis/ a -axis ratio changed by Li element addition [24] and lower CRSS of $\langle c+a \rangle$ slip than $\{10\text{--}10\} \langle a \rangle$ slip [23]. Having no evidence to support that the c -axis/ a -axis ratio was changed with the Ce addition so far, however,

there is still possible reason to believe that some physical properties of the Mg–Ce solute had been changed by Ce addition, such as the stacking fault of on basal and non-basal plane of Mg, which resulted in the changing of CRSS on non-basal plane. More investigated methods should be taken into account to clear the mechanism.

4 Conclusions

The in situ EBSD work shows few {10-12} extension twinning happened in Mg-0.5 wt%Ce alloy under tensile deformation. Contraction and double twinning are less than extension twinning at low strain. Besides basal slip and extension twinning, the trace analysis and EBSD data provide evidence for the operation of {10-11} and {11-22} slip, which contribute to the weakening of texture and the increase in ductility at room temperature of Mg-0.5 wt%Ce binary alloy as well.

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