Precipitation Behavior and Its Effects on Mechanical Properties in a Pre-Twinned Mg–6Al–1Zn Alloy

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In the present study, an extruded Mg–6Al–1Zn alloy was pre-compressed along extrusion direction to generate sufficient tensile twins. Afterward, the pre-twinned samples were annealed at 170°C for aging. Precipitation of the second-phase particles was found to preferentially occur in twins and twin boundaries. The precipitation phase is proved to be Al12Mg17. Moreover, precipitation behaviors in the two twin boundaries on both sides of every twin were very different: a considerable number of second-phase particles precipitated in one twin boundary, but only a few second-phase particles were present in the other twin boundary. The atomic structures or internal stresses of the two twin boundaries are assumed to be different. A theoretical model of atomic structure was constructed to analyze the difference between the two twin boundaries. This model shows that atomic densities of the two twin boundaries are different, thereby resulting in varying precipitation behaviors. Owing to precipitation in twins and twin boundaries, an obvious increase in yield stress was observed when the material was reloaded along the same direction. [doi:10.2320/matertrans.M2017406]

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1. Introduction

When magnesium and its alloys are subjected to plastic deformation, in addition to dislocation slip, twinning is always activated to accommodate macroscopic strain, especially in magnesium alloys with sharp initial texture.1–3) Apart from accommodating plastic deformation, twinning can change the microstructure of materials. The main effects of twinning on microstructure are summarized as follows: (i) twinning changes the orientation, thereby causing further slip6,7) and (ii) twin lamellae subdivide grains, thus leading to grain refinement.8) The twinning models of {10–11}, {10–13}, {30–34}, {10–14} and double twinning {10–11}-type twin boundary in magnesium and its alloys.9) However, the most frequent twinning mode is believed to be the {10–12} twinning, which is on {10–12} plane and parallel to {10–11} direction. {10–12} twinning has been reported to have an important influence on the mechanical properties of magnesium alloys, such as high asymmetry between compression and tension, low yield stress, and low elongation. Twinning boundary plane, also known as twinning plane, is important in identifying twinning mode. The twinning plane is always considered to be invariant during twinning. Theoretically, atoms on the twinning boundary remain on their initial position during twinning. When twinning is finished, matrix and twin are believed to be the twinning boundary. Owing to the importance of {10–12} type twinning, an atomic arrangement of twin boundary was observed by a few researchers, and a series of results was provided. Zhang et al.3) performed a high-resolution TEM observation on {10–12} twin boundaries in hcp alloys. Their results indicate that twin boundaries did not lie on the {10–12} twinning plane, that is, the twin boundaries did not coincide with the twinning plane. However, their investigation on the {10–11} type twin boundary structure showed that the twinning plane was a flat and mirror plane.10) The shuffling domi
AZ61 were reported. A theoretical twin boundary structure was constructed to explain the precipitation behaviors. Moreover, effects of the precipitation behaviors on mechanical properties were discussed.

2. Research method

The starting material used in the present study is a commercially hot-extruded Mg–6Al–1Zn (AZ61) bar. The initial material was annealed at 400°C for 10 hours to obtain a homogeneous microstructure. The annealed material exhibits nearly equiaxed grains around 40 µm (Fig. 1(a)) and a strong ring fiber texture (Fig. 2(a)). Pre-compression along the extrusion direction (ED) was conducted to generate deformation twins and obtain pre-twinned samples. Samples for pre-compressive deformation with a length of 24 mm and 16 mm in diameter were cut from the annealed rod. Pre-compression were carried out on a CMT5105 material test machine at a constant rate of $10^{-3}$ s$^{-1}$. After pre-compression, samples were annealed at 170°C for aging at different times. Pre-twinned samples were cut by wire-electrode cutting for the preparation of cylindrical compression samples (8 mm in diameter and 12 mm in height) to investigate the effects of aging on mechanical properties. Afterward, mechanical tests were carried out on a CMT5105 material test machine at a constant rate of $10^{-3}$ s$^{-1}$. The yield strength for samples without obvious yield point was measured as 0.2% proof stress $\sigma_{0.2}$. Microstructures were observed using an accelerating voltage of 20 kV after careful polishing and etching with an acetic picral solution containing 5 ml acetic acid, 6 g picric acid, 10 ml H$_2$O, and 100 ml ethanol. The pole figures in this paper were measured using a Rigaku D/max-2500 X-ray diffraction to observe the longitudinal section via Cu $k_\alpha$ radiation (Wave length $\lambda = 0.15406$ nm) at 45 kv and 150 mA with a sample tilt angle ranging from $0^\circ$–$80^\circ$. And also, XRD patterns were obtained through the same machine at 40 kv and 200 mA for identifying the precipitation phase. Electron backscattered diffraction (EBSD) analysis was carried out to identify the twins on FEI Nova Nano SEM 450 scanning electron

Fig. 1 Microstructures of samples subjected to different plastic deformation levels: (a) 0%, (b) 2%, (c) 6%, and (d) 8%.

Fig. 2 {0002} and {10-10} pole figures for (a) extruded magnesium alloy AZ61 and (b) sample subjected to approximately 6% compression parallel to ED.
microscope equipped with a TSL OIM-EBSD system using a step size of 1.0 µm. Samples for EBSD were mechanically ground followed by electrochemical polishing in a commercial AC2 solution. The TSL OIM analysis software was utilized to process the data obtained from the electron backscattered diffraction. The indexing percentage in our experiment is 80%.

3. Results and Discussion

3.1 Structure and texture evolution during pre-compression

As shown in Fig. 1(a), no twin is evident in the samples before pre-compression. When the samples were subjected to 2% pre-compression along ED, Fig. 1(b) shows the appearance of a few twins in the materials. When pre-compression was increased to 6%, many twins appeared in the OM micrograph. However, when the prestrain continued to increase, the matrices were nearly consumed and the twin amount apparently decreased from the OM micrograph, as shown in Fig. 1(d). Many previous reports16–19 have indicated this trend in wrought magnesium alloys when they were subjected to compression along extrusion or rolling direction.

Figure 2 shows the inverse pole figure maps of samples with and without pre-compression. Figure 2(a) shows that when the sample was not subjected to pre-compression, nearly all the basal planes of the material were parallel to ED. EBSD analysis technology was used to identify the twin model in this study. The EBSD analysis results are shown in Fig. 3, which illustrates an obvious 86.3 ± 5° misorientation angle that points to the {10–12}-type twinning in magnesium. Owing to the occurrence of {10–12} twinning, the crystals were reorientated to a new orientation by nearly 90°, which is nearly perpendicular to the initial orientation. Therefore, Fig. 2(b) shows that when the sample was subjected to 6% pre-compression along ED, most of the basal planes were changed to be perpendicular to ED via twinning.

3.2 Precipitation behavior of the twinned material during aging and its effect factors

This study aims to investigate the precipitation behavior of the second phase and its effects on mechanical properties in a pre-twinned magnesium alloy AZ61; therefore, the selected aging temperature was 170°C to avoid recrystallization. According to previous studies,20,21 the twin structure, grain size, and texture can be well maintained under the selected temperature or a slightly high one. Therefore, during 170°C annealing, all the twin structures would not be destroyed, and the effects of grain growth and texture evolution on mechanical properties can be ignored. Furthermore, the precipitation behavior of the second phase in twins and twin boundaries can be investigated because the twin structures are well maintained.

Figure 4(a) illustrates the twin morphologies of a pre-twinned AZ61 alloy that was not subjected to 170°C annealing. Many lenticular shaped {10–12} twins exist, and no obvious precipitation was found in the sample. However, the other pre-twinned samples, which were subjected to annealing at 170°C for different time, exhibited different microstructures. When the pre-twinned sample was annealed at 170°C for three hours, a small quantity of second phase accrued in the twin boundary, as shown in Fig. 4(b). When the aging time was extended to 10 hours, many white second-phase particles precipitated in the twins and twin boundaries, as illustrated in Fig. 4(c). In order to investigate the chemical position of second phase, two samples were selected for XRD analysis.
analysis. XRD patterns of the initial extruded sample and the sample subjected to 6% pre-compression and then annealing at 170°C for 10 hours were shown in Fig. 5. It can be found that the second phase in this study is Al12Mg17. It is also illustrated that there is an increase in the second phase diffraction intensity if the sample is subjected to pre-strain and then annealing at 170°C. That indicates the increase in Al12Mg17 amount after pre-strain and low-temperature annealing. Although precipitates appear in twins and twin boundaries, it can be seen that no obvious precipitation of second phase accrued in the matrices under this heat treatment. Twinning was completed by atomic shearing and shuffling of magnesium and its alloys.\(^11,22-24\)

Theoretically, atoms in matrices can be considered immovable during twinning. However, atoms in twins move to new sites to complete twinning. Atoms are significantly accepted to be divided into shearing and shuffling atoms during twinning.\(^25,26\) Shuffling atoms are forced to move to new sites by shearing atoms and exhibit irregular movements during twinning. Thus, atomic arrangements in twins and twin boundaries are more disordered than matrices. Furthermore, internal stress often exist between atoms in twins, thereby leading to a good precipitation condition. Therefore, precipitation of second-phase particles preferentially accrued in twins and twin boundaries when the material was subjected to annealing at 170°C. As shown in Fig. 4(d), if the annealing time was prolonged to 20 hours, then additional second-phase particles will precipitate in the twins and twin boundaries.

Focusing on the precipitation behavior of the second phase shown in Figs. 4(c) and 4(d), different precipitation behaviors can be found in the two twin boundaries for a certain twin. As shown in Fig. 4, two twin boundaries are present for every twin. As shown in Figs. 4(c) and 4(d), a considerable number of white particles precipitated in one twin boundary for every twin, but only a few white second-phase particles are present in the other twin boundary. This phenomenon appeared in every twin presented in Fig. 4, which indicates that the atomic structure or stress condition may be different in the two twin boundaries for every twin. In general, the internal stress condition is often a function of atomic arrangement. Therefore, analyzing the atomic structures of the two twin boundaries is very important. An atomic model was constructed to compare the atomic structures for the two twin boundaries and understand their differences. First, a new rectangular coordinate system was adopted in a magnesium crystal lattice, as illustrated in Fig. 6(a). In this new coordinate, x-axis is along direction \([1011]\), y-axis is normal to the twin plane \((1102)\), and z-axis is along \([11\overline{2}0]\). Afterward, magnesium crystal lattices were projected from
the z-axis, as shown in Fig. 6(b). The black balls stand for the A-type atoms in the well-known stacking sequence $\ldots$ABAB$\ldots$ in hcp structure and the grey balls stand for the B-type atoms. After twinning, atoms in the twinned area moved to new sites to construct symmetric relationships relative to atoms of the matrix. Atomic motion vectors are indicated by arrows in Fig. 7(a). The black balls stand for the new sites of atoms after twinning. Figure 7(b) shows the theoretical symmetric relationship of atoms in twins and matrices. Regarding the appearance of many twins in materials, two twin boundaries are present for every twin. Atomic motion during twinning is shown in Fig. 8(a). The new sites of atoms after twinning are indicated by arrows in Fig. 8(a). A matrix-twin-matrix structure is formed when deformation is completed, as shown in Fig. 8(b). A total of two units were selected to analyze the difference between the two twin boundaries, as bolded in Fig. 9(b). The two units are signed as I and II. Every twin boundary can be formed by reduplicate stacking of the unit on the twinning plane. Distinctly, atomic volume in unit II is bigger than unit I, and a similar situation can be found in the three-dimensional space. That is, atomic stacking and internal stresses in the two twin boundaries are different. Hence, precipitation behaviors in the two twin boundaries are different. It is needed to note that the conclusion above is obtained just from theoretical analysis. Atomic arrangement in real twin boundary will be more complicated because there is always interaction of slip and twinning during deformation.

3.3 Effects of precipitation on mechanical properties

Annealing at 170°C for 10 hours was done first to a material without pre-compression to investigate the effect of aging on a material without pre-compression. As shown in Fig. 10(a), the two samples with and without 170°C annealing share the same yield stress when materials were not subjected to pre-compression, that is, annealing at 170°C for 10 hours has few effects on the mechanical property of an untwinned sample. The yield stresses for these samples are approximately 130 MPa. However, a difference in mechanical properties existed for materials subjected to annealing at 170°C for different times when the samples were subjected to pre-compression. Figure 10(b) shows the stress–strain curves of pre-compressive samples annealed under 170°C for different times. The yield stress of 6% pre-twinned sample is approximately 175 MPa, as illustrated in Fig. 10(b). Previous reports$^{16,27,28}$ have indicated that twin growth dominates deformation after twin nucleation and requires high stress to expand. Twin nucleation was completed during pre-compression. When the pre-twinned samples were reloaded along the same direction, twin growth dominated deformation.
As shown in Fig. 10(b), annealing has no obvious effect on the mechanical property when the pre-twinned sample was annealed under 170°C for three hours. The micrograph shown in Fig. 4(b) illustrates that no obvious precipitation accrued in the sample when the pre-twinned sample was annealed under 170°C for three hours. Therefore, no obvious effect was observed on mechanical property. However, an obvious increase in yield stress was observed when the pre-twinned sample was annealed under 170°C for 10 hours. This increase reached a value of approximately 215 MPa and was approximately 40 MPa higher than that of the sample without aging. As shown in Fig. 10(b), a slight but inconspicuous increase in yield stress compared to 10 hours was observed when the annealing time increased to 20 hours.

According to the results shown in Figs. 4 and 9, precipitation of the second phase in the pre-twinned materials has obvious effects on mechanical properties. Nie et al. reported an annealing hardening phenomenon in magnesium alloys due to the pinning effect of solute atoms on twin boundaries. In the present study, the second-phase particles precipitated in twins and twin boundaries, thereby blocking twin growth during recompression.

4. Conclusions

Pre-compression was operated in an extruded Mg–6Al–1Zn alloy to generate sufficient tensile twins. Afterward, the pre-twinned samples were annealed under 170°C for different aging times. Precipitation behavior and its effects on mechanical properties were investigated. The major conclusions are summarized as follows.

(1) When the pre-twinned samples were annealed under 170°C, the precipitation of second-phase particles preferentially occurred in twins and twin boundaries. Precipitation behaviors are different for the two twin boundaries on both sides of every twin.

(2) Theoretical analysis shows that atomic densities of the two twin boundaries on both sides of every twin are different: atomic density in one twin boundary is higher than that of the other twin boundary. Therefore, the precipitation behaviors in the two twin boundaries are different.

(3) Precipitation of the second-phase particles in twins and twin boundaries hinders twin expansion, thereby leading to an increase in yield stress when the samples were reloaded along the same direction.

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