Materials Science & Engineering A 644 (2015) 365–373

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

The different effects of solute segregation at twin boundaries on mechanical behaviors of twinning and detwinning

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ARTICLE INFO

Article history:
Received 15 June 2015
Received in revised form
16 July 2015
Accepted 17 July 2015
Available online 18 July 2015

Keywords:
Mg alloy
Solute segregation
Twinning
Detwinning
Hardening

ABSTRACT

In the present study, a comparative study about the hardening effect of solute segregation at $\{10\bar{1}2\}$ twin boundaries (TBs) on a $\{10\bar{1}2\}$ twinning predominant deformation and a detwinning predominant one was carried out. The influence of the pre-straining and subsequent annealing conditions on mechanical behavior was systematically addressed. Our results show that solute segregation at TBs can occur even at 100 °C. The annealing at 100 °C for 20 min induces a partial segregation at TBs, while that at 150 °C or higher temperature for 20 min can induce a complete solute segregation. The annealing conditions and pre-strain levels generate quite different effects on deformation by twinning and that by detwinning. Both annealing hardening and annealing softening might happen during the twinning predominant recompression. Annealing hardening occurs only with pre-strains of 3.0% and 5.5% after annealing at 100 °C for 6 h. A higher pre-strain or a higher annealing temperature or a longer annealing time generate a higher annealing softening effect. However, during the detwinning predominant recompression, all used annealing treatments generate hardening effect in all the pre-strained samples. With a complete solute segregation at TBs, a hardening of about 11–25 MPa is generally achieved. It is also found that solute segregation at TBs reduces the strain hardening rate of deformation by TBs migration.

1. Introduction

Twin boundaries (TBs) play an important role in plastic deformation and ultimately in controlling the working ability and mechanical properties of many engineering materials [1]. Notable examples are Mg alloys which are attractive for weight saving constructions in transportation and aerospace industries. Due to the limited active slip systems at room temperature, twinning is one of the main deformation modes. The main role of twinning in plastic deformation involves accommodating strain along the *c*-axis direction [2,3], rotation of crystallographic orientations [4] and relaxing stress concentration [4,5]. Recently, pre-straining and subsequent annealing are extensively used to tailor mechanical performance of Mg alloys [6–8]. It was found that a large number of $\{10\bar{1}2\}$ TBs generated by pre-straining can effectively enhance yield strength of a hot-rolled AZ31 plate without a compromise of ductility [6]. Xin et al. reported that reorientation by $\{10\bar{1}2\}$ twinning can greatly improve the hot rolling ability or reduce the tension-compression yield asymmetry [9,10]. Pre-straining was also used to modify damping capacity, fatigue performance, stretch ability and extrusion microstructure of Mg alloys [7,8,11,12].

Recently, Nie et al. reported that a periodic segregation of solute at coherent TBs of Mg alloys would take place after suitable annealing treatments. The solute segregation at TBs will pin TBs, leading to hardening during subsequent reloading (a $\{10\bar{1}2\}$ twinning predominant deformation) [13]. In our previous work, it was also found that this solute segregation at $\{10\bar{1}2\}$ TBs of a pre-twinned Mg AZ31 plate also resulted in an enhanced activation stress for detwinning of $\{10\bar{1}2\}$ twins. Some atomic simulations were also carried out to understand the solute segregation process at TBs and its strengthening effect on twinning dislocations [14,15]. A twinning deformation is constituted of both twin nucleation and twin growth, while detwinning is only a TBs migration process. In fact, it was considered that the TBs migration during a twin growth also differs from that during detwinning. Generally, the twin growth involves the nucleation of new twinning dislocations, twinning dislocations subsequently gliding along coherent TBs and climbing along basal-prismatic facets [16]. However, the nucleation of twinning dislocations is not necessary during detwinning. Although the experimental results have shown that solute segregation at TBs can harden both twinning and detwinning, this solute segregation may generate different hardening effects on the deformation dominated by twinning and that controlled by detwinning. To our best knowledge, there is no

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http://dx.doi.org/10.1016/j.msea.2015.07.049
0921-5093/

comparative study addressing this possible different effects. In addition, solute segregation at TBs is closely related to annealing temperature and time. However, a systematical investigation about the influence of annealing conditions on annealing hardening effect was not conducted yet.

In the present study, a comparative study about the hardening effect of solute segregation at $\{10\bar{1}2\}$ TBs on the $\{10\bar{1}2\}$ twinning predominant recompression and the detwinning predominant one was carried out. The influence of pre-strain levels and annealing conditions on mechanical behavior was systematically addressed. The corresponding mechanisms were studied and discussed. This study deepens understanding about deformation mechanism of Mg alloy containing pre-existing twins and contributes to the applications of TBs to tailor mechanical behavior.

2. Experiments and methods

2.1. Pre-straining, annealing and mechanical tests

A hot-rolled Mg AZ31 thick plate with fully recrystallized grains was used. As seen in Fig. 1, the initial plate has a mean grain size of about 16 μm and a typical basal texture with the (0002) poles largely parallel to the ND. To generate $\{10\bar{1}2\}$ TBs, blocks of 20 mm (ND) × 30 mm (TD) × 24 mm (RD) were compressed along the TD to 1.5%, 3.0% and 5.5% at room temperature and designated as PR1.5%, PR3.0% and PR5.5%, respectively. Here, RD, TD and ND represent rolling direction, transverse direction and normal direction of the initial plate, respectively. Different pre-strains were used to prepare the samples with different fractions of $\{10\bar{1}2\}$ twins. Blocks with a dimension of 10 mm × 8 mm × 8 mm were cut from the center of pre-strained samples for subsequent reloading. A part of the pre-strained specimens were annealed using 6 different treatments: 100 °C for 20 min, 100 °C for 6 h, 150 °C for 20 min, 150 °C for 6 h, 200 °C for 20 min and 200 °C for 6 h. Mechanical behavior of pre-strained samples under recompression along the TD (to continue $\{10\bar{1}2\}$ twinning) and that along the ND (to initiate detwinning of $\{10\bar{1}2\}$ twins) at room temperature were both tested on a Shimadzu AG-X mechanical testing system using a strain rate of 0.001 s^{-1}. Each test was repeated three times. Graphite was used to reduce friction between the heap and the samples.

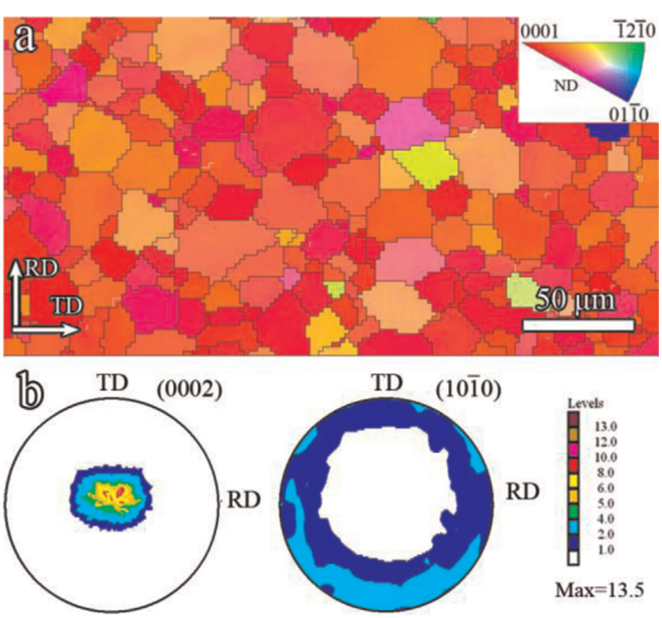

Fig. 1. (a) Inverse pole figure maps and (b) pole figures (acquired by XRD) of the hot-rolled Mg AZ31 plate used in the present study.

2.2. Microstructure and texture measurement

For microstructure examination by optical microscopy, the specimens were carefully ground and chemically etched in an acetic picral solution (2 ml acetic acid + 1 g picric acid + 2 ml H_2O + 16 ml ethanol). Pole figures were measured using an X-ray diffraction (XRD, Rigaku D/max-2500PC) meter. The measured incomplete pole figures were analyzed to determine the orientation distribution function and the complete pole figures were reconstructed. To reveal the microstructure and their crystallographic orientations, electron back-scattered diffraction (EBSD) mapping using a step size of 0.5 μm was conducted on a scanning electron microscope (SEM, Zeiss AURIGA) equipped with a HKL-EBSD system. Samples for EBSD mapping were mechanically ground followed by electrochemical polishing in the AC2 electrolyte. The EBSD data was analyzed using the Channel 5 software.

3. Results

3.1. Mechanical behavior

Stress–strain curves illustrating the compression behaviors along the ND and along the TD are shown in Figs. 2 and 3. The yield stresses derived from those stress–strain curves are listed in Tables 1 and 2. Under ND-compression (Fig. 2a–c), all the curves have a plateau shape, the typical feature of detwinning predominant deformation in Mg alloys [8,17] and the annealing treatment greatly changes the shape of stress–strain curves. An obvious yield elongation exist in annealed samples under ND compression except in those annealed at 100 °C for 20 min. As seen in Tables 1 and 2, the annealing at 100 °C for 20 min only generate a slight hardening during ND recompression, that at 150 °C for 20 min or 200 °C for 20 min generate more pronounced and similar hardening effects (about 15–25 MPa). Increasing the annealing time from 20 min to 6 h at 100 °C further enhances the annealing hardening effect in ND-compression. However, increasing annealing durations at 150 °C or 200 °C hardly varies the hardening effect.

The influence of annealing conditions on mechanical properties in TD recompression seems more complex. For PR1.5%, no hardening is observed in all the annealing conditions. For both PR3.0% and PR5.5%, a 20 min annealing at 100 °C or 150 °C hardly changes the yield stress. A slight hardening is noticed after annealing at 100 °C for 6 h. However, the annealing treatments at 200 °C for 6 h or at 150 °C for 6 h generate an obvious softening of PR5.5%. An annealing at 200 °C for 20 min already reduces yield stress of PR5.5% by 9 MPa.

The influence of annealing on strain hardening response of PR3.0% during TD recompression and ND re-compression were analyzed and the results are given in Fig. 4. All the strain hardening rate curves are characterized by a peak. Although annealing treatments hardly change the peak hardening rate, it seems that annealing treatments reduce hardening rate at the early stage after yielding (the regions denoted by the green arrows in Fig. 4).

3.2. Microstructure and texture

In order to confirm whether the as-used annealing treatments affect twins in pre-strained samples, optical microstructures of PR1.5% and PR5.5% before and after annealing were both examined and are presented in Fig. 5. Obviously, the twins well remain after annealing at 200 °C for 6 h. Therefore, it can be inferred that all the as-used annealing treatments do not damage the twin structure in