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Regulating twin boundary mobility by annealing in magnesium and its alloys

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ABSTRACT

We combine pre-compressive test, reverse tensile test, re-compressive test, *in-situ* electron back-scattered diffraction, and high-resolution transmission electron microscopy to systematically investigate the effect of annealing on the reciprocal motion of twin boundary (TB) in pure Mg and Mg alloys AZ31 and AZ91. We find that the twin boundary mobility (TBM) can be enhanced by decreasing the dislocation density and increasing the number of coherent TBs after annealing for a short time. On the other hand, after prolonged annealing in Mg alloys, TBM decreases since TBs are stabilized by segregated solute atoms and precipitates. As a result, the TBM significantly depends on both the alloying element content and the annealing time. We demonstrate, for the first time, that friction stress and back stress can be applied to clarify the variation of TBM during annealing in Mg alloys. Our findings show that the TBM can be regulated by annealing, opening up a novel avenue for developing Mg alloys with high damping capacity or enhanced mechanical properties.

1. Introduction

Ascribed to the insufficient slip systems during plastic deformation, twinning represents an important deformation mode in hexagonal close-packed (HCP) Mg alloys (Beyerlein and Tomé, 2008; Lu et al., 2008; Beyerlein et al., 2011a, 2011b; Mayama et al., 2011; Ma et al., 2012; Khosravani et al., 2013; Ghaffari Tari et al., 2014; Cheng and Ghosh, 2015; Kabirian et al., 2015; Khan et al., 2011; Lee et al., 2015a; Hama et al., 2016; Ishii et al., 2016; Kim et al., 2017; Balik et al., 2016; Wang and Agnew, 2016; Wang et al., 2016). In general, twin in Mg and its alloys is unstable and may either grow or shrink under further deformation (Muránsky et al., 2009; Proust et al., 2009; Hong et al., 2010; Beyerlein et al., 2011a,b; Li and Enoki, 2011; Lee et al., 2011; Yu et al., 2011; El Kadiri et al., 2013; Lee and Gharghouri, 2013; Wang et al., 2013a; Xiong et al., 2014; Qiao et al., 2015; Lee et al., 2015b; Wu et al., 2016). For this reason, twin boundary (TB) affects many aspects of performances of Mg alloys, such as the damping capacity (Cui et al., 2015), pseudoelasticity (Li et al., 2011; Cáceres et al., 2013; Wang et al., 2013b), strength (Nie et al., 2013) and fatigue (Yin et al., 2008; Liu et al., 2013). For example, the reciprocating motion of TB can effectively absorb vibration energy at a very low stress, resulting in enhanced damping capacity (Cui et al., 2015). The

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cyclic motion of TB is also closely related to the crack initiation and propagation and thus an important factor in determining low-cycle fatigue behavior of Mg alloys (Wu et al., 2008; Xu and Han, 2013). On the other hand, TBs significantly strengthen Mg alloys since segregation could markedly stabilize it after annealing (Nie et al., 2013).

In general, twin boundary mobility (TBM) depends on many factors, such as dislocation density (Wang et al., 2014; Cui et al., 2015), grain size (Tsai and Chang, 2013), twin size (Lee et al., 2014; Cui et al., 2017), solute atoms (Li and Enoki, 2007; Stanford and Barnett, 2013; Cui et al., 2017; Tahreen et al., 2015), precipitates (Gharghouri, 1996, 1998, 1999; Stanford and Barnett, 2009; Jain et al., 2010), and deformation state (Sarker et al., 2014). Of all these factors, annealing represents one of the most important ones (Xin et al., 2014). For example, TBs in Mg-Gd alloys can be stabilized significantly after annealing for a given duration due to the segregation of Gd and Zn atoms in TBs, resulting in an increased yield strength (Nie et al., 2013). Likewise, the stabilization of TBs in AZ91 alloy after annealing can be ascribed to the presence of precipitates in TB and matrix (Zeng, 2013), leading to an enhanced yield strength. Xin et al. (2014) revealed that the suppression of detwinning in AZ31 alloy after annealing correlates to the segregation of Al and Zn atoms in TBs. This finding, however, contravenes the current studies by Park et al. (2013) and Xin et al. (2015), who demonstrated that the inhibited detwinning in AZ31 alloy is intrinsically related to the removal of residual stress since the twin growth is facilitated even after prolonged annealing. Moreover, a recent study indicated that the reciprocal motion of TB was accelerated after annealing for a short span in AZ31 alloy. This is ascribed to the decreased dislocation density in TBs as well as to the transition from incoherent into coherent TBs after annealing (Cui et al., 2015). Conversely, no strengthening effect of TB was observed in pure Mg since segregation or precipitation didn't take place (Xin et al., 2014).

Despite the efforts above being devoted to investigating the effect of annealing on twin growth and/or detwinning in pure Mg and Mg alloys, there still lacks a systematic and fundamental research on how annealing influences TBM. Moreover, it is still unclear which factors govern TBM in Mg alloys. The aim of this study is to clarify the factors affecting TBM extensively and to understand how TBM varies upon annealing in pure Mg, Mg alloys AZ31 and AZ91. The present study demonstrates, for the first time, that TBM can be regulated by annealing, which varies significantly with alloying element concentration. The finding offers a systematic viewpoint in developing Mg alloys with either high damping capacity via mobilizing TBs or high strength through stabilizing TBs.

2. Experimental procedure

Extruded rods of pure Mg (3N) and Mg alloys AZ31 (Mg-3.1Al-0.9Zn-0.45Mn, mass%) and AZ91 (Mg-8.9Al-0.89Zn-0.42Mn, mass%) were used as-received. Cylindrical samples with a dimension of $\Phi 15 \times 50$ mm were cut from the extruded rods. Prior to the pre-compression at room temperature, all samples were subjected to solution treatment (ST) at 400 °C for 2 h, followed by water quench to obtain twin-free microstructures. Pre-compressions were carried out on the cylindrical samples at room temperature along extrusion direction (ED) to an engineering strain of 4% at a strain rate of 10^{-3} s^{-1} by using a computer-aided THERMECMASTOR-Z hot-forging simulator (Fuji Electronic Industrial Co., Japan). The subsequent annealing treatments were performed at 250 °C for 100–5000 s immediately after the compressive tests. To shed light on how annealing affects TBM, re-compression and reverse tensile tests were carried out at room temperature along ED at a strain rate of 10^{-3} s^{-1} on the pre-compressed samples after annealing using a THERMECMASTOR-Z hot-forging simulator to precisely capture the variations of load and displacement, and engineering stress-engineering strain curves were plotted. Re-compression tests were conducted directly on the pre-compressed cylindrical samples. The reverse tensile test was conducted on the sample shown in Fig. 1, which was machined from the pre-compressed cylindrical sample. To avoid the influence of mechanical machining on the microstructure of sample surface, careful polishing was conducted on the sample surface before the test.

Microstructures of pre-compressed pure Mg and Mg alloys AZ31 and AZ91 with and without annealing before and after re-compression or reverse-tension (to an engineering strain of 1%) were characterized in situ by focusing on the centre of samples with the electron back-scattered diffraction (EBSD) instrument equipped with a revised deformation device and data acquisition software (TSL-OIM 5.0, TSL Solutions, Japan). The re-compression and reverse tension were terminated in SEM at strain 1% for EBSD observation. The sample geometry for both re-compression and reverse tension in SEM is as same as that in our previous research (Cui et al., 2017). The textures of samples were evaluated by the inverse pole figures (IPFs), which were obtained by scanning all samples over a fixed region of 1000 × 1800 μm at a central area with a step size of 0.38 μm. We observed over 400 grains for various areas in pure Mg, Mg alloys AZ31 and AZ91 to obtain a statistic result. TEM samples were

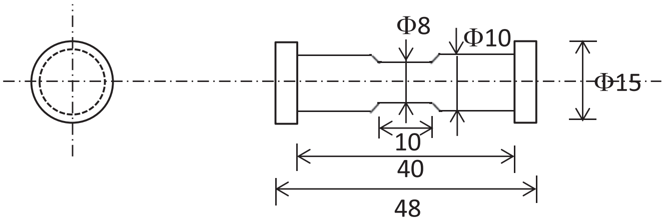

Fig. 1. Geometry of the sample for reverse tensile tests in Thermecmastor-Z simulator (mm).

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