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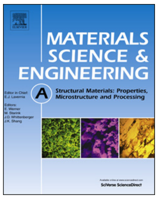

The role of dislocations in strain hardening of an extension twinning predominant deformation

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ABSTRACT

A $\{10\bar{1}2\}$ twinning predominant deformation of polycrystalline Mg alloys is often characterized by a low yield stress and a high degree of strain hardening. In this study, an interrupted compression-unloading-annealing-recompression experiment of pure Mg extruded rod was used to study the role of dislocations in strain hardening of compression along the extrusion direction. A quantified contribution of dislocations to strain hardening is analyzed. Our results show that dislocations associated mechanisms play an important role in strain hardening. Below a strain of 2.7%, the dislocation associated mechanisms are the main strain hardening mechanism, generating a hardening of about 8–10 MPa. Other mechanisms, e.g. grain refinement and texture hardening by $\{10\bar{1}2\}$ twinning, are not important at this stage. Upon further strain, both the work hardenings from the dislocations and other mechanisms rise. At a strain of 7.2%, the hardening from dislocations associated mechanisms reaches 22 MPa, accounting for 45% of the total hardening. It is also found that the grain refinement by twin boundaries is not an important mechanism in work hardening. The work hardening from texture hardening is important in the condition of a high twin volume fraction. With a twin fraction below 20%, the texture hardening mechanism hardly induces an obvious hardening.

1. Introduction

For magnesium alloys, the lack of easily-activated slips results in a poor deformation ability at room temperature, so deformation twinning is of great significance in plastic deformation [1–4]. There are mainly two types of twinning in Mg alloys: extension twinning (e.g. $\{10\bar{1}2\}$) and contraction twinning (e.g. $\{10\bar{1}1\}$ or $\{10\bar{1}3\}$) [5]. Due to the low critical resolved shear stress, $\{10\bar{1}2\}$ extension twinning is often the predominant deformation mechanism of textured polycrystalline Mg alloys under a tension stress along the *c*-axis or a compression stress perpendicular to the *c*-axis, e.g. compression along the extrusion direction (ED) of an extruded rod or compression along the transverse direction of a hot rolled plate at room temperature [1,6,7]. As seen in Fig. 1 showing a stress–strain curve under compression along ED of a pure Mg extruded rod, a $\{10\bar{1}2\}$ twinning predominant deformation is often characterized by a sigmoidal-shaped flow curve and a high degree strain hardening [8,9].

It is considered that the rapid hardening after yield in a $\{10\bar{1}2\}$ twinning predominant deformation mainly derives from the following mechanisms: (1) twin boundaries (TBs) dividing grains to produce Hall–Petch effect [9], (2) reorientation from a soft orientation to a hard orientation by $\{10\bar{1}2\}$ twinning (also referred to as texture hardening) [10,11] and (3) a glissile-to-sessile transformation of dislocations already present in the region experiencing the twinning shear transformation (also referred to as Basinski mechanism) [12]. In a $\{10\bar{1}2\}$ twinning predominant deformation, a certain amount of slips also involves in the plastic deformation as demonstrated in many simulations and experiments [10,13–15]. The enhanced work hardening by dislocation–TB interactions and/or dislocation accumulations at TBs is well demonstrated in deformation of cubic metals [16,17]. With regard to *hcp* metals, it is also found that the dislocation–TB interaction and dislocations accumulation can increase both the activating stresses for twin nucleation and TBs migration [18–20]. Although the influence of dislocations on strain hardening of a $\{10\bar{1}2\}$ twinning predominant deformation is discussed in many publications [15,20], an experimentally quantified evaluation of the role of dislocations to strain hardening is rare.

Recently, an interrupted compression-unloading-recompression experiment of a Mg–3Al–1Zn extruded rod indicated that the yield stress of recompression was identical to the peak stress of pre-compression [21]. This intrigues us to study the effect of dislocations on strain hardening of a $\{10\bar{1}2\}$ twinning predominant

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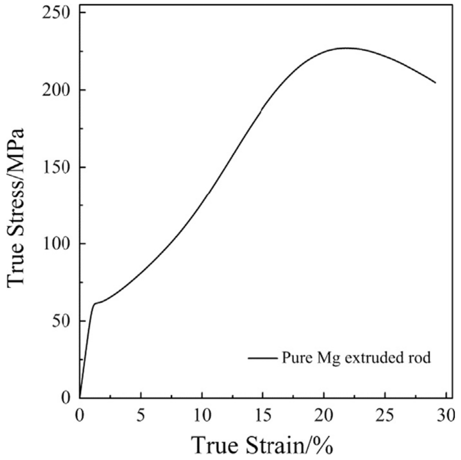

Fig. 1. Stress–strain curve of pure Mg extruded rod under compression along the extrusion direction at room temperature.

deformation by combining the usage of this interrupted loading–reloading process and annealing. If an annealing treatment prior to reloading can remove the dislocations in the pre-compressed sample whilst the twins are well remained, the contribution of dislocations to strain hardening can be deduced from the yield stress difference between the reloading without annealing and the reloading after annealing. In fact, the $\{10\bar{1}2\}$ twins in Mg alloys have a good thermal stability due to the low stored energy within them [7,22]. It is found that the $\{10\bar{1}2\}$ twins generated by ED compression of an AZ31 extruded rod are well remained even after an annealing at 250 °C for 3 h [22]. However, Nie et al. recently reported that annealing can induce a periodic segregation of solute atoms at TBs [23]. The segregated solutes at TBs can pin the TBs migration, leading to annealing hardening effect during reloading [23,24]. In the present study, the quantified contribution of dislocations to strain hardening of a $\{10\bar{1}2\}$ twinning predominant deformation was systematically studied by using of an interrupted compression-unloading-annealing-recompression experiment of pure Mg extruded rod. One thing to be highlighted is that the usage of pure Mg extruded rod can avoid the effect of solute segregation at TBs on reloading yield stress. In addition, the role of texture hardening and grain refinement by $\{10\bar{1}2\}$ twinning was also discussed in the present study. This work offers a quantified evaluation about contribution of different mechanisms to strain hardening of an extension twinning predominant deformation.

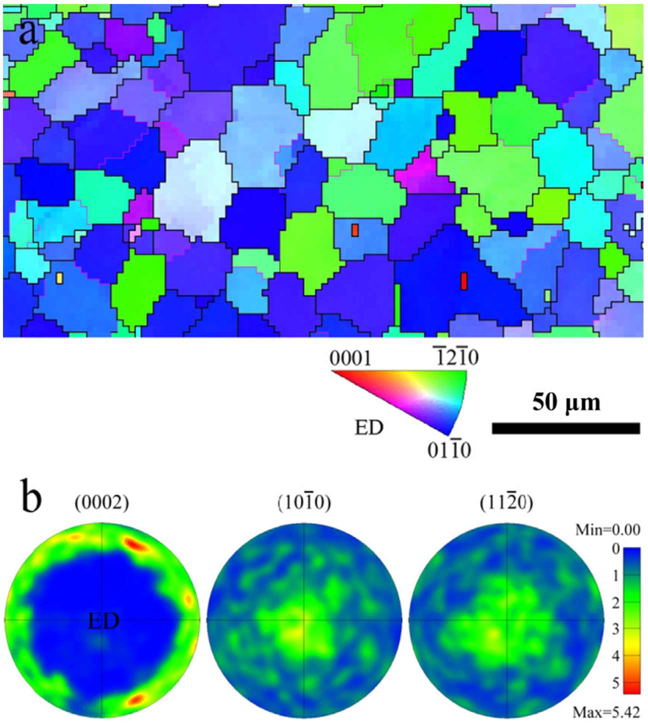

Fig. 2. (a) Inverse pole figure map and (b) pole figures of the as-used pure Mg extruded rod.

Table 1
Designation of the pre-compressed samples.

Pre-strain (%)	0	1.6	2.7	3.8	4.9	6.0	7.2
Without annealing	AR	PR1.6	PR2.7	PR3.8	PR4.9	PR6.0	PR7.2
With annealing		PR1.6A	PR2.7A	PA3.8A	PR4.9A	PR6.0A	PR7.2A

2. Experiments and methods

2.1. Mechanical tests

A pure Mg extruded rod (9 mm in diameter) with fully recrystallized structure was used in the present study. As shown in Fig. 2, the as-extruded rod has a grain size of about 22 μm and a typical extrusion texture with basal poles largely perpendicular to the ED. Cylindrical samples with a height of 12 mm were cut for compression tests. All compression tests were performed on a SHIMADZU AG-X50kN testing machine at room temperature using a strain rate of $10^{-3}\ s^{-1}$. As compression along the ED of a pure Mg extruded rod at room temperature is a typical $\{10\bar{1}2\}$ twinning predominant deformation, the as-cut samples were pre-compressed along the ED to a plastic strain of 1.6%, 2.7%, 3.8%, 4.9%, 6.0% and 7.2%, respectively, at room temperature. Some of the pre-compressed samples were re-compressed along the ED to failure immediately after unloading. In order to study the effect of dislocations generated during pre-compression on mechanical behavior, a part of pre-compressed samples were annealed and, then, re-compressed along the ED to failure. The grains of pure Mg are very easy to grow during annealing. This would greatly affect an accurate measurement of the contribution of dislocations to strain hardening. Therefore, several annealing regimes were tried to find a suitable annealing regime that can remove the dislocation as much as possible, while did not change the grains and twins. The annealing treatment was optimized to be at 100 °C for 48 h. The designations of pre-strained samples are shown in Table 1. Each mechanical test was repeated three times.

2.2. Microstructure and texture measurement

For optical microstructure examination, the specimens were mechanically ground and chemically etched in an acetic picral solution (2 ml acetic acid + 1 g picric acid + 2 ml H_2O + 16 ml ethanol). To examine the crystallographic orientations and twins in pre-strained samples, electron back-scattered diffraction (EBSD) analyses were carried out on an FEI Nova 400 SEM equipped with a HKL-EBSD system. All EBSD maps were recorded from the center of a cross section containing ED in the middle of cylinder. For each sample, two maps of 300 μm × 300 μm containing more than 400 grains were recorded. The samples for EBSD measurement were mechanically ground followed by an electrochemical polishing in an AC2 electrolyte at −30 °C and 20 V.