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Influence of precipitation on yield elongation in Mg-Zn alloys

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The effect of precipitation on yield elongation in an extruded Mg-4.5 wt% Zn alloy is investigated. The yield plateau is shown to disappear in the presence of precipitation and it is subsequently proposed that two criteria need to be satisfied for a distinct yield plateau: (1) the initial propagation of twins is autocatalytic in that twinning in one grain should stimulate twinning in an adjacent grain; (2) the twins that initially form should display an appreciable aspect ratio. These two criteria can also explain why the yield elongation decreases with an increase in grain size and deformation temperature.

Due to the limited number of available easy slip modes in magnesium, deformation twinning is readily activated [1–6]. The autocatalytic nature of twinning can cause twin cascades leading to a 'yield plateau' in the stress-strain curve [6–10]. Such a plateau increases the likelihood of instability during the load-bearing service of these alloys and is, therefore, undesirable [11,12]. Although grain refinement is an important tool to strengthen Mg alloys, it can accentuate the yield plateau [6,13,14]. The present work reports findings that show the yield plateau (yield elongation) decreases when Mg alloys are strengthened by precipitation hardening. An explanation for this observation is proposed which can also explain why the yield elongation has previously been seen to decrease with an increase in grain size and deformation temperature [8,15].

The phenomenon of yield elongation in magnesium alloys has been studied by a number of authors [7–10,16,17]. In compression testing of wrought materials, it is believed to arise from autocatalytic twinning where twins stimulate twinning events in neighboring grains. This results in an initial twin cascade over the sample. A particularly convincing study is that by Hazeli et al. [10] who employed digital image correlation combined with in-situ acoustic emission and ex-situ electron backscattered diffraction (EBSD) techniques. More recently, Timar et al. [16] applied a crystal plasticity finite element modelling framework to investigate the twin cascade effect and proposed that 'twin softening' could be the key to its occurrence. This 'twin softening' produces a twin cascade effect; once twinning has begun, further twinning activates more readily. The role of precipitation hardening on the twin cascade appears not to have been investigated. The present work examined precipitation hardening in an Mg-Zn alloy.

Mg-4.5 wt% Zn was cast with pure Mg (99.8%) and Zn (99.7%) using a resistance heating furnace at ~750 °C, after which a solution treatment (330 °C for 24 h and then 370 °C for 120 h) was applied. The alloy was then extruded at 350 °C with a reduction ratio of ~36 and a ram speed of 0.1 mm/s.

Compression samples were wire-cut into cylinders 6 mm high by 4 mm in diameter. Compression tests were carried out in a universal INSTRON machine coupled with a video extensometer at an initial strain rate of $2 \times 10^{-4}\ s^{-1}$ along the original extrusion direction (ED). This strain path is chosen so that deformation twinning dominates the stress-strain behavior [1,5,18]. For each condition, three repeated mechanical tests were performed. For one sample of each condition, the test was halted at an applied plastic strain of 0.012 (determined using the video extensometer) for microstructural analysis of twins.

The samples for EBSD were examined in a LEO 1530 SEM and were prepared in such a way that the plane parallel to the extrusion direction could be examined. They were ground to the center line, followed by mechanically polishing with the final step using active oxide polishing suspensions (OPS). For the un-deformed samples, a step size of 2 μm was used while for the deformed samples, a step size of 0.25 μm was selected to better characterize the deformation twins. The total area involved in the analysis in each of the deformed samples contained at least 1500 grains. The obtained EBSD patterns were analyzed with HKL Channel 5 software. Due to the high quality of the EBSD maps, the band contrast maps provided high accuracy and were used for the characterization and stereological analysis. Three parameters were directly obtained from the band contrast maps: (1) the twin area fraction using point counting, which is equal to the volume fraction V_V; (2) the

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number of twins intercepting a line of unit length, N_L; and (3) the number of twins per unit of observation area (twin number density), N_A. From these parameters, the twin aspect ratio q (the twin thickness divided by twin length) was calculated based on the equations in [7,8,19]. Transmission electron microscopy (TEM) samples were prepared from the plane perpendicular to ED following the same method detailed in [20]. A JEOL 2100 LaB6 with an accelerating voltage of 200 kV was used.

As shown in Fig. 1(a), the alloy consists of equiaxed grains with an average linear intercept grain size of ~20 μm and a typical fiber texture having most of the grains with their basal planes parallel to the ED. The extruded sample was aged for 84 h at 150 °C to the peak aged state. Fig. 1(b) shows a TEM micrograph of the peak aged sample, illustrating a uniform distribution of rod-shaped precipitates parallel to the c-axis of the grain. This is consistent with previous reports [20–22].

Fig. 2 shows the compressive true stress – true strain curves for the as-extruded (AE) and peak-aged (PA) samples. The solid lines represent the samples interrupted for EBSD observation at an applied plastic strain of 0.012, while the dotted lines represent samples tested to failure. Aging leads to an increase of yield stress (YS) of about 60 MPa, consistent with previous reports [20,23–25]. It is also evident that a yield plateau exists in the AE sample, but not after precipitation hardening.

Fig. 3(a) and (b) represents maps of band contrast and inverse pole figure (IPF) coloring for as-extruded and peak-aged samples compressed to an applied plastic strain of 0.012. The IPF contrast shows that the lenticularly shaped twins are $\{10\bar{1}2\}$ extension twins judging from their ~90° change in orientation with reference to their parent grains around an axis near <$1\bar{2}10$> [5]. It can be seen that noticeably thinner twins are not indexed in the map, especially for the peak aged condition. For this reason, the band contrast maps have been used to carry out the twin characterization of these two samples. It is also evident that the twins in the as-extruded sample are thicker and fewer in number than those in the peak aged condition.

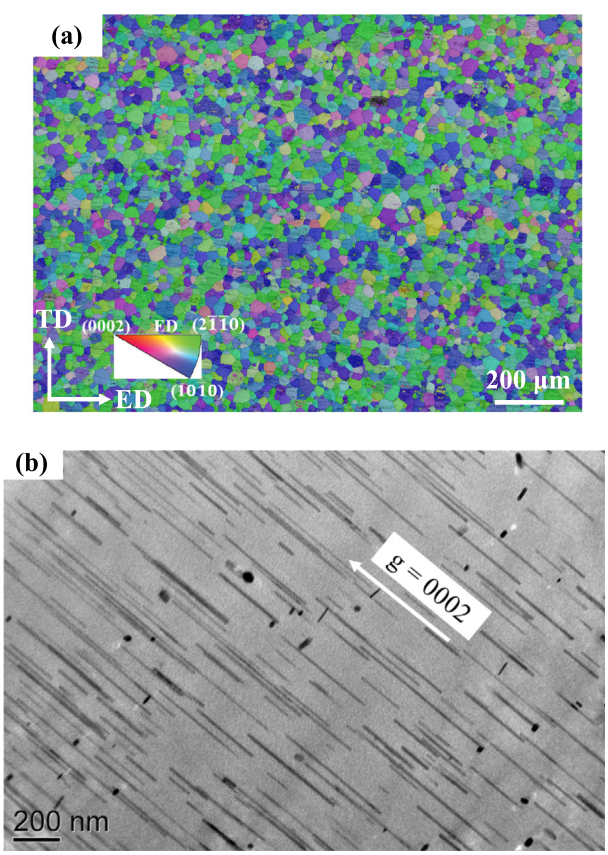

Fig. 1. Inverse pole figure (IPF) ED map of as-extruded Mg-4.5Zn alloy (a), and TEM micrograph showing the distribution of precipitates in the peak aged sample (b).

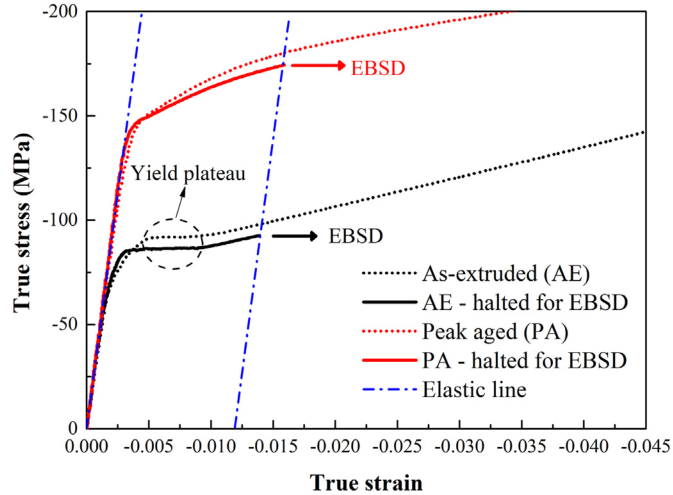

Fig. 2. True compressive stress – strain curves obtained from as-extruded (AE) and peak aged (PA) Mg-4.5Zn samples used for EBSD analysis and those without interrupting.

Fig. 3(c) shows that the twin aspect ratio of the as-extruded sample decreases from 0.05 to 0.03 after the precipitation hardening treatment, a decrease of around one third. However, the number density of twins per unit area doubles, increasing from 4400 mm^{-2} to 9300 mm^{-2} when the sample is age hardened. The considerable changes in the twin aspect ratio and twin number density between the as-extruded and the peak aged samples reveals a significant impact of precipitates on the twinning process. It is also evident in Fig. 3(d) that the twin connectivity fraction drops from 0.57 to 0.28 with aging.

We propose that two requirements must be met for the occurrence of a distinct yield plateau in the present material. The first is the initial propagation of twins should be autocatalytic in that twinning in one grain should stimulate twinning in an adjacent grain. This autocatalytic nature is evidenced by twin connectivity at the boundaries. The second is that twins should display an appreciable aspect ratio. If the twin aspect ratio is low, the contribution of the initial twin cascade to the macroscopic strain will be concomitantly low, and a small yield plateau will appear.

Despite the difference in the fraction of cross-boundary twins, the aged samples display the same number density of 'cross-boundary twins' to that observed in the as-extruded sample. Assuming that these represent the twins formed during an initial cascade of twinning in the sample, we conclude that in both conditions the initial twin cascade produced approximately one twin per grain in both samples. This result agrees with previous findings for alloy AZ31 [8]. However, the twin aspect ratio in the aged sample is significantly lower than that in the as-extruded sample, an observation which we propose explains the negligible yield plateau after aging.

It has been proposed [26] that the twin aspect ratio (q) for a stable twin in equilibrium with an imposed shear stress can be obtained from using a modified Eshelby approach:

$$\frac{1}{q} = \frac{2sG\beta'}{\tau - \tau_G^*} - \frac{11}{5} \quad (1)$$

where τ is the applied shear stress resolved on the twin shear system, τ_G^* is the friction stress for twin growth, s is the twin shear, G is the shear modulus and β' is a relaxation factor <1 (simplified from Eq. (15) in [26]). The relaxation factor captures the effect of plasticity induced in the matrix which relaxes the constraint placed on the twin by the surrounding matrix [26,27]. This plastic relaxation allows greater amounts of twin thickening for a given value of applied stress [26].