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Initiation of basal slip and tensile twinning in magnesium alloys during nanoindentation

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The present study addresses the challenge of determining the stress required for the initiation of deformation modes in Mg alloys. Nanoindentation is employed to detect the onset of basal slip and tensile twinning. A Mg-6 wt. % Zn alloy and a series of Mg-Gd binary alloys with concentrations between 0.3 and 4 wt. % Gd are examined in the extruded state. Nanoindentation tests were conducted on $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ crystal planes using a 5 μm radius spherical indenter. It is shown that the initial yielding point in the load trace corresponds to the appearance of basal slip lines on the sample surface. A pop-in event is seen to accompany the appearance of twinning. We find that the addition of Zn strengthens the basal slip but shows mild influence on twin initiation. For alloying with Gd, an impact on basal slip and twinning is only seen at the highest solute levels. A means of examining the alloying effect on twin growth is proposed and revealed that solute Zn has a greater impact on twin growth than initiation.

1. Introduction

Basal slip and $\{10\bar{1}2\}$ extension twinning frequently dominate the deformation of magnesium and often control yielding [1,2]. Understanding the respective critical resolved shear stress (CRSS) values of these two deformation modes – in the context of a polycrystal - is therefore of practical interest. A considerable range of values exist in the literature for both basal slip [1–4] and extension twinning [1,5]. This evidently reflects alloying effects [6–13] as well as potential variations due to the technique of measurement. The effect of Zn additions on twinning for example are in dispute, one of us has reported that the effect is minor [6] while others report it to be significant [9,10]. Gd has been shown to have a considerable impact on slip [12] whereas its impact on twinning is unknown. There is need for additional experimental clarification, particularly in regard to the separate processes of twin nucleation and growth. In the present work, we explore the extent to which nanoindentation technique can be usefully brought to bear on the problem, for the benefit of understanding rational alloy design for the micro- and nano- scaled structure.

Somekawa and Schuh [14] employed cube corner indenter (up to loads of 1 mN) to compare the hardness and pop-in stress for a range of fine grained solute strengthened magnesium alloys. It was found that the increments in hardness values due to solute addition (Ca, Zn, Y, Al, Li) qualitatively followed theoretical predictions based on Yasi et al. [15] for basal slip, but fell 1.5 to 3 times higher in magnitude. They found the pop-in stress to be considerably less sensitive to solute addition. However, no distinction was made between deformation modes.

Sanchez-Martin et al. [16] have recently taken some steps towards addressing this gap. They used crystal plasticity finite element (CPFE) modelling combined with Berkovich indentation (up to depth of ~300 nm) on electropolished surfaces. CPFE modelling was employed to map out the broad impact of relative CRSS values on the sensitivity of hardness to crystallographic indentation plane. The measurements are seen to be sensitive to the choice of CRSS values only within certain ranges of values. This, compared with the fact that in their model frame work three parameters are needed to describe the hardening for each of the four deformation modes, makes unique determination of characteristic CRSS values problematic. Their study also did not consider deformation twinning.

Catoor et al. [17] have examined the apparent CRSS value corresponding to pop-in events seen in their nanoindentation tests. They employed a 3 μm radius spherical tip indenter and examined indentation of three chemically polished crystallographic planes

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using pure Mg single crystals. Twinning was observed in their study but the initial pop-in event was interpreted in each case to be due to slip. Nevertheless, complete rationalization of the pop-in events based on the nanoindentation Schmid factor was not possible. It was concluded, however, that the pop-in event probably corresponded to the homogeneous nucleation of <a> dislocations at stresses between 0.6 and 1.2 GPa.

In a previous work, we addressed the issue of associating nanoindentation yielding events with different deformation modes [18]. For Mg alloy AZ31, we found that basal slip and $\{10\bar{1}2\}$ extension twinning events could be separated for spherical tip indentation (5 μm tip radius) on colloidal silica (OPS) polished planes containing the crystallographic c-axis. Atomic Force Microscopy (AFM) was employed to confirm that the initial yielding event corresponded to the appearance of copious basal slip lines on the surface, which is the onset of the plasticity during penetration. The apparent CRSS for the onset of basal slip for these experiments could be estimated using Hertzian elastic contact theory as [19]:

$$\tau_{yielding} = S\left(\frac{6P_yE^{*2}}{\pi^3R^2}\right)^{1/3} \tag{1}$$

where P_y is the load of yielding and R is the indenter radius. E^* is the reduced Young's modulus. S is defined as the elastic Indentation Schmid Factor (ISF) and the value is 0.18 for basal slip in $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ indentation [17,18].

Twins on the surface were only seen once a pop-in event had occurred after the yielding. Associating a CRSS value with the twinning event is more difficult in these tests because it relies on the estimation of the stresses under the indenter during plastic indentation immediately prior to the onset of twinning. Finite element crystal plasticity (CPFEM) simulations were employed to establish the relationship between indentation load and the resolved shear stress on twinning plane [18]. This was done for a range of candidate CRSS values for slip and the mean was recorded. With this relationship in hand, it is possible to convert the load corresponding to the pop-in to an estimate of the corresponding resolved shear stress on the most heavily stressed twinning system.

The present study employs nanoindentation on planes containing the crystallographic c-axis in an attempt to discern the sensitivity of the CRSS values corresponding to the onset of basal slip and twinning to the addition of Zn and Gd to pure Mg. Our first objective is to verify that our previous findings, made on alloy AZ31 [18], hold for Mg alloys in general (i.e. that the yielding is due to slip and the following pop-in is due to twinning).

2. Materials and experimental method

Cast Mg-6Zn billets ($\Phi = 59.5$ mm and h = 40 mm) were solution treated at 335 °C for 72 h and quenched in water. The billets were then extruded to plates 46 mm wide and 12 mm thick at 370 °C and a ram speed of 0.1 mm/s. Samples for nanoindentation tests were annealed at 340° C for 3 h. Four magnesium alloys containing 0.3, 1, 2.5 and 4 wt. % Gd were cast and cut into billets measuring 59.5 mm × 40 mm for extrusion. All the billets were homogenized at 450° C for 3 h. The extrusion schedule included preheating the billets at 400° C for 2 h, followed by extrusion at 400° C at a ram speed of 0.5 mm/s. The samples prepared for nanoindentation tests were annealed at 450 °C for another 3 h. As-received cast pure Mg billets with a diameter of 29.8 mm and height of 20 mm were extruded into rods 8 mm in diameter at 370° C at a strain rate of 15 mm/s. The samples were immediately annealed at 450 °C for 2 h followed by a water quench. The chemical compositions of all the materials used in the study are given in Table 1.

Table 1
Chemical composition of the materials in the present study (wt. %).

Material	Zn	Gd	Mn	Mg
Mg-6 Zn	6%	0.0415%	0.036%	balance
Mg-0.3 Gd	–	0.304%	0.0182%	balance
Mg-1Gd	–	1.09%	0.0178%	balance
Mg-2.5 Gd	–	2.51%	0.0162%	balance
Mg-4 Gd	–	4.08%	0.0153%	balance
Pure Mg	0.006%	–	0.0345%	99.7%

The dimension of the samples used for nanoindentation was 10 mm × 10 mm × 5 mm. The large surface was cut to be perpendicular to the extrusion direction, which was then selected as the testing surface. Similar to the previous experiment carried out on AZ31 in Ref. [18], all the tests were performed on planes within 5° of the $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ crystal planes. EBSD was employed on a pre-marked area to obtain the grain orientation data and the initial crystallographic texture (Fig. 1) by a field emission Leo 1530 Scanning Electron Microscope. The textures for pure Mg and Mg-6Zn were typical for extruded magnesium alloys with the basal planes aligned perpendicular to the extrusion direction. The texture in the Mg-Gd series alloys agreed with reference [20]. The $<11\bar{2}1>$ texture increased with Gd addition. The testing surfaces for EBSD mapping were first wet ground using 1200 grit SiC paper, cleaned in ethanol in an ultrasonic bath and dried with flowing air. Then, 9 μm, 6 μm and 3 μm diamond suspensions were used for rough polishing on a Struers DP-Pan cloth, followed immediately by etching in acetic-nitric acid (15 ml acetic acid, 5 ml nitric acid, 60 ml ethanol and 20 ml distilled water) for 20 s. Samples for EBSD mapping were polished with 10% of OPS (colloidal silica) for 10 min in the middle of a Struers OP-chem cloth at a rotating speed of 150 r/min. Before the nanoindentation tests, samples were polished another 3 min with OPS and 2 min with ethanol. The purpose for this step is to get rid of the oxidation film and clean the colloidal silica thoroughly. Two to seven grains were selected for each orientation, depending on the grain size and quantity of $\{10\bar{1}0\}$w and $\{11\bar{2}0\}$ oriented grains on the polished surface.

Nanoindentation tests were conducted using a UMIS ultra-micro indentation system (UMIS is the name of the machine manufactured by CSIRO Division of Applied Physics, Lindfield, Australia). A 5 μm radius diamond spherical tip are conical shaped with a spherical end, which were employed in the present work. Tests were performed under the closed loop control, ensuring reproducible and stable results. The loading rate was around 0.015 ± 0.005 mN/s (There is no influence of rate on the experimental results over this range.) The maximum applied load is 5 mN. On the indented surface, the grain boundaries are avoided (within ~10 indent diameters). Some tests were interrupted once the yielding or pop-in event was observed. The deformation around the indents was characterized using a Brucker Multimode 8 Atomic Force Microscopy (AFM) in the PeakForce error mode and analysed by software 'NanoScope Analysis'.

The occurrence of yielding and the pop-in events is sensitive to the surface damage caused by polishing [21,22]. Mechanical polishing can alter the dislocation density in the near-surface zone. We therefore examined the influence of mechanical polishing time on the yielding and pop-in load in a large grain in pure Mg. The tests were first carried out for chemical polished condition (60% ethanol, 20% distilled water, 5% nitric acid and 15% acetic acid for 5 min). It was found that a pop-in is the first and only event in 90% of the curves, which is consistent with the behaviour commonly seen in