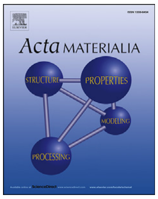
Acta Materialia 135 (2017) 411–421

Contents lists available at ScienceDirect

Acta Materialia

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



Full length article

Experimentally quantifying critical stresses associated with basal slip and twinning in magnesium using micropillars

Y. Liu [a], N. Li [b], M. Arul Kumar [a], S. Pathak [b, c], J. Wang [d, *], R.J. McCabe [a], N.A. Mara [b, e], C.N. Tomé [a, **]

[a] Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM, 87545, USA
[b] Center for Integrated Nanotechnologies, Los Alamos National Laboratory, Los Alamos, NM, 87545, USA
[c] Chemical and Materials Engineering, University of Nevada, Reno, NV, 89512, USA
[d] Department of Mechanical and Materials Engineering, University of Nebraska-Lincoln, Lincoln, NE 68583, USA
[e] Institute for Materials Science, Los Alamos National Laboratory, Los Alamos, NM, 87545, USA

ARTICLE INFO

Article history:
Received 23 March 2017
Received in revised form
16 May 2017
Accepted 5 June 2017
Available online 7 June 2017

Keywords:
Magnesium
Micropillar compression
Basal slip
Twin

ABSTRACT

Basal slip and $\{01\bar{1}2\}$ twinning are two major plastic deformation mechanisms in hexagonal closed-packed magnesium. Here we quantify the critical stresses associated with basal slip and twinning in single-crystal and bi-crystal magnesium samples by performing *in situ* compression of micropillars with different diameters in a scanning electron microscope. The micropillars are designed to favor either slip or twinning under uniaxial compression. Compression tests imply a negligible size effect related to basal slip and twinning as pillar diameter is greater than 10 μm. The critical resolved shear stresses are deduced to be 29 MPa for twinning and 6 MPa for basal slip from a series of micropillar compression tests. Employing full-field elasto-visco-plastic simulations, we further interpret the experimental observations in terms of the local stress distribution associated with multiple twinning, twin nucleation, and twin growth. Our simulation results suggest that the twinning features being studied should not be close to the top surface of the micropillar because of local stress perturbations induced by the hard indenter.

Published by Elsevier Ltd on behalf of Acta Materialia Inc.

1. Introduction

Magnesium (Mg) alloys have potential applications as structural components in the transportation industry due to their high strength-to-weight ratio [1]. Mg alloys have a hexagonal close-packed (hcp) structure and deform primarily via slip on the basal plane and $\{01\bar{1}2\}$ tensile twinning [2–9]. Quantifying experimentally the critical stresses associated with basal slip and twinning (nucleation, growth and interactions) enables the prediction of the mechanical response of Mg and Mg alloys [10–19]. For example, recent cyclic studies reveal that twin junctions in single-crystal Mg have a considerable impact on the critical stresses necessary for twinning and detwinning, and can become a source of fracture [19–23]. However, these twinning phenomena and their effects on mechanical behavior are not well characterized or understood.

More than a decade ago, the micrometer-scaled pillar (micropillar) compression technique opened the possibility of exploring mechanical properties of sub-micron and micro-sized microstructural features owing to its relatively simple stress-states compared to other techniques such as micro/nano-indentation [24–29]. However, it is often observed that the mechanical strength increases with decreasing micropillar diameter, similar to the feature-size strengthening identified in nano-scale systems [30–38]. This so-called size-scale effect is related to the pillar surface-to-volume ratio. For face-centered cubic and body-centered cubic metallic materials, a micropillar manifests bulk mechanical behavior when the diameter is on the order of tens of microns [24,27]. For hcp metals such as Mg, several *in situ* electron and optical microscopy micropillar compression experiments have shown an obvious size-effect for {0001} basal slip compared with bulk single crystal results. Kelly and Hosford's plane-strain compression on single-crystal bulk Mg measured the compression yield stress for basal slip to be 4 MPa [39]. Yu et al. stated that the yield strength for basal slip could reach 2000 MPa when the

---

* Corresponding author.
** Corresponding author.
E-mail addresses: jianwang@unl.edu (J. Wang), tome@lanl.gov (C.N. Tomé).

http://dx.doi.org/10.1016/j.actamat.2017.06.008
1359-6454/Published by Elsevier Ltd on behalf of Acta Materialia Inc.

pillar diameter is smaller than 0.1 μm [40]. Ye et al. measured the yield strength for pillar diameters varying from 0.25 μm to 1.6 μm, and reported results in the range of 300 MPa to 50 MPa [41]. Moreover, *ex situ* micropillar compression results (the onset of yielding could not be determined) have provided some insights as well. Byer and Ramesh reported that the response of single-crystal Mg pillars oriented along (0001) are less sensitive to dislocation density when the pillar diameter increased to 10 μm [42]. Prasad et al. compared the size-effects of both basal slip and $\{01\bar{1}2\}$ twinning by compressing micropillars (3 μm) and macropillars (3 mm). These results indicate a significant size-effect for basal slip but a negligible size-effect for twinning [43].

In spite of these micropillar studies on single-crystal Mg, several important questions remain unanswered. First, what is the critical micropillar diameter that can reproduce similar yield strength for basal slip as the bulk single crystal counterpart? Second, what is the critical micropillar diameter associated with $\{01\bar{1}2\}$ twinning? Third, what are the critical stresses associated with basal slip and twinning? To address these issues, we employ *in situ* scanning electron microscopy (SEM) micropillar compression. We aim at quantifying the critical stresses associated with basal slip and twin growth (twin boundary migration). Twin nucleation could be identified but the corresponding nucleation stress is related to stochastic factors, especially local stress or strain concentration. Corresponding to these goals, we design micropillars with specific orientations and microstructures. The critical resolved shear stress (CRSS) values deduced here for a 10 μm pillar are: twin boundary migration (29 MPa) and basal slip (6 MPa). The CRSS ratio of twin growth and basal slip in this study is estimated as 5, which is the same as Kelly & Hosford plane-strain compression results and comparable to various bulk measurements in the range of 2.5–4.4 in the literature [7]. The use of local plasticity simulations provides us with insight about the non-homogeneous stress field inside the pillar and allows us to understand the correlation between the micropillar test conditions and the observed twinning configurations in terms of local stress fields.

2. Experimental details

Mg single crystals were grown using the Bridgman method and $\{01\bar{1}2\}$ deformation twins were introduced by pre-loading in tension along the c-axis to a total strain of 1%. Standard metallographic techniques were used to polish specimens. A solution of 10% HNO_3 and 90% H_2O was used to chemically polish each specimen to remove any residual surface damage. An FEI XL30 with an accelerating voltage of 25 kV was used for electron backscatter diffraction (EBSD) to obtain crystal orientation for both parent and twin phases. An FEI Helios 600 focused ion beam (FIB) with accelerating voltage 30 kV was used to prepare micropillars. The length and diameter of the micropillars were measured by FIB/SEM before and after straining. The length-to-diameter ratio was 2.5-to-1 and the taper angle is within 2-to-5°. Such geometrical dimensions ensure uniform deformation [27]. *In situ* SEM analyses were conducted on a FEI Magellan 400 and an FEI Helios 660 SEM with an accelerating voltage of 1–2 kV (The low kV in secondary electron mode allows better surface detail such as revealing slip steps.). *In situ* micropillar compression was performed using a Hysitron PI-85 and PI-85 × R SEM PicoIndenter. The micropillar compression tests were conducted with a 20 μm flat punch tip at a strain rate of $1 \times 10^{-3} s^{-1}$. During the *in situ* testing, the indenter was controlled to minimize the misalignment between the tip and the top surface of the pillars, and a minimum of 3 tests were performed for each type of pillar to ensure the reproducibility of results. All of the micropillars were compressed to a maximum of 10% of the pillar height and no *ex situ* compression was performed. We plotted stress-strain curves to show mechanical response. The stress was calculated by using a full width at half maximum (FWHM) approach [44], where the diameter in the middle of the pillar is estimated from known (1) top surface diameter, (2) taper angle and (3) total length of the pillar. The strain was estimated by displacement/height of the pillar. The displacement is considered once the indenter contacts the pillar. Postmortem transmission electron microscope (TEM) specimens were prepared using an FEI DB235 dual-beam FIB with an accelerating voltage of 30 kV. An FEI Tecnai F30 field emission transmission electron microscope with accelerating voltage of 300 kV was used for TEM imaging.

3. Results and discussion

3.1. Micropillar design

Micropillar compression experiments were performed along $<2\bar{1}\bar{1}3>$ (basal slip favored) and $<10\bar{1}0>$ (twinning favored) orientation in single-crystal Mg, and also in bi-crystals that consist of the previous parent orientation and a $\{01\bar{1}2\}$ twin. Fig. 1 shows EBSD images of pre-strained samples and reveals the crystal orientation of both the single-crystal pillars (1 and 3) and bi-crystal pillars (2 and 4). The three-dimensional (3D) geometry of the four micropillars (1–4) determined from the EBSD analysis is schematically illustrated in Fig. 2, together with the maximum Schmid factors for basal slip (m_b) and twinning (m_t). Based on the Schmid factors, pillars 1 and 2 favor basal slip while pillars 3 and 4 favor twinning.

3.2. Micropillars with 10 μm diameter reproduce bulk single crystal values

By varying the diameter of pillar 1 (basal slip preferred single-crystal pillar with m_b~0.49), we deduce that the minimum diameter necessary to obtain a similar yield strength for basal slip to bulk Mg single crystal with similar m_b is 10 μm. Fig. 3a shows an undeformed micropillar with slight tapering (the diameter difference between the top surface and the base is less than 5%). The height of all pillars is about 2.5 times the pillar diameter in order to avoid barreling or buckling effects [27]. Postmortem SEM images of pillar 1 with diameters of 3 μm (Fig. 3b), 5 μm (Fig. 3c) and 10 μm (Fig. 3d) reveal that obvious basal slip occurs at the upper edge of these micropillars, and that it is repeatable. Fig. 3e compares the stress-strain curves of these micropillars and shows multiple stress-drops corresponding to multiple events of basal slip. The elastic slope is affected by incomplete contact of the indenter with the top of the pillar during the initial loading. The taper angle is within 2–5°. The true stress was estimated using the FWHM approach. The *in situ* nature of the experiment allows us to directly correlate a discrete slip event with the corresponding load. We thus directly calculate the local resolved shear stress for the slip event at the corresponding load value (given by the *in situ* movie and snapshots). The corresponding stress is marked by stars on each curve in Fig. 3e. We determine the yield strength both when the stress starts to deviate unambiguously from linearity and when slip traces are detected. The yield strength for these micropillars manifests a clear size-effect. Pillars with smaller diameter exhibit higher onset yield strength. By comparing with literature results for both *ex situ* and *in situ* pillar compression with similar m_b, we determine the critical size that represents the mechanical responses of bulk Mg. Fig. 3f (compared to *ex situ* literature) shows the measured flow stress as a function of pillar diameter compares well with *ex situ* micropillar compression with similar pillar diameters [42]. Fig. 3g (compared to *in situ* literature) shows the comparison of the onset yield strength as a function of pillar diameter with other *in situ*