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Effects of the initial dislocation density on size effects in single-crystal magnesium

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Abstract

Single-crystal magnesium micropillars, ranging in diameter from approximately 600 nm to $10 \mu m$, are fabricated using focused ion beam machining and are loaded in uniaxial compression along either the $[0001]$ or $[2\bar{3}14]$ axis. The influence of initial dislocation density on size effects is investigated for compression along the [0 0 0 1] axis using two distinct initial dislocation densities. Separately, at fixed low dislocation density, the influence of orientation on size effects is examined by comparing compression along the $[2\bar{3}14]$ and $[0001]$ directions. Our microcompression results show that decreasing the initial dislocation density results in a stronger size effect in terms of both increased strength and stochasticity with decreasing pillar size. Comparison with a probability-based model shows good agreement between theoretical predictions and experimental observations. Our results demonstrate that the properties of magnesium micropillars depend on the specimen diameter, the initial dislocation density and the orientation of the basal planes with respect to the loading axis. - 2013 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Magnesium; Microcompression; Size effects; Dislocation density; Single crystal

1. Introduction

Magnesium is becoming an increasingly popular material for engineering applications because of its high strength-to-weight ratio. With a low density of 1740 kg m^{-3} for pure Mg, the hexagonal close-packed (hcp) material and its alloys are very attractive for aerospace and automotive applications, where weight is closely related to energy efficiency. However, because of the low symmetry in the crystal structure, deformation mechanisms in magnesium can be much more complex than those of the more commonly studied high-symmetry body-centered cubic (bcc) and face-centered cubic (fcc) materials. These deformation mechanisms need to be better understood before magnesium can be extensively used for industrial applications. We note that magnesium's c/a ratio of 1.624 is close to the ideal hcp crystal's ratio of 1.633, making it

also a good representative element for research studies on hcp materials.

The typical slip systems of hexagonal close-packed materials (shown in [Fig. 1](#page-1-0)) consist of the basal ${0002}$ and prismatic $\{01\overline{1}0\}$ planes, as well as the $\{11\overline{2}2\}$, $\{01\bar{1}1\}$ and $\{10\bar{1}2\}$ pyramidal planes [\[1\]](#page--1-0), but there is still debate over which systems are active at various temperatures [\[2\]](#page--1-0). Reed-Hill and Robertson [\[3\]](#page--1-0) strained single crystals of magnesium in tension parallel to the basal plane to observe nonbasal slip at various temperatures and strain rates. They observed $\{10\bar{1}0\}$ prismatic slip at 85 K and 298 K, but $\{10\bar{1}1\}$ pyramidal slip at 423 K and 559 K. Obara et al. [\[4\]](#page--1-0) strained crystals in compression along the c-axis and found that the pyramidal slip system $\{11\overline{2}2\}\langle\overline{1}\overline{1}23\rangle$ operated at all temperatures from room temperature to 773 K. Kitahara et al. [\[5\]](#page--1-0) also performed c-axis compression and observed $\{11\overline{2}2\}\langle11\overline{2}3\rangle$ pyramidal slip in the temperature range of 77–473 K. Chapuis and Driver [\[2\]](#page--1-0) found that this $\{11\bar{2}2\}\{\bar{1}123\}$ slip system only occurred above 573 K, contrary to the observations of Kitahara et al. [\[5\]](#page--1-0) and Obara et al. [\[4\]](#page--1-0). This discrepancy

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Fig. 1. Slip planes of hcp materials. The unit cell on the left illustrates the $\{0002\}$ basal and $\{01\bar{1}0\}$ prismatic planes. The unit cell on the right illustrates the families of pyramidal planes: $\{11\bar{2}2\}$ in yellow, $\{01\bar{1}1\}$ in red and $\{10\bar{1}2\}$ in blue. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

may be attributed to the different loading conditions: Chapuis and Driver used channel die compression, while Kitahara and Obara used uniaxial compression.

While slip is a common mode of deformation in magnesium, basal and prismatic slip are unable to accommodate tension or compression along the c -axis, and some studies indicate pyramidal slip may be difficult to activate due to temperature dependence and its higher critical resolved shear stress [\[2\].](#page--1-0) Therefore, to accommodate global plasticity, twinning is activated as an additional mode of deformation. The most common twinning mode results from an extension of the *c*-axis and is $\{10\bar{1}2\}$ "tensile" twinning [\[6\].](#page--1-0) As with slip, however, the literature does not always agree upon the conditions that activate each twinning system, especially when the c-axis is in compression. Reed-Hill and Robertson [\[6\]](#page--1-0) strained single crystals in tension parallel to the basal planes (putting the c -axis in compression) and observed $\{30\bar{3}4\}$ and $\{10\bar{1}3\}$ compression twinning. Yoshinaga et al. [\[7,8\]](#page--1-0) investigated twinning deformation as a result of c-axis compression and observed not only $\{30\bar{3}4\}$ and $\{10\bar{1}3\}$ twinning, but also $\{10\bar{1}1\}$, $\{10\bar{1}5\}$ and {1124} twinning. Chapuis and Driver [\[2\]](#page--1-0) performed plane strain compression experiments and determined that the $\{10\bar{1}2\}$ "tensile" twinning mode was not temperature dependent, but that the $\{10\overline{1}1\}$ and $\{10\overline{1}3\}$ "compressive" twinning modes were. Thus the twinning modes also provide a rich set of deformation mechanisms. These deformation mechanisms (both slip and twinning) result in significant anisotropy in magnesium, so an understanding of both along with their interactions (as discussed in detail by Zhang and Joshi [\[9\]\)](#page--1-0) is important for understanding the macroscopic behavior of magnesium.

Studies show that there is also a size effect with respect to the grain size on deformation processes in polycrystal-line magnesium [\[10–17\]](#page--1-0). This suggests a possible dependence of the active deformation mechanisms on the overall specimen size as well (e.g. in microcompression experiments). If these size-dependent and orientationdependent mechanisms could be better understood, the microstructure of magnesium might perhaps be manipulated in such a way as to optimize its properties.

"Size effects" generally refers to the phenomena that occur when the structural length scale approaches that of the deformation mechanisms and encompasses such trends as increasing flow stress and strain hardening with decreasing specimen size. The microcompression technique, first introduced by Uchic and his colleagues in 2004 [\[18\]](#page--1-0), has become a popular technique for investigating these size effects, allowing experimentalists to perform uniaxial compression tests $[19]$ in the μ m and sub- μ m regimes $[20-23]$. Although originally applied to fcc single crystals [\[18\],](#page--1-0) this technique has been extended to study materials including (but not limited to) bcc [\[24\]](#page--1-0) and hcp [\[25\]](#page--1-0) crystals, alloys [\[26\],](#page--1-0) intermetallics [\[18\],](#page--1-0) metallic glasses [\[27\]](#page--1-0), nanoporous foams [\[28\]](#page--1-0), nanolaminates [\[29\]](#page--1-0) and nanocrystalline [\[30\]](#page--1-0) materials.

In 2010, Byer et al. [\[25\]](#page--1-0) and Lilleodden [\[31\]](#page--1-0) both published results from microcompression experiments on single-crystal magnesium. In Lilleodden's experiments [\[31\],](#page--1-0) increasing yield stresses were observed with decreasing pillar diameter, providing an apparent size effect, and she observed shear instabilities oriented with the basal planes. Lilleodden defined the primary deformation mechanisms to be slip on the six equivalent pyramidal $\{11\bar{2}2\}$ systems. Byer et al. [\[25\]](#page--1-0), however, observed that the mechanical

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