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Orientation-dependent indentation response of magnesium single crystals: Modeling and experiments

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Abstract—We investigate the orientation-dependent characteristics of magnesium single crystals under localized contact using nanoindentation experiments and crystal plasticity finite element (CPFE) simulations. Nanoindentation experiments on (0001) and $(11\bar{2}0)$ planes exhibited distinct load–depth responses. Atomic force microscopy revealed material pile-up with sixfold symmetry in the former case and a sink-in phenomenon with twofold symmetry in the latter case. Our corresponding detailed CPFE simulations uncover the evolution of deformation activity in the indented volume, thereby providing insight into the interacting effects that cause the pile-up and sink-in phenomena. The simulations indicate the occurrence of $\{10\bar{1}2\}$ extension twins in both cases, although their spatial locations are different. These observations strongly corroborate with our transmission electron microscopic analysis of the indented samples. Finally, our simulations also indicate that, depending upon the crystal orientation, elastic recovery upon unloading may play important role in final surface morphology around the indented region.

Keywords: Nanoindentation; Elasto-plastic contact; Magnesium; Crystal plasticity; Pile-up

1. Introduction

The hexagonal closed-packed (hcp) crystal structure of magnesium is host to about 50 slip and twinning systems, nearly 30 of which are commonly reported in experiments [1]. However, the stresses required to activate these systems vary over a wide range. For example, the critical resolved shear stress (CRSS) required to activate the plastically softest $(0001)\,\langle 11\bar{2}0\rangle$ basal slip is nearly two orders of magnitude lower than the plastically hard non-basal slip modes [2,3]. The low-symmetry hcp crystal structure of Mg often limits the number of independent slip systems available to accommodate plastic deformation, thereby promoting twinning. Under nominally uniform loading, the dominance of particular slip or twin systems within a single crystal of Mg is typically determined by the orientation and sense of the loading directions with respect to its *c*-axis. For instance, *c*-axis extension results in the prevalence of the plastically soft $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ extension twinning; on the other hand, *c*-axis contraction results in the dominance of the much harder $\{11\bar{2}2\}\langle 11\bar{2}3\rangle$ pyramidal $\langle c+a\rangle$ slip. However, given the extremely low basal slip CRSS, a small misalignment between the loading axis and the crystallographic *c*-axis can cause profuse basal slip activity, leading to interacting effects even under simple loading conditions. The scenario can become significantly more complicated if the loading condition is complex. An important situation is that of an Mg crystal subjected to localized contact, e.g. indentation. The complexity of the stress state around an indenter, which depends on the geometric characteristics of the indenter and the crystal orientation, may give rise to rich interactions between the various deformation modes. These interactions may influence the local material flow and the macroscopic load–deformation characteristics. While there are several detailed experimental reports and computational efforts that probe the micro–macro nexus in Mg single crystals under simpler loading conditions [2,3], there is much less fundamental understanding regarding the nature of plastic deformation activity under such localized loading conditions.

Nanoindentation is a popular protocol for experimentally evaluating the mechanical properties of various classes of materials ranging from bulk materials to thin films [4–16]. The triaxial stress state induced during indentation varies spatially and evolves with increasing depth of indentation.

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Such a systematic, but highly heterogeneous, stress state offers a platform for the extraction of quantitative information regarding the deformation mechanisms as a function of varying levels of triaxiality. Furthermore, it is a valuable method, which may broadly resemble the complex stress states within individual grains of polycrystalline Mg subjected to intricate deformation processes. In particular, indentation of single crystals can provide insight into the orientation-dependent interacting effects arising from the dominant deformation mechanisms in their highly textured polycrystalline analogs. Although the micromechanics that prevails during indentation is reasonably well understood for face-centered cubic [17–21] and body-centered cubic crystals [22,23], similar detailed analysis for hcp single crystals in general [24,25], and Mg in particular [24,26–30], is nascent. Notably, none of the hcp-CPFE indentation simulations to date account for the role of deformation twinning [24,25,30]. A recent investigation on Mg single crystals included nanoindentation experiments and a theoretical analysis based on anisotropic elastic contact theory to rationalize the observed slip modes [28].

In this work, we present a detailed micromechanical analysis of the orientation-dependent Mg single crystal indentation response. To that end, we performed systematic three-dimensional finite-element-based hcp crystal plasticity (CPFE) simulations and nanoindentation experiments on Mg single crystals with different orientations. It is well known that twinning is one of the most important deformation modes in Mg and needs to be taken into account [29,31–33]. Therefore, we adopted an hcp-CPFE framework that includes constitutive descriptions of both slip and twinning (extension as well as contraction twinning) modes [3] to elucidate the manner in which plastic deformation evolves in the neighborhood of an indenter as a function of the crystal orientation. Recent nanoindentation experiments indicate a possible dichotomy with regards to the occurrence of extension twinning: the cono-spherical indentation experiments of Shin et al. [29] exhibited extension twinning when indented on both the basal and the $\{11\bar{2}0\}$ prismatic planes. On the other hand, the spherical nanoindentation experiments of Catoor et al. [28] showed extension twinning only in the case of indentation on the $\{10\bar{1}0\}$ plane, but no twinning in the case of indentation on the basal plane. By corroborating our simulations with experiments, we provide insight into these observations. We also assess the implications of the slip and twinning evolution on the load–depth curves, and the pile-up and sink-in behaviors.

In the following, we first discuss the experimental method adopted for indentation experiments on two distinct crystallographic planes of Mg single crystals: the (0001) basal plane and the $(11\bar{2}0)$ second-order prismatic plane. These experiments give primary information that includes the indentation force vs. depth $(P-\delta)$ response and the indentation-induced surface deformation characteristics (pile-up/sink-in) as a function of the crystal orientation. Further, we also discuss the observations from transmission electron microscopy (TEM) analysis performed on these indented crystals that highlight the deformation characteristics induced in the vicinity of the indenter. We corroborate these and other recent experimental observations with the predictions from our hcp-CPFE simulations.

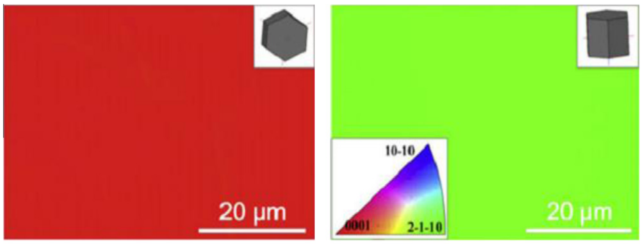
2. Experimental procedure

Single-crystalline Mg was fabricated by a modified Bridgman method, whereby 99.9% pure polycrystalline Mg was heated in a boron-nitride-sprayed graphite crucible in an argon atmosphere above its melting point. The fabricated single-crystal Mg was then sliced with a low-speed cutting wheel along two planes with different crystallographic orientations: (0001) basal plane and $(11\bar{2}0)$ second-order prismatic plane. Using electron backscatter diffraction (EBSD; HKL Nordlys Channel 5), we confirmed that the misorientation in the samples deviates less than 5° from the desired orientations (Fig. 1a and b). For the nanoindentation test, the samples were prepared by a standard metallographic grinding and polishing procedure, finishing with a 0.1 μm diamond suspension followed by electropolishing with a 5% perchloric acid to remove the mechanically damaged layer. The surface roughness was measured at less than 90 nm. Finally, the samples were annealed at 350 °C under an argon atmosphere to relieve the possible residual stress. Additional EBSD analysis showed no signs of recrystallization.

Using a Hysitron TriboLab® 750 Ubi nanoindenter equipped with a cono-spherical diamond tip of radius equal to 663 nm, series of load-controlled nanoindentation tests were performed on the basal and prismatic planes. The single crystals were loaded at a nominally constant indentation rate of 30 μN/s. Multiple indentation tests were performed decreasing the peak load from 6000 μN to 50 μN. For the indentation on (0001) plane, the measured indentation depths were 178 nm and 1300 nm for the peak loads of 6000 μN and 50 μN, respectively. Likewise, for the indentation on the $(11\bar{2}0)$ plane, the indentation depths were 178 nm (at 50 μN peak load) and 1500 nm at 6000 μN peak load. Some of the indents were sectioned using focused ion beam milling (FIB; Nova Nanolab 200, FEI) and these regions were observed using TEM (JEM-3000F). The surface morphologies of the indentation sites were analyzed using AFM (XE-70, Park Systems Co.) in the non-contact mode. A commercial cantilever (PPP-NCHR, NANOSENSORS™) having tip radius of curvature of less than 10 nm was used for this measurement. The measured area and scan resolution were 10 × 10 mm^{2}and 512 × 512 squared pixels, respectively.

3. Numerical simulation procedure

We performed three-dimensional finite element (FE) simulations using ABAQUS/STANDARD®. The next subsection describes the key numerical aspects of the FE model and SubSection 3.2 briefly discusses the underlying single CP constitutive model that includes slip and twinning.



(a) (b)

Fig. 1. EBSD orientation maps for (a) (0001) basal plane, (b) $\{11\bar{2}0\}$ second-order prismatic plane.