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Incipient plasticity and deformation mechanisms in single-crystal Mg during spherical nanoindentation

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Abstract

Incipient plasticity in Mg single crystals was investigated using the pop-ins generated during spherical nanoindentation on (0001), (10−12) and (10−10) surfaces. Representative deformed regions extracted from underneath indents by means of focused ion beam machining were examined by transmission electron microscopy (TEM) to identify the deformation mechanisms. Anisotropic elastic Hertzian contact theory was used to calculate indentation Schmid factors and the relevant resolved shear stresses at pop-in from the load–displacement curves. The pop-in statistics in conjunction with the TEM analysis showed that the most likely deformation mechanism responsible for pop-in is slip via ⟨a⟩ dislocations even in the case of indentation along the *c*-axis.

Keywords: Mg single crystal; Nanoindentation pop-in; Dislocation nucleation

1. Introduction

Consistent with the theoretical strengths measured in tensile tests of dislocation-free whiskers in the 1950s [1], nanoindentation [2–7] and micropillar compression tests [8] have in the last 15 years shown that theoretical strengths can be achieved in these geometries if small enough volumes are probed where the likelihood of finding pre-existing dislocations or dislocation sources that can be activated is minimal. The mechanism by which elasticity gives way to plasticity under these conditions is believed to be the nucleation of dislocations when the shear stresses approach theoretical limits ($\sim G/30$–$G/5$, where G is the shear modulus). In a load-controlled indentation test, the elastic–plastic transition is manifested as a sudden displacement excursion (pop-in) on an otherwise smooth load–displacement curve [3,4,9]. The pop-in phenomenon thus offers a unique opportunity to quantitatively study dislocation nucleation, a stress-assisted, thermally activated process [3,10]. Nucleation of dislocations can be homogeneous (in defect-free volumes [11]) or heterogeneous (i.e. assisted by the presence of defects such as surfaces [12], inclusions or precipitates [13], vacancies [14] or grain boundaries [15,16]). In addition, pop-ins can also occur at stresses much below the theoretical stress when sufficiently large volumes are probed due, presumably, to the activation of pre-existing dislocations [17–19]. As a practical matter, it may not always be possible, based simply on the relative magnitudes of the pop-in stresses, to differentiate between pop-ins that are the result of heterogeneous nucleation and those resulting from the activation of pre-existing dislocations. Nevertheless, it is a useful conceptual distinction to keep in mind.

The elastic and plastic anisotropy inherent in crystalline materials together with the complexity of the indentation

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stress fields make it difficult to determine the pertinent shear stress components responsible for the elastic–plastic transition. Therefore, pop-in studies typically employ the simpler approach of using isotropic linear elasticity to estimate the maximum shear stress under the indenter on a plane at 45° to the indenter axis, a plane with no particular significance since slip need not occur on such a plane in the general single-crystal case. The effect of crystallographic orientation, or which slip systems are likely to be activated first, cannot be determined within this framework. The problem becomes especially pronounced in the case of hexagonal close-packed (hcp) metals and, probably as a result of these difficulties, the geometrically simpler case of micropillars in compression has received greater attention in the literature [20–22]. Although a few isolated indentation studies have been performed on polycrystals [24,25], the pop-in behavior of single crystals of hcp metals such as Ti, Mg, Zr and Zn has not been thoroughly examined. The nanoindentation responses of hexagonal ceramics such as sapphire, and device materials such as GaAs, have been extensively studied. For example, Lloyd et al. [23] identified the active deformation mechanisms in these materials under Berkovich indentations. However, their main focus was not on pop-in behavior or the determination of dislocation nucleation stresses from pop-in loads.

Recently, Gao and Pharr [26] and Li et al. [27] have used anisotropic elastic contact theory to calculate indentation moduli, Hertzian stress fields and resolved shear stresses for any given crystallographic loading direction, thus bringing the mechanics of indentation in hcp crystals within reach. hcp metals exhibit several deformation modes such as slip via the $\langle a \rangle$ dislocations ($1/2\langle 1\,1\,{-2}\,0\rangle$) on the (0 0 0 1) basal and {1 0 −1 0} prism planes, via the $\langle c + a \rangle$ ($1/3\langle 1\,1\,{-2}\,3\rangle$) dislocations on the {1 1 −2 2} second-order pyramidal planes, as well as multiple twinning modes: e.g. $\{1\,0\,{-1}\,2\}\langle 1\,0\,{-1}\,1\rangle$ "tension" twinning and $\{1\,0\,{-1}\,1\}\langle 1\,0\,{-1}\,2\rangle$ "compression twinning". For uniaxial loading in relatively large specimens, their relative contributions of these twinning modes to plasticity can be ascertained by comparing the resolved shear stresses on different slip or twinning systems with the critical levels required for activation. However, for the case of indentation where the stress states are highly complex, a similar understanding is currently lacking at any length scale for hcp metals. Furthermore, if the indented volume is defect-free, as is often the case in nanoindentation experiments, incipient plasticity is controlled by dislocation nucleation as opposed to dislocation source activation, and the shear stresses on different types of slip systems can simultaneously approach theoretical limits at the elastic–plastic transition. It is unclear in such cases which deformation mechanism(s) will activate or dominate, and what factors determine the outcome of this competition.

To better understand some of these issues, we have investigated the elastic–plastic transition and the early stages of plasticity in Mg single crystals, a model hcp metal with a near ideal *c*/*a* ratio, by measuring the pop-in loads during spherical nanoindentation on three different crystallographic planes: (0 0 0 1), (1 0 −1 2) and (1 0 −1 0).

The plane normals in the two latter orientations are at 43° and 90° to the *c*-axis, respectively. The active deformation mechanisms were identified by performing a transmission electron microscopy (TEM) examination of the representative deformed regions underneath a few selected indentations. Using linear elasticity theory and Hertzian contact mechanics applied to anisotropic solids as in Li et al. [27], we analyzed the load–displacement curves and calculated the resolved shear stresses corresponding to the pop-in loads for the possible slip systems. Of the many different deformation mechanisms that become active during indentation, analysis of the pop-in data combined with TEM evidence was used to identify the most likely deformation mechanism responsible for the transition from elastic to plastic behavior.

2. Experimental procedure

2.1. Material and surface preparation

A Mg single crystal was grown using a modified vertical Bridgman method in a Crystalox CGX65 crystal growth furnace as described elsewhere [28]. Three approximately rectangular specimens, 2–3 mm in thickness, were spark cut from this crystal with surfaces parallel to (0 0 0 1), (1 0 −1 2) and (1 0 −1 0); these will be referred to hereafter by their respective Miller indices. The specimens were carefully ground using 1200, 2400 and 4000 grit SiC, and then hand-polished using oil-based diamond suspensions and a viscous blend of alumina and silica. Finally, an ethanol-based chemical polishing solution (4 ml HNO_3 + 6.5 ml HCl + 100 ml ethanol) was used to remove the mechanical damage resulting from the above grinding and polishing operations. A 3 min immersion in the solution and agitation of the samples was observed to dissolve at least 30 μm from the surface. The amount of material removed was estimated by making relatively large indents with a Vickers indenter and measuring the impression sizes before and after chemical dissolution.

2.2. Nanoindentation

Nanoindentation tests were performed using a spherical diamond indenter with a radius of 3.3 μm mounted in an MTS XP nanoindenter. The measurements were made at an exponential loading rate or a constant $\dot{P}/P = 0.05\ \mathrm{s}^{-1}$ (where P is the load and $\dot{P}$ is its derivative w.r.t. time). The radius of the indenter tip was calculated by fitting the elastic regions of the load–displacement curves from nanoindentation experiments on single-crystal tungsten to curves predicted by Hertzian contact theory [29]. For each specimen in a given condition, more than 100 indentations spaced at least 25 μm apart were made.

Earlier studies have shown that surface finish and near-surface damage have a significant effect on the pop-in