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Continuum modeling of dislocation starvation and subsequent nucleation in nano-pillar compressions

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The mechanical behavior of single crystalline aluminum nano-pillars under uniaxial compression differs from bulk Al in that the former is characterized by a smoother transition from elasticity to plasticity. We propose an extension of the phenomenological model of dislocation starvation originally proposed in [Greer and Nix, Phys. Rev. B 73 (2006) 245410] additionally accounting for dislocation nucleation. The calibrated and validated continuum model successfully captures the intrinsic mechanisms leading to the transition from dislocation starvation to dislocation nucleation in fcc nano-pillars.

Keywords: Continuum model; Nanopillar; Crystal plasticity; Dislocation starvation

The enormous advances in fabrication processes, computational modeling, and experimental characterization methods at the submicron scale have catalyzed the emergence of a new era in materials science, specifically in the area of structural materials. Namely, the development of novel material systems with radically superior properties will be achieved through architectural control at the appropriate microstructural scales. This will result in the ability to develop structural materials with vastly superior properties [1].

In order to utilize these principles towards insertion into relevant structural applications, it is essential to assess mechanical properties and deformation mechanisms in surface-dominated structures with reduced dimensions (i.e. not via nanoindentation), as they will likely comprise the aforementioned architectural constituents. Recently, it has been shown that at the micron- and submicron scales, the presence of free surfaces in small-scale samples dramatically affects crystalline strength [2–5]. In these studies, cylindrical nano-pillars were fabricated mainly by the use of the focused ion beam (FIB) and, remarkably, the results of all of these reports for fcc metals show the same power-law dependence between the flow stress and sample size, implying that this scaling might be universal [6].

To date, a fundamental understanding of nano-scale mechanical response in surface dominated structures with sub-micron dimensions is still elusive [7–10]. Because of the intrinsic nature of the continuum approach *a priori* contradicting the stochastic and highly size dependent nature of nanoscale mechanics, continuum finite element methods have been scarcely used to model nanopillar compressions. Among these efforts, Zhang et al. used an isotropic plasticity finite element model to provide a set of guidelines to minimize the experimental artifacts [11]. They mostly emphasize proper choice of pillar diameters and aspect ratios while minimizing both the vertical taper angle and indenter head-pillar top misalignment. Raabe et al. followed up on this work by making use of a crystal plasticity model focusing on studying the effect of crystal orientation and its stability on the deformation mechanisms [12]. Schuster et al. also built up on Zhang's efforts by applying their model to metallic glass and evaluating the effect of specimen taper on the compressive strength [13]. This was further described by Wu et al. by specifically studying the geometrical constraint dependent stress distribution [14]. Shade et al. emphasized the non-constitutive law related hardening caused by the lateral stiffness of the indenter by means of crystal plasticity [15], and Chen et al. recently focused on local stress concentration in metallic glass pillar bending, using an isotropic plasticity model [16]. Finally, Hurtado and Ortiz proposed a non-local model accounting for surface effects during pillar compression [17], and Zhang and Aifantis developed a strain gradient model capturing the strain bursts in micropillars [18].

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Setting the burst-ridden stochastic nature of nano-deformation aside, we propose a model capable of capturing the macroscopic stress–strain behavior of single crystalline Al nano-pillar compressions. This involves the consideration of the phenomenological 'dislocation starvation' model originally proposed by Greer et al. [19,20], which rationalizes the initial stage of yielding by a starvation of the initially present mobile dislocations. A crystal plasticity model accounting for size-dependent dislocation starvation and subsequent nucleation is thus proposed, calibrated and validated against experiments.

It has now been ubiquitously shown that even in the absence of strong strain gradients, small-scale uniaxial compression and tension experiments reveal a so-called size effect, or size-induced strengthening of single crystals in micron- and sub-micron structures. Deeply in the sub-micron regime, this size dependence has been at least in part rationalized by dislocation starvation followed by nucleation mechanism [2–4,6].

Based on the classical work of Johnston and Gilman [21,22], Greer and Nix proposed a phenomenological model [19,20] in which mobile dislocations initially present in the sub-micron pillar annihilate in the vicinity of a free surface. A follow-up on this approach accounting for the kinetics of nucleation was recently proposed by Nix and Lee [23]. Accounting for the resistance stress necessary to overcome the elastic dislocation interactions, and lengthening and bowing out of dislocations in their slip planes, the average dislocation resistance shear stress associated to the dislocation starvation is given by [20]:

$$s_{starv} = 0.5\mu b\sqrt{\rho} + 1.4\frac{\mu b}{4\pi a(1-\nu)}\ln\left(\frac{\alpha a}{b}\right) \tag{1}$$

where μ, ν, b and α are the shear modulus, the Poisson's ratio, the Burgers vector and a constant of order unity, and where the instantaneous pillar diameter a and density ρ are given by:

$$\begin{cases} a = a_0(1-\epsilon)^{0.5} \\ \rho = \rho_0 + \frac{\left(\delta - \frac{1}{a}\right)}{b}\frac{\epsilon_p}{M} \end{cases} \tag{2}$$

where ρ_0 and a_0 are the initial dislocation density and pillar diameter, ϵ and ϵ_p are the engineering overall strain and plastic strain, M is the Schmid factor, and δ is the breeding coefficient (representing the inverse of the distance a dislocation travels before replicating itself).

We postulate here that the starvation of pre-existing dislocations and nucleation of new ones should be independently characterized by two reference critical resolved shear stresses (CRSSs). We thus propose another model based on Eq. (1) taking into account a reference (initial) starvation CRSS $s_{0,starv}$ and a reference nucleation CRSS $s_{0,nucl}$ such that, for each slip system i:

$$s_0^i = \mathrm{Min}\left(\left(1-\frac{\epsilon_p}{\epsilon_p^{starv}}\right)s_{0,starv} + \frac{\epsilon_p}{\epsilon_p^{starv}}s_{0,nucl}, s_{0,nucl}\right) \tag{3}$$

where

$$s_{0,starv} = 0.5\mu b\sqrt{\rho_0} + 1.4\frac{\mu b}{4\pi a_0(1-\nu)}\ln\left(\frac{\alpha a_0}{b}\right) \tag{4}$$

and where ϵ_p^{starv} is a model parameter corresponding to the plastic strain for which nucleation dislocation is more favorable than dislocation starvation, and at which the nucleation CRSS $s_{0,nucl}$ is reached. In this model, the CRSS is equal to the starvation CRSS at initial yielding, and then linearly increases as a function of ϵ_p as the mobile dislocations are moving towards the free surface, while nucleation processes are becoming increasingly more prevalent. Once all mobile dislocations have been annihilated, plasticity is fully nucleation driven. The linear dependence has been chosen as a simple first approximation and is justified below.

The rate-independent constitutive model adopted here is following Ref. [24]. The reader is invited to consult this reference for a complete description.

In the following, we make the hypothesis that the crystal is small enough so that dislocations exit the crystal before interacting significantly (i.e. no hardening). As a consequence $s^i = s_0^i$ at all time (see Eq. (3)).

For both the calibration and the validation, experimental results of mono-crystalline pillar compressions are chosen as reference [5]. These pillars are made of high purity aluminum (5N purity, ESPI Metals), annealed under vacuum at 350 °C overnight and electropolished by Able Electropolishing. Grain orientation maps were obtained by automatic indexing of electron backscatter diffraction (EBSD) patterns using a Zeiss1550 VP field emission scanning electron microscope equipped with an EBSD system from Oxford Instruments. Once a suitable grain was chosen, nanopillar samples were fabricated using a FEI Nova 200 scanning electron microscope with a focused ion beam (FIB), see Ref. [5] for more details.

For each considered nano-pillar, the top diameter and tapering angle were extracted by means of a scanning electron microscopy (SEM). The height being the most ambiguous geometric parameter due to FIB undercutting, it was determined by a stiffness-matching method [25]. The average diameter calculated along with this height was also calculated. The geometrical parameters of the two chosen pillars, as well as their finite element characteristics are gathered in Table 1.

Pillar A (see Table 1) was chosen for calibration. The elastic constants for aluminum at room temperature, the Burgers vector, the initial dislocation density for nano-pillar (approximating its value for aluminum by the one for gold), and α were taken from the literature [20,26,27]. The two remaining parameters $s_{0,nucl}$ and ϵ_p^{starv} were then independently adjusted to fit the experimental stress strain curve of the nanopillar indentation. Note that, by definition, the two calibrated parameters modify the final strength and the plastic strain at which this final strength is reached; as a consequence, their unicity is guaranteed.

The simulations were done using Abaqus/Explicit [28] under quasi-static loading, clamping the base of the nanopillar, while applying a free slip boundary condition on the top surface. Both temporal and spatial convergence were checked. The parameters are given in Table 2, the stress–strain curve in Figure 1, and the resulting Von Mises stress field in Figure 2.

As previously observed in other efforts [5,13], the experimental stress–strain curve appears to be abnormally compliant during the initial part of loading. This