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Surface effects and the size-dependent hardening and strengthening of nickel micropillars

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ABSTRACT

We evaluate the extent to which two mechanisms contribute to the observed size effect of the ultimate yield strength of micropillars of diameters in the range of $1-30\ \mu m$: dislocation pile-ups, modeled by means of a physically based non-local single-crystal plasticity model; and the short-range interaction of dislocations with the free surface of the micropillars, e.g., through the formation of surface steps. To this end, we formulate a crystal-plasticity model that accounts for the self-energy of geometrically necessary dislocations and the formation energy of dislocation steps at the boundary of the solid. These two additional sources of energy have the effect of rendering the internal energy of the solid non-local, thereby introducing the possibility of size effects. By way of validation of the model, we simulate the uniaxial compression tests on [269] nickel micropillars of Dimiduk et al. (2005). The calculated dependence of the ultimate strength of the micropillars exhibits strong power-law behavior, and is in good agreement with observation. Our analysis suggests that non-local hardening due to the self-energy of geometrically necessary dislocations does not suffice to account for the observed size effect of the ultimate yield strength of micropillars, and that surface effects, such as resulting from the formation energy of dislocation steps, contribute significantly to that size effect.

1. Introduction

A vast and long-standing body of experimental evidence conclusively establishes the fact that the yield strength of crystals is size-dependent. Indeed, marked size effects are found in conventional polycrystals (Dao et al., 2007), in the form of the classical Hall–Petch effect (Hall, 1951; Petch, 1953), indentation tests on single crystals (Stelmashenko et al., 1993), torsion tests on microwires (Fleck et al., 1994), microbend tests on thin films (Stolken and Evans, 1998), tension tests in Fe, Cu and Ag whiskers (Brenner, 1956), and more recently in compression tests of Ni (Uchic et al., 2004), Au (Greer et al., 2005), Cu (Kiener et al., 2006) and Al (Ng and Ngan, 2008) micropillars. This size dependence can be exploited to fabricate materials combining both high strength and ductility, e.g., by the equal channel angular extrusion (ECAE) process (Segal, 1995; Zhu and Lowe, 2000; Beyerlein et al., 2003), and in other ways.

The size effect of the yield strength in conventional polycrystals has been extensively studied and modeled. In particular, the Hall–Petch effect has been variously attributed to the formation of dislocation pile-ups in grains

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(Hirth and Lothe, 1982) and the formation of sub-grain dislocation structures (Ortiz and Repetto, 1999; Ortiz et al., 2000; Aubry and Ortiz, 2003; Conti and Ortiz, 2005). In these models, the increase in strength ultimately arises from the self-energy of the dislocations, which has the effect of introducing a length scale into the response of the crystal. Thus, dislocation line-tension results in Taylor scaling of the yield strength, $\sigma \sim \mu b\sqrt{\rho}$ (Taylor, 1934, 1938), and in an optimal mean dislocation density $\rho \sim 1/bd$ which scales in inverse proportion to the size d of the crystal (Ortiz and Repetto, 1999; Aubry and Ortiz, 2003; Conti and Ortiz, 2005). Here μ denotes a reference shear modulus of the crystal and b the size of the Burgers vector. These two scaling relations combine to give the Hall–Petch relation $\sigma \sim \mu\sqrt{b/d}$. Hall–Petch scaling is extraordinarily robust over crystal sizes spanning many orders of magnitude, but tends to break down at the nano-scale as a result of the activation of additional deformation mechanisms such as grain-boundary sliding (Schiotz et al., 1998). It bears emphasis that the dislocation pile-up mechanism and the formation of sub-grain structures can operate under conditions of nominal uniform deformation. However, deformation gradients can in effect introduce additional length scales and result in size effects. This geometrical scaling is the basis of strain-gradient plasticity theories (Fleck and Hutchinson, 1993, 1997; Fleck et al., 1994; Nix and Gao, 1998).

The origins of the experimentally observed size effect in nano and micropillars is comparatively less well-understood at present and the subject of some scientific controversy (Dou and Derby, 2009; Norfleet et al., 2008; Kiener et al., 2006). Unlike torsion tests, the uniaxial tension and compression tests of pillars involve conditions of nominal uniform deformation and, hence, strain-gradient plasticity theories do not readily apply. Uchic et al. (2004) attributed the increase in strength in single-slip Ni micropillars to reductions in defect multiplication and storage as the sample diameter decreases. Greer et al. (2005) found a similar trend in multislip compression tests of Au nanopillars, which they explained by recourse to the concept of dislocation starvation. In the dislocation-starvation model, the diameter of the pillar is assumed to be comparable to the mean-internal path for dislocation multiplication by double cross-slip (Gilman, 1969), a dominant mechanism of dislocation multiplication in crystals. Under these conditions, dislocations may exit the crystal before breeding may take place, leaving the crystal dislocation-starved. Dislocation generation can then only occur through the activation of fixed sources, which requires the application of high stresses to the crystal. Norfleet et al. (2008) studied the formation of dislocation structures in nickel micropillars after compression testing and measured dislocation densities for pillar diameters ranging from 1 to 20 μm. Based on their observations, they concluded that the combined effects of lattice friction, source-truncation hardening, and forest hardening predict strength values much smaller than the observed ones, which suggests that other mechanisms also contribute significantly to the size effect. Dou and Derby (2009) compiled experimental data available in the literature on compression tests of micropillars. For a variety of face-centered-cubic (FCC) metals, including Ni, Au, Cu and Al, this compilation is suggestive of universal power-law scaling of the yield strength normalized by the material shear modulus with the pillar diameter normalized by the Burgers vector length. This universal power-law behavior points to dislocations as the origin of the size effect of yield strength in micropillars, but does not identify the precise mechanism by which dislocations cause the size effect.

The effect of surface mechanisms on the nano and micropillar strength has been comparatively less studied. Most micropillar experiments reported in the literature consider samples fabricated using the focussed-ion-beam (FIB) technique, which consists in Ga^+ ion bombardment of bulk crystals to obtain the desired shape. This fabrication process necessarily results in ion implantation during milling, thus altering the chemical composition and mechanical properties of the sample near the surface. To investigate FIB surface damage, Kiener et al. (2007) measured the Ga^+ concentration on Cu samples. The formation of an amorphous layer due to Ga^+ implantation was observed, with penetration depths up to 50 nm and concentrations of 20 at.% close to the sample surface, depending on the incidence angle, ion energy and ion dose. Similar observations have been made for Au, Pt and W specimens (Arnold et al., 2003; Machalett et al., 2000). Despite this extensive experimental evidence, the effect of FIB damage as regards the mechanical properties of micropillars appears to have eluded theoretical investigation to date.

As pointed out by Needleman (2000) and Needleman and Van der Giessen (2001), direct atomistic simulations and discrete dislocation dynamics (DDD) simulations prove insightful in the understanding of crystal plasticity. The initial dislocation structures, nucleation of dislocations from Frank-read sources, annihilation and dislocation interaction can be directly modeled using DDD, and their role in size effects found in single- and poly-crystalline materials has been studied using DDD simulations (Guruprasad and Benzerga, 2008; Balint et al., 2006). For example, Deshpande et al. (2005) carried out 2D simulations to study the influence of the rotation of the loading axis in the size-dependent strength of micropillars under uniform loading conditions. From their results, they concluded that for unconstrained rotation of loading axis the major size effect was related to the high stresses needed to nucleate dislocations from sources, as dislocations rapidly escape the crystal before encountering obstacles, thus supporting the dislocation starvation mechanism. A similar conclusion was obtained from 3D micropillar DDD simulations of micropillars by Tang et al. (2007).

Despite major advances in DDD simulations during the last decade, they remain a computationally demanding technology for large-scale problems. Moreover, DDD simulations necessarily involve relatively high strain rates, which are several orders of magnitude higher than the strain rates found in micropillar compression experiments. These issues can be easily addressed by dislocation-based continuum crystal plasticity theories, thus they play a crucial role in the feasibility of computational multiscale simulations of micromaterials. Finite-element simulations using traditional size-independent crystal plasticity constitutive models have been employed in the study of the effect of the initial orientation, sample geometry and lateral constraints (Raabe et al., 2007; Shade et al., 2009) on micropillar compression tests. Phenomenological continuum dislocation methods have recently been employed to study the effect of dislocation sources