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IN-SITU INVESTIGATION of PLASTICITY at NANO-SCALE

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Summary
Mechanical behavior of crystals is dictated by dislocation motion in response to applied force. While it is extremely difficult to directly observe the motion of individual dislocations, several correlations can be made between the microscopic stress-strain behavior and dislocation activity. Here, we present for the first time the differences observed between mechanical behavior in two fundamental types of crystals: face-centered cubic, fcc (Au, Cu, Al, Ni, etc.) and body-centered cubic, bcc (W, Cr, Mo, Nb, etc.) with sub-micron dimensions subjected to *in-situ* micro-compression in SEM chamber. In a striking deviation from classical mechanics, there is a significant increase in strength as crystal size is reduced to 100nm; however in gold crystals (fcc) the highest strength achieved represents 44% of its theoretical strength while in molybdenum crystals (bcc) it is only 7%. Moreover, unlike in bulk where plasticity commences in a smooth fashion, both nano-crystals exhibit numerous discrete strain bursts during plastic deformation. These remarkable differences in mechanical response of fcc and bcc crystals to uniaxial micro-compression challenge the applicability of conventional strain-hardening to nano-scale crystals. We postulate that they arise from significant differences in dislocation behavior between fcc and bcc crystals at nanoscale and serve as the fundamental reason for the observed differences in their plastic deformation. Namely, dislocation starvation is the predominant mechanism of plasticity in nano-scale fcc crystals while junction formation and subsequent hardening characterize bcc plasticity, as confirmed by the microstructural electron microscopy. Experimentally obtained stress-strain curves together with video frames during deformation and cross-sectional TEM analysis are presented, and a statistical analysis of avalanche-like strain bursts is performed for both crystals and compared with stochastic models.

In this work we investigate flow stress as a function of diameter in several fcc metals (gold, aluminum, and copper) and bcc metals (molybdenum, niobium, and tungsten) single crystal nano-pillars subjected to uniaxial micro-compression. The results that follow suggest that fcc and bcc crystals have fundamentally different plasticity mechanisms when reduced to nano-scale with significant strain-hardening present in the latter and virtually none in the former. The attainment of nearly 50% of the theoretical strength in Au suggests that plasticity is likely controlled by nucleation of new dislocations rather than by interactions of the pre-existing ones. On the contrary, the smallest Mo nano-pillar achieves only ~7% of its theoretical strength, implying that plasticity is likely driven by the intricate motion and interactions of dislocations inside the pillar rather than by nucleation events.

Single crystal nano-pillars described in this work were fabricated via Focused Ion Beam (FIB) and subsequently uniaxially compressed along <001> direction with a flat punch tip of 30 μm diameter. The specifics of nano-pillar fabrication are based on the authors' previous work [1], however, unlike previously reported, the testing was performed *in-situ* in "SEMentor," a one-of-a-kind instrument, which combines the strengths of SEM (for visualization) and Nanoindenter (precise loading control). SEM images of gold (initial diameter = 185 nm) nano-pillar before and after compression showing homogeneous deformation are shown in Fig. 1(a) and (b), and a compressed Mo pillar with clear multiple crystallographic slip lines is shown in Fig. 1(c).

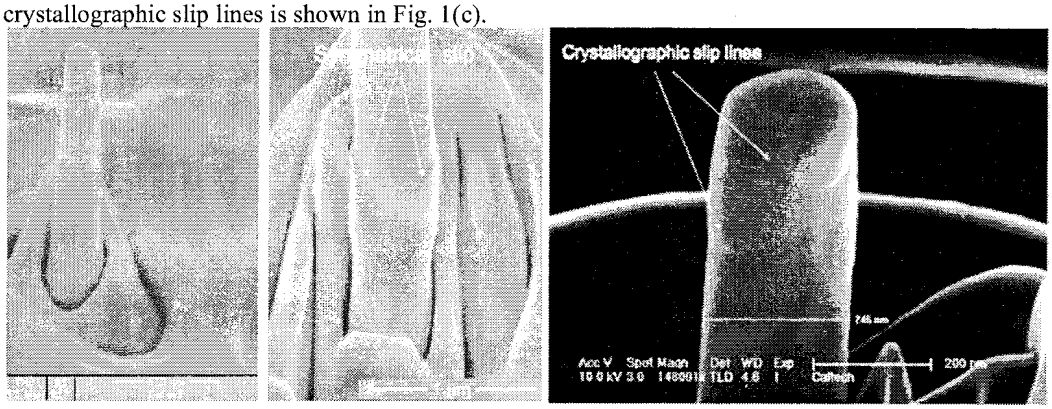

Figure 1. (a) Au <001>-oriented pillar before compression with initial diameter of 185 nm.
(b) Mid-section of a representative gold pillar after compression showing symmetric double-slip.
(c) Mo <001>-oriented pillar with initial diameter of 205 nm after compression (52° tilt angle for all).

Load-displacement data collected during the compressions were subsequently converted into uniaxial stress-plastic strain relationship, and some of the representative curves for both crystals are shown in Fig. 2.

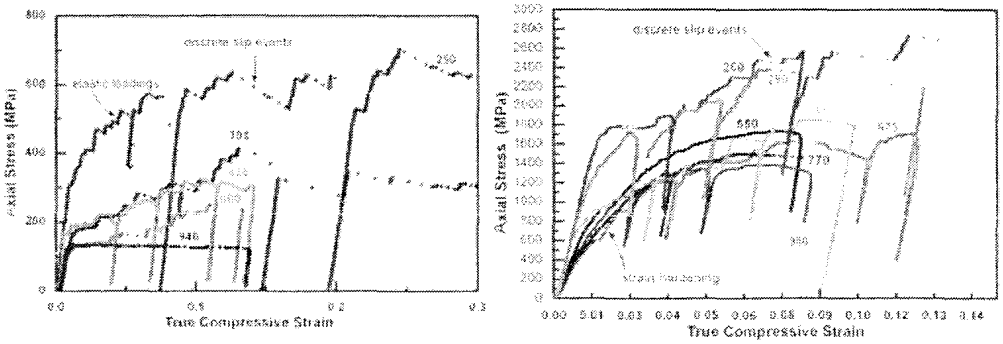

Figure 2. Some representative stress vs. strain curves for (a) fcc Au and (b) bcc Mo nano-pillars. Some pillars were intentionally unloaded and re-loaded several times.

An interesting feature of these curves is that unlike in a typical smooth stress-strain plot of bulk single crystals, the stress-strain relationship for smaller pillars does not undergo Stage II hardening, but instead is composed exclusively of elastic loading segments followed by discrete displacement bursts. These bursts are likely correlated with the initiation of dislocation avalanches, activated in response to the shear stress in the glide plane. Contrary to the very discontinuous stress–strain behavior in gold, the results for molybdenum nano-pillars subjected to the same uniaxial deformation are drastically different, as shown in Fig. 2(b). In the early stages of post-yield deformation clear strain-hardening is observed for most curves, which is manifested through the non-linearity of the stress vs. strain relationship. Interestingly, larger pillars exhibit a smoother and much more continuous deformation compared with smaller pillars, which show several discrete bursts throughout the process. The scaling relationship between attained stresses at a particular strain and diameter for both types of crystals clearly shows a power-law type dependence, as can be seen in the log-log plot in Fig. 3. While both crystals exhibit a power-law trend, the scaling slopes are vastly different: -0.97 for gold and -0.47 for molybdenum, indicating that the size effect is much more pronounced in fcc crystals compared with bcc. In addition, the smallest (205nm) Mo nano-pillar reaches the stress of 2.85 GPa (7% of theoretical strength) compared with ~800 MPa (44% of ideal strength) for a 200nm Au at equivalent strain. These nano-pillars can sustain strengths much higher than their bulk equivalents, of ~450MPa for Mo[2] and 25MPa for Au[3].

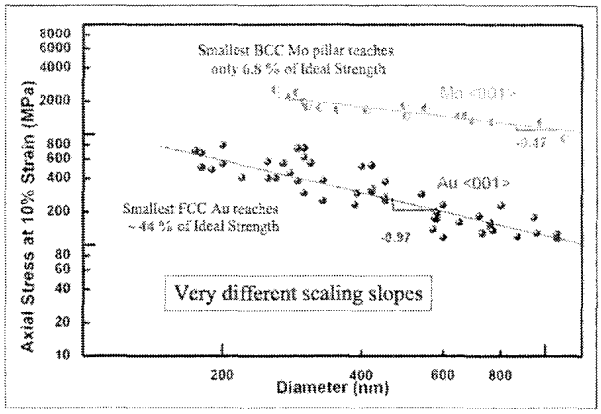

Figure 3. Log-log plot of flow stress as a function of initial diameter representing scaling laws for Mo and Au. The strengthening slope for gold (fcc) is ~ 2x higher than that for molybdenum (bcc).

These findings suggest that fundamentally different dislocation motion mechanisms might be operating in fcc and bcc crystals at nano-scale. The experimental results reported here are consistent with recent computational findings by molecular dynamics (MD) and 2-D and 3-D dislocation dynamics (DD) simulations [4-6]. Moreover, post-mortem cross-sectional TEM analysis of the deformed pillars reveals that Au crystals are nearly free of dislocations while Mo has ubiquitous dislocation loops concentrated primarily near the surface. Avalanche-like displacement burst behavior prevalent in all deformation curves is found to be independent of crystal structure and of specimen size, however the probability of slip events smaller than ~ 10 Burgers vectors is found to be lower than previously predicted.

References

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