

## Comment on “Effects of focused ion beam milling on the nanomechanical behavior of a molybdenum-alloy single crystal” *Appl. Phys. Lett.* 91, 111915 (2007)

Julia R. Greer,<sup>1,a)</sup> Horacio Espinosa,<sup>2</sup> K. T. Ramesh,<sup>3</sup> and Edward Nadgorny<sup>4</sup>

<sup>1</sup>Materials Science Department, California Institute of Technology, Pasadena, California 91125, USA

<sup>2</sup>Department of Mechanical Engineering, Northwestern University, Evanston, Illinois 60208, USA

<sup>3</sup>Department of Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland 21218, USA

<sup>4</sup>Physics Department, Michigan Technological University, Houghton, Michigan 49931, USA

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While this article provides insight into differences in mechanics between Ga<sup>+</sup>-irradiated and “pure” surfaces of molybdenum, there are several statements that are either inaccurate or poorly stated. It is clear that when a surface is directly irradiated by orthogonal ion beam (0.07–0.21 mW), a focused ion beam (FIB) damage layer will likely form and affect the strength. However, this finding does not provide adequate foundation for raising the question of FIB-induced hardening in nanopillars, given the vast differences between these experiments and procedure used in pillar fabrication. These issues would cause considerable confusion and result in disservice to mechanical testing community if not clarified.

1. In reviewing previously published work, the authors state that “none of the measurements have come close to the theoretical strength, even though some of the pillar diameters were small enough (submicron) that they would be expected to contain few or no dislocations.” This statement is not true—for example, in the work of Greer and Nix as well as of Volkert and Lilleodden, the smallest gold pillars (diameter ~300 nm) deformed at the stress of ~800 MPa, which represents 44.4% of the theoretical strength and is  $\sim\mu/27$ .<sup>1,2</sup> The authors claim that their “micropillars all yielded at shear strengths close to the theoretical  $\sim\mu/25$ .” In addition, this estimate of theoretical strength is relatively old as it dates back to Frenkel in 1926,<sup>3</sup> and since then many calculations and models have provided more specific estimates different for different materials, assumptions, and potentials. For instance, estimates of the theoretical shear strength of the Au crystal range from 0.74 to 10.9 GPa, i.e., roughly from  $\mu/33$  to  $\mu/2$ .<sup>4</sup>

2. The authors arrive at the conclusion that their FIB-damaged layer is different from a nondamaged layer by performing a series of nanoindentation tests on FIB irradiated versus pristine sample surface. The authors then infer from that data that the results from FIB-machined samples reported up to date are questionable. However, the experimental conditions described in this work are vastly different from those used in pillar fabrication. In the reported study, the

beam is directed orthogonally to the surface. However, when a pillar is fabricated using the FIB, the ion beam is oriented at grazing incidence to the feature and Never directly onto the pillar. Although the FIB damage cannot be completely excluded, the beam intensity distribution is angular, resulting in a very small fraction of the Ga ions actually producing damage by implantation into the pillar sides.<sup>5</sup> Moreover, the top of the pillar is never subjected to the Ga<sup>+</sup> ions. The Auger profile studies showing how little Ga implantation is present in the pillars are described in detail in Ref. 1. Therefore, the authors’ data do not provide significant information on the possible damage due to the ion implantation in real micropillar specimens.

In addition, size effects have been seen in other geometries dominated by the free surfaces, for example, freestanding thin films and 3D Discrete Dislocation Dynamics (DDD) studies on pillars, which clearly demonstrate such size effects and were not prepared by using the FIB.<sup>6–10</sup> The latest DDD results based on Frank networks also demonstrate the “staircaselike” stress-strain behavior and provide an explanation for the starvation argument first proposed in Ref. 1

3. The current and voltage settings of the ion beam reported here are at least an order of magnitude higher than those used in nanopillar fabrication. For example, the beam power used in the final steps of gold pillar fabrication is  $\sim 3 \times 10^{-4}$  mW while that used by Bei *et al.* varies between 0.07 and 0.21 mW.

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<sup>a)</sup>Electronic mail: jrgreer@caltech.edu.