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The Contribution of Back Stress to Strength in Nanomaterials

A J. Wagner[1], E.D. Hintsala[1], U.R. Kortshagen[2], W.W. Gerberich[1], and K.A. Mkhoyan[1]

[1.] Department of Chemical Engineering & Materials Science, University of Minnesota, Minneapolis, US.
[2.] Department of Mechanical Engineering, University of Minnesota, Minneapolis, US.

The effects of length scale on mechanical properties are still not fully understood despite being an area of active research for several years. Previous results have shown hardened structures approaching or exceeding their theoretical strength. The high contact stress of 40-100 nm Si nanospheres was explained as the result of a pressure effect which would also increase the modulus of elasticity [1], however with strains as high as 30% the contact stress should be well into the plasticity regime and reduce the flow stress. Comparable results were found for AFM nanoindentation of Si with sub-5 nm penetration [2]. Thin films of Permalloy 2.5 nm to 35 nm thick demonstrated an experimental loading stiffness two to three times larger than predicted using Hertzian contact theory [3]. While the higher modulus of the sapphire substrate and pile-up around the indenter may partially account for these results, it is proposed that back stresses from confined dislocations play the largest role in the high strengths observed. Understanding this behavior is critical for optimization of materials properties as nanoscale technologies mature.

To explain these findings we have performed *in-situ* compression of H-terminated and oxide-passivated crystalline Si nanocubes (NCs) inside the TEM. Defect-free Si nanocrystals composed of H-terminated [001] facets were synthesized in a flow-through plasma reactor producing NCs predominately 30 nm in size but ranging from 20 nm to 75 nm [4]. Nanocrystals were compressed with a Hysitron PI95 PicoIndenter in an FEI Tecnai T12 TEM operating at 120 keV while imaged with a TV-rate camera. Specimens were transferred from the reactor to the microscope within one hour of growth and remained nominally oxide-free. Within one day the H-terminated NCs became passivated by a 23 Å oxide layer. NCs were compressed along their [001] direction in displacement control with rates of 0.5 to 2.0 nm/sec. The electron beam was oriented along either the [100] or [110] zone axis providing for contrast of the $\{111\}\langle 1\bar{1}0\rangle$ silicon slip system.

A series of displacement excursions following initial elastic deformation were observed during uniaxial compression (Figure 1). Discontinuous yielding in the load-displacement curves for 30 nm oxide-passivated Si NCs (Figure 2) which can be associated with dislocation emission complement the excursions seen while imaging. It is proposed that the increases in flow strength are comprised of an effective stress associated with repeated dislocation nucleation and a back stress associated with the interaction of dislocations contained within the constrained volume. By estimating the number of dislocations necessary to produce each displacement excursion, we can determine the associated back stress based on a pile-up model as given in [5]. Results for 30-60 nm NCs suggest that the contribution of back stress increases with confinement while the effective stress for dislocation motion remains constant. As confinement increases the constrained plastic flow builds up large back stresses which contribute to the high flow stress observed here.

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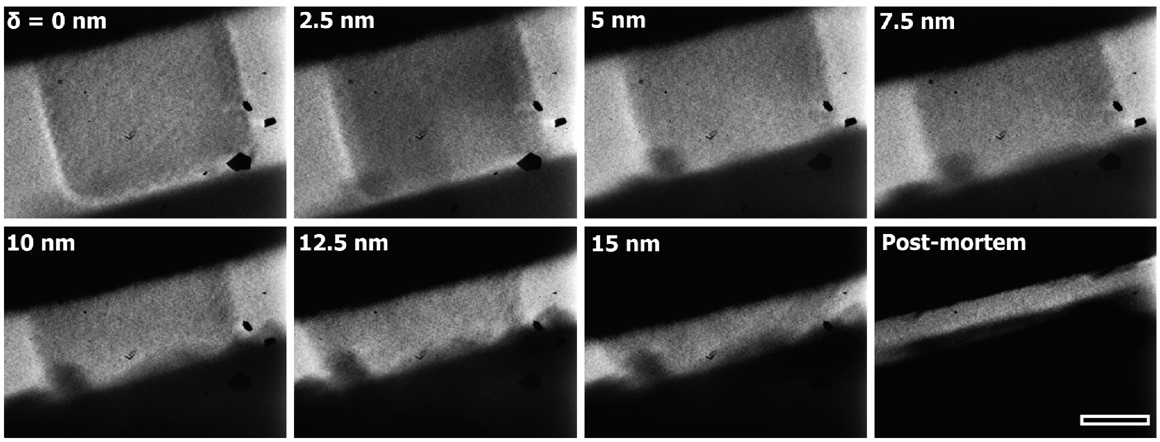

Figure 1. In-situ [100]-oriented BF images of 31 nm Si NC during 0.8 nm/sec compression. Little contrast is visible suggesting dislocations have Burgers vectors invisible in this orientation. Dimension of the NC at the indenter surface stays largely constant suggesting high friction forces while particle expands at the substrate interface. The final frame shows the remaining 3 nm thick NC adhered to the diamond indenter. Scale bar is 10 nm.

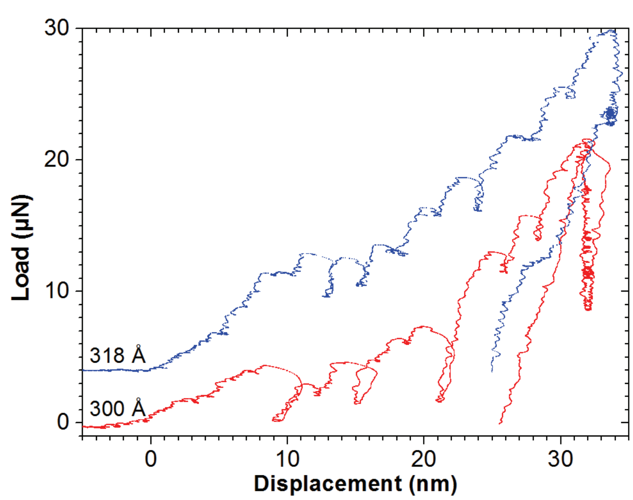

Figure 2. Load-displacement curves for ~30 nm Si NCs passivated with 2.3 nm oxide on Si substrate. A series of load drops with displacement excursions on the order of 2 to 3 nm are attributed to dislocation nucleation. Residual displacements between 4-7 nm agree well with post-mortem imaging of compressed NC.

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