APPLICATION NOTE

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In-Situ Compression of Nanopillars

Nanomechanical testing in the transmission electron microscope

The unusually high strength exhibited by many nanoscale materials has been a topic of discussion since the phenomenon was observed in metal whiskers in the 1950s. While the role of dislocations in the mechanical properties of bulk materials is fairly well known, the theories developed to predict and explain the properties of bulk materials cannot always be extended to nanoscale volumes, and testing these properties directly can be troublesome.

One major obstacle in evaluating the mechanical properties of nanoscale materials using traditional testing methods is the inability to see a deformation event as it occurs. Since contact with a nanoscale structure can induce deformation, it is necessary to determine the location of the object without making contact prior to testing.

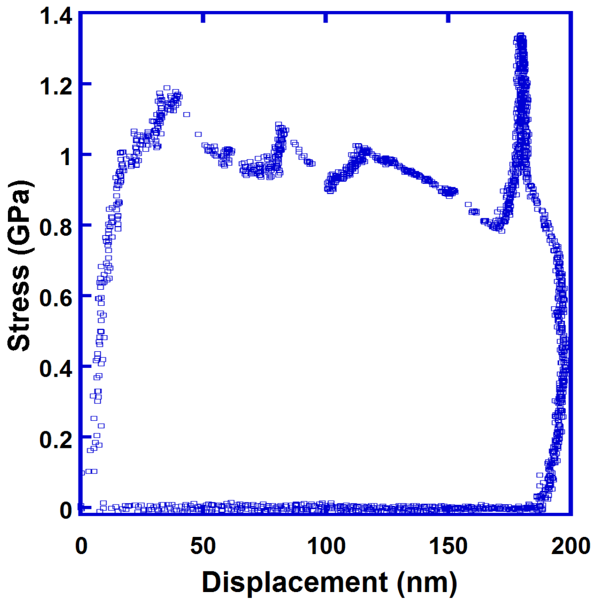

Figure 2: Stress vs. Displacement curve from the compression test in Figure 1. Stress is calculated from the applied load and the measured instantaneous contact area.

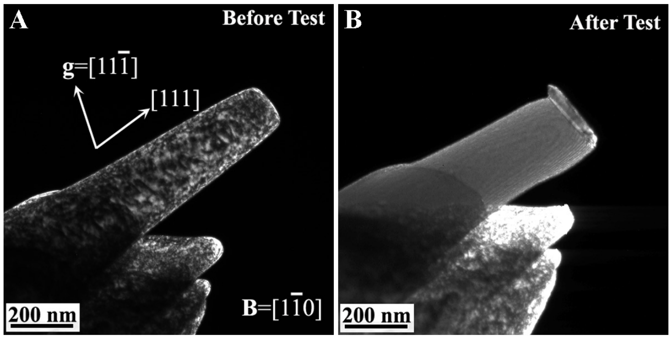

Figure 1: Dark-field TEM images of a Ni nanopillar before compression (A), and after compression (B).

The **PI 95 TEM PicoIndenter®** offers a unique method of circumventing these issues. With the **PicoIndenter**, it is possible to manipulate individual nanostructures while simultaneously viewing them in the TEM. Since both the sample and the tip can be monitored using the TEM, sample–tip contact can be limited to only during the test. Further, *in-situ* testing allows the user to determine the orientation of the crystal using diffraction techniques and to monitor the presence, formation, and propagation of defects.

Ni pillars were fabricated from a bulk single crystal using a focused-ion beam. Prior to testing the pillars had free-end diameters ranging from 150-290 nm in diameter and a sidewall taper angle of ~4°. The as-prepared sample was then mounted in a **TEM PicoIndenter** for examination in the TEM. This **PicoIndenter** was equipped with a diamond flat-punch tip, doped with boron for conductivity in the TEM.

Individual pillars were first oriented with respect to the electron beam. The pillars were then compressed with the diamond probe while simultaneously acquiring TEM video and load–displacement curves.

Prior to compression, the Ni pillars were observed to contain a large number of dislocations (Figure 1A and 3A). The pillar in Figure 1 had an initial free-end diameter of ~160 nm, and was tested under load control. Upon contact with the diamond probe this pillar yielded and upon further compression the pre-existing defects disappeared (Figure 1B). The resulting structure was essentially a dislocation-free single-crystal pillar. This phenomenon, referred to as "mechanical annealing," is consistent with the proposed dislocation starvation mechanism. The increased strength of nanoscale structures can be explained by the exhaustion of dislocation sources; higher stresses are then required to activate new dislocation sources.

One of the benefits of performing this study *in-situ* is that the instantaneous contact area can be measured from individual video frames. With these measurements, coupled with the quantitative force data, it is possible to calculate the contact stresses throughout the test (force divided by instantaneous contact area). A plot of the calculated stress vs. displacement for the pillar in Figure 1 is shown in Figure 2. The physical phenomena observed using the TEM can be correlated directly with events in the curve. After the initial deformation, subsequent dislocation bursts were observed which correspond to the load fluctuations. The sharp increase in load at a displacement of ~170 nm corresponded with primarily elastic behavior. For this pillar, stresses as high as 1.3 GPa were sustained at peak load during the test.

The extent of mechanical annealing observed was found to be consistent with smaller structures being stronger. Compression of a second pillar, which had a larger initial free-end diameter of ~290 nm, is shown in Figure 3A. This pillar was tested under displacement rate control. Upon initial loading the pillar yielded and a number of the pre-existing dislocations disappeared. The pillar was then observed to shear through the activation of a single slip system. When this source was exhausted, the stress in the pillar built up to a calculated value of 2.6 GPa and then punched in to its bulk-Ni substrate. This event corresponds to the peak followed by a sudden drop off at ~8 seconds in the force-displacement curve in Figure 3C. The punch-in, which occurred despite the large number of remaining dislocations, is also visible in the TEM images by the relative change in position of the free end of the pillar between Figure 3A and Figure 3B.

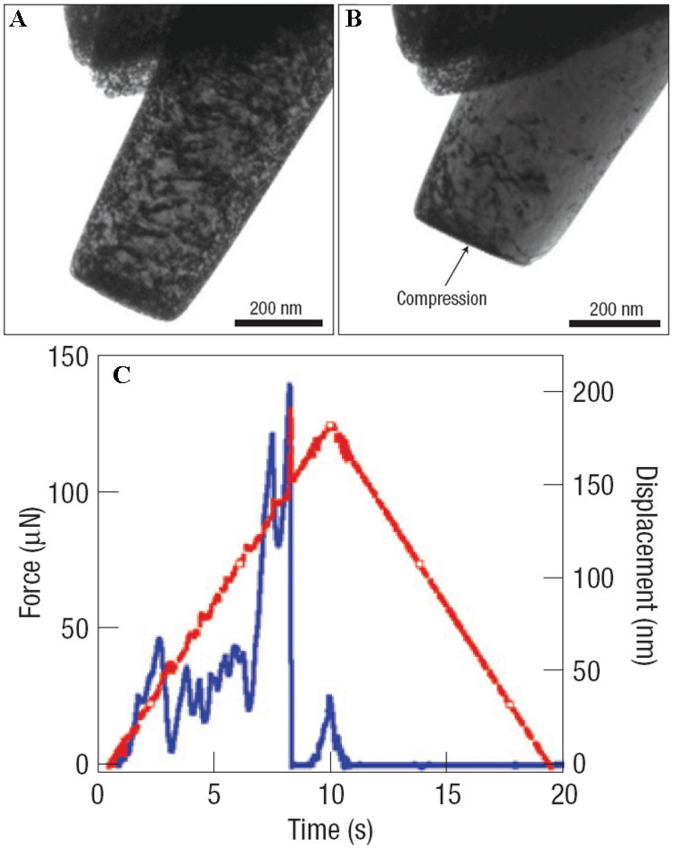

Figure 3. Bright-field TEM images of a Ni nanopillar before (A) and after (B) compression, and the corresponding load-displacement curve (C).

The **PI 95 TEM PicoIndenter** was used to investigate the mechanical properties of individual Ni nanopillars. The initially high dislocation density in the pillars was observed to decrease or even disappear upon compression.

This *in-situ* study illustrates the coupling of two high-resolution techniques which together make it possible to acquire quantitative mechanical data while simultaneously monitoring the microstructural evolution of the sample. The correlation of the two types of data provides unique insight into the phenomena responsible for the mechanical properties of these nanoscale structures.