

A New Perspective on Mechanical Testing: *In Situ* Compression in the TEM

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Testing the mechanical properties of nanoscale materials faces a number of inherent challenges. As the size of the “target” decreases, the inability to actually *see* what occurs during testing can be troublesome. While a number of studies have attempted to characterize the mechanical response of nanoscale materials using traditional nanoindentation techniques, certain lingering questions have always remained. For example, the nature and sequence of failure events in the case of multilayer films has long been the subject of scientific debate. Similarly, in the case of individual nanoparticles, it is often difficult to ascertain that contact was both made and maintained throughout the test. Further, the crystallographic orientation of the sample and/or the presence of preexisting defects, which can have a significant influence on mechanical properties at the nanoscale, typically remain unknown.

The transmission electron microscope (TEM) is an ideal tool for determining these unknowns. In particular, with careful attention to sample preparation, the TEM can be used to characterize samples after mechanical testing. With the TEM it is fairly straightforward to determine the sample orientation and to examine residual plastic deformation. However, these “*post mortem*” studies suffer from their own unknowns, in many ways complimentary to those faced by nanomechanical testing. For example, it is impossible to characterize any purely elastic deformation that occurred during testing. In addition, determining at what point during testing certain defects were formed is out of the question. A simultaneous combination of the two techniques has some clear advantages over a sequential approach. Here we present a discussion of the development of the *in situ* nanoindentation holder and of its application to characterizing the mechanical behavior of nanoscale materials.

The evolution of the *in situ* nanoindenter for the TEM has a long and varied history. In one of its early incarnations, developed in the 1960s, the *in situ* indenter was a piezo-driven indentation stage capable of loads on the order of 10^{-2} to 10^{-6} N [1]. Sample holders capable of indentation did not emerge until a few decades later, most notably from Lawrence Berkeley National Laboratory [2-4]. These holders employed a piezoelectric actuator for indentation, but lacked a force sensor. As such, they were essentially qualitative in nature, though quantitative displacement information could be extracted from the TEM images. This type of holder resulted in a number of fundamental studies characterizing deformation of nanoscale materials such as thin films and nanoparticles [5-8]. Until quite recently however, the major aspect of *in situ* indentation that had been lacking was the capability of determining both load and displacement *quantitatively* during a test. We have developed a system capable of both: the TEM PicoIndenter™ [9].

With the PicoIndenter it is possible to acquire quantitative mechanical data while simultaneously monitoring the microstructural evolution of the sample with the TEM. In the PicoIndenter,

the sample is mounted directly opposite a boron-doped diamond indentation tip, such that the indentation direction is along the length of the holder and perpendicular to the electron beam (Figure 1). Using the relative foci of the tip and the sample, the tip can be brought into the same plane as the sample using the coarse (manual) and fine (piezoelectric actuator) controls. In addition, the PicoIndenter incorporates a patent-pending miniature transducer for electrostatic actuation and capacitive displacement sensing [10]. The transducer is the heart of the PicoIndenter, making it capable of producing quantitative load–displacement curves with load or displacement controlled as a function of time. The PicoIndenter, in its earliest form, has been used to characterize the onset of plasticity during the deformation of Al [11], to study dislocation–grain boundary interactions and dislocation motion in Mg–Al alloys [12, 13], and to quantify the mechanical behavior of nanoporous Au films [14].

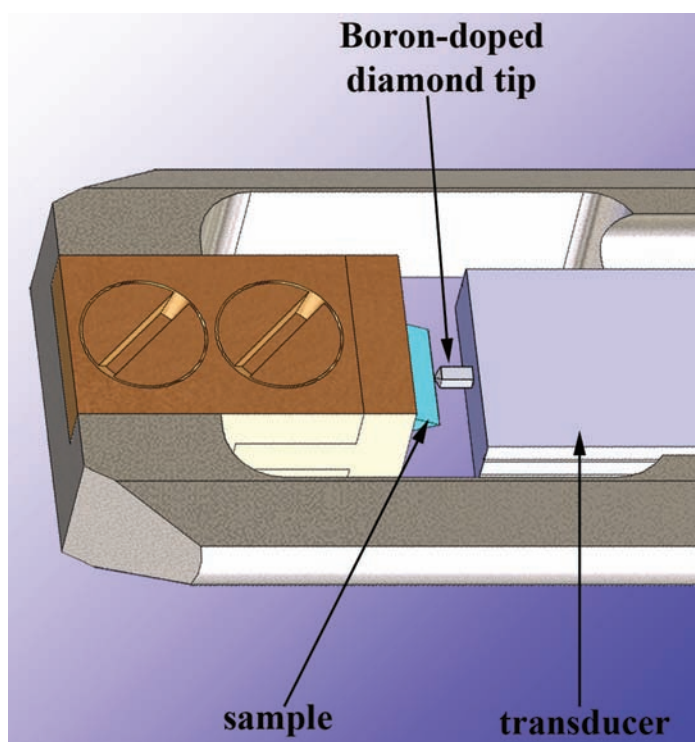


Figure 1: Schematic illustrating the geometry at the tip of the holder. The electron beam is perpendicular to the plane formed by the sample and the diamond tip.

More recently, the PicoIndenter has been used to study the mechanical response of individual Ni pillars, with astounding results [15-17]. The Ni pillars were fabricated from a bulk single crystal using a focused-ion beam (FIB) such that their long axis was oriented along a [111] direction. Prior to *in situ* testing the pillars had free-end diameters ranging from 150–290 nm with a sidewall taper angle of $\sim 4^\circ$. The prepared sample was then mounted in the PicoIndenter for examination in the TEM. This PicoIndenter was equipped with a diamond flat-punch tip, doped with boron for conductivity in the electron microscope. With this system it is possible to align the tip with the area of interest and begin the test without making contact with the sample prior to testing—an important consideration as dislocation bursts have been observed in metals at near noise-level loads [11, 18]. Individual pillars were first oriented with respect to the electron beam and then compressed

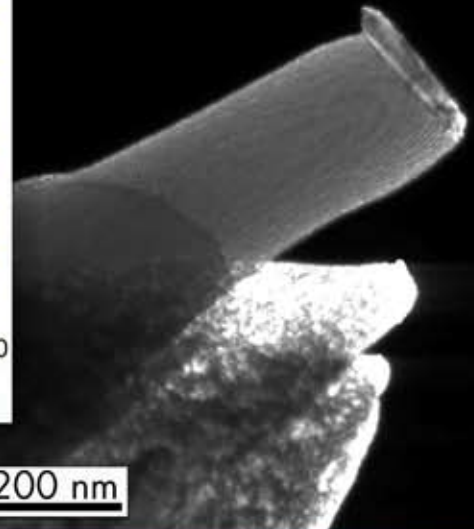
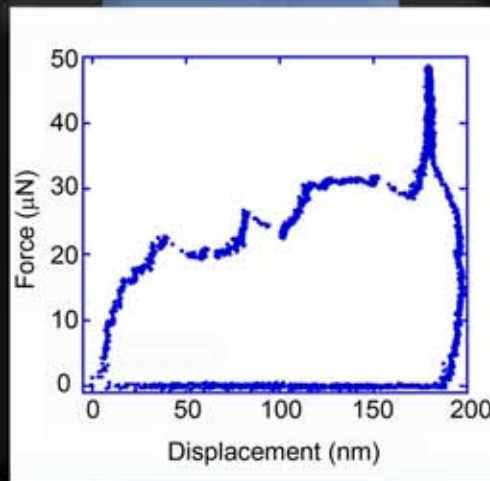
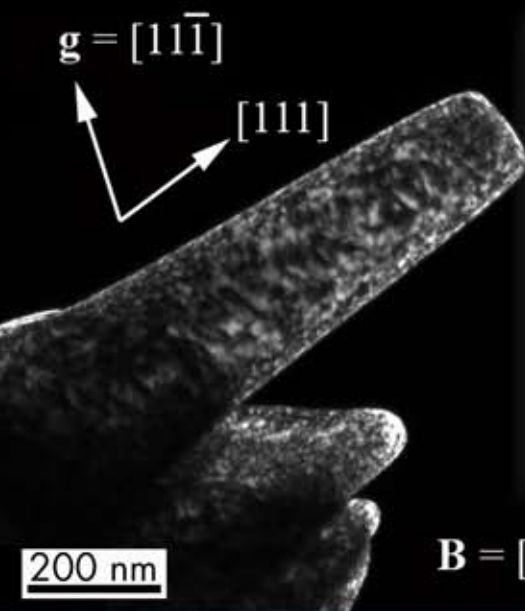
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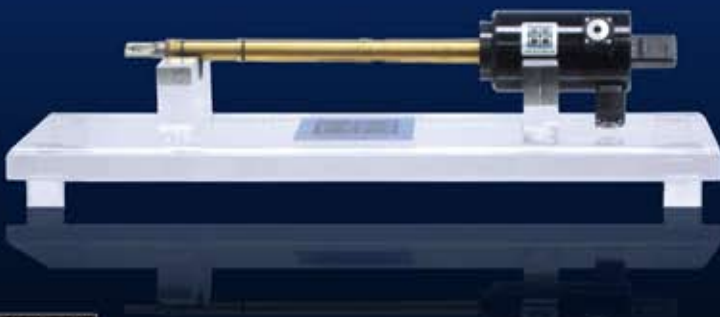
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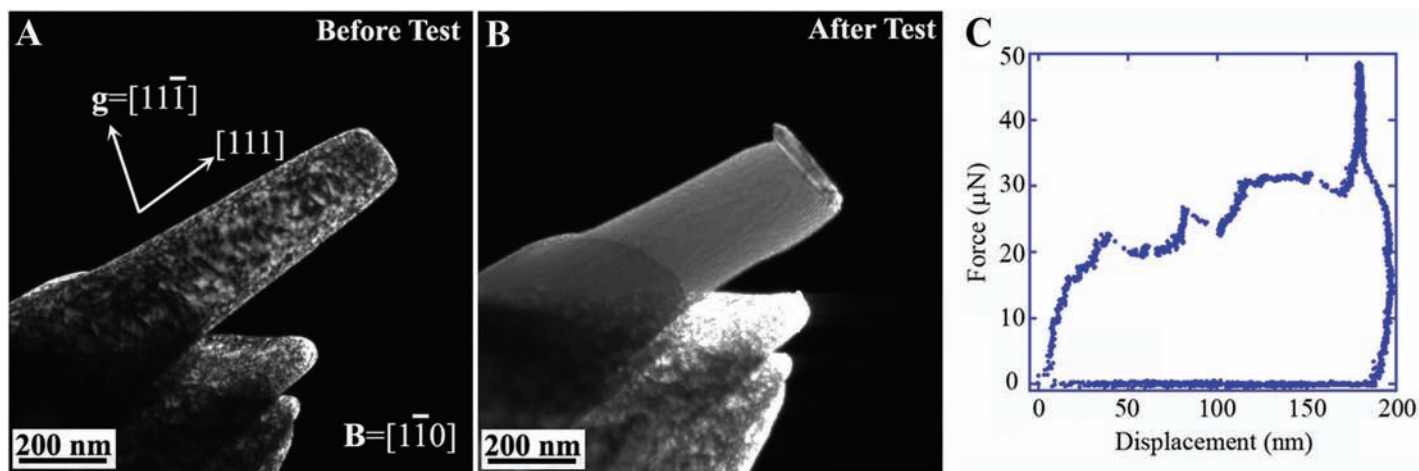


Figure 2: Dark-field TEM images of a Ni nanopillar before compression (A) and after compression (B), and the associated force vs. displacement curve (C).

with the diamond probe. During compression, both TEM video and load–displacement curves were acquired simultaneously. While we commonly refer to the holder as an “indentation holder”, in this case it is more accurate to describe the test as a compression test due to the relative sizes of the sample and probe.

Prior to compression the Ni pillars were observed to contain a large number of dislocations (Figure 2A), but upon contact with the diamond probe the pillars yielded and upon further compression these pre-existing defects disappeared. The resulting structure was essentially a dislocation-free single-crystal pillar (Figure 2B), as determined by dark-field imaging under a number of two-beam conditions. This phenomenon, dubbed “mechanical annealing,” is consistent with a dislocation starvation mechanism. The initially high dislocation density drops to zero and further deformation is controlled by the activation of new dislocation sources, taking place at higher stresses. The sharp load increase to nearly 50 μN shown in the load-displacement plot (Figure 2C) coincided with primarily elastic behavior. By measuring the instantaneous contact area from still frames of the recorded video, one of the advantages of performing the test in the TEM, it was possible to calculate the instantaneous contact stresses throughout the test. For this pillar, stresses as high as 1.3 GPa were sustained at peak load.

This *in situ* study represents the first direct observation of the dislocation starvation mechanism responsible for the strengthening of these nanopillars and gives new insight into the phenomena involved in the atypical mechanical properties associated with nanoscale structures. However, this work is really only the tip of the iceberg. The origins of the enhanced properties of materials in their nanoscale form are still poorly understood. Clearly, this technique could be extended to materials of different chemistries and morphologies. For example, it has already been used to explore the properties of metallic-glass pillars [19]. In the future, *in situ* TEM studies will undoubtedly benefit from the coupling of nanomechanical testing with other techniques. Sample holders which can test electrical or thermal properties in conjunction with quantitative nanomechanical testing will certainly contribute to a better understanding of how certain properties are coupled. The capability of investigating these phenomena *in situ* presents truly amazing possibilities. ■

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References

- Gane, N., *The direct measurement of the strength of metals on a sub-micrometer scale*. Proc. R. Soc. London, Ser. A, 1970. **317**: p. 367-391.
- Wall, M.A., T.W. Barber, and U. Dahmen, *Techniques for in situ HVEM mechanical deformation of nanostructured materials*. 53rd Annual MSA Meeting, 1995: p. 240-241.
- Wall, M.A. and U. Dahmen, *Development of an in-situ nanoindentation specimen holder for the high voltage electron microscope*. Microsc. Microanal., 1997. **3**(suppl. 2): p. 593-594.
- Stach, E.A., et al., *Development of a nanoindenter for in situ transmission electron microscopy*. Microsc. Microanal., 2001. **7**: p. 507-517.
- Deneen, J., et al., *In situ deformation of silicon nanospheres*. J. Mater. Sci., 2006. **41**: p. 4477-4483.
- Deneen Nowak, J., et al., *Fracturing a nanoparticle*. Philos. Mag., 2007. **87**(1): p. 29-37.
- Jin, M., et al., *Study of deformation behavior of ultrafine-grained materials through in situ nanoindentation in a transmission electron microscope*. J. Mater. Res., 2005. **20**(7): p. 1735-1740.
- Minor, A.M., et al., *In-situ transmission electron microscopy study of the nano-indentation behavior of Al*. J. Electron. Mater., 2002. **31**(10): p. 958-964.
- www.hysitron.com.
- US Patent Application No. 20070180924.
- Minor, A.M., et al., *A new view of the onset of plasticity during the nanoindentation of aluminium*. Nature Materials, 2006. **5**(9): p. 697-702.
- De Hosson, J.T.M., et al., *In situ TEM nanoindentation and dislocation-grain boundary interactions: a tribute to David Brandon*. J. Mater. Sci., 2006. **41**: p. 7704-7719.
- Soer, W.A., et al., *Incipient plasticity in metallic thin films*. Appl. Phys. Lett., 2007. **90**(18): p. 181924-3.
- Sun, Y., et al., *The mechanical behavior of nanoporous gold thin films*. JOM, 2007. **59**(9): p. 54-58.
- Shan, Z.W., et al., *Mechanical annealing and source-limited deformation in submicrometre-diameter Ni crystals*. Nature Materials, 2008. **7**(2): p. 115-119.
- Shan, Z.W., et al., *Exploring the mechanical properties of nanostructures and nanomaterials inside a TEM*. Microsc. Microanal., 2008. **14**(Suppl. 2): p. in press.
- Hemker, K.J. and W.D. Nix, *Nanoscale deformation: Seeing is believing*. Nature Materials, 2008. **7**(2): p. 97-98.
- Warren, O.L., et al., *In situ nanoindentation in the TEM*. Materials Today, 2007. **10**(4): p. 59-60.
- Shan, Z.W., et al., *Plastic flow and resistance of metallic glass: Insight from in situ compression of nanopillars*. Phys. Rev. B, 2008. **77**: p. 155419.

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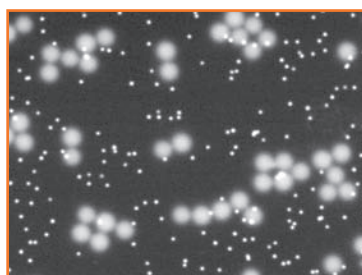
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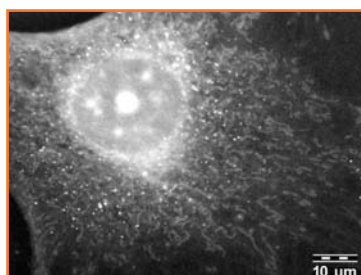
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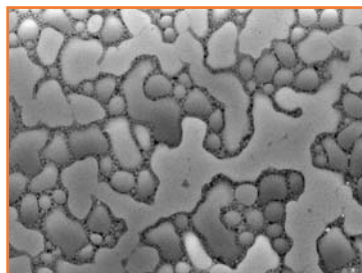
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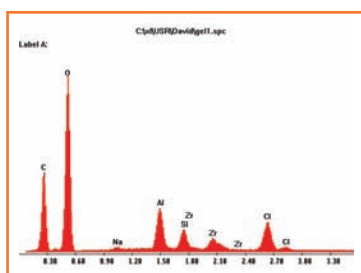
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