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In-Situ TEM Compression of MgO Nanocubes

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In-situ indentation is a powerful technique for studying the fundamental mechanical behavior of TEM compatible materials. It is capable of probing several different mechanical properties of materials, depending on the exact methodology utilized. When combined with TEM imaging and structural characterization techniques, structure-property relationships and real-time monitoring of deformation mechanisms can be explored. Working with nano-constrained volumes allows both for TEM compatibility and detection of discrete mechanical events, such as dislocation nucleation and crack formation and propagation.

MgO serves as a model material for exploring a wide variety of mechanical response, as it has been shown to exhibit significant plasticity as well as brittle fracture at room temperature [1]. By varying parameters such as particle size and strain-rate, fundamental behavior such as the brittle-to-ductile transition (BDT), strain hardening according to pile-up mechanisms, and energies for plasticity and fracture can be studied. This is of interest for establishing links between atomistic and multiscale modeling [2], and even in the geological community for fault formation [3]. From an applications standpoint, as nanoscale devices grow in popularity, complexity, and functionality, understanding of length-scale effects on mechanical behavior is needed [4]. Ductile ceramics, if possible, could provide high toughness insulation for a variety of nanodevices.

MgO nanoparticles were synthesized by burning commercially pure Mg turnings and collecting the resulting smoke. These particles were suspended in isoproponal and ultrasonicated to separate agglomerated particles, then deposited onto prepared sapphire wedges. These samples were mounted to a Hysitron PI-95 PicoIndenter TEM holder and studied FEI F30 TEM operating at 100 keV. The PicoIndenter was outfitted with a 500 nm radius of curvature diamond cube corner tip. The particles were compressed uniaxially under displacement control with a rate of 5 nm/s. Bright Field, Dark Field imaging and selected area diffraction were performed on the particles and videos were taken during indentations (Figure 1).

Results showed largely brittle fracture of MgO nanocubes, however, this was observed in tandem with other interesting activity. In Figure 1(b) contrast bands can be observed occurring during the first peak in the load-displacement curve (Figure 2) that could correspond to either plasticity or phase change. They appear well-aligned with the typical {111} slip plane for this material, however, contrast changes in the particle post fracture could signify some other activity. From the estimated stress-strain curve in Figure 2(b), the elastic modulus of 270 GPa and fracture stress of 6.8 GPa were estimated.

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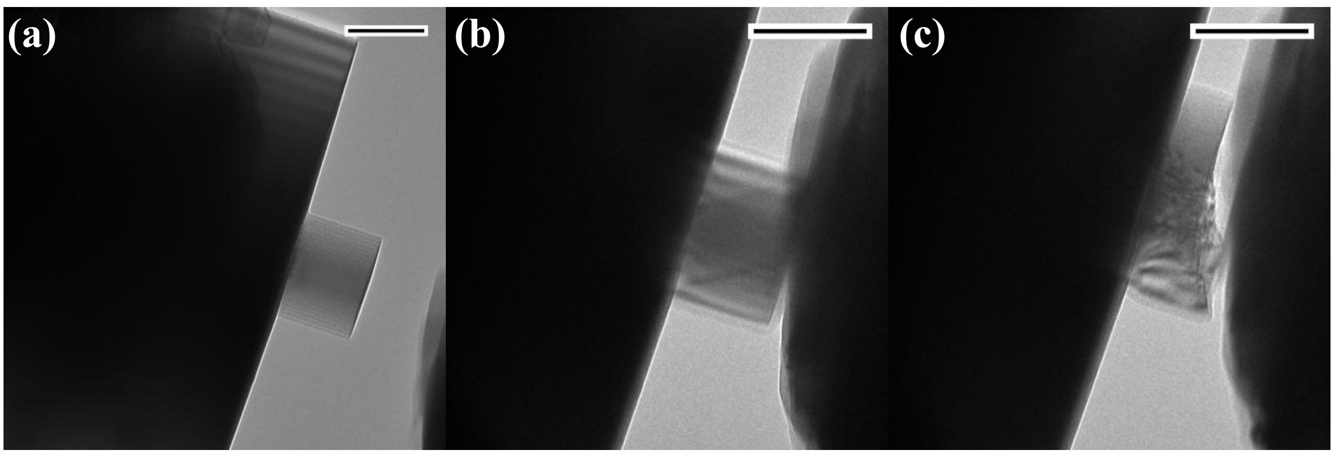

Figure 1. TEM Bright field video images of MgO nanocube (a) pre, (b) during, and (c) post compression testing. 1(b) is labeled in mechanical data below. Scale bar represents 100 nm in all images.

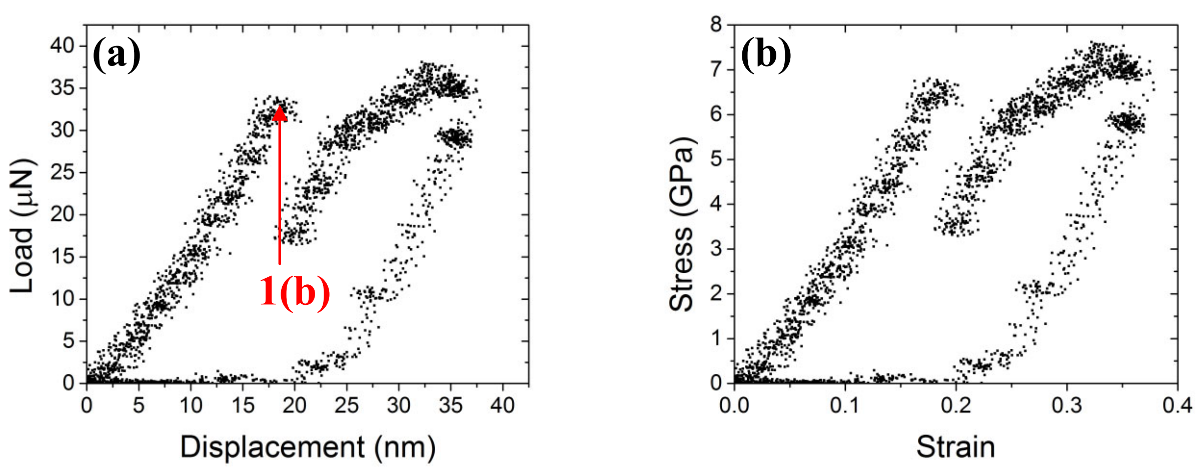

Figure 2. Corresponding mechanical data from nanocube in Figure 1: (a) measured load-displacement curve, (b) estimated engineering stress-strain from image correlation

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