APPLICATION NOTE

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Nanoparticle Fracture

In-Situ Nanomechanical Testing of Hollow CdS Nanospheres

Introduction

Nanoscale particles are expected to exhibit enhanced strength compared to their bulk counterparts because their small size limits the number of defects they can contain. This high strength makes them desirable for a number of industrial applications. However, despite this enhanced strength, nanoparticles often fail mechanically due to the large internal strains they encounter during loading, a limitation that ultimately hinders their application in engineered materials.

Structural hierarchy in bulk materials is known to play an important role in determining their mechanical properties. A similar effect might be expected for nanoscale materials exhibiting a multi-scale structure, though the fabrication and testing of these structures is generally more complex. In this work, hollow nanocrystalline CdS nanoparticles were tested *in situ* in the transmission electron microscope (TEM) using a **PI 95 TEM PicoIndenter®**, which allows the particles to be imaged and mechanically tested simultaneously.

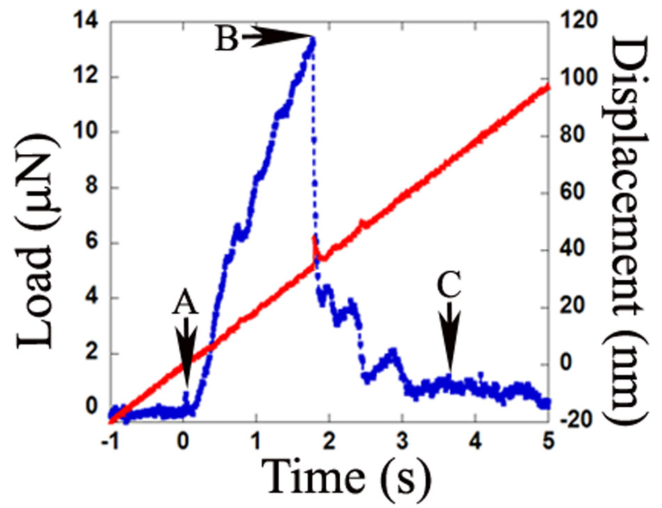

Figure 1: Load and Displacement vs. time. Load is shown in blue, displacement is shown in red. Data is from the compression test of the particle shown in Figure 2. Points A, B, and C correspond to images A, B, and C in Figure 2.

Procedure

The CdS spheres studied in this work were hollow and ranged in diameter from 200-450 nm with shell thicknesses ~13% of their outer diameter. The particles were polycrystalline with

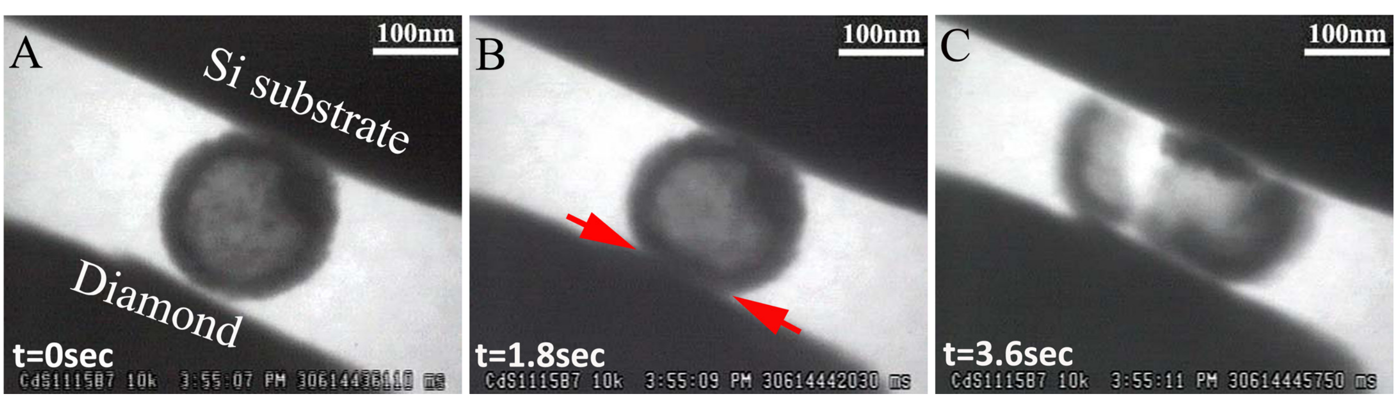

Figure 2: An individual CdS nanoparticle Stress vs. Displacement curve associated with the compression test in Figure 1. Stress is calculated from the applied load and the measured instantaneous contact area.

a grain size on the order of a few nanometers. The particles were deposited on a Si substrate with a 1 µm wide plateau on its surface; the particles on the plateau are unobstructed and can be probed relatively easily.

The sample was then mounted in a **PI 95 TEM PicoIndenter** and individual particles were compressed in the TEM. This **PicoIndenter** was equipped with a flat-punch shaped diamond tip.

Results and Discussion

The load-displacement curve associated with the displacement-rate controlled compression of an isolated nanoparticle is shown in Figure 1. The particle has an outer diameter of 210 nm and a shell thickness of 30 nm. Three frames from the TEM video acquired during the test are shown in Figure 2. Because the video and the curve are time-correlated, discrete events in the curve can be linked directly to events observed in the video. The three points labeled A, B, and C in Figure 1 correspond to images A, B, and C in Figure 2. Point A is just prior to contact with the diamond tip. On contact the load increases up to point B, where the maximum load is reached. This peak load corresponds to a displacement of 34 nm, an effective strain of over 16%. By point C the particle has suffered multiple fractures, which are illustrated by the uneven nature of the curve after the initial load drop.

Because the particle can be imaged throughout the test it is possible to measure the instantaneous contact diameter very accurately. If a circular contact is assumed, it is also possible to calculate the contact pressure at peak load (instantaneous load divided by instantaneous contact area). For this particle, with a peak load of 13.2 µN and a measured contact diameter of 128 nm, the contact pressure was approximately 1 GPa. Both high strength (~3% of the bulk Young's modulus) and ductility (up to ~20% strain) were observed in a number of particles. The graph in Figure 3 shows the calculated contact pressure plotted with respect to strain for several different particles.

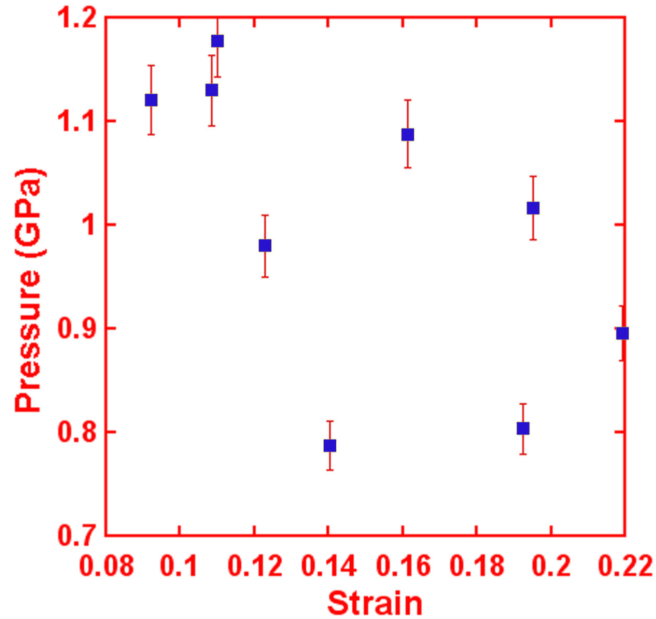

Figure 3. Plot of contact pressure vs. strain. Pressure is calculated using the peak load in the load-displacement curve and the measured contact diameter from the TEM images.

The high stresses and strains sustained by the particles are related to their structure. The small grain size aids in strengthening the particle and the hollow morphology allows for high effective strains.

Conclusions

The mechanical properties of hollow CdS nanoparticles with a unique hierarchical structure were tested *in situ* using a **PI 95 TEM PicoIndenter**. The particles were found to exhibit ultra-high strength and considerable strain to failure, which can be correlated directly with their structure. The high ductility, unusual for nanoparticles, makes these particles potentially useful as components in engineered materials.

The combination of the quantitative mechanical data and the TEM video allows for precise calculations of contact pressure, which would be not be as accurate with either technique independently.

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