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Developments in *In Situ* Nanomechanical Testing

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The past decade has seen a notable interest in *in situ* studies in the scanning and transmission electron microscopes (SEM and TEM), which has in turn driven a number of advancements in *in situ* testing systems. The miniaturization of certain critical components has transformed historically qualitative techniques into fully quantitative complementary test systems. This miniaturization is important because the physical constraints of the SEM and TEM are quite demanding, making compact systems not only desirable, but also geometrically necessary.

Of particular interest, small-scale transducers which can accurately measure force and displacement have been developed, which can make what would otherwise be a simple *in situ* nanomanipulator a fully quantitative nanomechanical test instrument [1]. With these systems it is possible to pair the high sensitivity of nanoindentation with the high spatial resolution of electron microscopy. Here we report on the development of a series of compact vacuum-compatible nanomechanical test instruments for the SEM and TEM [1-2]. Nanoscale structures (such as the nanoparticles shown in Fig 1) are often difficult to test because of the inherent challenges imposed by the size of the samples involved and the scale at which the phenomena in question occur, but with *in situ* techniques simultaneous imaging and nanomechanical testing is possible.

One notable advancement is the integration of simultaneous electrical contact resistance (ECR) measurements (Fig 1). Using a conductive indenter probe and a current/voltage source, a time-based correlation between force, displacement, current, and voltage measurements can be determined. Other new developments include an *in situ* tensile apparatus and a miniaturized heating stage has been designed for simultaneous *in situ* heating during mechanical testing with rapid heating rates and extremely low thermal drift. Depending on microscope configuration, tests can be further coupled with complementary techniques such as XEDS and EBSD [3].

The present work will describe new developments in *in situ* testing techniques, highlighted by application examples including compression studies of nanoparticles [4-5] and nanopillars [6], bending studies of nanowhiskers, as well as thin film nanoindentation [2].

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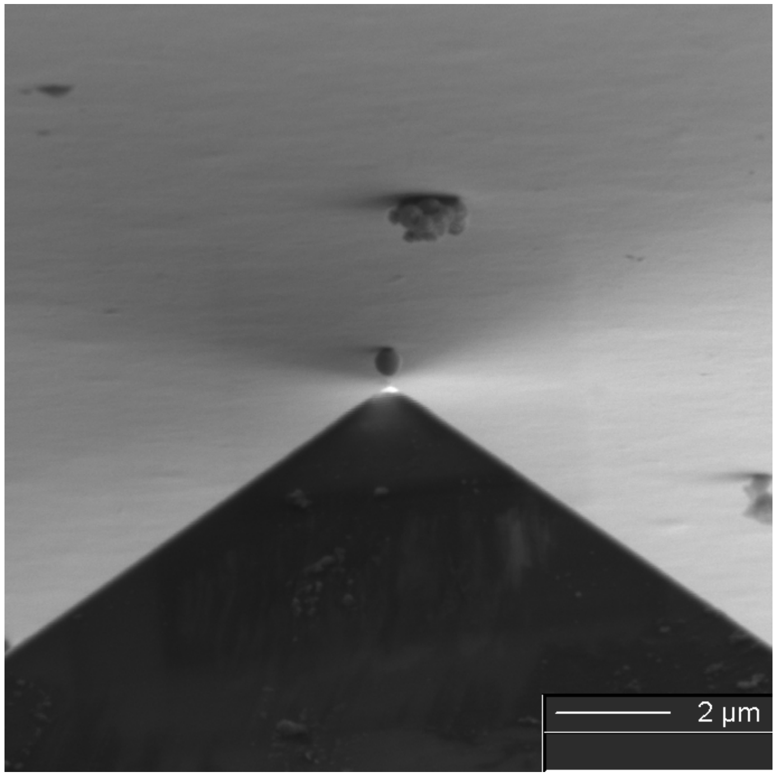

FIG. 1. SEM image of a Cu nanoparticle just prior to *in situ* compression in the electron microscope.

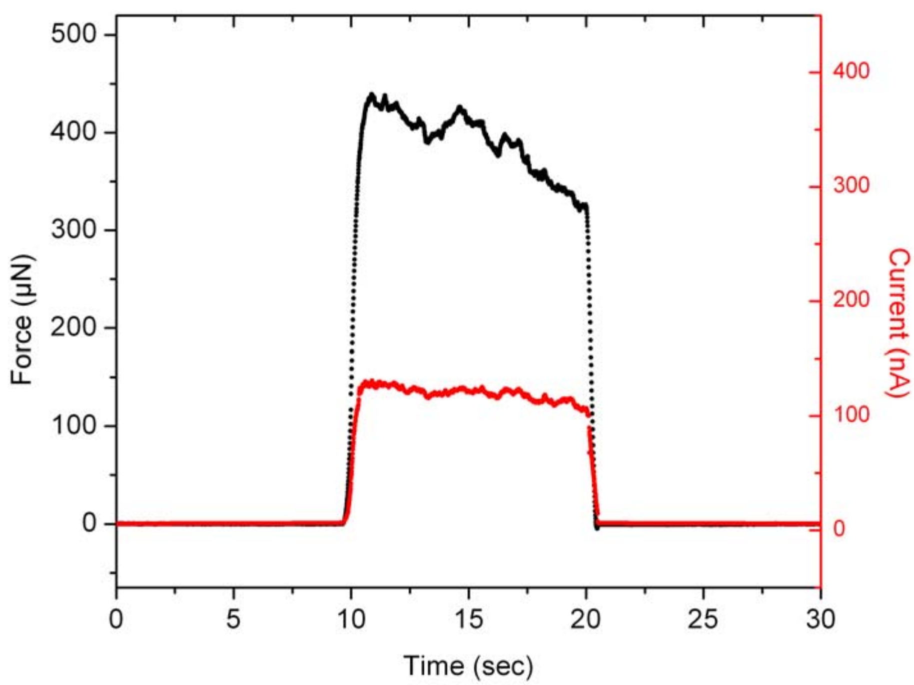

FIG. 2. Plot of current and force as a function of time acquired during an *in-situ* indentation test in the TEM on <100> p-type Si. A 200 mV bias was applied during the measurement.

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