Environmental and In Situ SEM/TEM

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In situ tensile tests of single crystal metal nanowires inside the SEM and TEM

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It is well known that the mechanical properties of nanoscale metals differ from those of macroscale samples, in particular the strength increases with decreasing sample size. A number of studies on sub-micron sized metals have been performed which attempt to reveal the mechanisms controlling deformation at small length scales. Machine compliance and complex stress states are often major problems in nanomechanical experiments that make the interpretation of the results challenging.

Here we present a combination of two complementary in situ electron microscopy studies: qualitative TEM tensile tests, which allow direct observation of the defect evolution, and quantitative testing inside the SEM, where the stress-strain behavior of the wires is determined. The use of the two methods makes the realization of very stiff set-ups possible and tests on metal whiskers show highly reproducible results both in TEM and SEM.

To test the wires in the TEM a standard straining holder is used. Wires are transferred with the aid of a micromanipulator and electron beam assisted Pt deposition onto a CuBe frame leaving the wires undeformed (see Figure 1). The homogeneous deformation of the gauge length of the frame allows the realization of deformation steps <1nm. During deformation, movies as well as high resolution TEM images are recorded to monitor the defect evolution and investigate the fundamental mechanisms of deformation.

The tensile stage for the SEM consists of two piezoelectric positioners (x- and y-direction) on the one side, with a TEM-grid mounted on top. On the opposite side, a MEMS based force-sensor is mounted on top of a third positioner (z-direction). Nanowires from the same batch are transferred in an analogous manner to stretch from the grid to the tip of the force-sensor (see Figure 2). Strains are obtained by digital image correlation of SEM images taken during testing and stresses are calculated from the measured force and the wire area as measured by a FIB cross-section. Post-mortem TEM investigations of the failed wire segments allow the deformation morphologies of the SEM and TEM tested wires to be compared.

Investigations show that single crystal Au nanowires with diameters ranging from 40 to 250 nm deform at stresses in excess of 1 GPa by homogeneous nucleation of partial dislocations creating stacking faults on {111} planes. The stacking faults thicken into nanotwins through the sequential activation of partial dislocations on neighboring {111} planes. Post-deformation TEM studies show that fracture occurs within the nanotwins.

A quantitative model based on classical nucleation theory will be presented which explains the observed difference in deformation mode. Implications for different materials and loading geometries will be discussed. In addition, preliminary studies of the effect of surface structure and gas ambient on the deformation behavior of the nanowires will be introduced.

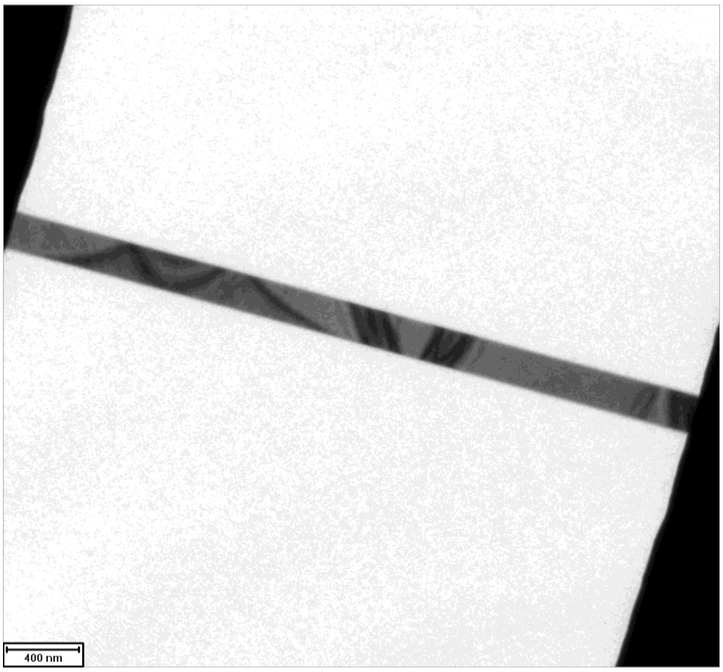

Figure 1. Bright field image of a transferred single crystal Au wire. Only bending contrast is visible indicating that the transfer process leaves the wire undeformed.

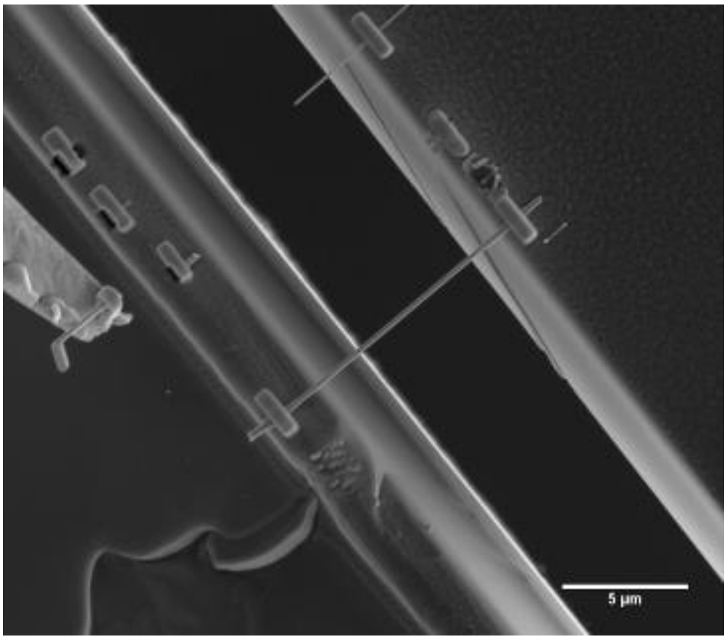

Figure 2. SEM image of a wire glued with aid of a micromanipulator and electron beam assisted Pt deposition to a TEM-grid (lower left corner) and the tip of the force sensor (upper right corner).