

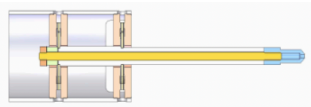
Bruker/Hysitron PI 95 JEOL TEM PicoIndenter

- A Nanomechanical Test Instrument in (MC)2

The PI 95 PicoIndenter makes it possible to perform nanomechanical testing while simultaneously imaging in a transmission electron microscope (TEM). Load or displacement-controlled testing modes enable indentation, compression, bending, tensile and fatigue tests to directly observe dislocation nucleation and motion, deformation twinning, phase transformations, and fracture at TEM resolution. The most challenging part of the tests could be sample preparation. First of all, the sample should be thin enough to be electron transparent in a TEM. Second the sample should be properly mounted to achieve the best test results.

Indenter Setup

- The PI 95 PicoIndenter is designed to integrate a miniature transducer and a sample mount into a TEM holder for in-situ imaging.
- The PI 95 PicoIndenter in MC2 is only compatible with a JEOL TEM.
- To exchange tip, the front-end needs to be removed from the outer-tube, then a probe can be carefully screwed onto a threaded post.
- Sample mount is attached to the front end of the PI 95 holder with four screws.



Transducer

- Miniature design with 1.5 mN max. load / 5 μm max. displacement
- <200 nN force sensitivity / <1 nm displacement sensitivity

Probes

- Conductive probes include flat end (1 μm , 5 μm , 20 μm radius), cube corner (<50nm radius of curvature) and cono-spherical (tip radius <1 μm) probes.
- Probes for PI 89 and PI 95 are interchangeable between the two PicoIndenters.

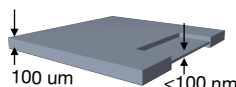
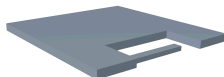
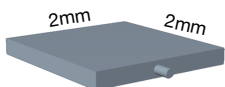
Sample Mounts

- Half-grid or bulk sample (a)
- Si wedge (b)
- Push-to-Pull device (c): a consumable, multi-use MEMS-fabricated apparatus for in-situ tensile testing; compatible with both SEM PI 89 and TEM PI 95 Bruker/Hysitron PicoIndenters.

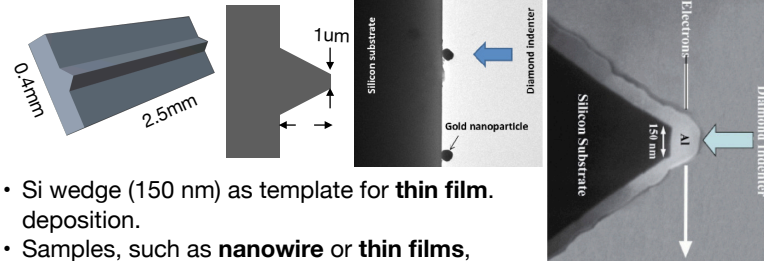


Sample Preparation

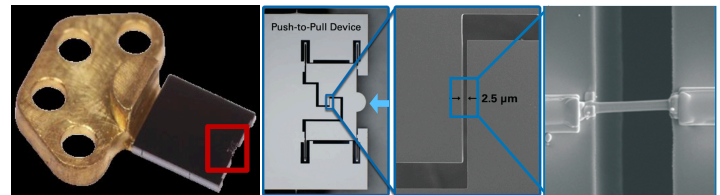
- A variety of forms of samples could be used for PI 95 testing when properly mounted.
- Half-grid with FIB lift out sample.
- Bulk sample** with thin features (pillar, bending bar, or just a thin area) prepared by focused ion beam (FIB).



- Si wedge (1 μm plateau) as substrates for **nanoparticle** deposition.



- Si wedge (150 nm) as template for **thin film** deposition.
- Samples, such as **nanowire** or **thin films**, attached to push-to-pull device.



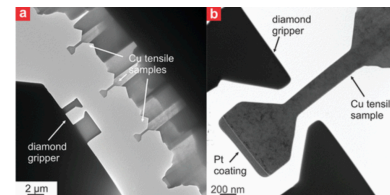
Images from presentation by Sanjit Bhowmick, Ph.D. | Bruker 2018

Tip Positioning

- Three levels of control for tip positioning and mechanical testing.
- Manual: x,y,z knobs at the end of the holder (μm to mm)
- Piezoelectric: x,y,z for fine positioning only (nm to μm)
- Transducer: indentation direction only (nm to μm)

In Situ Testing Techniques in TEM

- Indentation:** Usually a sharp cube corner probe is used to indent a sample with either load or displacement control.
- Compression:** Usually a cono-spherical or flat end probe is used to compress particles or pillars.
- Direct Pull Tensile Test:** Usually a dog bone sample is firstly made by FIB. The sample should be thin enough to be electron beam transparent.
- Push to Pull Tensile Test:** three different stiffness values (15, 150, and 450 N/m) available for use with a variety of materials and sizes.



D. Kiener and A. M. Minor, *Nano Lett.* 11,9(2011)

Software and Data Analysis

- TriboScan software for stage and probe control and data acquisition. Real-time plotting of force vs. displacement and control variable vs. time.
- Integrated TEM video capture and synchronization for side-by-side analysis.