

In-situ material testing devices and methods

St. Fahlbusch, L. Philippe, B. Moser and J. Michler

Empa, Laboratory for Materials Technology, CH-3602 Thun, Switzerland

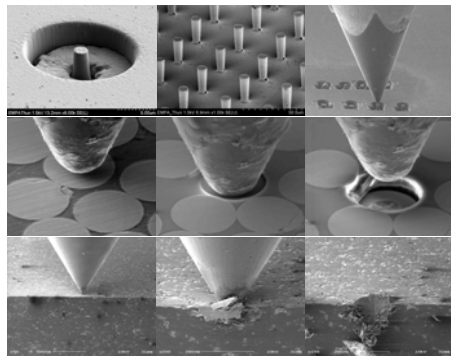


Materials Science & Technology



Introduction

- The understanding of small scale deformation mechanisms is important for the reliable design of miniature/micro products.
 - Deformation on a small scale may differ greatly from large scale deformation.
 - The scanning electron microscope (SEM) offers high resolution, large depth of field and analytical capabilities for time-resolved observation and investigation of small scale deformation mechanisms.
 - The combination with a quantitative mechanical test makes this a very interesting tool.
- In-situ SEM material testing yields additional information that is not available from ex-situ investigations.



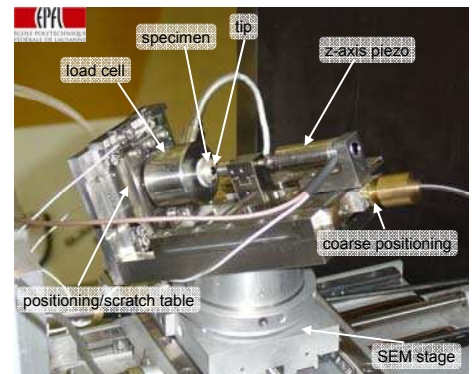
Micro-tensile Testing Stage



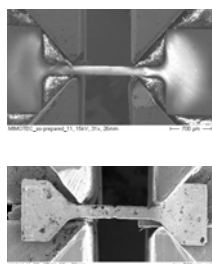
- Tensile stage adapted for the use with micro-tensile specimens.
- Fits on the SEM sample stage just like an oversized specimen.
- Load ranges from 0...0.2 N up to 0...10 kN
- Deformation speed range: 0.1 to 100 $\mu\text{m}/\text{sec}$
- An LVDT is used for the measurement of the cross-head displacement. Machine compliance induced by, e.g., the load cell is measured, too.
- A non-contact linear proximity measuring system is used for measuring highly reproducible displacement values.
- The system control and data acquisition is based on LabVIEW from National Instruments.

Scanning Electron Microscope Indenter

- Indentation axis operated at an inclined angle with respect to the SEM column; this allows looking at the surface around the indent during indentation.
- Variable load ranges: 500 mN, 1.5 N, 10 N and 50 N load cells.
- Piezo driven indentation axis: range: 20 μm closed-loop
- Stick-slip stage for x-axis: positioning and scratching, several mm travel
- Stack piezo for y-axis: positioning and scratching, range: 20 μm open-loop
- LabVIEW based system control and data acquisition
- Tips compatible with MTS NanoXP indenter
- System calibrated against MTS NanoXP indenter

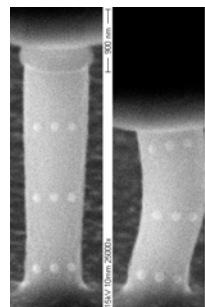


Micro-tensile Testing



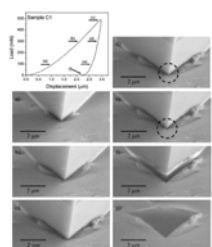
- Tensile testing in general is the most important mechanical testing method.
- A specimen is subjected to a continually increasing uniaxial load and the elongation is measured. The obtained load-elongation curves are translated into stress-strain curves.
- The most important advantage of in-situ micro-tensile testing is, apart from the measurement of the complete load-displacement curve of micro-sized specimens, the quasi real-time observation at the same time.
- Strain can be extracted from the video capture by special vision algorithms.

Micro-compression and Bending



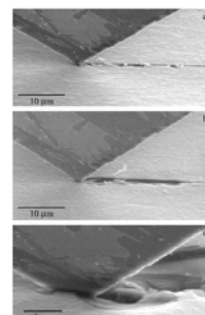
- The micro-compression test method consists of performing instrumented compression tests on pillars inside the SEM. This gives access to information concerning the deformation and failure mode such as buckling and/or cracking.
- Efficient testing of a large number of specimens.
- Sample dimensions can be scaled down to below one micrometer in diameter.
- The direct SEM-observation during the testing allows for very efficient positioning, assessment of the failure mechanism and automation of the test.
- Strain measurement using the SEM video capture is possible.

Indentation



- Crack and pile-up formation can be directly observed with sub-micrometer resolution and linked to the simultaneously recorded load-displacement data.
- SEM indentation made on a 1.5 μm thin TiN/SiNx coating deposited on Si; 13 at.% Si content.
- The loading/unloading cycle has been stopped several times to record SEM images of the indentation region (indicated by arrows in the load-displacement graph).
- During unloading the formation of a small crack can be observed (indicated by the circle in figures (d) and (e)).

Scratching



- Observation of the initiation and propagation of cracks with sub-micrometer resolution and the flow of the material near the indenter (piling-up and sinking-in).
- Study chip and particle formation mechanisms during microscratching.
- Yields information not available from conventional microscratch experiments.
- SEM images recorded during scratches with 5 mN (a), 10 mN (b), and 150 mN (c) applied normal load on a 3 μm DLC coating on a steel substrate.