Determination of micro-mechanical properties: in-situ compression, tension and fracture testing within the SEM

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The ongoing miniaturization of devices and components in many fields of modern technologies, e.g. medical devices or micro-electronic mechanical systems (MEMS), requires the knowledge of material properties in small scales. Known size effects, i.e. the mechanical properties changes with principle specimen size, prohibits the usage of macroscopic material properties. Novel miniaturized test methods have to be applied to measure these small-scale properties.

With the advent of the focused ion beam (FIB) technique in material science, the preparation of micro-sized samples has become straightforward. This has led to an increasing number of micro- and nano-mechanical experiments in the last years, predominantly inspired by the micro compression tests performed by Uchic et al. [1]. Nowadays, micro-compression, -tension [2], -bending [3] and -fracture tests are performed. For a proper alignment of specimen and loading device, as well as for a direct observation of deformation and fracture processes, in situ testing within a scanning electron microscope (SEM) has become more and more important, recently. In the current work we introduce novel techniques to measure mechanical properties in small dimensions and to study deformation and fracture processes at this small length scale.

Micro-sized samples made of e.g. copper, tungsten single crystals or amorphous silicon oxy-nitrides, with typical dimensions ranging from 500 nm to ~10 µm were prepared with the FIB technique. In principle, almost any specimen geometry can be realized by this technique, however, in the following it will be concentrated on micro-tension and micro-bending specimens, where examples are depicted in Fig. 1a and 1b, respectively. The micro-tension specimens were prepared on the top of a single crystalline needle that were electro-polished prior the FIB milling. This set-up enables easy handling of the specimens (no lift-out is necessary) and loading with an appropriate gadget fitting to the sample's "head" in the right side. The micro-bending specimens were prepared in the vicinity of one of the sample's edge or at the top of a wedge prepared by Ar^+ milling allowing a FIB milling from two sides. For fracture tests, the bending beams were notched subsequently (see Fig. 1b). An in situ micro indenter performs the loading of the beams by prescribing a displacement or load at the free end of the beams (see for example Fig. 2).

Load vs. displacement data as well as images from the sample surface are recorded during the in-situ testing. For the tensile tests, size effects in flow stress, i.e. the increase in flow stress with decreasing sample diameter, are studied. It is shown that the flow stress at 10% plastic strain increases from 50 MPa up to 200 MPa if the sample width is decreased from 7.5 µm down to 500 nm for <-234> copper single crystals. Load drops (strain bursts) in the stress vs. strain curve can be associated with distinct slip events in the sample. Furthermore, the influence of experimental constraints, e.g. lateral stiffness of the sample-

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indenter system, misalignment between sample and indenter, etc., on the results is discussed. In the case of in situ fracture tests, the fracture behavior of (almost) ideal brittle dielectric materials (silicon oxy-nitrides) and single crystalline tungsten are compared. Where the macroscopic brittle tungsten shows a significant amount of plasticity and stable crack growth at the micron scale, the dielectric material fails in an ideal brittle mode. The measured fracture toughness values are close to the macroscopic ones for the dielectric materials.

This presentation should envisage the possibilities and advantages of in situ micro-mechanical testing, which enables easy alignment, tracking of deformation and damage evolution during loading, direct crack length measurement in fracture tests, etc.

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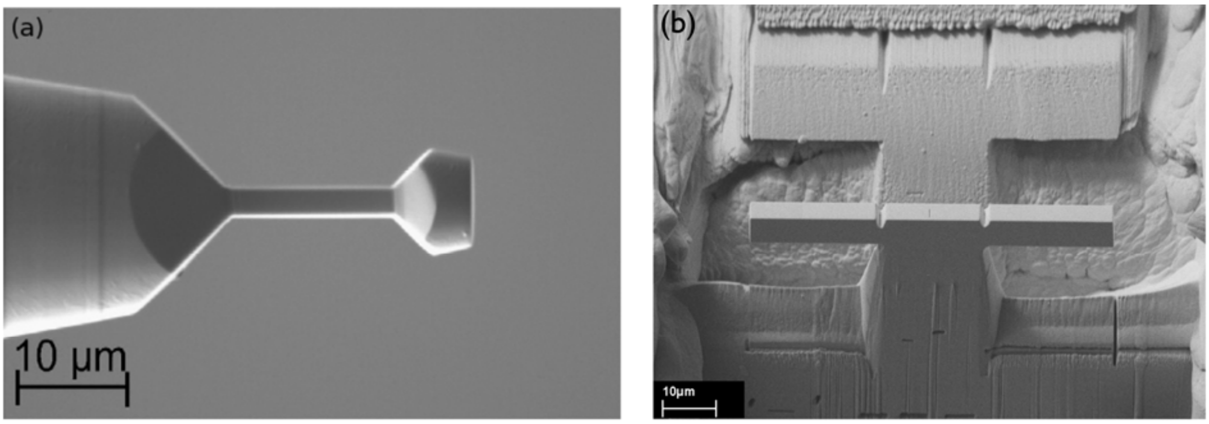

Figure 1. Examples of a micro tension specimen (a) and 2 notched micro-bending samples used for fracture tests (b) prepared by the FIB technique.

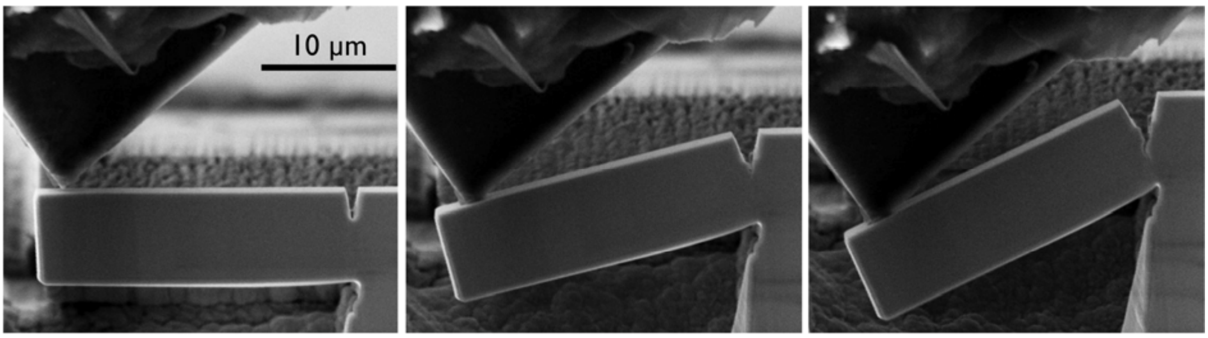

Figure 2. Example of an in situ micro fracture test carried out on a notched micro-bending beam. The loading is performed with an in situ micro indenter. A series of 3 SEM micrographs at different loading stages is presented.

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