A Novel Preparation Route to Obtain Micro-Bending Beams for In-situ TEM Studies

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Ultra-thin beams are widely used in micro-electro-mechanical systems (MEMS), sensors, and electrical actuators [1, 2]. The thickness of these beams is typically below 10µm. Within this size range the mechanical properties, especially the yield strength and the flow stress, become strongly size dependent.

The focused ion beam (FIB) workstation is a popular tool for preparing TEM samples as well as for fabricating micro-mechanical test structures to study the influence of small sample dimensions on the mechanical properties [3-7]. However, the microstructure of the sample can be critically altered by FIB milling, as studied for Cu in details in [8, 9]. The Ga implanted and damaged regions in Cu were found to be several nm thick depending on the ion dose and accelerating voltage.

Here, we outline a procedure for preparing micro-beams for cross-sectional TEM studies from single crystal Cu using a combination of conventional preparation and FIB milling to keep ion damage and FIB milling time as small as possible.

The preparation route includes the following steps: Firstly, a disc with a diameter of ~ 3mm was cut out using an ultrasonic disc cutter (Gatan). Secondly, the sample was ground from both sides down to a final thickness of ~100µm using diamond polishing papers “Ultraprep” (grade 15, 6 and 1 µm) and polished using “Masterprep” suspension 0.05µm (Buehler). As a next step, approximately 2/3 from the disc was cut off (Fig 1a). After slicing the sample, a large fraction of the surface was covered with the acid resistant lacquer “Lacomit” (Fig. 1b). For electrolytic thinning, the sample was immersed in a liquid electrolyte D2 (Struess) and acted as an anode. A Pt wire was used as a cathode. An electrical potential of ~2V was applied, which ensures anodic dissolution of the Cu specimen. The specimen was gently moved by hand so that the electrolyte builds up at the uncovered Cu surface, resulting in uniform thinning. The advantage of this technique, especially for elemental metals, is that the surface quality is superior to ion-thinned samples. The result of these preparation steps is a wedge shaped sample, where the region in vicinity of the prepared edge shows electron transparency. Finally, the bending beams were cut by FIB (Ga^+-ions, 30 keV, 20 to 200 pA ion current) on a LEO XB 1540 workstation. Thereby, a rectangle with a size of approx. 4 x 2 µm is milled under perpendicular ion beam incidence leaving a thin “bridge” left on the sample. Subsequently, this bridge is cut free on one side resulting in a bending beam as can be seen in Fig. 2b and Fig. 3. These bending beams can be used in subsequent in situ loading analyses. The main advantage of this preparation method is the low ion damage of the bending beams compared to pure FIB preparation.

1. K. Chau, W. Allegretto, L. J. Ristic, Sensors and Materials, 2 (1991) 253.
2. H. Geijselaseres, H. Tijdeman, Sensors and Actuators 29 (1991) 37.
3. J. R. Greer, W. C. Oliver, W. D. Nix, Acta Mat 53 (2005) 1821.

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4. D. Dimiduk, M. Uchic, T. Parthasarathy, Acta Mat 53 (2005) 4065.
5. C. Motz. T. Schöberl, R. Pippan, Acta Mat 53 (2005) 4269.
6. C. A. Volkert, E.T. Lilleodden, Phil Mag 86 (2006) 5567.
7. D. Kiener, C. Motz, T. Schöberl, M. Jenko, G. Dehm, Adv Eng Mat 8 (2006) 1110.
8. D. Kiener, C. Motz, M. Rester, M. Jenko, G. Dehm, Mater Sci Eng A 459 (2007) 262.
9. J. Marien, J. Plitzko, R. Spolenak, R.-M. Keller, J. Mayer, J Microsc 194 (1999) 71.

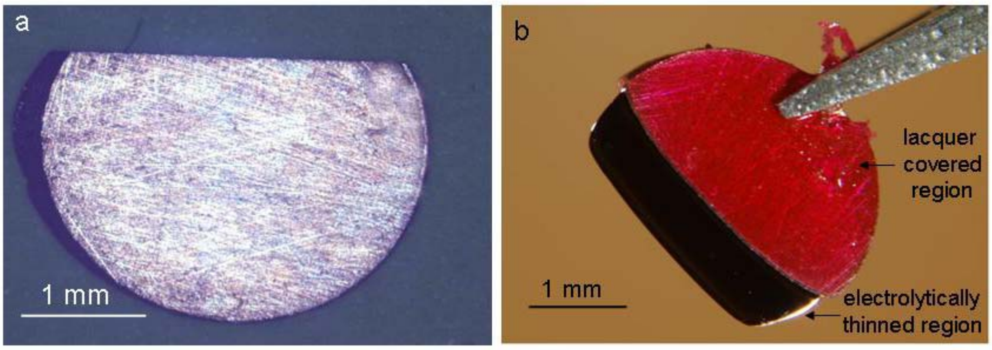

Figure 1. Optical micrographs of a) a 3 mm Cu disc, where 1/3 has been cut off using a wire saw and b) the specimen after having it covered with the lacquer and electrolytical thinning. The front part is thinned in a wedge shape which is electron transparent at the front end.

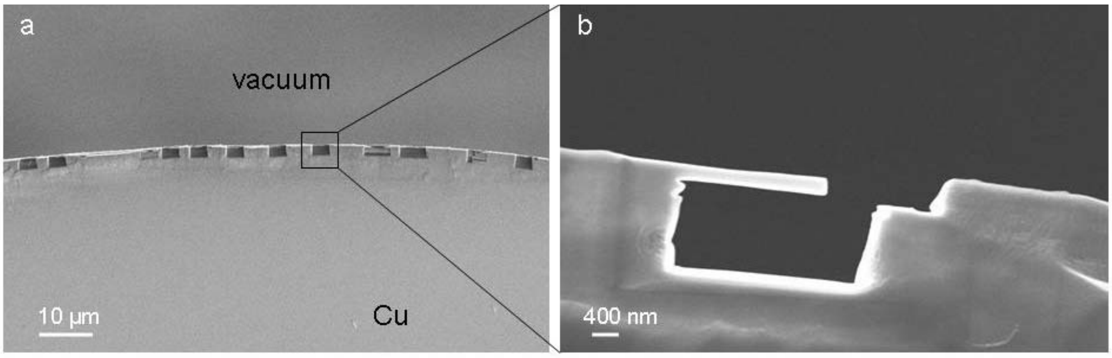

Figure 2. Scanning electron microscopy images of FIB milled bending beams. a) In the electrolytically thinned region several electron transparent beams were cut by FIB milling. b) One bending beam imaged at higher magnification.

Figure 3. Bright-field TEM image of a micro beam prior to mechanical testing.

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