FRACTURE BEHAVIOR OF METALLIC THIN FILMS AS EVALUATED BY BULGE-TESTS AND IN SITU TEM DEFORMATION EXPERIMENTS

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Metallic thin films generally show a fracture toughness which is considerably lower than that of bulk samples. Although this has been evidenced by several groups, a conclusive understanding of this low fracture toughness is still missing and open questions related with the difficulty of reliably testing very thin films often remain. Bulge testing is a very suitable method allowing reliable investigations of the fracture toughness of thin films by introducing a slit in a freestanding membrane by focused ion beam (FIB) milling. With such tests the fracture toughness of silver and gold films in the thickness range of 100 nm have been determined to be around 2 MPa $m^{1/2}$ confirming earlier results obtained with other testing techniques on similar metallic thin films. Recent investigations by Preiss et al. [1] gave an explanation for this extremely low fracture toughness based on in-situ observations of the crack tip region by atomic force microscopy (AFM). The AFM scans show stable crack growth mainly along grain boundaries and sliding of grains. Plastic deformation is localized in a very narrow corridor in front of the crack tip and a large plastic zone, as one would typically expect under plane stress, is not observed. We conclude that the spatial confinement of the plastic deformation is the primary reason for the low fracture toughness of metallic thin films.

More detailed observations of the deformation mechanisms are of particular interest and are enabled by *in situ* transmission electron microscopy (TEM). For this a new flexible method for the preparation of thin film samples for *in situ* mechanical testing in a TEM has been developed [2], which is based on a combination of focused ion beam (FIB) shadow milling and electron-beam-assisted etching with Xenon difluoride precursor gas. Loading of the specimens is performed by a TEM Nanoindenter combined with a Push-to-Pull conversion device. In contrast to existing FIB-based preparation approaches, the area of interest is never exposed to ion beam irradiation and a pristine microstructure is preserved.

With this method nanotwinned Cu and Cu-Al thin films were tested *in situ* in the TEM. Al is an effective element to reduce the stacking fault energy in Cu alloys and leads to increased amount of twinning and detwinning events. The films are tested until final fracture and different deformation mechanism as sliding of grains, twinning and dislocation activity can be correlated with the captured stress-strain curves from the experiment. The fracture behavior of these films will be discussed in the presentation and compared to the bulge-test results.

References

[1] E. I. Preiß, B. Merle, M. Göken; Understanding the extremely low fracture toughness of freestanding gold thin films by in-situ bulge testing in an AFM; Mat. Sci. Eng. A691 (2017) 2018-2025

[2] J.P. Liebig, M. Göken, G. Richter, M. Mačković, T. Przybilla, E. Spiecker, O.N. Pierron, B. Merle; A flexible method for the preparation of thin film samples for in situ TEM characterization combining shadow-FIB milling and electron-beam-assisted etching; Ultramicroscopy 171 (2016) 82–88