In-situ TEM study of micro/nanoscaled amorphous or nanocrystalline freestanding films under stress

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Basic understanding of the mechanical properties of amorphous or nanocrystalline materials is essential for their future potential application. Consequently, various established measurements as well as new developed methods have been used to deepen the knowledge of the deformation behaviour and processes of such materials. In the present work we investigated amorphous or nanocrystalline freestanding thin films using transmission electron microscopic (TEM) methods to study their elastic and plastic behaviour under tensile stress condition.

Freestanding microscaled amorphous TiAl and nanocrystalline Al thin films were produced on MEMS devices using DC magnetron sputtering. The MEMS based tensile devices have built-in force and displacement sensing gauges to measure macroscopic stress and strain during in-situ tensile deformation (cf. Fig. 1a). The in-situ TEM study was performed with a Philips CM200 microscope operating at 80-200kV and a Philips straining holder. Bright-field and diffraction images were acquired with a Gatan Orius™ SC600 at different stress states of the film.

Fig. 1b shows the stress-strain curves of a nanocrystalline Al sample taken under different imaging conditions. The black curve (first cycle) corresponds to the stress-strain values measured at the gauges without irradiating the samples whereas the red, green and blue curves show the behaviour during imaging using 80, 120 and 200kV, respectively. The analysis of the curves in combination with bright-field imaging of dislocations at different stress states (cf. Fig. 1c) indicates a clear electron beam-induced stress relaxation caused by enhanced activation of dislocations [1]. The effect is more pronounced at lower accelerating voltages and leads to local sample necking at the irradiated area. Nevertheless, in-situ TEM can be used to correlate the stress-strain curve with the grain size dependent activity of dislocations and to understand the deviation from linearity during unloading (Bauschinger effect).

Fig. 2a shows the diffraction image of sputter deposited TiAl. The broad intense ring is characteristic for an amorphous structure. Under stress the circular ring becomes elliptic as illustrated schematically in Fig. 2a. By measuring the position of the intensity maxima (as a function of the azimuthal angle χ) and fitting an ellipse the local atomic-level strain can be calculated (cf. Fig. 2b). The method, evaluation and calculation using our Digital Micrograph™ script are described in [2]. By plotting principal atomic-level elastic strains as a function of stress mechanical properties (Young's modulus, Poisson's ratio) of the thin film can be calculated. The comparison with macroscopic data reveals the presence of anelasticity of the TiAl thin film. In addition, strain maps or time-dependent strain evolution under stress can be obtained by this method [see C. Ebner, this conference].

Ref:
[1] R. Sarkar, C. Rentenberger, J. Rajagopalan, Scientific Reports **5** (2015) 16345.
[2] C. Ebner et al., Ultramicroscopy **165** (2016) 51-58.
[3] C.E. and C.R. acknowledge financial support by the Austrian Science Fund (FWF): [I1309].

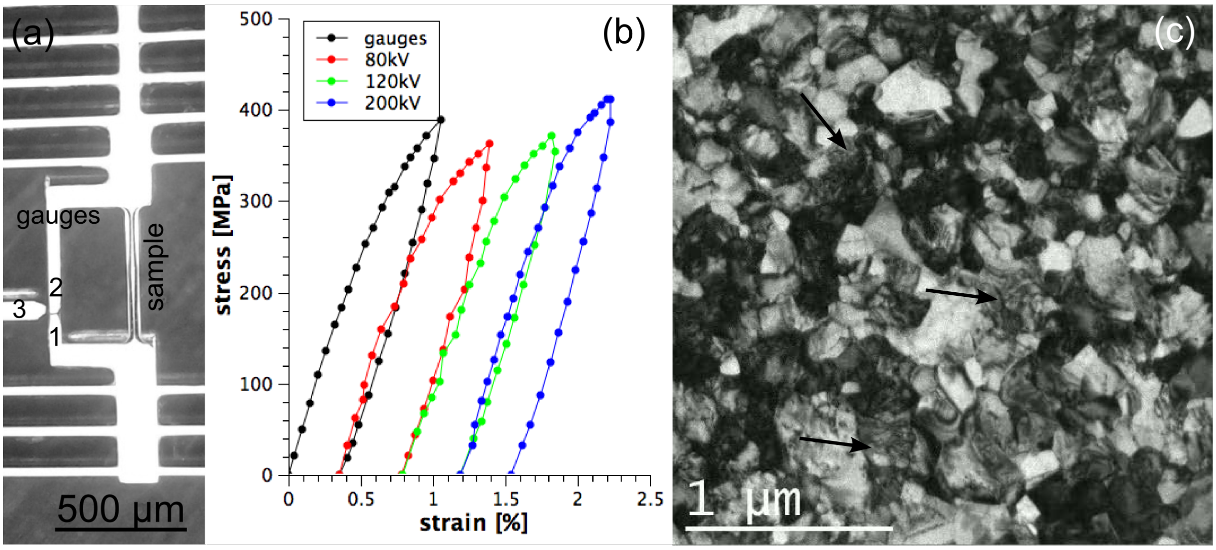

Figure 1: Nanocrystalline Al. (a) Freestanding thin film and stress-strain gauges prepared on a MEMS device. (b) Stress strain-curves measured under different illumination conditions. (c) Bright-field image, sudden contrast changes indicate dislocation activity.

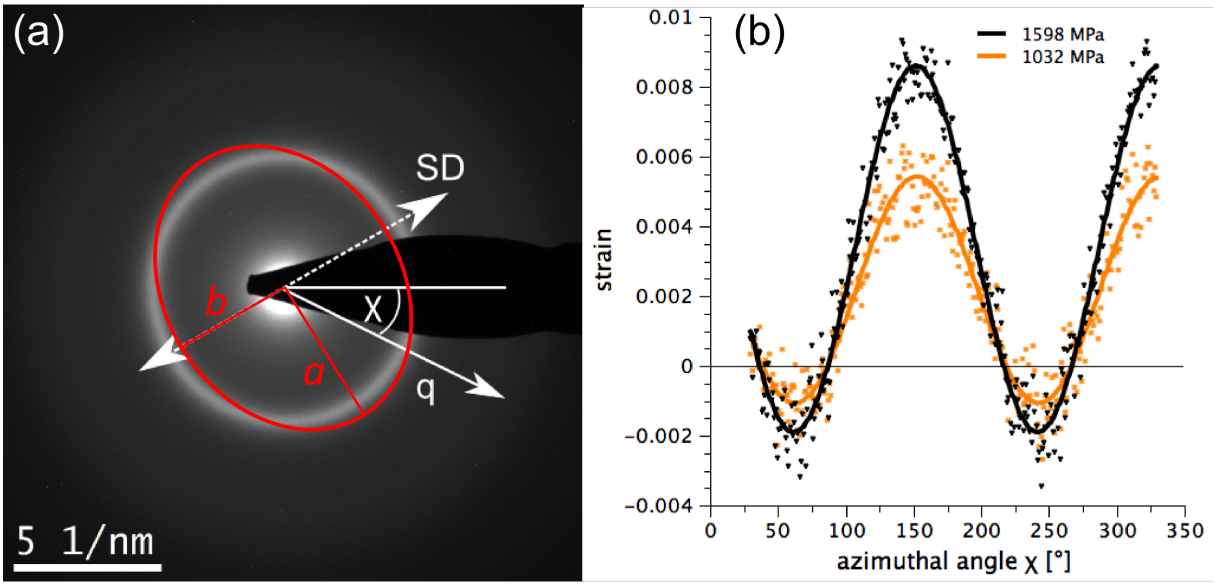

Figure 2: Amorphous TiAl. (a) Diffraction pattern becomes distorted under stress as illustrated by the ellipse (shear direction SD, scattering vector q). (b) Atomic-level elastic strain: maxima and minima correspond to the principal elastic strains.