	Experiment title: Quantitative diffraction on single nano-structures for mechanical, photonic and electronic applications	Experiment number: HS-4670
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Report:

Within the framework of the project ANR MecaniX and this long-term proposal (HS-4670) we developed a scanning force microscope for *in situ* nanofocused X-ray diffraction studies (SFINX) [1]. After having demonstrated its capabilities in combination with coherent X-ray diffraction at the ID01 beamline in June 2013 (see last report), the tool was installed at BM32 in March 2014. During this experiment *in situ* three-points bending tests on single self-suspended Au nanowires were performed in combination with µLaue diffraction.

***In situ* µLaue diffraction**

A photograph and a schematic of the experimental setup at BM32 are presented in Figs. 1(a) and (b), respectively. Using the recently installed KB mirrors, the polychromatic X-ray beam which covers an energy range of 5 – 22 keV was focused down to 400 nm x 700 nm in vertical and horizontal direction, respectively. The X-rays diffracted by the sample were monitored by a MarCCD mounted at 90° in a distance of 70 mm from the sample stage. Additionally, the X-ray fluorescence was monitored by means of a ROENTEC energy dispersive detector.

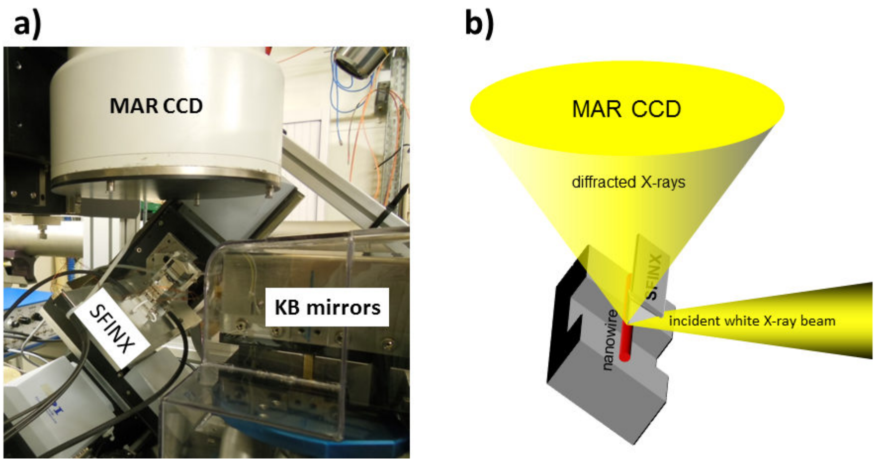

Fig. 1: a) Photograph and b) schematic of the experimental setup at the BM32 beamline including SFINX.

In this experiment, for the first time, self-suspended Au nanowires were mechanically loaded by means of the AFM-tip and the deformation was monitored by µLaue diffraction. For this purpose, nanowires have been placed across micro-trenches which were created by UV lithography on a Si(001) wafer. The resulting self-suspended nanobridges were mechanically loaded in their center employing the AFM tip and the deformation of the nanowire was followed in-situ by µLaue diffraction. A scanning electron micrograph, an *in situ* AFM topography image, and an *in situ* fluorescence map of the Au nanowire which was later-on mechanically loaded are presented in Fig. 2(a), (b), and (c).

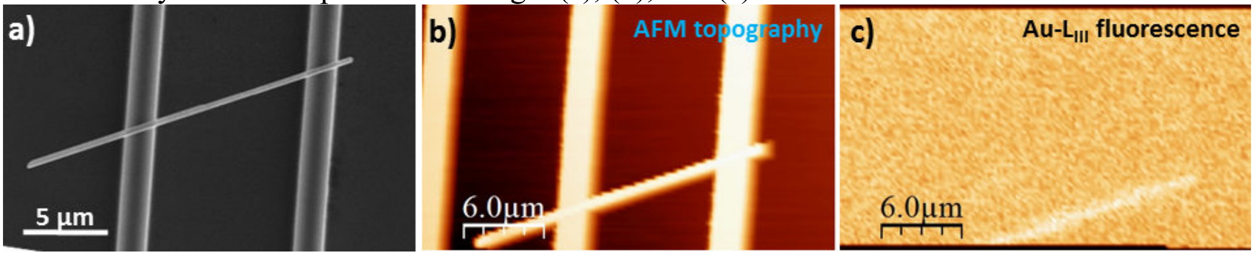

Fig. 2: a) Scanning electron microscopy image, b) *in situ* AFM image, and c) *in situ* fluorescence map of a single Au nanowires crossing a Si micro-trench.

During the bending of the nanowire with the AFM-tip µLaue diffraction patterns were recorded. A sequence of µLaue diffraction patterns of the Si001 and Au222 Laue spot is displayed in Fig. 3(a). While the two Laue spots are superimposed with each other for the pristine nanowire, the Au222 Laue spot moves on the detector during the bending process and it returns to its original position after complete unloading of the wire. Thus, the mechanical test remains in the elastic regime of the nanowires. From the positions of the Laue spots on the detector, the orientation (UB) matrices for the Au nanowire were calculated employing the LaueTools software developed by the staff of the BM32 beamline. By this means the bending of the wire during the mechanical test was computed. The bending angle as a function of the movement of the piezostage is presented in Fig. 3(b) revealing a bending of about 3.3 ° at the highest load. Finite element method simulations using COMSOL Multiphysics revealed that the stress in the nanowire amounts to ~ 500 MPa exceeding by far the yield strength of bulk gold which amounts to about 1 MPa. Thus, this experiment on nominally defect free nanowires proves that nanowires are stronger than their bulk counterparts in agreement with literature on the same topic.

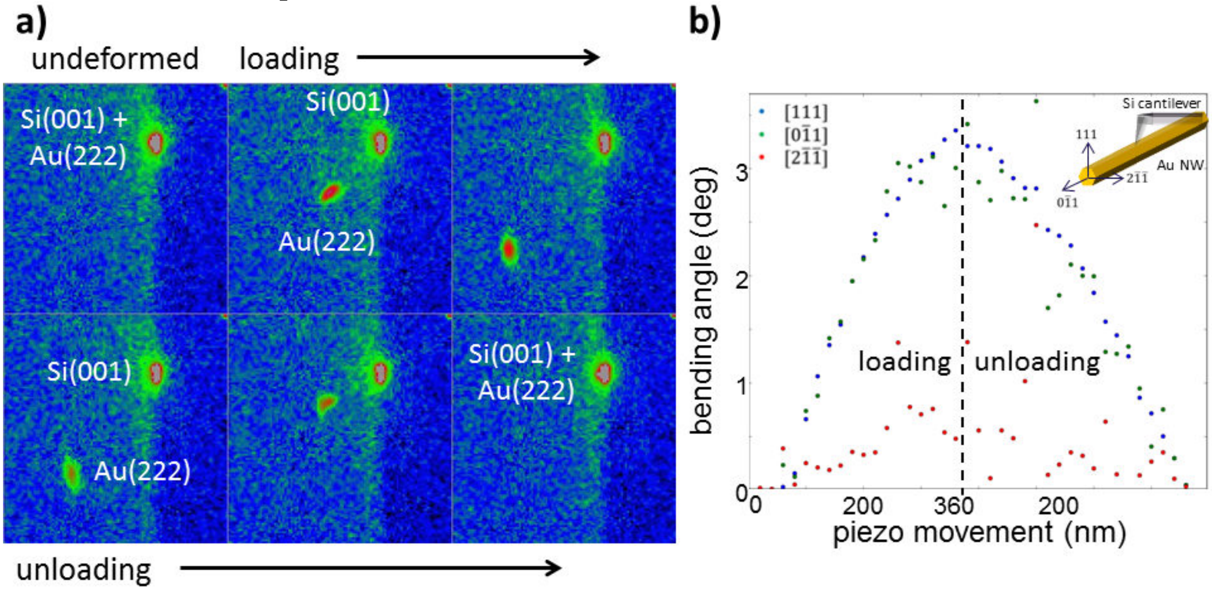

Fig. 3: a) *In situ* µLaue diffraction patterns recorded during the bending of a Au nanowire and b) bending angle computed from the position of the Laue spots.

This experiment demonstrates the successful coupling of contact atomic force microscopy and µLaue diffraction to monitor the deformation of a nanowire *in situ*. It is the first *in situ* µLaue diffraction experiment so far which has been performed on the mechanical deformation of sub-micrometer structures. This work has been presented as an oral contribution at the XTOP 2014 conference in Villard de Lans and published in J. Appl; Crystallogr. [2].

References

[1] Z. Ren, F. Mastropietro, S. Langlais, A. Davydok, M.-I. Richard, O. Thomas, M. Dupraz, M. Verdier, G. Beutier, P. Boesecke, T.W. Cornelius, J. Synchrotron Radiat. 21 (2014) 1128 – 1133

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