# Standard Project

## *Experimental Report template*

| **Proposal title:** **In-situ mechanical testing on mechanically deformed Cu nanowires by µLaue X-ray diffraction and Atomic Force Microscopy** | | **Proposal number:** 32-02 760 |
|---|---|---|
| **Beamline:** BM32 | **Date(s) of experiment:** from: 24/07/2014 to: 28/07/2014 | **Date of report:** 10/02/2015 |
| **Shifts:** 12 | **Local contact(s):** Odile Robach | *Date of submission:* |

**Objective & expected results** (less than 10 lines):

The goal of this study was the *in situ* mechanical testing of single self-suspended nanowires by three-points bending tests using a newly developed *in situ* AFM in combination with µLaue diffraction. The deformation of the wire was monitored by the position of the Laue spots on the detector and the shape of the Laue spots may give access to strain and defects induced in the nanostructure. Thus, the results will improve the understanding of the elastic and plastic properties of quasi one-dimensional nanomaterials.

**Results and the conclusions of the study** (main part):

While *in situ* mechanical tests on Cu nanowires were planned, we decided to use Au nanowires instead since they were nominally defect free in contrast to the Cu wires which were studied in experiment 20120033 revealing plenty of stacking faults and twins. Single crystalline gold nanowires were grown by physical vapor deposition on carbon coated tungsten substrates under ultrahigh vacuum conditions at elevated temperatures. For three-points bending tests, Au nanowires were placed across 10 µm wide and 1.5 µm deep micro-trenches forming self-suspended nano-bridges (Fig. 1(a)). The micro-trenches were fabricated on Si(001) wafers by a combination of UV lithography and reactive ion etching. In order to avoid any sliding of the wire during mechanical testing, the wires were thoroughly clamped at the two supports by electron beam induced deposition of carbon from the residual gas in the scanning electron microscope chamber.

The polychromatic X-ray beam covering an energy range of 5 – 25 keV was focused down to 500 x 700 $nm^2$ using KB mirrors. For aligning the AFM-tip and the nanostructure with respect to the focused X-ray beam, *in situ* AFM topography images and Au-$L_{III}$ fluorescence maps were recorded simultaneously. After alignment of the two probes, the AFM-tip was positioned above the center of the self-suspended Au nano-bridge. For mechanical loading, the AFM-tip was moved down with a constant speed of 5 nm/s and µLaue diffraction patterns with an exposure time of 1 s were recorded *in situ*. After every 50 nm of piezo movement, the movement of the AFM-tip was interrupted and a complete profile along the bent nanowire was recorded. In order to avoid any vibrations induced by motors of the sample stage, the X-ray beam was scanned along the wire by tilting the KB mirrors mounted on a hexapod stage. The mirror tilting resulted in a displacement of the beam spot on the sample. The calibration of the beam displacement as a function of the mirror tilting is presented in Fig. 1(b) revealing a linear relationship. The full width at half maximum of the vertical beam focus increases from about ~800 nm to about 1.2 µm for a displacement of 12 µm on the sample. This degradation of the focus does not affect the experiment, only slightly reducing the diffracted intensity. The evolution of the central Laue spots of Au111 along the nanowire and the Si001 substrate beneath are presented in Fig. 1(c) for different stages of deformation. From the displacement of the Laue spots on the detector the orientation of the nanowire, i.e. bending and rotation of the crystal was inferred employing the LaueTools software. The bending angle as a function of the piezo movement is presented in Fig. 1(d). The experimental data are in good accordance with finite element method simulations using COMSOL Multiphysics represented by the red curve. The sudden jump of the bending angle at $\Delta z$ ~900 nm is probably caused by a plastic event in the nanowire such as an avalanche of dislocations.

At the position where the wire is supported, the Laue spots are strongly streaked even without application of any force indicating that strain and/or defects may be induced by the support and the additional carbon clamping. This effect shall be further investigated by coherent diffraction methods which are highly sensitive to both strain and defects.

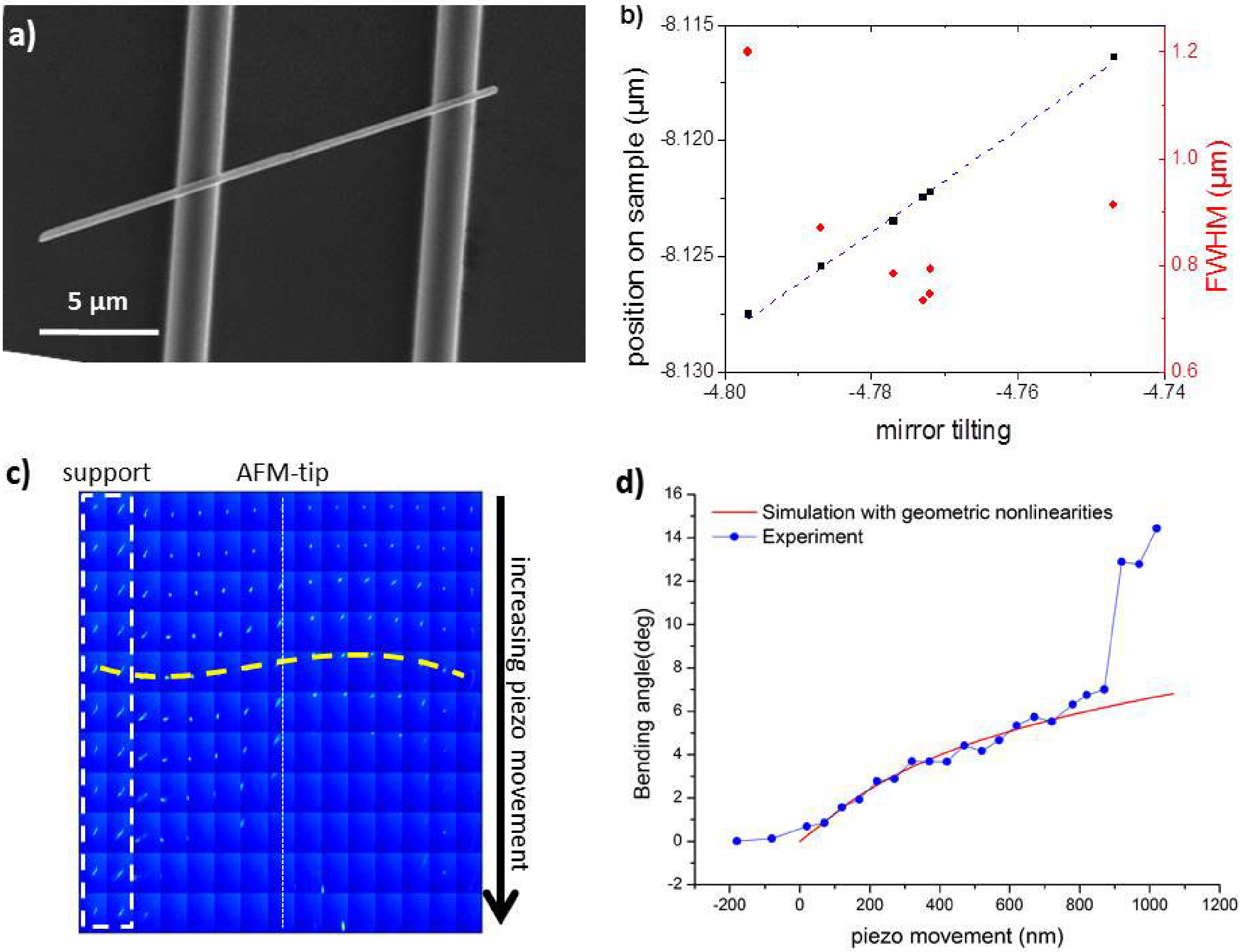


*Fig. 1: a) Scanning electron microscopy image of a self-suspended Au nanowire. b) Calibration of the displacement of the focused X-ray beam on the sample when tilting the KB mirrors. c) Au111 Laue spot along the self-suspended nanowire recorded during mechanical loading. d) Bending angle as a function of the piezo movement inferred from the displacement of Laue spots on the detector during mechanical loading. The red curve represents finite element method simulations of the bending of the wire.*

In addition to the experiments presented above, an energy dispersive pnCCD was installed and energy dispersive µLaue diffraction patterns along nanowires which were plastically deformed during the prior *in situ* bending tests were recorded.

The experiments presented in this report demonstrate for the first time measurements of the profile of a nanostructure during mechanical deformation. The wire profile gives access to the boundary conditions, i.e. whether the wire is thoroughly clamped at the supports being important for determining the elastic constants of the quasi one-dimensional nanostructure.

**Justification and comments about the use of beam time** (5 lines max.):

This experiment allowed for the first time to follow the evolution of the nanowire profile during a three-points bending test. This new *in situ* technique will be an important asset for the beamline community.

Due to the time consuming installation of the energy dispersive pnCCD, the beamline staff granted us with 3 additional shifts.

**Publication(s):**

- Z. Ren, C. Leclere, T.W. Cornelius, A. Davydok, J.-S. Micha, O. Robach, G. Richter, L. Belliard, O. Thomas, ESRF User Meeting, poster