

# Standard Project

## Experimental Report template

<b>Proposal title: Mechanical properties of single Fe nanowires studied by in situ three-points bending tests combined with <math>\mu</math>Laue diffraction</b>		<b>Proposal number:</b> 32-02 786
<b>Beamline:</b> BM32	<b>Date(s) of experiment:</b> from: 13/04/2016 to: 17/04/2016	<b>Date of report:</b> 26/08/16
<b>Shifts:</b> 12	<b>Local contact(s):</b> O. Robach	<i>Date of submission:</i>

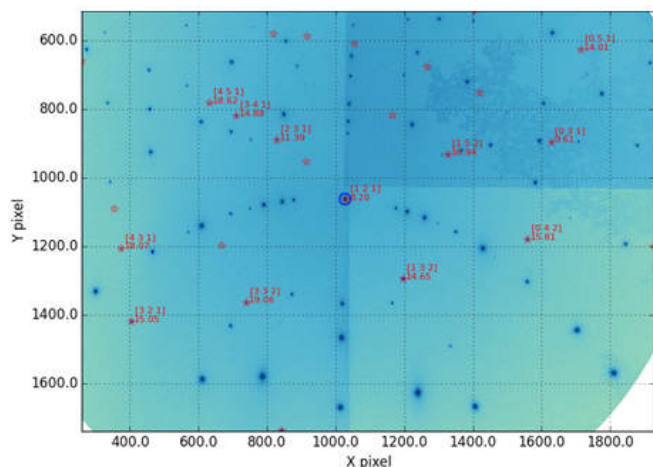
### Objective & expected results (less than 10 lines):

The goal of this experiment was the study of the mechanical properties of Fe nanowires by three-point bending tests using the in-situ AFM “SFINX” in combination with simultaneous  $\mu$ Laue diffraction. While the mechanical properties of FCC metals are rather well studied even on the nanoscale, experiments on BCC metals are lacking. In addition, the nucleation and storage of defects and dislocations are fundamentally different in FCC and BCC structures. The three-point bending tests on single self-suspended iron nanowires were expected to give access to the elastic and plastic behavior and to elucidate the storage of dislocations in BCC nanostructures in comparison to FCC nanostructures such as Au nanowires studied during former beamtimes.

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

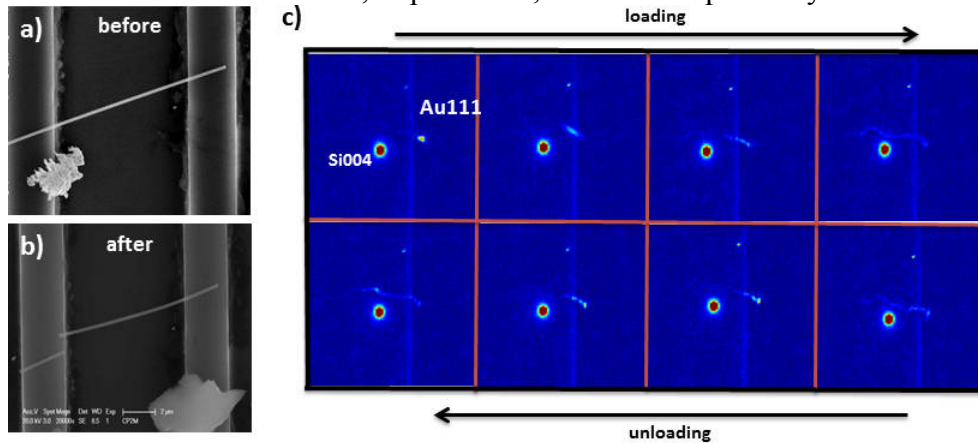
Due to the fact that the length of the as-grown nanowires was  $< 10 \mu\text{m}$  we were not able to prepare self-suspended Fe nanowires. Thus, on the one hand, we concentrated on the crystalline structure and orientation of the Fe nanowires and, on the other hand, we performed three-point bending tests on Au/Al<sub>2</sub>O<sub>3</sub> core/shell nanowires. In addition, we used this beamtime to improve and to optimize the KB scanning method where the focused polychromatic X-ray beam is scanned across the stationary sample in order to measure the profile of the deformed nanowire in-situ.

Preliminary  $\mu$ Laue experiments concentrated on the crystalline structure of Fe nanowires. The  $\mu$ Laue diffraction patterns exhibit well-defined and sharp Laue spots indicating that no geometrically necessary dislocations are stored in these iron nanowires (Fig. 1). Moreover, while the growth direction of the iron nanowires is along the  $\langle 110 \rangle$  direction,  $\mu$ Laue diffraction revealed that the wires lie either on their (211) or on their (200) facet. These different orientations are important to know due to the anisotropic mechanical behavior of the material.



**Fig. 1: Laue microdiffraction pattern of a Fe nanowire.**

Instead of mechanically testing Fe nanowires, we loaded Au/Al<sub>2</sub>O<sub>3</sub> core/shell nanowires using the in-situ AFM and monitoring the deformation by Laue microdiffraction. Figures 2(a) and (b) show scanning electron micrographs of a self-suspended Au/Al<sub>2</sub>O<sub>3</sub> core/shell nanowire before and after the mechanical test revealing a rupture at the left-hand side of the nanowire. The evolution of the Au 111 Laue spot during the mechanical test is shown in Fig. 2(c). During the deformation the spot becomes elongated and is displaced on the detector. After complete unloading the Laue spot does not return to its original position on the detector and remains streaked indicating irreversible deformations, i.e. plasticity which is in agreement with the SEM observations. These experiments on cladded nanowires will pave the way to study surface effects on the mechanical behaviour, in particular, the onset of plasticity and the nucleation of defects.



**Fig. 2:**

During this beamtime we also tested and improved the KB scanning method where the focused polychromatic X-ray beam is scanned across a stationary sample. Different measurement protocols were tested in order to minimize drifts and increase the experiment stability. We finally obtained uncertainties below 0.35  $\mu\text{m}$  which is smaller than the beam size on the sample surface. These improved measurement protocols will facilitate faster and more accurate measurement of the profile of deformed nanostructures in the future. They will be accessible for the whole user community of the BM32 beamline at ESRF.

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

Originally we intended to deform Fe nanowires by three-point bending tests. The limited length of the Fe nanowires did not allow us for preparing self-suspended nano-bridges. Thus, we concentrated on studying the crystalline structure and orientation of the Fe nanowires revealing that they are free of defects. In addition, we mechanically loaded Au/Al<sub>2</sub>O<sub>3</sub> core-shell nanowires using the in-situ AFM. These experiments pave the way to study cladded nanowires, thus enabling us to investigate the influence of the nanostructure surface on the mechanical behavior.

**Publication(s):**

- 
-