| ESRF | Experiment title: Quantitative diffraction on single nano-structures for mechanical, photonic and electronic applications | Experiment number: HS-4670 |
|-------------------|--|------------------------------------|
| Beamline: BM32 | Date of experiment: from: 27 Nov 2015 to: 30 Nov 2015 | Date of report: 2 February 2016 |
| Shifts: | Local contact(s): O. Robach | Received at ESRF: |

Names and affiliations of applicants (* indicates experimentalists):

T.W. Cornelius*, Z. Ren*, O. Thomas*, Im2np (UMR 7334) CNRS, Aix-Marseille University, 13397 Marseille, France

- V. Favre-Nicolin, CEA Grenoble, INAC SP2M, Grenoble, France
- J. Eymery, CEA Grenoble INAC SP2M/NPSC, Grenoble, France
- O. Robach, CEA Grenoble INAC SP2M/NRS, Grenoble, France

Report:

Within this long term project we successfully coupled the in situ AFM "SFINX" with µLaue diffraction for in situ three-point bending tests on individual self-suspended nanowires. While the experiments so far concentrated on Au nanowires which exhibit a face centered cubic (FCC) crystalline structure, we extended the measurements to Fe nanowires which possess a body centered cubic (BCC) crystalline structure. In general, the mechanical properties of FCC metal nano-objects are rather well studied, whereas investigations on BCC metal and, in particular, on Fe nanowires are lacking.

Preliminary μ Laue experiments concentrated on the crystalline structure of Fe nanowires prepared as self-suspended nano-bridges crossing Si micro-trenches for future nano-mechanical tests (Fig. 1(a)). The μ Laue diffraction patterns exhibit well-defined and sharp Laue spots indicating that no geometrically necessary dislocations are stored in this iron nanowire (Fig. 1(b)). Moreover, while the growth direction of the iron nanowires is along the <110> direction, μ 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.

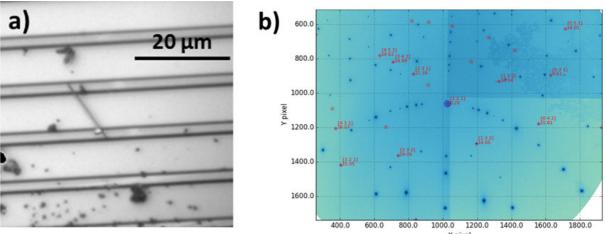


Fig. 1: a) Optical micrograph and b) µLaue diffraction pattern a self-suspended Fe nanowire crossing a Si microtrench.

In addition, we coupled for the very first time a MEMS based tensile testing stage with µLaue diffraction which allows for in situ tensile tests on individual nanowires. An optical microscopy image of a thermally

actuated tensile testing stage is presented in Fig. 2(a). The scanning electron micrograph in Fig. 2(b) shows an individual nanowire bridging the gap between the two grips being clamped by electron beam induced deposited Pt at each end. For locating the nanowire the sample was scanned with respect to the focused X-ray beam and the fluorescence yield of the Au L α_1 and the Pt L α_1 fluorescence was monitored. A scanning fluorescence map showing the Au nanowires and the Pt clamps is displayed in Fig. 2(c). Individual Au nanowires were tensile strained using this MEMS based tensile testing stage and μ Laue diffraction patterns were recorded simultaneously. The data recorded during these in situ tensile tests are currently being analyzed.

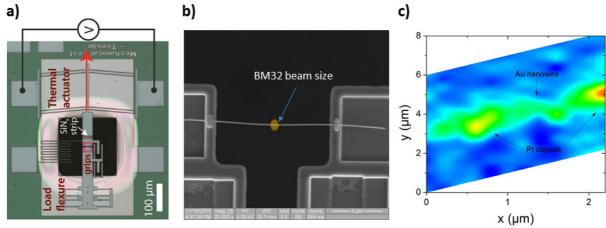


Fig. 2: a) Thermally actuated tensile testing stage onto which an individual gold nanowire (b) is manipulated. The comb features to the side of the sample grips may be used for tracking displacements of the load cell and actuator. c) Fluorescence map of the Au nanowire and its Pt clamps.

Besides in situ tensile tests on Au nanowires, on ex situ tensile tested nanowires were studied by μ Laue diffraction. Transmission electron microscopy revealed planar defects which are oriented along the long axis of the nanowire (see Fig. 3(a)). A μ Laue diffraction pattern and a zoom of the Au 222 and the Au 131 Laue spots are presented in Fig. 3(b) and (c), respectively. While the Au 222 Laue spot is very sharp, the Au 131 spot exhibits fringes which indicate the presence of stacking faults within the nanowire. The fact that no fringes are observed for the Au 222 spot originates from the orientation of the defects with respect to the (222) atomic planes.

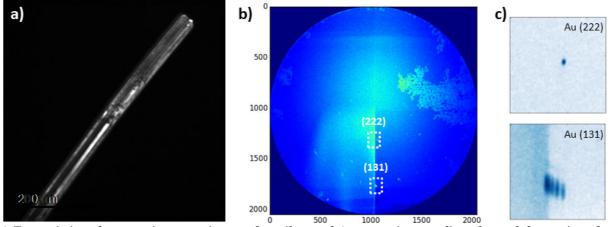


Fig. 3: a) Transmission electron microscopy image of tensile tested Au nanowire revealing planar defects oriented along the long axis of the nanowire. b) µLaue diffraction of the same nanowire as in (a). c) Zoom on the Au 222 and Au 131 Laue spots.

These experiments open up new possibilities in nano-mechanical studies covering FCC as well as BCC nanostructures and they pave the way to *in situ* mechanical tests both by three-point bending tests and by tensile tests.