ESRF	Experiment title: Studying nanoscale deformation mechanisms in individual coated and uncoated Au nanowires via <i>in situ</i> tensile testing with coherent X-ray diffraction	Experiment number : MA-3065
Beamline: BM32/ID01	Date of experiment:from: Nov 06th, 2016to: Nov 13th 2016	Date of report:
Shifts: 21(6+15)	Local contact(s): Jean-Sébastien Micha, Marie-Ingrid Richard	Received at ESRF:
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Report:

Series of in-situ tensile tests were performed on a gold nanowire to study the fundamental deformation mechanism of nanomaterials. The gold nanowires were grown on tungsten substrates via physical vapor deposition with molecular beam epitaxy method. These nominally defect-free nanowires were then individually transferred and fixed on a MEMS based tensile stage with Pt using electron beam induced deposition (See Fig.1-a, b, and Fig.4). While the growth direction of the nanowires is well known to be along the <110> direction, the nanowire orientation on the MEMS based tensile testing stage is a priori unknown. Previous attempts on in-situ test were mainly obstructed by this difficulty finding a desirable Bragg peak of the nanowire on the stage. We solved this issue by Laue microdiffraction at the BM32 (Fig. 1-c) beamline prior to the in-situ Bragg coherent diffraction imaging experiment at ID01 which allowed us for precisely indexing the orientation of the as-manipulated wire relative to the tensile stage as shown in Figure 1-d.



Figure 1. a) Thermally actuated nanotensile stage b) and the close up view. c) Sample mounted at BM32 d) CCD image of Laue pattern of the Au nanowire. Red is indexed Au peaks and the <0-11> wire zone axis is labeled in yellow line.

At BM32, series of μ Laue patterns were recorded along the entire nanowire. This analysis revealed that the nanowire on the stage was bowed and twisted by 1.0° and 1.3°, respectively, as depicted in the Figure 2-b. With the appropriate transformation of all the axes from BM32 to ID01, we were able to find the exact Bragg peak that we have targeted which was Au {311} peak. Figure 2-c shows the two-dimensional

projection of the 3D coherent diffraction image of the Au 311 peak recorded at ID01 before straining the wire, and Figure 2-d shows the reconstructed phase.



Figure 2. a) Sum of {311} peak along the wire measured in BM32 b) schematic of as-manipulated nanowire understood by the micro-Laue analysis c) {311} Bragg peak measured in ID01 before straining d) 2D reconstruction of the wire

The tensile stage was actuated by applying voltage to the actuator beam (Fig1-a). At each voltage step a rocking scan was performed, thus recording a 3D coherent diffraction pattern of the Au 311 Bragg peak. Figures 3a and b display the overlay of the applied voltage and the tensile strain along the <110> direction computed from the compressive strain along the 311 axis (determined from the variation of the Au 311 Bragg peak along Qz) and the tilt of the nanowire calculated from the angular change of the peak in reciprocal space. The shape as well as the position of the {311} peak evolved during the tensile test as presented in Fig. 3c. The bending decreases as the nanowire gets strained while the torsion remains nominally unchanged. The Au nanowire yielded at around 2.1% tensile strain which corresponds to about 1.7GPa by using the elastic modulus along the <110> direction for bulk Au single crystal for the calculation.



Figure 3. Applied voltage (actuation) is overlaid with a) <110> tensile strain and b) angular movement of $\{311\}$ peak in reciprocal space. c) Qz sum of the $\{311\}$ Bragg peak during the tensile tests

Figure 4 shows scanning electron micrographs of the nanowire before and after the in-situ tensile tests evidencing plastic deformation of the nanowire. Also we should notice that the contamination was more concentrated on where the beam was staying during the experiment.



Figure 4. a) SEM image of the nanowire before and b) after the in situ tensile tests at ID01. The rough beam size and the position is marked in red squared box.

To our knowledge this experiment reported here is the first successful systematic in-situ tensile test on a single nanowire in Bragg coherent x-ray imaging condition.