ESRF	**Experiment title:** Interdiffusion in Ag-Au core-shell nanowires: an *ex* situ anomalous coherent X-ray imaging study	**Experiment number**: HC-1134
Beamline: ID13	**Date of experiment**: from: 31/01/2014 to: 04/02/2014	**Date of report**: 1 March 2014
Shifts: 12	**Local contact(s)**: Emanuela Di Cola	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

M.I. Richard*, S. Fernandez*, S. Labat*, T. Cornelius*, C. Leclere*, O. Thomas* (Aix-Marseille University, IM2NP-CNRS)

O. Mandula*, V. Favre-Nicolin (UJF-CEA-Grenoble)

L. Chen*, P. Jungo Shin*, D. Gianola (Department of Materials Science and Engineering, University of Pennsylvania, Philadelphia, USA)

G. Richter, D. Floetotto (Max Planck Institute for Intelligent Systems, Sttutgart, Germany)

Report:

The goal of this experiment was to characterize *single* and *isolated* core-shell nanowires (NWs) by using coherent diffraction imaging in both forward scattering and Bragg diffraction geometries in order to determine their shape, composition and strain fields. Isolated Ge-Si and Ag-Au core-shell nanowires, grown by chemical vapor deposition (CVD) [1], were measured. Before the experiment, some NWs were selected from their growth substrate thanks to an ultrasonic bath and then deposited on a Si_3N_4 membrane (see Fig. 1**a**). A spiky surface morphology was observed on the Ag-Au core-shell nanowires, when measured under atmospheric conditions (see Fig. 1**b**). A small chamber, allowing measurements in inert gas (He) flow, was developed (see Fig. 1**c**) to host the Si_3N_4 membrane and avoid beam damage (spiky surface morphology or C-overlayer formation).

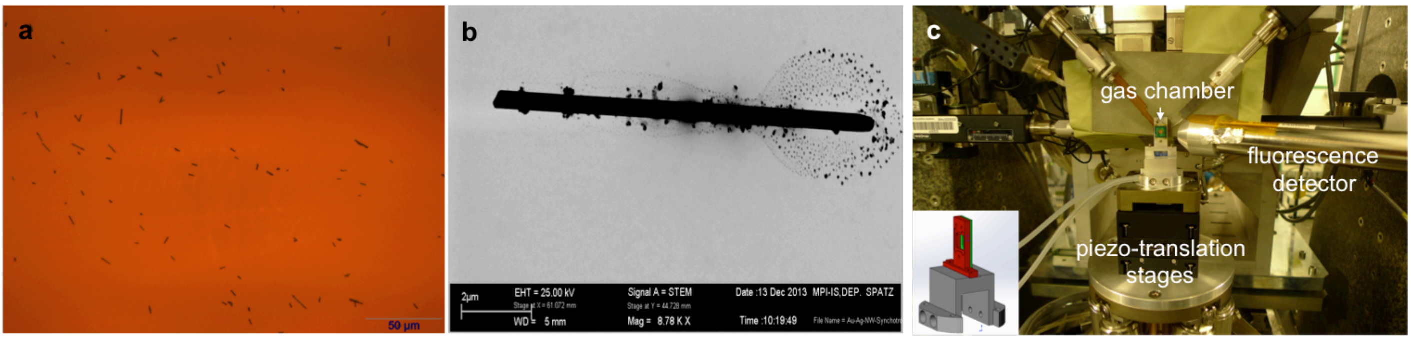

Fig. 1: (**a**) Isolated Ag-Au core-shell nanowires deposited on a Si_3N_4 membrane measured using the optical microscope available at ID13. (**b**) Scanning electron microscopy (SEM) image of a Ag-Au core-shell nanowire after X-ray measurements under atmospheric conditions. (**c**) Newly developed chamber allowing measurements in inert gas flow at ID13.

The X-ray beam was monochromatized to an energy of 14.9 keV and focused down to 200 nm using Si compound refractive lenses. The nanowires were located on the membrane with the help of the optical microscope and the fluorescence detector, mapping the Ge or Au fluorescence signal for the Ge-Si or Ag-Au core-shell nanowires. Coherent diffraction patterns in forward scattering have been recorded for several Ge-Si and Ag-Au NWs using a two-dimensional (2D) Maxipix detector.
Coherent scattering signal from Ge-Si nanowires was not strong enough to allow performing 2D ptychography or tomography of a single isolated NW. In contrast, we succeeded to measure the forward scattering signal of isolated Ag-Au NWs for different tomographic orientations by rotating the sample

azimuth angle, θ, over a range of 55° (see Figs. 2**a**-**b**). Fringes are observed along the streaks originating from the facets of the NWs. These patterns do not always show clear evidence of a beat-frenquency generated by the core-shell structure. A "*pre-selection*" of the nanowires should be performed for next experiments to insure their core-shell structure. A nanowire has been characterized by 2D ptychographic measurements in forward scattering geometry. The (002)Au Bragg peak of an isolated Ag-Au nanowire has also been measured (see Fig. 2**c**). The Bragg peak is quite complex, probably due to the presence of defects, and reconstruction may turn out complicated in Bragg geometry.

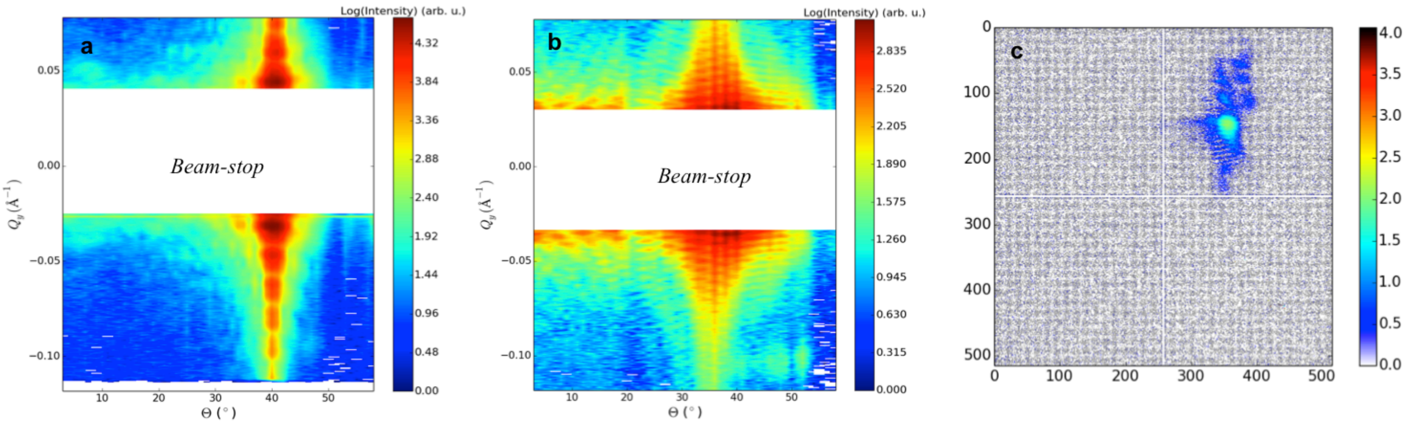

Fig. 2: (**a**)-(**b**) Forward scattering patterns of isolated Ag-Au NWs for different tomographic orientations. (**c**) Maxipix image of the (002)Au Bragg peak of one Ag-Au NW.

Moreover, we performed 2D ptychographic measurements on a Siemens star in order to characterize accurately the beam size. A first estimation of the beam size gives 200 (V) x 210 (H) nm^2. As previous experiments revealed that too many data were missing (due to the size of the beam stop) for the reconstruction to be possible, we recorded coherent patterns with and without beam stop, placing the camera far from the sample position to overcome saturation problems on the recorded images.

Finally, a preliminary test has been performed on a single Pd NW mounted on a MEMS-based tensile stage [2] (see Fig. 3**a**). Imaging in both forward scattering (Fig. 3**b-c**) and Bragg diffraction (Fig. 3**d**) geometries with the sample is possible. The nanowire could not be located with the fluorescence detector owing to the too high (or too low) Pd-edge value but we succeeded to locate it with X-ray beam. Next experiments on this nanostructure should consider some modifications on the chip, like fluorescence markers to locate the NW, but diffraction results show a high critalline quality of the NW.

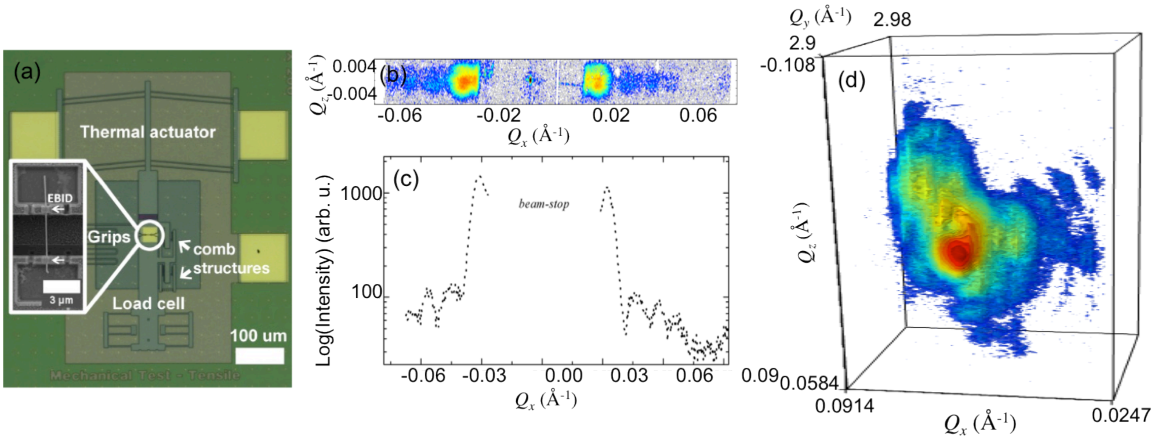

Fig. 3 (**a**) Thermally actuated tensile testing stage onto which a Pd NW is manipulated. The comb features to the side of the sample grips may be used for tracking displacements of the load cell and actuator. Diffraction pattern (**b**) and diffraction line profile (**c**) of the Pd NW in forward direction. (**d**) Pd200 Bragg peak of the Pd NW.

References:

[1] G. Richter, K. Hillerich, D. S. Gianola, R. Mönig, O. Kraft, and C. A. Volkert, Nano Lett. **9**, 3048 (2009).

[2] L. Y. Chen, G. Richter, J. P. Sullivan, and D. S. Gianola, Phys. Rev. Lett. **109**, (2012).