ESRF

Experiment title: In situ anomalous coherent X-ray diffraction of single core-shell nanowires during heating.		**Experiment number:** SI-2521
Beamline: ID03	**Date of experiment:** from: 20/02/2013 to: 26/02/2013	**Date of report:** 1 March 2013
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Report:

Our goal was to study intermixing in Ag-Au core-shell nanostructures by anomalous coherent X-ray diffraction in Bragg geometry. The combination of the two methods should allow for the non-destructive, simultaneous characterization of the strain fields and compositional gradients.

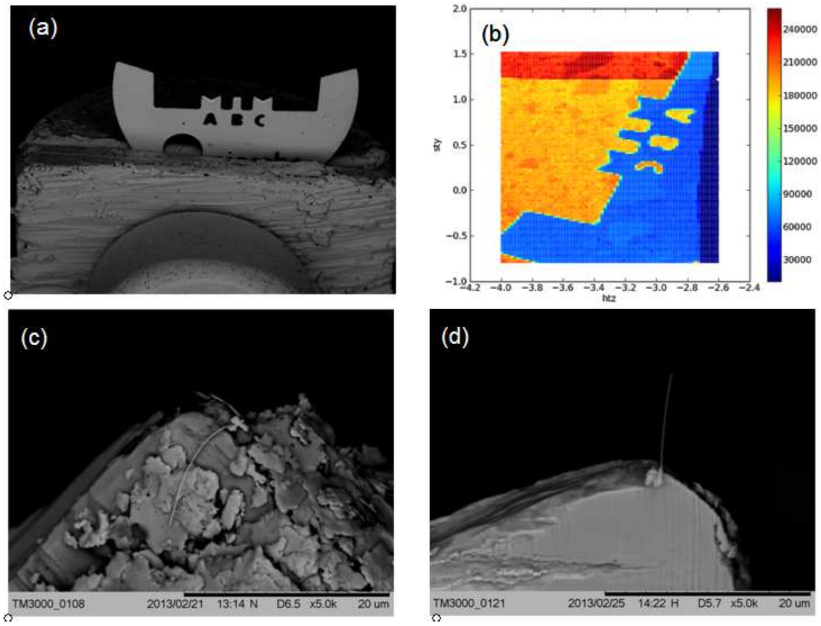

Fig. 1 (a) Scanning electron microscopy (SEM) image of the Mo TEM grid. (b) Image of the TEM grid obtained by scanning it in the direct beam. (c)-(d) SEM images of single nanowires on top of the TEM posts.

We investigated Ag/Au core-shell nanorods prepared by physical vapor deposition [1]. Before the experiment, single Ag/Au nanorods with diameters of ~100-200 nm (Figs. 1(c-d)) were selected from their growth substrate and glued on Mo TEM grids (see Fig. 1a) with Pt in a combined SEM/FIB instrument. The TEM grid was first aligned in the direct beam (see Fig. 1b). Sample preparation is very challenging as well as the handling of the TEM grid. Figure 1c shows a nanowire unintendedly covered by silver paint, which was used to glue the TEM grid to a sample holder during transportation.
For the anomalous measurements, the beam energy was adjusted to the Au L_{III} edge (11.92 keV). The KB system was used for beam focusing. The beam size at the sample position was relatively large (~7 µm (H) x ~10 µm (V)). A multichannel fluorescence detector was employed to locate the nanorod in

the beam using the Au fluorescence signal. The initially used mini UHV chamber with a Be dome (from ID01), set under vacuum for the measurements to avoid beam damage or the formation of a C-overlayer around the nanowire, had to be replaced by another stage (Anton Paar DHS-900 with a polymer dome) from BM32, as the Be dome was unluckily contaminated with Zn (with the same peak position in fluorescence as Au).

To find a 111 reflection (the (111) planes are parallel to the <110> rod axis), the Maxipix detector was placed close to the sample for covering a large solid angle. After successfully aligning the rod in diffraction condition, the detector distance was increased to ~1 m for better resolution. Figure 2 displays two-dimensional sections of a rocking-curve around the 111 Ag reflection of the NW shown in Fig. 1d. The asymmetry of the patterns is caused by an inhomogeneous strain field. The curved pattern, which is observed in the Q_x-Q_z plane, indicates that the NW is bent, which hinders a possible reconstruction using phase retrieval algorithms [2,3]. Finite element simulations will be performed to analyse the curvature of the NW.

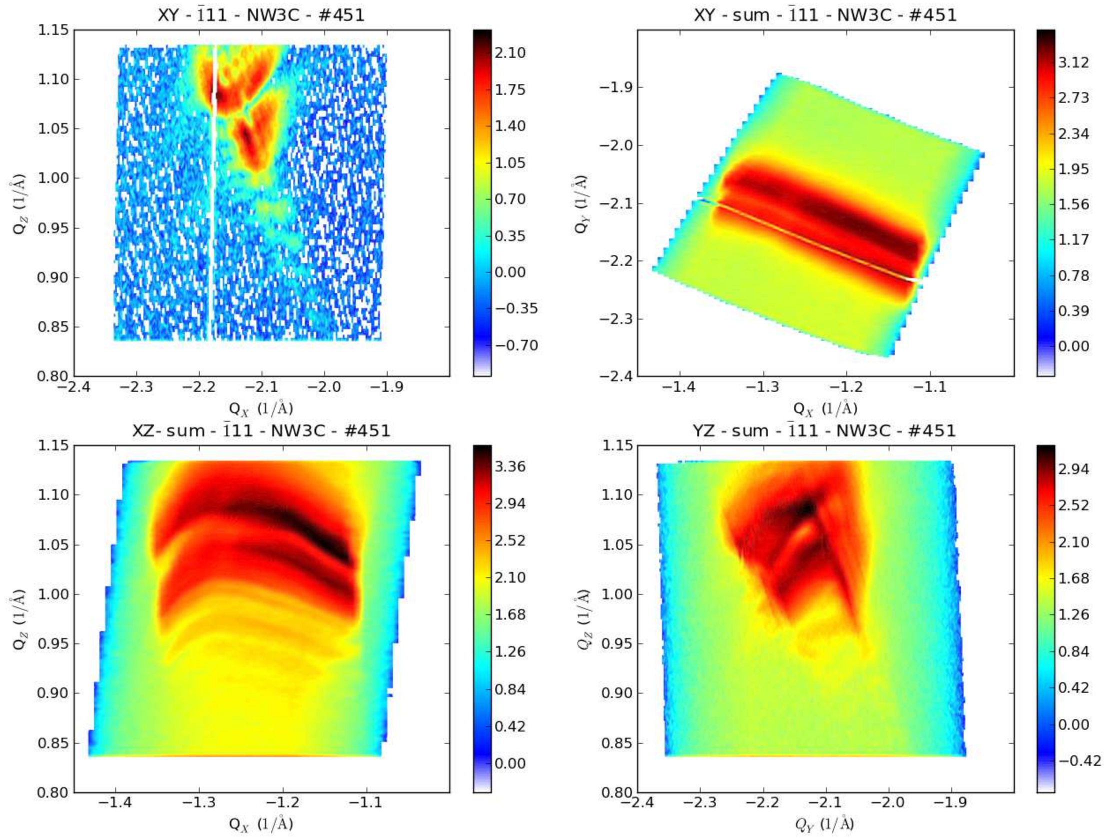

Fig. 2 Two-dimensional cuts of a three-dimensional rocking-curve around the 111 Ag Bragg reflection of a Ag-Au core-shell nanowire (see Fig. 1d).

In conclusion, the diffraction signal of one Ag-Au core-shell nanowire was collected using a coherent and focused beam. The diffraction patterns clearly demonstrate that the NW is curved. The associated large strain field of the curved NW makes difficult its phase retrieval analysis. To overcome this problem, coherent X-ray imaging in forward scattering geometry with a smaller beam can be performed. This will allow probing the evolution of the chemical intermixing in nanostructures with core-shell geometry, getting rid of the effects of strain in the diffraction patterns. Moreover, a smaller X-ray nanobeam would allow to probe only a small part of a nanowire and therefore be insensitive to a bending of the wire induced by the core/shell synthesis.

References:

[1] G. Richter et al., Nano Lett. 9, 3048 (2009).
[2] J. R. Fienup, Appl. Optics 21, 2758 (1982).
[3] R. W. Gerchberg et al., Optik 35, 237 (1972).