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Report:

Our goal was to study intermixing in core-shell nanostructures by coherent diffraction imaging in Bragg diffraction geometry in order to determine shape, composition and strain fields of a single isolated nanowire.



Fig. 1 Measured intensity distribution around the 111 Ge Bragg reflection. The Ge substrate Bragg peak is observed as well as the scattering from the Ge-Si NW and the vertical (along Q_z) CTR.

Ge-Si core-shell nanowires (NWs) grown on a Ge(111) substrate were investigated by coherent x-ray diffraction in Bragg condition using the nano-focused x-ray beam at the ID01 beamline [1]. The Si shell thickness was 50 nm, and the Ge NW core radius was varied from 50 to 200 nm. The required spatial resolution was obtained with a circular Fresnel Zone plate, giving a measured focal size of $130 \times 380 \text{ nm}^2$ (vertical × horizontal). The nano-diffraction experiment was carried out at a beam energy of 8 keV, so that the **111** Ge and Si Bragg peaks were accessible at a scattering angle of 27.44° and 28.63°, respectively. Reciprocal space maps (RSMs) were collected around the **111** Bragg reflection for the different *single* NWs with various core radii. Figure 1 displays the intensity distribution as a function of different components of the momentum transfer. The Ge substrate creates a sharp and intense Bragg peak, superimposed by a vertical streak corresponding to the crystal truncation rod (CTR) originating from the surface of the substrate. Applying phase retrieval algorithms on the recorded data was challenging because of the presence of the CTR and the difficulty to isolate one single NW on the Ge(111) substrate. To overcome these problems, a single Ge-Si core-shell NW was isolated by gluing the it at the top of a Cu pin with a nano-manipulator using focused ion beam (FIB) (see Figure 2a). The nominal diameter of the studied nanowire was 250 nm, and its length was about 7 μ m (see Figure 2b).

The isolated nanowire has been located with the optical microscope of the beamline and then using the quicK-Mapping technique developed at the ID01 beamline [2]. Coherent diffraction patterns around the **111** and **220** Ge reflection of the isolated NW were recorded (see Fig. 3), so that the spatial resolution in direct space, when applying phase retrieval algoritms, should be less than 6 nm (see Fig. 3).



Fig. 2 (a) SEM image showing the isolation process for CDI experiments. (b) SEM image of the studied nanowire.

The fact that there is no measured signal from the Si-shell might be due to its polycristalline nature (in agreement with some transmission electron microscopy measurements), implying that the Si grains scatter incohenrently. The tickness fringes reveal a diameter of 350 nm of the Ge-core, and the splitted Bragg peak (see Fig. 3b) and 2d)) could arise from defects or from bending [3] of the NW.



Fig. 3 Measured intensity distribution around the 111 Ge Bragg reflection (a) and (b), and around the 220 Bragg peak (c) of the Ge-Si core-shell nanowire.

The high-quality data allows to reconstruct the sample even when there are defects in the NW. The first 3D reconstructions of the phase can be seen in Fig. 4; the hexagonal shape of the NW has been succesfully retrieved. The wrapped phase need to be analyzed in more detail for a better understanding.



During the experiment, we also performed ptyhography measurements on the wire and we succesfully test the code developped by F. Mastroprieto *et al.* [4] that allows to simulate and to reconstruct the wavefront.

References:

- [1] S. Labat, M.-I. Richard, G. Beutier, M. Gailhanou, M. Dupraz, M. Verdier, F. Mastropietro, T. W. Cornelius, T. Schülli, J. Eymery, and O. Thomas, ACS Nano (2015).
- [2] G. A. Chahine, M.-I. Richard, R. A. Homs-Regojo, T. N. Tran-Caliste, D. Carbone, V. L. R. Jacques, R. Grifone, P. Boesecke, J. Katzer, I. Costina, H. Djazouli, T. Schroeder, and T. U. Schülli, J. Appl. Crystallogr. 47, 762 (2014).
- [3] X. Shi, J. N. Clark, G. Xiong, X. Huang, R. Harder, and I. K. Robinson, New J. Phys. 15, 123007 (2013).
- [4] F. Mastropietro, D. Carbone, J. Eymery, A. Sentenac, T. H. Metzger, V. Chamard, and V. Favre-Nicolin, Opt. Express 19, 19223 (2011).