ESRF	**Experiment title:** Multi-wavelength Anomalous Dispersion approach applied to Bragg Coherent X-ray Diffraction to unambiguously solve the phase problem.	**Experiment number**: HC-2614
Beamline: ID01	**Date of experiment**: from: February 1st, 2017 to: February 6th 2017	**Date of report**:
Shifts: 18	**Local contact(s)**: Marie-Ingrid Richard	*Received at ESRF:*
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Report:

The samples consist in Ge-Si core-shell nanowires (NWs). They were grown by chemical vapor deposition using vapor-liquid-solid (VLS) phase epitaxy on a Si(111) substrate at the CEA-Grenoble. The Si shell thickness was 10 nm, while the Ge NW core radius was 300 nm. The growth axis is <111>. Figure 1 shows a scanning electron microscopy (SEM) image of one of these NWs.

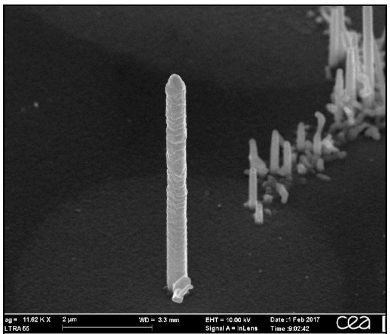

Fig. 1: SEM image of one nanowire

During the experiment, we measured individual as-grown and *ex situ* annealed (at 750°C) Ge-Si core-shell nanowires by Bragg coherent X-ray diffraction imaging (Bragg-CDI). The X-ray beam energy was set to 9 keV. A KB mirror focusing optic was used to focus down the beam to 150 nm (V) x 350 nm (H). Figure 2 displays the measured coherent diffraction patterns (the **111** Ge Bragg reflection was measured) as well as results obtained applying phase retrieval algorithms, *i.e.* the retrieved intensity, retrieved density and retrieved phase, for the as-grown (Fig. 2(a)) and annealed (Fig. 2(b)) nanowires.

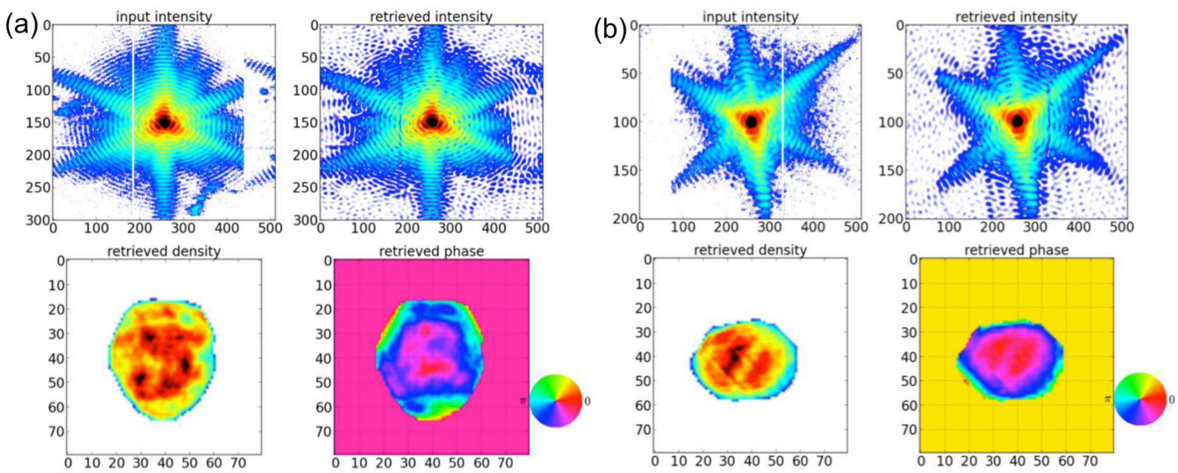

Fig. 2: Input intensity (measured at the **111** Ge Bragg reflection), retrieved intensity, retrieved density and retrieved phase for an as-grown nanowire (a) and a different annealed nanowire (b).

Interestingly, the phase, which is directly proportional to the atomic displacement, shows a three-fold symmetry. It is known that during the growth process, Au diffuses along the nanowires and only decorates three of the six {112} sidewalls facets [1]. This leads to nanowires with a shape in the form of an asymmetric hexagon. Au-decoration on only three of the six facets may also explain the three-fold symmetry observed for the phase, *i.e.* the atomic displacement field.

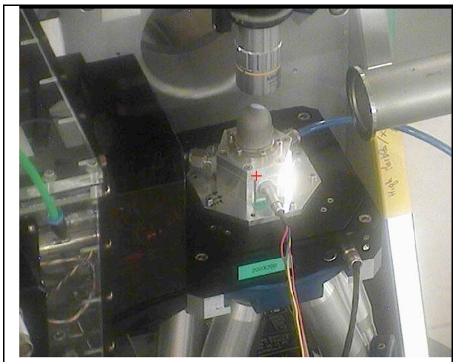

Fig. 3: Photograph of the furnace used for the *in situ* annealing experiment.

To follow atomic diffusion in a single Ge-Si core-shell nanowire, we performed coherent X-ray diffraction using a compact and lightweight (< 300 g) furnace compatible with the use of a nano-focused coherent X-ray beam (see Fig.3). We succeeded to anneal the nanowires at a temperature up to 750°C during 1 hour. Figure 4 displays coherent X-ray measurements and results obtained for two nanowires with different morphologies of their core. Interestingly, intermixing tends to occur depending on the initial shape of the core. We observed no intermixing when the Ge core has a hexagonal faceted shape bounded by {112} facets (see Figs. 1(c-d)), despite Si and Ge are known to be highly miscible. At 800°C, the diffusivity values for Si in Ge are around 10^{-14} cm^2s^{-1} [2], implying that Si atoms can diffuse as far as 60 nm during 1 hour in bulk Ge. In the case of a dodecagonal cross-section, drastic changes in the coherent diffraction patterns are observed after annealing (see Fig. 1(a-b)).

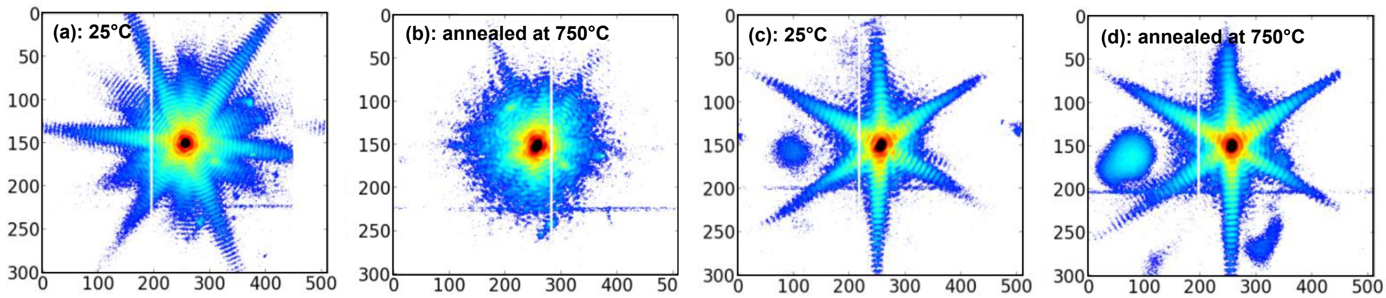

Fig. 4: Two-dimensional reciprocal space maps measured at the **111** Ge Bragg reflection of two Ge-Si core-shell nanowires (diameter of ≈350 nm) measured at room temperature (a)-(c) and at 750°C (b) and (d). Drastic changes are observed for the first nanowire after annealing (see (a)-(c)); while no changes are observed for the second nanowire (c-d).

Due to numerous occurences of beam lost during the experiment (see Fig. 5) , we did not have time to successfully perform anomalous coherent X-ray diffraction and look at different core shapes and sizes. These preliminary results raise, however, very interesting questions on the influence of the nanowire initial shape on the core-shell interdiffusion. Further experiments are clearly needed to confirm these findings and in particular anomalous coherent diffraction at the Ge K-edge could help resolve the concentration profile at the core-shell interface.

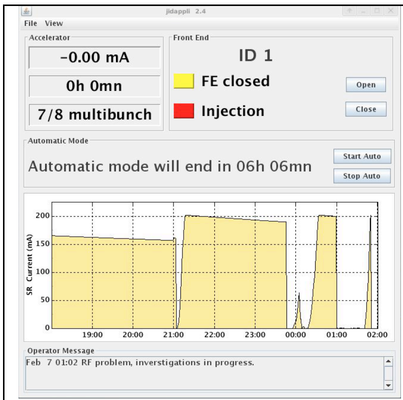

Fig. 5: Synchrotron Ring Current

References:

[1] L. Vincent, R. Boukhicha, C. Gardès, C. Renard, V. Yam, F. Fossard, G. Patriarche, and D. Bouchier, J. Mater. Sci. **47**, 1609 (2012).

[2] M. Gavelle, E. M. Bazizi, E. Scheid, P. F. Fazzini, F. Cristiano, C. Armand, W. Lerch, S. Paul, Y. Campidelli, and A. Halimaoui, J. Appl. Phys. **104**, 113524 (2008).