ESRF	**Experiment title:** Tomographic coherent X-ray imaging of Ge-Si core-shell nanowires	**Experiment number**: HC-3444
Beamline: ID10	**Date of experiment**: from: October 19th, 2017 to: October 24th, 2017	**Date of report**: 23/03/2018
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Report:

The aim of the experiment was to study intermixing in core-shell Ge-Si nanostructures. Diffusion at the nanoscale is expected to be very different from what it is in the bulk because of the vicinity of surfaces, which may act as sinks for point defects. Considering the importance of core-shell nanostructures for various applications it is crucial to understand and characterize atomic transport in these objects.

We have studied single Ge-Si core-shell nanowires (NWs) by tomographic full-field coherent diffractive imaging at the ID10 beamline of ESRF. Ge-Si NWs with a Ge core and a Si shell were grown by chemical vapor deposition using vapor-liquid-solid phase epitaxy at CEA-Grenoble. The Si shell thickness was either 50 nm or 100 nm, and the Ge NW core radius varied from 100 to 200 nm. Before the experiment, single NWs with a length of 2-3 µm were selected from their growth substrate and deposited on a Si_3N_4 membrane. The core-shell nanostructures was then imaged by phasing a diffraction volume measured at 8.1 keV photon energy. The beam was focused by two Be compound refractive lenses of radius 200 µm. The beam defining roller-blade slits of 10 µm x10 µm opening placed 500 mm from the sample ensured the coherent illumination of the isolated core-shell nanowires. The two-dimensional (2D) MAXIPIX detector (512 x 512 pixels with a pixel size of 55 µm) was positioned at 2.4 m from the sample. This leads to a resolution in reciprocal space of $\delta q = (2\pi/\lambda)(55 \times 10^{-6}) = 2.26\text{x}10^{-4}$ Å^{-1}. This implies a voxel size of $2\pi/(512 \times \delta q)$=5.4 nm (smaller than the thickness of the shell, which is 50 or 100 nm) and the maximum size that can be retrieved, taking into account an oversampling of 2, is $2\pi/(2 \times \delta q) = 1.4$ µm.

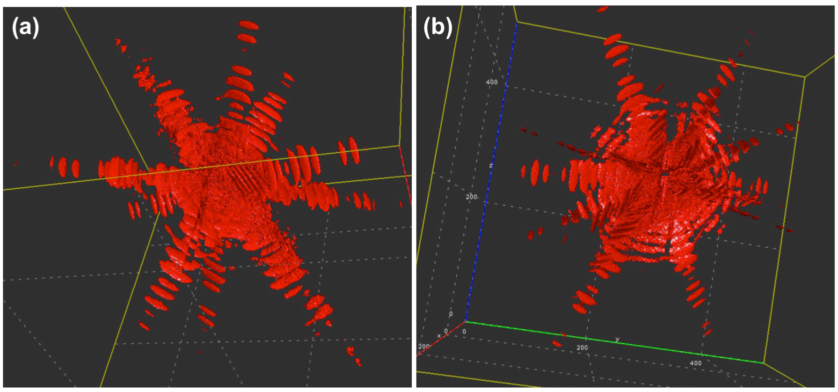

Figure 1: Three-dimensional full-field coherent diffractive imaging of two different Ge-Si core-shell nanowires

For five different samples (pristine and *ex situ* annealed), we have recored 800 diffraction patterns spanning approximatively 160° (-82° to 78°), as illustrated in Fig. 1, which demonstrates that single isolated nano-sized objects can be successfully measured by tomographic coherent X-ray imaging at the ID10 beamline. Thickness fringes from the NW diameter and beat frequencies from core/shell interferences are well observed.

The good contrast between Si (Z_{Si} = 14) and Ge (Z_{Ge} = 32) should allow reconstruction of both the core and shell of the NWs. Tomographic coherent diffractive imaging allowed to quantitatively reconstruct the electron density of the bi-material nanowires by iterative schemes before and after annealing.

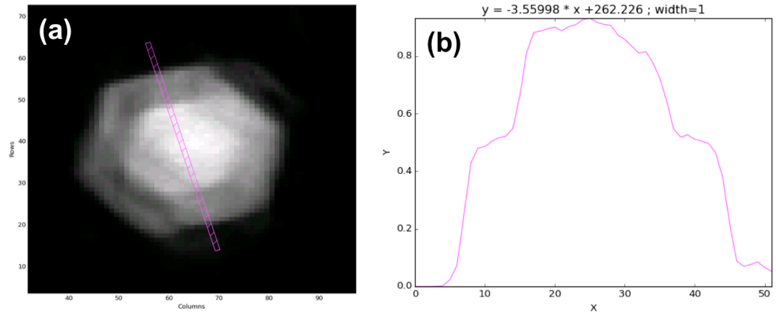

Figure 2: (a) Reconstructed electron density of a Ge-Si core-shell nanowire ex situ annealed at 700°C. (b) One-dimesnional cut of its electron density.

Then, two Ge-Si core-shell nanowires have been *ex situ* annealed at four different temperatures of 700°C, 750°C, 800°C and 850°C using the furnace available at the Sample Environment Laboratory of ESRF. For each temperature, the annealing time was 1 hour. After annealing, the samples were immediately quenched to room temperature. The NWs dit not move on their supporting membrane during the annealing process. It was straightforward to find back the nanowires after annealing. Figure 3 displays cross-sections of the coherent scattered signal of a core-shell nanowire (200 nm Ge core and 100 nm Si shell). Streaks coming from the hexagonal shape of the nanowire can be observed as well as beat frequencies.

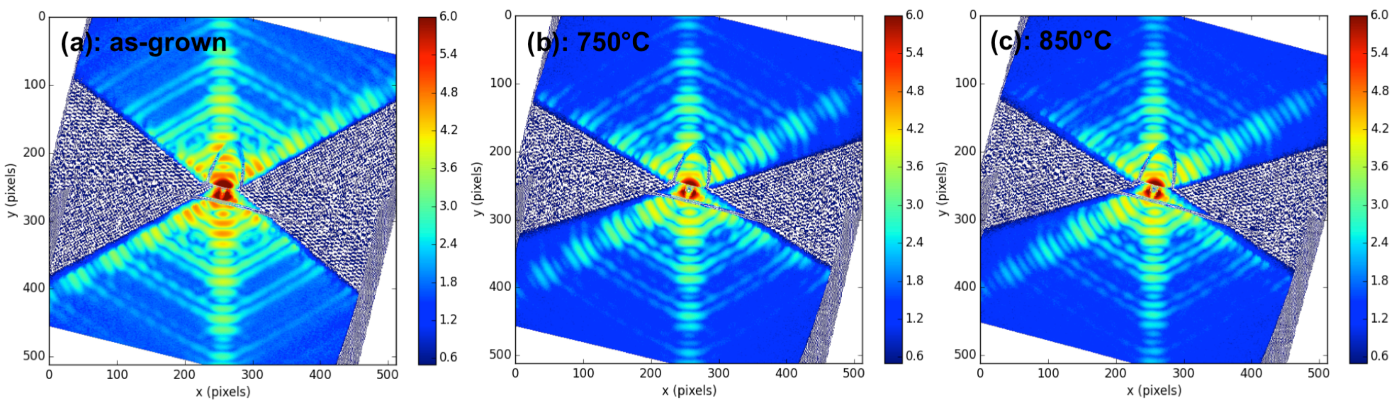

Figure 3: Cross-sections of the coherent scattered signal of a core-shell nanowire (200 nm Ge core and 100 nm Si shell) (a) as-grown and after ex situ annealing at 750°C and 850°C.

At 800°C, the diffusivity value for Si in Ge is around 10^{-14} cm^2s^{-1} [1], implying that Si atoms can diffuse as far as 60 nm during 1 hour in Ge bulk. The diffraction patterns show weak evolutions after annealing at 750°C or 850°C. Beat frequencies are always observed at 750°C or 850°C. Our results demonstrate an absence of intermixing at 850°C contrary to bi-material bulk or thin film.

References

[1] 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).