ESRF	**Experiment title:** **Coherent microdiffraction on single semiconductor nanowires.**	**Experiment number**: HS3223
Beamline:	**Date of experiment**: from: 18/04/2007 to: 24/04/2007	**Date of report**: 31/08/2007
Shifts:	**Local contact(s)**: Oier Bikondoa	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Joël Eymery (*)	CEA Grenoble, DRFMC/SP2M
Vincent Favre-Nicolin (*)	CEA Grenoble, DRFMC/SP2M & Université Joseph Fourier
Robert Koester (*)	CEA Grenoble, DRFMC/SP2M

Abstract:

We have carried out X-ray coherent scattering measurements on single nanowires (NWs), on the ID01 beamline. The 3D intensity collected has been successfully used to obtain the real-space structure of the wires, for the first time for single nanowires with a diameter less than 100 nm.

Experimental setup:

For this experiment we used NWs that were randomly dispersed on the surface of a substrate, either naturally (for samples not grown by epitaxy), or by collecting the NWs on a pure silicon substrate. The sample was then put on a goniometer head which allowed translations with a ~2µm precision. We then translated the surface of the sample in the beam (8x10 μm^2 at E=10keV, focused using Be Compound Refractive Lenses (CRL)), until one NW passed through the beam with the correct orientation to diffract. The 2D detector (a direct-illumination CCD was used for a better resolution) was set at the Bragg angle corresponding to one reflection of the NW's crystal structure. In order to collect the 3D scattered data, we rotated the diffracting wire around one axis of the goniometer (typically of ± 1°), collecting images every 0.01-0.05° step.

The individual images are then aggregated to form a 3D array of scattered intensity and then interpolated into a 3D array with regular (q_x,q_y,q_z) coordinates in $Å^{-1}$.

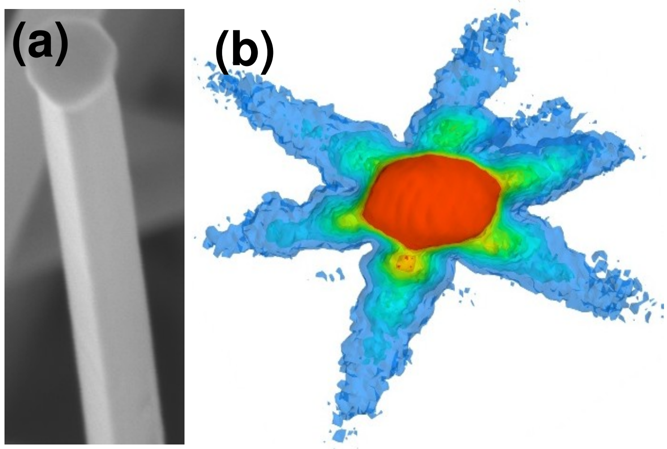

Figure 1: (a) a GaP wire with a triangular section and (b) the corresponding collected diffracted intensity. Each facet of the wire produces a streak on both sides so that the diffraction appears with an hexagonal symmetry. Note the difference in the scattered intensity distribution with respect to an hexagonal wire (see fig. 2)

We used a variety of samples (GaP, GaN, Si, Si/Ge) and obtained good results for the wires which had the smallest number of stacking faults: GaP (fig.1) and Si (fig.2).

The collected images exhibited inteference fringes (due to the facets of the wires) up to the 3rd order. The limited range of these oscillations is due the small size of the wire as well as the weak scattering power from the element of our NW (many studies of coherent diffraction on single objects have been conducted on gold or tungsten objects, which are much stronger scatterers). This range was nevertheless sufficient to obtain a real-space image of the wires.

Data analysis:

The analysis is still ongoing but we have already successfully “inverted” the 3D scattered intensity.

Using coherent diffraction on single objects ensure that the 3D scattered signal is directly the Fourier transform of the object's electronic density (=its shape, for homogeneous objects).

However only the intensity is collected so that the phase of the scattered amplitude is lost and therefore no direct inverse Fourier transform can be performed.

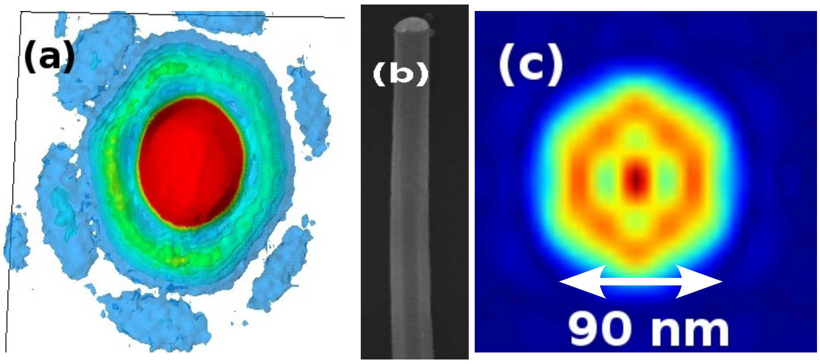

Figure 2: (a) 3D coherent diffraction image of (b) a single Si nanowire. The hexagonal symmetry of the wire is visible in the 3D diffraction image. Once the phase of the scattered intensity is recovered [6,7] the shape (c) of the nano-wire can be recovered, with a size less than 100 nm and a resolution of 5 to 10 nm.

This is a long-standing problem in crystallography, and can be solved in the case of coherent diffraction using oversampling (e.g. see [1-3]). In order to achieve this we used the now well-known “Fourier recycling” algorithms [1-4]:

1. affect a random phase to each point of the 3D data, its amplitude being the square root of the collected intensity
2. calculate the FT of the resulting array – if the phases are correct we obtain then the real electronic density of the object
3. apply “density modification” using real-space constraints: (i) the electronic density must be real and positive and (ii) only part of the 3D real space (the “support”) should be non-null. All points outside the support or with non-real positive are modified (see [1-3]) using a choice of algorithms [4]
4. calculate the FT of the modified object and associate the new set of phases with the observed scattered amplitudes. Go back to step (2)

We successfully used this method to invert the structure of hexagonal silicon wires – the inverted cross-section of the wire is shown in fig. 2c). The size of the wire is estimated to ~90nm , with a resolution around 5nm. The density inside the wire is not perfectly constant, which is due to the truncation of the data to the 3rd fringes. The hexagonal shape is nevertheless clearly visible.

Future work:

Only a preliminary analysis has been presented here. We are now working to also invert the 3D data from the triangular GaP wire, and the resolution of the data indicates that it will also be possible with a similar resolution of about 5nm. Other data involving several wires or broken wires are also under investigation.

Although the results obtained are very good (we believe the first successful coherent diffraction and inversion for a single nanowire less than 100nm), the experiment would have benefited from a more intense and more coherent beam. It is known that vibrations in the optics have limited the size and coherence of the beam. This lack of coherence could be seen when studying InAs/InP wires, for which stacking faults normally induce a “speckle” pattern instead of a Bragg peak, which was not visible during our experiment.

Due to the upgrade of the ID01 optics during the summer 2007, we expect to obtain a much more stable and coherent beam for new experiments, allowing to measure coherently scattered intensity to a higher resolution and with more intensity.

References:

[1] Robinson IK, Vartanyants IA, Williams GJ, et al. *Phys. Rev. Lett.* **87** (2001), 195505

[2] I. A. Vartaniants, I. K. Robinson, J. Onken, M. A. Pfeifer, G. J. Williams, F. Pfeiffer, T. H. Metzger, Z. Zhong and G. Bauer, *Phys. Rev. B* ***71***(2005) 245302

[3] Pfeifer MA, Williams GJ, Vartanyants IA, et al. *Nature* **442** (7098): 63-66 JUL 6 2006

[4] Wu J.S. and Spence J.C.H., *Acta Cryst.* **A61**(2005), 194-200