



**Experiment title: Catalytic growth of confined silicon nanowires investigated in-situ by grazing incidence diffraction**

**Experiment number:**  
SI-2013

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**Report:**

**Aim of the experiment :**

We have investigate the structural behaviour of confined catalytic silicon nanowires (SiNW). These SiNW were grown in a Vapor Liquid Solid (VLS) catalytic mode in a Chemical Vapor Deposition (CVD) reactor with colloidal gold used as a catalyst. Preliminary to the confined growth in a alumina template, we have performed measurements first, on nanoporous alumina template alone, and next on silicon nanowires (SiNW) alone. Both were deposited on a <111> orientated silicon substrate. The aim of this experiment is to investigate the morphology of the alumina template as well as the morphology of SiNW with a constant diameter value.

**Measurements :**

We have performed Grazing Incidence Small Angle X ray Scattering (GISAXS) and Grazing Incidence X ray Diffraction (GIXD) measurements on SiNW on a silicon substrate. The selected energy was  $E = 11.0$  keV corresponding to a wavelength  $\lambda = 0.112713$  nm. At this energy, the critical angle of alumina is  $\alpha_c = 0.13^\circ$  and for silicon  $\alpha_c = 0.16^\circ$ . The experimental set-up was a multi-circles goniometer, the detector was a linear Position Sensitive Detector for GIXD and a 2D CCD camera for GISAXS. For alumina template, two samples were investigated with a cell parameter (center to center pore distance) of about 92nm. For SiNW, three samples were investigated of three different diameter (50, 100 and 200nm).

**Results :**

We have performed GISAXS observation of nanoporous alumina (Figure 1). As we can see the GISAXS image reveal periodic scattering coming from the porous structure. The scattering particles are cylindrical

pores, embedded in a amorphous template of Alumina. Considering the Babinet's principle it is possible to observe interferences fringes coming from the porous structure. A cross section, corresponding to the structure factor is plotted on Figure 1b. Briefly this observation allow us to measure the distance between two adjacent pores (distance between two peaks), the size of the domain of the hexagonal structure (width of a peak). We can also estimate the diameter of the pore with the form factor which envelop this function. Thus, we estimate the following values: pore diameter  $\approx 80$  nm, cell size  $\approx 93$ nm, and domain size  $\approx 2.5\mu\text{m}$ .

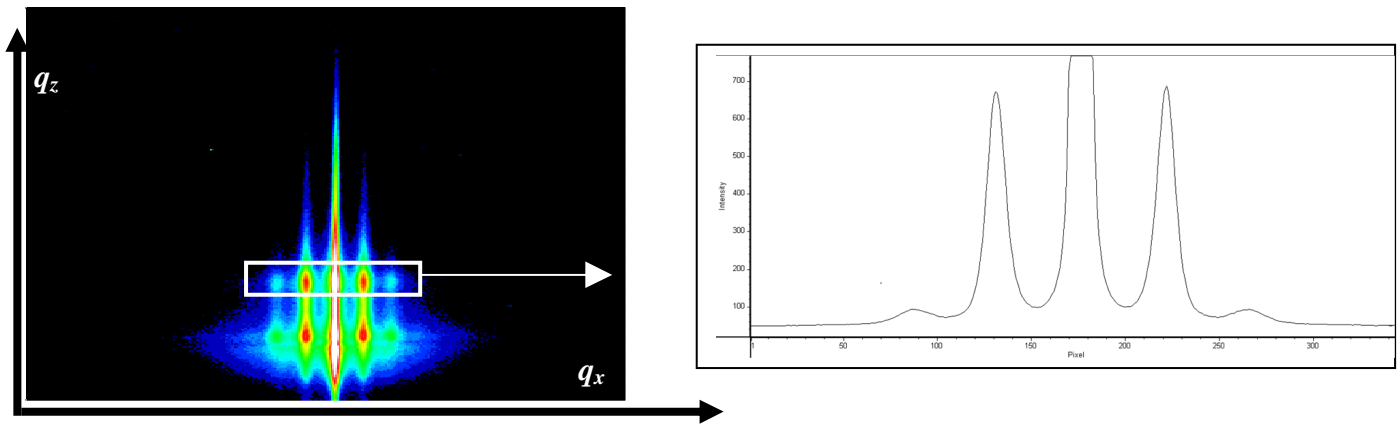


Figure 1

We present on Figure 2 GISAXS from SiNW grown with colloidal gold used as a catalyst: Figure 2 (a) correspond to SiNW with a diameter of 50 nm and Figure 2b with a diameter of 100 nm respectively. The incident angle was  $0.30^\circ$

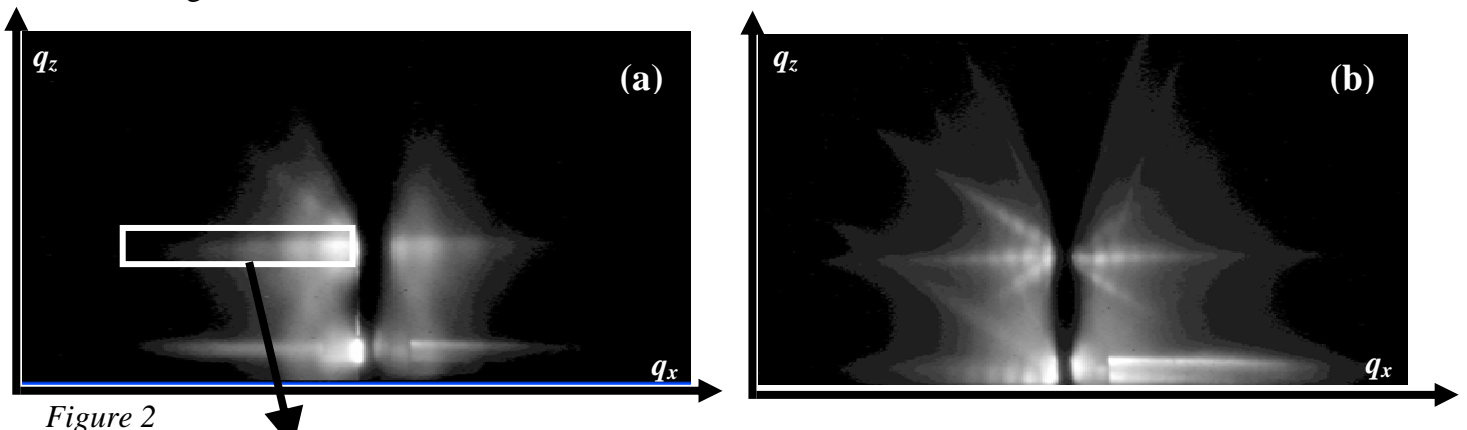
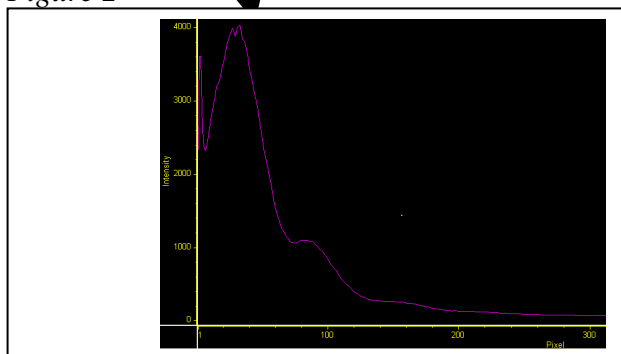


Figure 2



For all samples we observed scattered streaks coming from the side of the silicon nanowires. We also observed periodic interferences fringes produce by the very cylindrical wires with a quasi perfect constant diameter. The length of the wires is few  $\mu\text{m}$ . The cross section of Figure 1(a) present these periodic modulations and correspond to the form factor of the structures.

From the distance between two adjacent peaks we measure the diameter of the wires. We measure  $D \approx 47$ nm,  $D \approx 95$ nm,

$D \approx 194$ nm, respectively for Silicon nanowires called "50, 100 and 200nm".

## Conclusion

In conclusion, the form and structure factor have been measured from these experiments and they will be analyzed further. The regularity of the nanoporous alumina template, and the regularity of the diameter of the SiNW allow to accurately measure the morphology of such systems. We have also performed GIXD and GISAXS measurements by varying the *in-plane* angle in order to investigate this structure along  $120^\circ$ .