ESRF	**Experiment title:** Structural x-ray investigation during in-situ annealing of silicon nanowires encapsulated in a porous alumina template	**Experiment number**: 32 03 662
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Report:

Aim of the experiment :

The aim of this experiment is to perform a structural characterization of catalytic VLS growth silicon nanowires [1] during various stages of the sample preparation. This is supposed to yield important the knowledge about the growth characteristics like epitaxial and crystalline properties as texture, orientation, lattice parameter, strain, and shape/faceting of the silicon nanowires.

Measurements :

We have performed Grazing Incidence X-Ray Diffraction (GIXRD) using a position sensitive detector (PSD) during the annealing of the sample. The porous alumina structure was investigated as well as the structure of the nanowires. Reciprocal space maps were performed at the early nucleation stages and also for fully-grown silicon nanowires on bulk silicon substrate. These nanowires were also investigated by Grazing Incidence Small Angle X-ray Scattering (GISAXS) using a 1152x1242 pixels low-noise 16 bits CCD detector from Princeton.

Results :

We report the results of X-ray experiments on silicon nanowires grown by VLS method on (111) oriented silicon substrate. Gold nanoparticles were used as catalysts. The figure 1 shows a GIXRD map of the scattered intensity around the (220) peak of silicon. A peak is present at the center of the figure, which indicates the single crystal character of nanowires. Their hexagonal section is here confirmed because the six sides of these nanowires produce the six trails around the peak. As this map is the scattered intensity coming from an entire population of nanowires, we are also sure that these nanowires are all oriented in the same direction; otherwise the peak would have a usual round shape. This ensures nanowires are in epitaxy with the substrate. The angle between two trails around the peak is 60° and the directions of the six sides of the nanowires can be deduced from the directions of these trails on the map. So it appears that the directions of the six sides of the nanowires are (112), (121), (211), (112), (121) and (211). Similarly, the directions of the edges are (110) and the five other equivalent directions. These results are well consistent with previous

electronic microscopy observations [2]. The same experiment was made on another sample which was realized exactly in the same way except for the duration of the growth which was very short in order to obtain short nanowires at the very beginning of their growth. The similar map (not shown) had a round shape and we can thus say the sides were either not yet formed or too small to be visible on the map. This confirms the unusual shape of the peak observed from the sides of the nanowires.

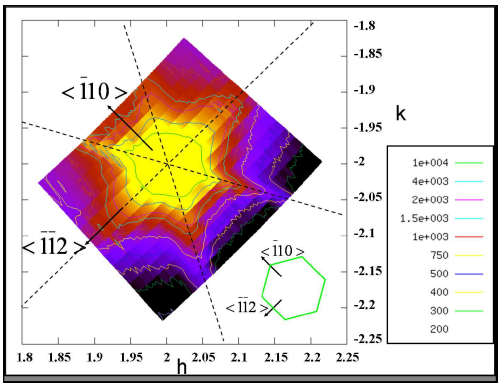

Figure 1: GIXRD map of scattered intensity (arb. units) around (220) peak of silicon. h and k are the reciprocal space coordinates. The unusual shape of the peak's surroundings is due to the shape of the scattering objects: The nanowires. The intensity peak present six shoulderings regularly spread each 60° around it. These shoulderings reflect the six sides of the hexagonal-sectioned nanowires and thus allow the determination of their directions.

Grazing Incidence Small-Angle X-ray Scattering experiments have highlighted a fine saw-tooth faceting of the sides of nanowires (Figure 2).

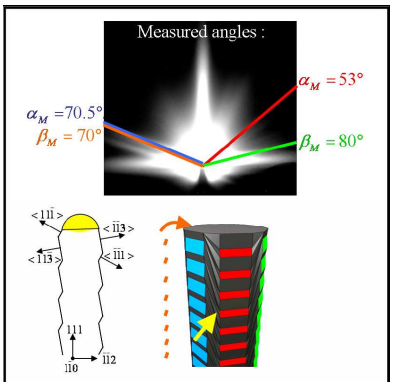

Figure 2: Origin of the dissymmetry in GISAXS images: Only upward facets scatter light. Each kind of facet (either the 'small' ones (S) or the 'large' ones (L)) produce rods at different angles on the right and left of the GISAXS image; Depending on the in-plane angle they make with the incident beam and thus whether a correction is needed or not.

This fine saw-tooth faceting turned out to appear alternatively upward and downward on each side of the nanowires, which shows the trigonal nature of nanowires. Moreover, a few nanowires (especially big ones, with diameter above 200 nm) present six additional faces truncating the edge of the usually hexagonal-sectionned nanowires. These additionnal faces present also a saw-tooth faceting which is tilted with respect to the horizontal and seem to be present only around the top of nanowires. This could indicate their relative stability towards the usual saw-tooth facets of hexagonal nanowires is dependent on the gold coverage of the surface of the nanowire. The crystallographic orientations of some of all these facets have been determined whereas some of them are not so clear yet.

Conclusion

In conclusion, we have investigated morphological and structural properties of silicon nanowires catalytic grown by CVD/VLS with a good epitaxy on a (111) oriented silicon substrate using X-Ray grazing incidence Diffraction. The nanowires have proven to be in epitaxy on the substrate, grow in the (111) direction and have a hexagonal section with sides being in (112) directions (in average over the whole sample). GISAXS results give evidence of a saw-tooth faceting. We have determined the direction of these little saw-tooth facets (111) and (113) and it seems to us to appear on every side of the nanowires (when their diameter is over 200 nm at least) and not only on one side in two.

References

[1] R. S. Wagner and W. C. Ellis, Appl. Phys. Lett. 4, 89 (1964).
[2] M. Den Hertog, unpublished results.
[3] F.M. Ross, J. Tersoff and M.C. Reuter, Phys. Rev. Lett. 95 (2005) 146104.