



Experiment title: Thermal history dependence of the solid-liquid transition in a single AuSi eutectic nano-droplet.		Experiment number: SI-2024
Beamline: ID01	Date of experiment: from: 28/04/2010 to: 04/05/2010	Date of report: 10/03/2011
Shifts: 18	Local contact(s): Tobias U. Schülli	<i>Received at ESRF:</i>
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Report:

Semiconductor (SC) nanowires (NWs) are produced via the vapour-liquid-solid (VLS) mechanism [1] in which a low temperature semiconductor/metal eutectic is used to enable their nucleation and their growth. The base of this mechanism is the liquid state of these alloys which present in general a deep eutectic point that confers catalytic properties even at low temperature. As the state of the catalyst is the base to control the growth parameters, it is of prime importance to investigate in details the mechanism and depict at the atomistic scale this intricate solid-liquid-solid transition. For that sake, we intensively investigated deposits of Au on Si substrates which compose the most used metal-semiconductor eutectic ($\text{Au}_{81}\text{Si}_{19}$) catalyst for the VLS growth of Si NWs.

We employed *in situ* Grazing Incidence X-ray Scattering (GIXS) in ultra-high vacuum (UHV) (at BM32) and reported that the supercooling is enhanced in presence of the Au-(6x6) surface reconstruction [2]. This surface structure displays pentagonal [3] sites which may induce icosahedral short range order in the liquid droplet and thus prevent crystallization. One of the fundamental questions arising from this tricky system is whether or not the reconstruction is present at the interface (i.e between the Si substrate and the droplet). This question can be solved using a nanometre-sized beam which allows probing only one droplet at a time instead of averaging over a wide area of the sample and therefore, selecting the signal coming from the bare substrate or from the Au/Si droplet to substrate interface. Then, positioning the sample and the detector into condition of (6x6) diffraction and scanning the surface with the synchrotron beam by a translation of the sample in the real space, it would be possible to record the intensity variation (or not) coming from the (6x6) signal and see if this latter is uniformly distributed (or not) on the surface.

The first step for this experiment on ID01 was to characterize the spot size at the focus point. The sample holder was replaced by a gold cross composed of two gold wires. This latter was then placed at several positions along the beam direction (y) and for each position, two scans were performed: one was a translation of the cross along x until the beam intensity was cut by the edges, the other was the same but along done along z. The FWHM of the derivate of each scan gives the size of the spot in the direction of the measurements; the results are plotted on Figure 1(a). It shows, that the focus is at $y \approx 13\text{mm}$ (with arbitrary

origin) and that its size is about $1.7 \times 1 \mu\text{m}^2$. Note here that assuming the droplet size to be around 100 nm, the beam size is still too large to clearly achieve the aim of the study. Despite many efforts, we were not able to decrease the spot size more than this value because of external disruptions (temperature variations, mechanical vibrations, etc...) that could not be ride out. In addition, as ID01 is not equipped with an UHV chamber, we had to work in a vacuum that could not be better than 10^{-7} mbar. As a consequence, despite many tries and thermal cycles, we were not able to obtain a Au-(6x6) reconstruction on our samples as this latter needs a particular clean Si surface to form. Therefore, during this experiment, no supercooling phenomena were recorded.

We thus decided to scan the sample surface in Au(222) reflection conditions. The recorded map is $240 \times 240 \mu\text{m}^2$ and is plot on Figure 1(b). The red points corresponding to a detection of high intensity show the location of the gold island. This experiment was perhaps performed a bit too early as there were no hexapod nor UHV chamber on the ID01 beamline at this time. With the expected upgrades that will be established very soon, there is no doubt that this experiment will be possible in a close future.

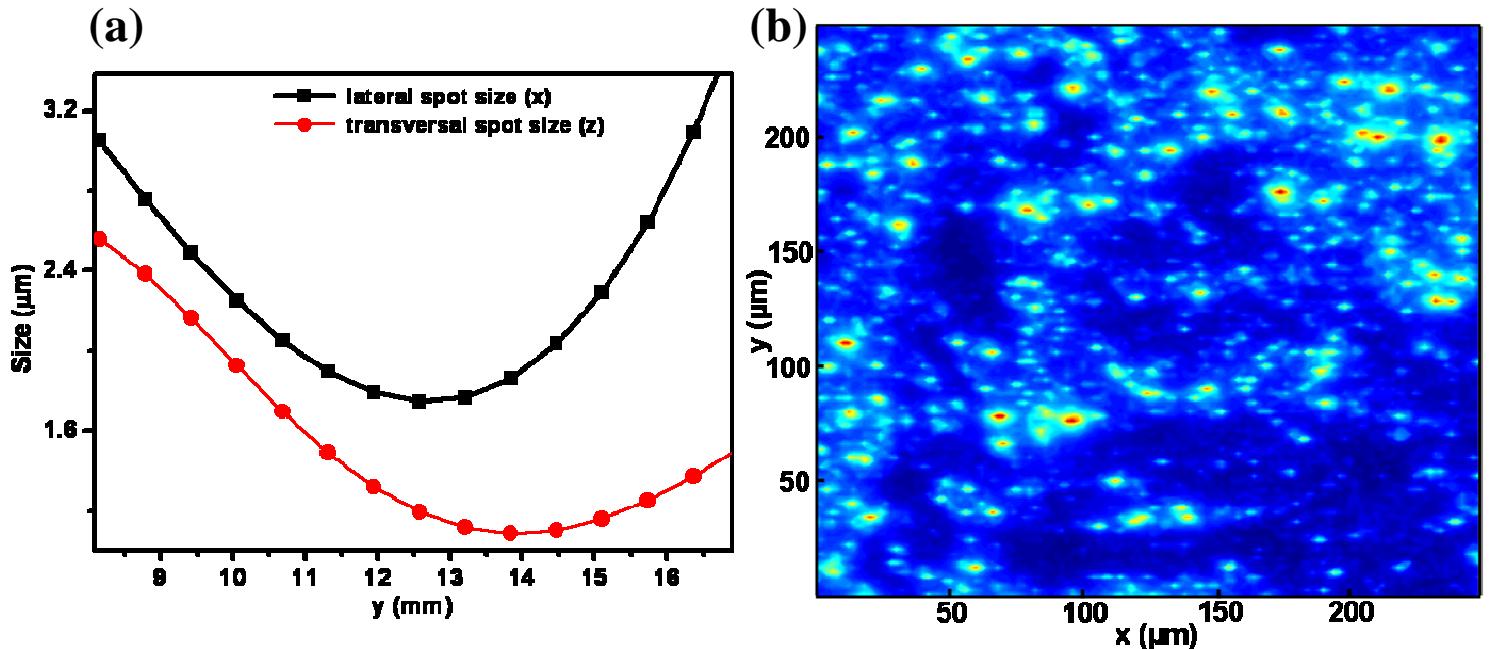


Figure 1 : (a) Variation of the lateral (black/squares) and vertical (red/circle) spot size in function of the position along the beam direction (y). (b) Au(222) reflection intensity map recorded by translating the sample along x for different value of y, the scanned surface is $240 \times 240 \mu\text{m}^2$. Blue, yellow and red colors correspond to low, intermediate and high intensities, respectively. The high intensity spots thus represent the location of the gold islands.

References:

- [1] R. S. Wagner, W. C. Ellis, *Appl. Phys. Lett.* **4**, 89-90 (1964).
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