| | **Experiment title:** Self-organized 2D nanostructures onto vicinal sapphire substrates. A coupled GISAXS and XRD study on D2AM | **Experiment number:** **02-02- 802** |
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| **Beamline:** BM02 | **Date of experiment:** from: 29/03/13 to: 01/04/13 | **Date of report:** September 2013 |
| **Shifts:** **18** | **Local contact(s):** Mireille Maret | *Received at ESRF:* |

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**Objective & expected results** :

The goals of this proposal were (i) to test the new setup of the BM02 beamline allowing coupled GISAXS and XRD measurements (ii) to perform GISAXS measurements in order to determine the shape of 2D networks realized by a high temperature annealing of vicical sapphire substrates by recording 3D reciprocal space maps.

The sapphire substrats studied here were obtained from polished wafers with a miscut angle of 10° from the (001) planes towards the [110] direction.The samples were annealed under oxygen at 1250°C with an annealing duration of 8h, 16h and 32h. We have previoulsly observed during our first experiment made using the old configuration of the D2AM beamline (proposal 02-01-815) that the azimutal angle φ, which correponds to the direction of the incident X-ray beam respect to the step edges, has an important effect on the GISAXS patterns. That's why, a part of the current proposal was devoted to elaboration of an accurate orientation process.

**Results and conclusions of the study**:

The GISAXS experiments were performed at 9,659 keV, with an incidence angle of $\alpha_i \approx 0.23°$ and a sample-to-detector distance of 4910 mm. Before the GISAXS experiments, we used XRD to precisely orientate the sample. The sample was put onto the diffractometer sample holder equipped with a motorized goniometric head. X-ray diffraction of diffracting planes not parallel to the (001) sapphire planes was used to determined the azimutal angle with an accuracy of few thousandth of degree. Here, the azimutal orientation was obtained by realizing the diffraction

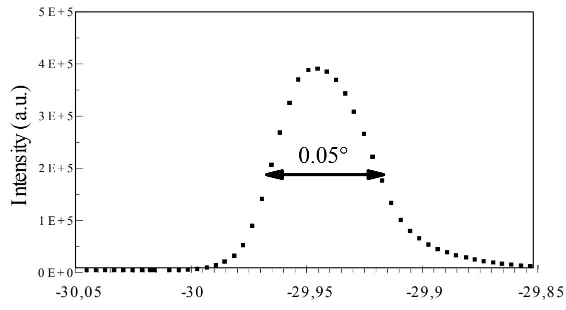
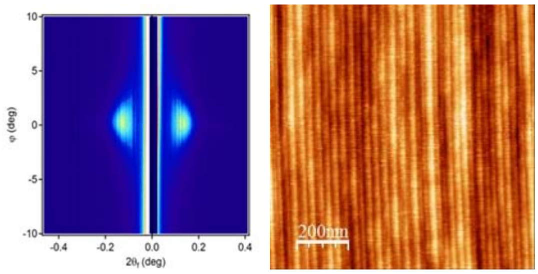


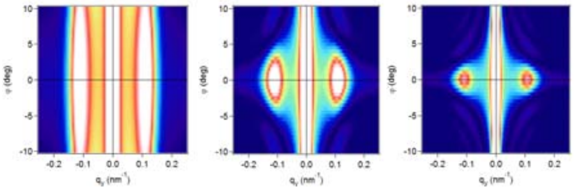

*Fig.1. evolution of the integrated diffracted intensity of the (11 12) planes*

of the asymmetric planes (1 1 12) of the sapphire (see figure 1). Then, once the orientation of the sample is completed, the rotation axis φ and the normal of the mean surface are colinear so whatever the azimutal angle φ is, the incident angle on the surface stay the same. The 3D reciprocal space maps have been recorded by the acquisition of set of ($q_y$,$q_z$) maps, on a 2D detector (XPAD), as a function of the azimutal angle φ with a step of 0.05°.This procedure has been used for all the samples.




*(a) (b)*

***Fig.2. (a) experimental ($q_y$, φ) map and (b) AFM image of the sapphire substrate treated at 1250°C during 8h under oxygen.***

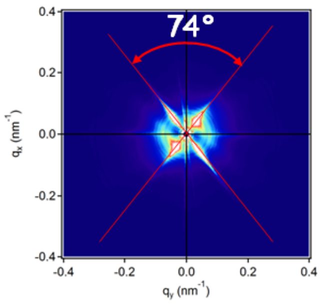


*(a) (b) (c)*

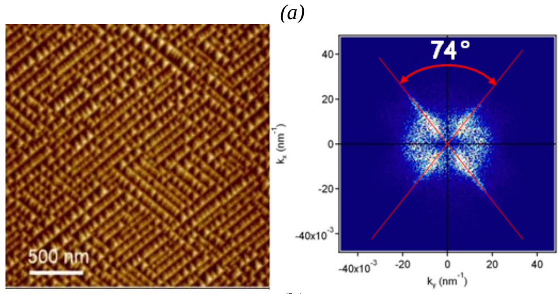
***Fig. 3: calculated ($q_y$, φ) maps for different values of lenght Dx of the steps***
***(a) Dx = 100 nm, (b) Dx = 500 nm and (c) Dx = 1000nm***

The first studied sample was annealed during 8h and presents a 1D organization. The 3D reciprocal space map was recorded for an angular range of 210° with an angular step respect to φ axis equal to 1°. We observed that scattered signal is only present when the azimutal angle is close to 0°, which correspond to the situation where the incident X-ray beam and the step edges are in the same direction. We have thus, the recorded a map close to φ = 0° with a smaller step, this map is presented in the figure 2. Assuming that the surface is made of adjacent prims with a rectangular base and triangular profil, a serie of ($q_y$,φ) maps corresponding to different value of length ot the prims were calculated (see figure 3). Finally comparing with the experiment map, the characteristic size of the prims was determined.

The second sample, annealed 32h, presents an organization in two different directions. For this sample, we realized a 3D reciprocal space map for an agular range of 360° with a step of 0.5° which is presented fig. 4 with the AFM image of the sample and her Fourier transform. On the Fourier transform of the AFM image of this surface, we can clearly observe two distinct directions in the same directions than on the experimental ($q_x$,$q_y$) map. Simulations have helped us to determined the shape of the surface and of the 2D network. It seems that the network is centered rectangular and the scattering objects have a truncated tetraedron shape.




*(a)*




*(b)*

***Fig. 4: (a) experimental ($q_x$,$q_y$) map, (b) AFM image and her Fourier transform obtained on a sapphire substrate treated at 1250°C during 32h under oxygen***

The last studied sample has been annealed during 16h and has a 2D organization. Analyses of the data, in order to determine the shape of the objects and the 2D network, are still in progress for this sample but it seems that the 2D network is different from the 2D network obtained on the sample treated during 32h. This lets us think that there may be an evolution of the 2D network during the thermal treatment.