ESRF	**Experiment title:** Formation of 2D networks onto sapphire vicinal surfaces probed by GISAXS: influence of the azimuthal disorientation	**Experiment number**: 02-02-817
Beamline: BM02	**Date of experiment**: from: 12/06/14 to: 17/06/14	**Date of report**:
Shifts: **15**	**Local contact(s)**: M. Maret	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr David Babonneau[1]*, Pr René Guinebretière[2]*, Ms Caroline Matringe[2]*

[1] Institut PPRIME, Poitiers, France

[2] Laboratoire Science des Procédés Céramiques et Traitement de Surfaces (SPCTS), Limoges, France

Objective & expected results:

This experiment is part of a large study devoted to the self-organization of vicinal surfaces of sapphire through thermal treatment. We have demonstrated previously that according to the duration and the atmosphere of the treatment 1D or 2D lattices can be obtained. The goal of this experiment was to determine the influence of the azimuthal disorientation onto both the kinetic of the formation of the 2D lattices and the shape of this lattice.

In the general framework of the QMAX ANR project, the BM02 beamline has been completely renewed and the measurements realized during this experiment fully used the new configuration of the beamline. The GISAXS measurements were performed at 8 keV, with an incidence angle α_i close to 0.3°. According to a procedure developed last year during the 02-02-802 experiment, the samples were put onto the kappa goniometer and oriented respect to the goniometer axis and the primary beam direction. The scattering signal close to the central point of the reciprocal space, i.e. the GISAXS signal, was recorded using a 2D detector (XPAD) located at 5235 mm from the sample. The 3D reciprocal space maps were recorded by the acquisition of set of (q_y-q_z) maps, on a 2D detector, as a function of the azimuthal angle φ.

Results and conclusions of the study:

The sapphire samples studied here were obtained from polished wafers with a miscut angle of 10° from the (001) planes towards the [110] direction. The influence of the azimuthal angle was checked using 3 different samples having an azimuthal angle equal respectively to 0.5°, 1° or 2° respect to the [110] direction. In each case, samples were annealed in oxygen at 1250°C with an annealing duration of 8h or 32h before the GISAXS measurements.

The first set of samples has been annealed during 8h. Such thermal treatment duration induces on sample without azimuthal the formation of well-ordered 1D organization (see our reports on previous experiments). For each samples, partial 3D reciprocal space maps were recorded with an angular φ step of 0.05° for an angular range of 20° from -10° to +10° respect to the [110]

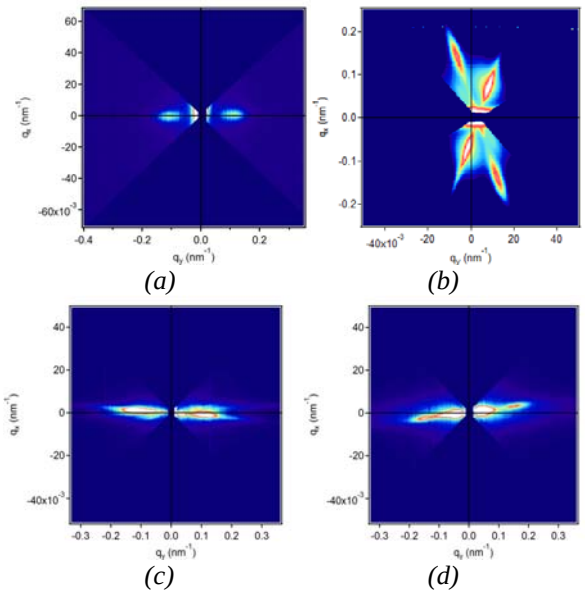

Fig. 1: (q_x-q_y) maps obtained for samples treated 8h with (a) φ = 0°, (b) φ = 0.5°, (c) φ = 1° and (d) φ = 2°

direction. q_x-q_y sections of those maps are reported fig. 1. The presence of 1D ordering is well evidenced for an azimuth equal to 0 (fig. 1a). A very low value of azimuthal angle (0.5°) seems to favor the formation of a 2D lattice. On the contrary, when this azimuthal angle is equal 1° or 2° this 2D arrangement is not observed. Another remarkable thing is the fact that for those two samples, the scattering directions observed on the section reported fig.1c and 1d are no longer aligned with the q_y axis. Moreover, this disorientation δ increases with the azimuthal angle value (δ = 0.4° for φ = 1° and δ = -1.1° for φ = 2°). Looking more carefully on the fig1c and 1d one can see that the diffuse scattering signal presents in each case 2 maxima in both side of the map. This can be more clearly seen on the q_y-q_z map reported fig.2. This observation may be related to a second order effect or more probably to the apparition of a super-period.

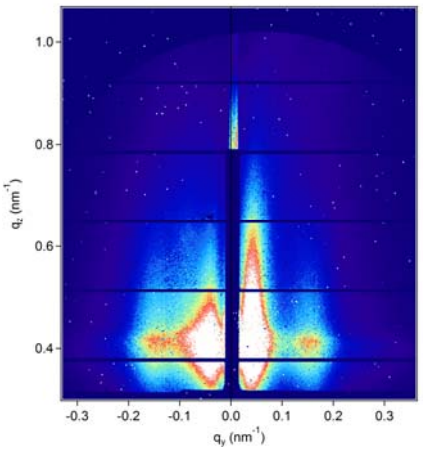

Fig. 2. (q_y-q_z) map obtained at φ = -1.1° for the sample with an azimuthal angle equal to 2° and treated during 8h.

A second set of samples has been studied. Those samples were treated during 32h, which is the thermal treatment leading to a 2D-ordering for samples without any azimuthal angle. q_x-q_y sections of the 3D reciprocal maps are reported fig. 3. The first remark is that clearly the presence of an azimuthal angle does not favor the formation of 2D lattice! Similarly to that we observed for "short" thermal treatment, a small disorientation of the scattering directions is evidenced when φ=1° or φ=2°. This disorientation δ is almost the same than the one measured for the short thermal treatment.

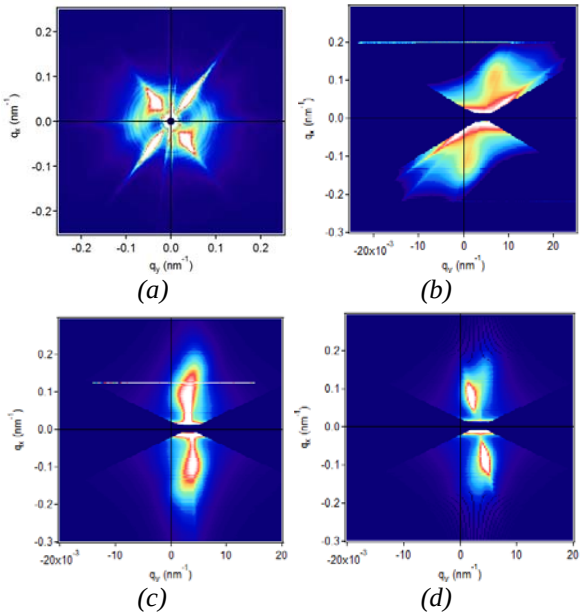

Fig. 3: (q_x-q_y) maps obtained for samples treated 32h with (a) φ = 0°, (b) φ = 0.5°, (c) φ = 1° and (d) φ = 2°

The determination of the actual shape of the scattered objects constituting the ordered surfaces is under progress using the FitGISAXS software. After this new experience we are convinced that the complete understanding of the surface evolution through the diffusion process activated by the thermal treatment requires in-situ GISAXS measurements during the thermal treatment. This could be realized thank to the development of a convenient high temperature set-up which has been built at the BM02 beamline as a part of the QMAX project.