ESRF	**Experiment title:** Etude par diffusion centrale des rayons X sous incidence rasante de l'auto-organisation de surfaces vicinales de saphir	**Experiment number**: 02-01-815
Beamline: BM02	**Date of experiment**: from: 24/09/2010 to: 27/09/2010	**Date of report**: July 2011
Shifts: 9	**Local contact(s)**: Jean-Paul Simon, Mireille Maret	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr David Babonneau*, Mr. Ahmad Fakih*, Pr René Guinebretiere*, Dr Mireille Maret*, Dr Jean-Paul Simon*

Report:

The goal of this proposal was to perform GISAXS measurements in order to determine quantitatively the structural evolution of vicinal surfaces of sapphire subjected to a thermal treatment. The sapphire substrates used in this study were obtained from polished wafers with a miscut angle of 1°, 5°, or 10° from the (0 0 1) planes towards the [1 1 0] direction. Before annealing, the samples were cleaned by immersing in acetone, ethanol and rinsed in extra-pure de-ionised water. Each sample was then placed into a furnace and annealed in air, O_2, or Ar at temperatures ranging from 1000°C to 1500°C, with the annealing duration being varied between 1h and 48h. The results constitute a significant part of the PhD thesis of A. Fakih, which will be defended at the end of 2011. This project is supported by the ANR P3N program through the QMAX project (2010-2014).

The GISAXS experiments were performed on the D2AM beamline at 10 keV, with an incidence angle $\alpha_i \approx 0.25°$ and a sample-to-detector distance of 2620 mm. It should be also noted that, for some selected samples, series of GISAXS patterns were possibly collected at different azimuthal angles φ (i.e., rotating the sample from φ = -10° to φ = 100°) in order to obtain a full 3D intensity map of the nanostructured surface. For this purpose, the samples were put onto the 7-circle diffraction goniometer even though the SAXS 2D detector was located onto the granite SAXS bench. Such a configuration allows to achieve an accurate sample positionning and to reach long sample to detector distances lying between 2 and 4 meters.
As a typical example, Fig. 1 shows the effect of the treatment duration on the 2D GISAXS patterns of vicinal surfaces of sapphire with a miscut angle of 10° subjected to annealing at 1250°C in O_2. The formation of self-organized steps and terraces is confirmed by the presence of intense and sharp vertical streaks at φ = 0° (Figs. 1a-1c). The quantitative analysis of the data, which is performed in the framework of the distorted wave-Born approximation assuming an asymmetric sawtooth surface profile, shows unambiguously that the period, the height, the degree of order, and the asymmetry of the surface profile increase with the treatment duration. In addition, while only unidimensional nanostructures are formed after 8h of treatment, the GISAXS maps recorded at φ = 90° clearly demonstrate the formation of a bidimensional pattern for higher treatment durations (Figs. 1d-1e), as confirmed by atomic force microscopy (Fig. 1f). Actually, the analysis of the complete 3D map for the sample treated during 48h suggests the formation of a nanostructured pattern corresponding to a centered rectangular lattice of pyramids.

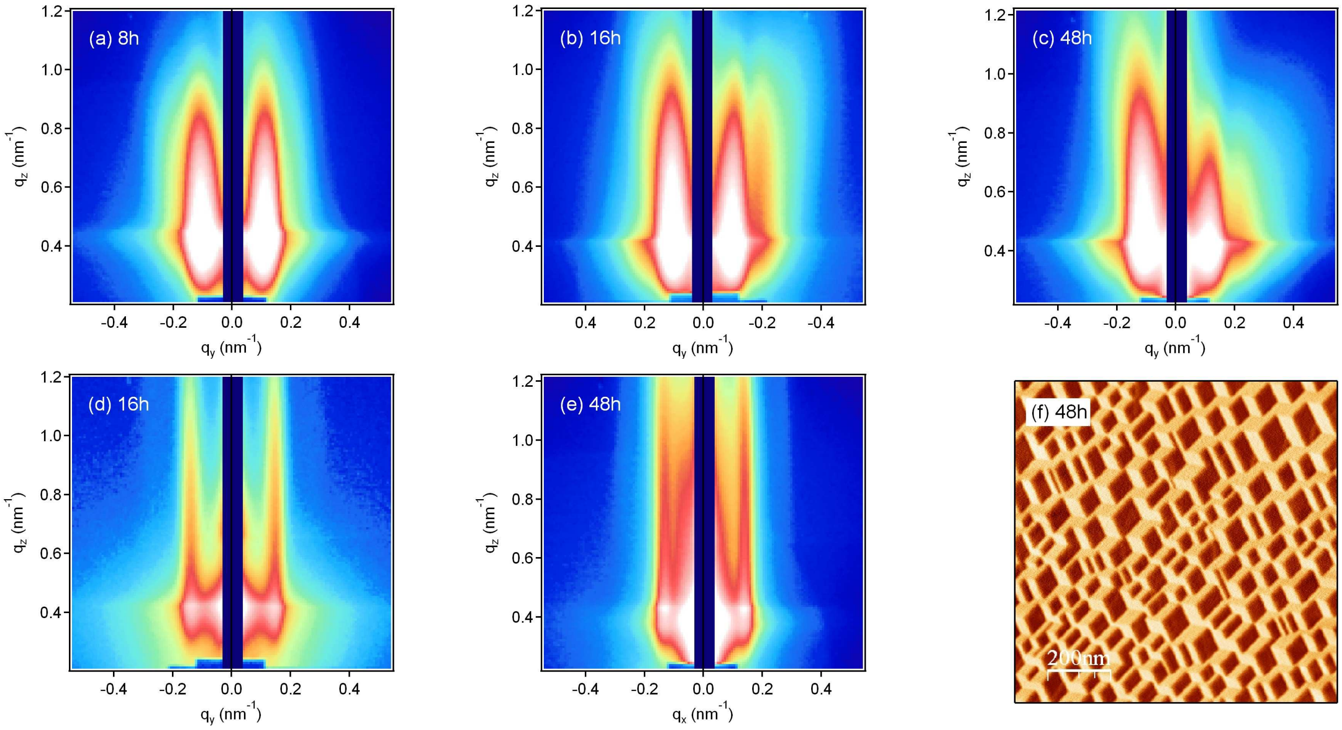

Figure 1: 2D GISAXS patterns of vicinal surfaces of sapphire with a miscut angle of 10° subjected to annealing at 1250°C in O_2 during different durations. (a) 8h, $\varphi = 0°$, (b) 16h, $\varphi = 0°$, (c) 48h, $\varphi = 0°$, (d) 16h, $\varphi = 90°$, (e) 48h, $\varphi = 90°$. (f) AFM image of the surface treated during 48h.

It is worth noting that such unidimensional and bidimensional patterns can also be obtained by annealing in air. However, in that case, the bidimensional nanostructures are formed at higher annealing temperature and duration than in O_2. These results show that the kinetics of pattern formation is strongly influenced by the oxygen partial pressure. Moreover, our GISAXS experiments show that both the period and the kinetics for the unidimensional patterns are strongly influenced by the miscut angle of the vicinal surfaces of sapphire. Increase of the miscut angle clearly results in a decrease of the period of the pattern (see Fig. 2).

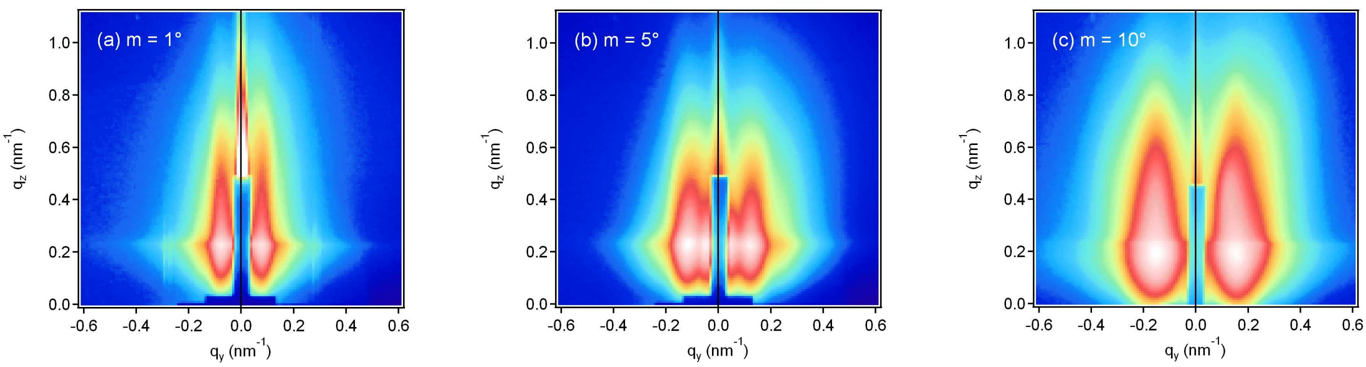

Figure 2: Influence of the miscut angle on the 2D GISAXS patterns of vicinal surfaces of sapphire treated at 1250°C in air during 1h. (a) m = 1°, (b) m = 5°, (c) m = 10°.

As far as we know, these experiments are the first observations of 2D nanosized patterns elaborated through high temperature thermal treatment of vicinal surfaces. Because, the GISAXS signal is due to the contribution of millimetre sized zones of the sample, we are confident that this feature is representative of the sample surface microstructure. Starting from these results, not only the typical size but also the symmetry of the elaborated 2D network can be managed changing the miscut angle, the type of crystallographic planes and of course the nature of the single crystal under consideration.

In term of measurements, it is clear that quantitative characterization of such pattern need a convenient positioning of the sample and particularly it must be possible to rotate the surface around his normal accurately. On the D2AM beamline, coupling of the SAXS setup with the 7-circle diffractometre is a convenient way to achieve this goal.