ESRF	**Experiment title:** **GISAXS study on the formation of bubbles induced by He implantation in Si and 4H-SiC**	**Experiment number**: HS-2183
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Report:

The main objective of this project was to investigate the morphology of nanocavities induced by He implantation in Si at various temperatures by means of grazing incidence small-angle x-ray scattering (GISAXS). 5 implanted Si samples have been prepared with the energy of the implanted ions being fixed at 50 keV and the implanted dose at 5×10^{16} cm^{-2}, whereas the implantation temperature was varied from 100°C to 550°C. In order to characterize in detail the depth distribution of the cavities, GISAXS experiments were carried out by varying the angle of incidence α_i of the X-ray beam and thus the analysis depth. As a typical example, the 2D GISAXS pattern of the sample implanted at 100°C obtained with $\alpha_i = 0.3°$ is shown in fig. 1. The corresponding signal, resulting from cavities buried at 500 nm under the surface, is isotropic and characteristic of spherical scattering objects. Before analysis, GISAXS patterns were corrected to refraction, absorption and transmission effects.

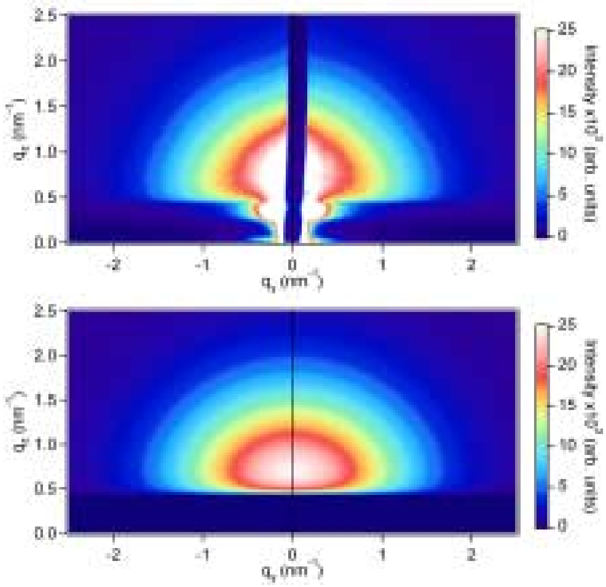

Fig. 1 : 2D GISAXS patterns of the Si sample implanted at 100°C with $\alpha_i = 0.3°$: experimental (up) and simulated (down).

Then, 1D scattering profiles were obtained by integrating large sectors in the intensity maps. Finally, the 1D profiles were fitted assuming a

Angle of incidence	Penetration depth	Mean diameter	fwhm
(a) 0.18°	48 nm	1.19 nm	1.35 nm
(a) 0.20°	207 nm	2.02 nm	1.47 nm
(a) 0.23°	336 nm	2.41 nm	1.34 nm
(a) 0.30°	561 nm	2.50 nm	1.31 nm
(b) 0.30°	561 nm	3.63 nm	2.31 nm
(c) 0.30°	561 nm	4.03 nm	4.45 nm
(a) 0.40°	834 nm	2.38 nm	1.38 nm
(a) 0.50°	1089 nm	2.27 nm	1.45 nm

Table 1 : GISAXS results for the Si samples implanted at (a) 100°C, (b) 200°C, and (c) 300°C.

spherical shape for the cavities and a log-normal size disribution function (described by a mean diameter and a full width at half maximum fwhm). The results obtained for the sample implanted at 100°C are summarized in Table 1. It can be deduced that very small cavities (diameter ~1 nm) are present in the superficial region of the sample while 2.5 nm cavities are formed at higher depth.

By increasing the implantation temperature up to 300°C, same qualitative results have been obtained, with an increase of the mean diameter as well as of the width of the size distribution being evidenced. In strong contrast, as seen in fig. 2, GISAXS patterns clearly show the presence of (111) faceted cavities [angle of 54.7° with the (001) surface] and (113) ribbon or rod-like defects [angle of 25.2° with the (001) surface] when He^+ ions are implanted in Si at temperatures higher than 400°C. However, we were not able to well reproduce the experimental data using a model of polydisperse faceted spheres. This suggest that faceting occurs for cavities larger than a critical size and that a shape dispersion exists in these samples. Accordingly, transmission electron microscopy experiments are in progress, which should confirm the actual qualitative results and permit a better quantitative modelling of the GISAXS data.

The effect of a post-implantation annealing at 800°C has also been studied for samples implanted at 100°C, 200°C, and 300°C. Whatever the implantation temperature, similar GISAXS results have been obtained showing an increase of the cavity size (from 2-4 nm to 8 nm), with neither (111) faceting nor (113) defects formation as seen in fig. 3.

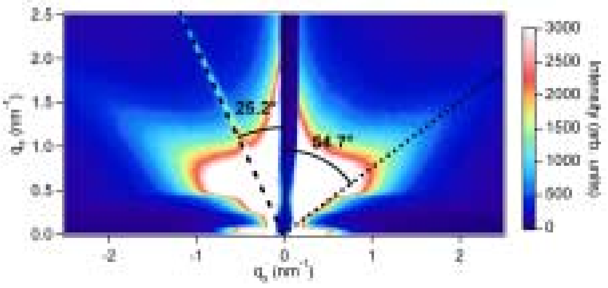

Fig. 2 : 2D GISAXS pattern of the Si sample implanted at 550°C with $\alpha_i = 0.3°$.

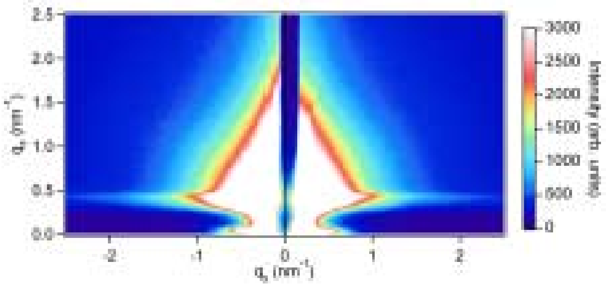

Fig. 3 : 2D GISAXS pattern of the Si sample implanted at 100°C and annealed at 800°C.

Finally, first tests have been carried out with a 4H-SiC sample implanted at 300°C. The GISAXS pattern presented in fig. 4 shows a diffuse scattering ring characteristic of very small objects (1.5 nm in diameter) with a short-range ordering (intercavity distance around 3 nm).

In conclusion, the very high flux delivered on D2AM allowed us to detect signals resulting from cavities buried in Si and SiC, and to characterize their morphology and organization.

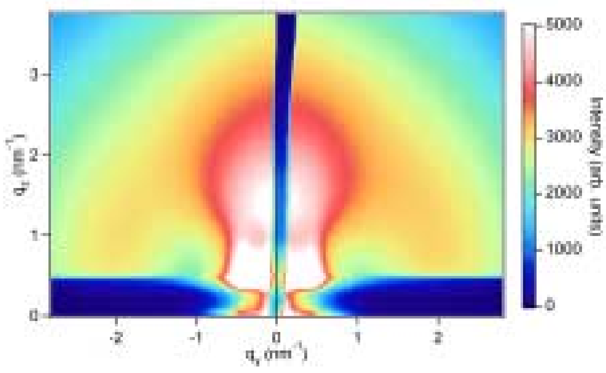

Fig. 4 : 2D GISAXS pattern of the SiC sample implanted at 300°C with $\alpha_i = 0.3°$.