ESRF	**Experiment title: GISAXS study on the formation of bubbles induced by He implantation in Si and 4H-SiC**	**Experiment number**: 02-01-653
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Names and affiliations of applicants (* indicates experimentalists):

D. Babonneau*, J.F. Barbot, M.F. Beaufort, M.L. David, A. Declémy, D. Lantiat*, S. Leclerc*, E. Oliviero, S. Peripolli*

LMP, UMR 6630 CNRS, Poitiers, France

V. Charmard, J.P. Simon*

LTPCM, UMR 5614 CNRS, Grenoble, France

Report:

The aim of this project was to study the microstructure of nanocavities (size, shape, arrangement, depth distribution) formed after implantation in semiconductors. Grazing incidence small-angle x-ray scattering (GISAXS) experiments have been perfomed on the D2AM beamline at 8950 eV using a 2D CCD detector fixed in front of the direct beam. The beamsize was 0.5×0.1 mm^2 and the angle of incidence α_i was varied from 0.3° to 0.5° in order to characterize the depth profile of the cavities from the near surface to deeply buried regions up to 1 μm. The influence of the implantation parameters on the formation of the nanocavities has been studied through the analysis of 3 types of samples:

- Si(001) implanted with He ions (50 keV, 5×10^{16} He$^+$.cm^{-2}) at different temperatures (200-600 °C) subjected to further annealing at 400, 600, and 800 °C.
- Si(001) implanted with Ne ions (50 keV) at different fluences (0.1-5×10^{16} cm^{-2}) and different temperatures (250-800 °C)
- SiC implanted with He ions (50 keV, 300 °C) at different fluences (3-10×10^{16} cm^{-2})

1) Helium implantation in silicon

As a typical example, figure 1(a) shows the GISAXS pattern of a Si(001) sample implanted with He ions at 200 °C and annealed at 600 °C for 1h. The angle of incidence was $\alpha_i = 0.4°$ and the incident x-ray beam was parallel to the [110] direction (φ = 0°). Furthermore, the length of the sample (30 mm) was adjusted in order to be able to perform absolute intensity measurements. An isotropic scattering signal without interference

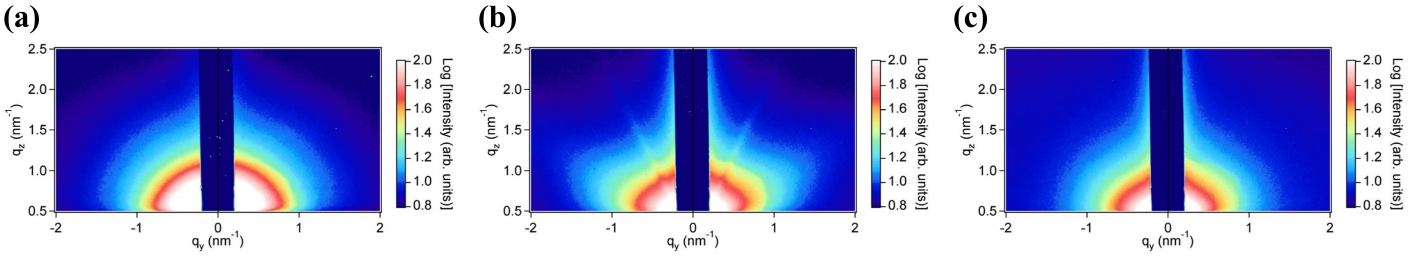

Figure 1: (a) GISAXS pattern of Si(001) implanted at 200 °C and annealed at 600 °C for 1h with $\alpha_i = 0.4°$.
(b) GISAXS pattern of Si(001) implanted at 600 °C with $\alpha_i = 0.4°$ and $\varphi = 0°$.
(c) GISAXS pattern of Si(001) implanted at 600 °C with $\alpha_i = 0.4°$ and $\varphi = 26.6°$.

maximum is clearly seen in figure 1(a), indicating a random distribution of spherical cavities with average size of about 5 nm as deduced from a simple Guinier plot. The quantitative analysis of the experimental data is under progress and will permit to determine the evolution of the volume occupied by the cavities as a function of the annealing temperature.

In contrast, the GISAXS pattern of a Si(001) sample implanted with He ions at 600 °C [figure 1(b)]) shows diffuse rods oriented at 54.7° from the surface normal (corresponding to <111> directions) and intense rods oriented at 25.2°, i.e. along <113> directions. To study the symmetry properties of the corresponding scattering objects, experiments at different azimuthal angles φ have been performed [see figure 1(c)]. The results reveal the existence of {111} facets on the cavities and the presence of {113} defects of ribbon-like type.

2) Neon implantation in silicon

It is worth noting that neither facetting of the cavities nor formation of {113} defects was observed for neon implantations. On the other hand, no GISAXS signal was detected for fluences lower than 2×10^{16} Ne^+.cm^{-2}. As examples, Figure 2(a) and 2(b) display the GISAXS patterns of Si(001) samples implanted at 250 °C with fluences of 2×10^{16} and 5×10^{16} Ne^+.cm^{-2} respectively. The quantitative analysis of the GISAXS data [figure 2(c)] shows that the average diameter of the spherical cavities increases with the temperature whereas the size distribution broadens as the fluence increases.

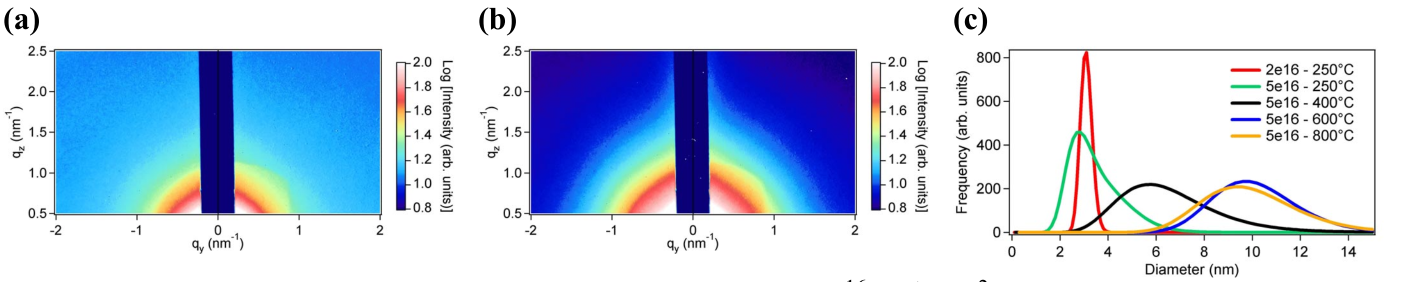

Figure 2: (a) GISAXS pattern of Si(001) implanted at 2×10^{16} Ne^+.cm^{-2} and 250 °C with $\alpha_i = 0.4°$.
(b) GISAXS pattern of Si(001) implanted at 5×10^{16} Ne^+.cm^{-2} and 250 °C with $\alpha_i = 0.4°$.
(c) Size distribution of the cavities as a function of the fluence and the temperature.

3) Helium implantation in silicon carbide

While no GISAXS signal was detected at 3×10^{16} He^+.cm^{-2}, a diffuse ring characteristics of very small (< 1.5 nm) cavities with a short-range ordering (intercavity distance around 3 nm) was observed at 1×10^{17} He^+.cm^{-2} in SiC. In conclusion, our experiments clearly show the capabilities of the GISAXS technique combined with the synchrotron brilliance for the study of buried defects induced by implantation in Si and SiC.