ESRF	**Experiment title:** Formation and growth of helium nanocavities in monocrystalline silicon during heat treatments using Small Angle X-ray Scattering	**Experiment number**: 02-01-683
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Names and affiliations of applicants:

Dr. Myriam Dumont (TECSEN, Université Paul Cézanne Aix-Marseille III, France)

Dr. Gabrielle Regula (TECSEN, Université Paul Cézanne Aix-Marseille III, France)

Dr. Marie-Vanessa Coulet (TECSEN, Université Paul Cézanne Aix-Marseille III, France)

Pr Alexis Deschamps (LTPCM-ENSEEG, INPG, Grenoble, France)

Report:

Synchrotron radiation was used to investigate the formation of nanocavities in monocrystalline silicon obtained by high dose ion implantation which is a promising technique for applications in localised lifetime engineering, metal gettering, point defect diffusion barrier, help in Si 1-x Gex buffer layer /(100) Si relaxation for further growth of strained Si, low K material manufactering etc. In particular, the nanocavities created during this process offer an efficient method to control the impurity concentration in the active area of silicon-based devices, by trapping impurities especially monovalent atoms such as Cu, Ag, Pt or Au.
The nanocavities are created by high multi-energy implantation of helium atoms. Helium is introduced with doses sufficiently high enough to form the so-called nano-pores initially filled with helium. Annealing of the samples at high temperatures (700-900°C) leads to the formation of a cavity band of 1 to 5 μm wide, depending on the implantation energy, and buried at 2 μm under the surface, which can be observed in transmission electron microscopy. However the as-implanted condition and early stages of growth of the nanocavities are difficult to investigate by high resolution electron microscopy due to the size and the low contrast of the objects and the strain induced by the He pressure.
The aim of this experiment was to investigate the existence of nano-pores in the as-implanted state and at low temperatures using Small Angle X-ray Scattering on BM02. In fact they are expected to be visible by SAXS due to the high electronic density contrast between silicon and helium. The second goal was to study in-situ their growth / coarsening kinetics at higher temperatures.

Experimental method

Two kinds of Si-He samples have been investigated.

- First of all, we have measured the SAXS response from a well-characterised sample (hereafter called *post-mortem* sample). This sample implanted at 5 Mev and annealed at 700°C, has already been investigated by high resolution microscopy, in particular in terms of density and distribution of the nanocavities. Those measurements would allow us to validate the method and to compare both techniques. One should note that one challenge of the present experiment was to get signal from a limited zone of the sample : only 1 to 5% of the thickness investigated was in fact implanted and may give rise to significant scattering.

- In a second time, as-implanted samples have been measured prior to further heat treatments consisting of isotherms at temperatures varying from room temperature to 700°C. These samples, 100µm wide in total, were implanted in the KeV range and then exhibit an implantation width estimated to around 1µm from TEM observations. For thermal treatments, we used the furnace designed at the LTPCM laboratory in collaboration with D2AM beam line, specially designed for in-situ SAXS experiments.

Regarding the small scattering volume, the high flux of the synchrotron radiation was needed to obtain good statistics in a reasonable time of measurements during in-situ heat treatments. Experiments were performed with a 300*300 µm² beam at 9.6 KeV. A small-angle set-up was chosen to well characterise nano-cavities in the 10-100 Å size range (in terms of Guinier radius), i.e. in a q-range varying from 0.02 to 0.25 $Å^{-1}$. For each measurement, volume fraction in the implanted region and Guinier radius were estimated.

<u>*Results*</u>

- *Post-mortem measurements – Comparison with TEM observations*

The results of the measurements on the reference sample (MeV annealed at 700°C) as well as the as-implanted KeV sample (prior to in-situ annealing) are displayed on figure 1 in the Iq² versus q plot. The existence of a peak in this plot vouches for the presence of nano-cavities in both samples in the studied q-range, in particular in the as-implanted sample in which no TEM observations can attest their existence.
In the annealed sample, the size and volume fraction of the nano-cavities has been estimated to 88 Å in terms of Guinier radius and to 2.2% respectively, to be compared to 135 Å diameter and 4% porosity results obtained from TEM observations (refer to proposal 02-01-683).

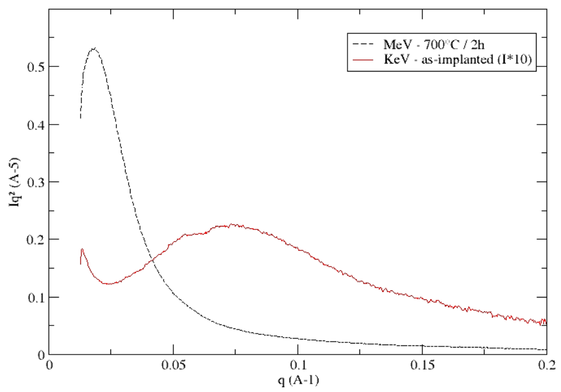

Figure 1: Iq²=f(q) plot from annealed sample and as-implanted sample. Note that the intensity for the as-implanted sample has been multiplied by 10 to be of observable magnitude.

- *In-situ experiments*

The results of in-situ measurements performed on a sample implanted in the KeV range at 700°C are displayed in figure 2 in the Iq² versus q plot.
One can observe that the initial peak is shifting towards smaller q-values, reflecting the coarsening of the precipitates during annealing. Moreover the area under the peak is increasing, this can be interpreted in terms of evolution of the porosity during the heat treatment. These effects remain to be quantified.

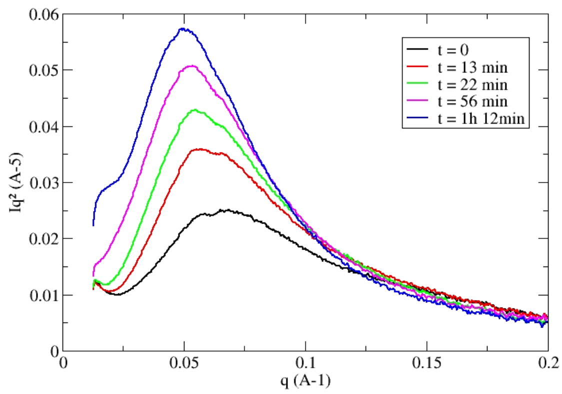

Figure 1: Iq²=f(q) plot from a KeV sample during in-situ annealing at 700°C.

<u>*Conclusion and acknowledgements*</u>

These successful results completed with TEM observations will be the subjet of publication in the next months. We acknowledge LTPCM for providing the useful furnace used for in-situ experiments. We are grateful to the ESRF/CRG staff, especially on the BM02/D2AM beam line for giving us the opportunity to perform this challenging experiment and for their help during the experiment. Special acknowledgement is addressed to Dr. Françoise Bley for her support before, during and after the experiment.