ESRF	**Experiment title:** **X-ray diffraction study of Kr and Xe clusters in implanted semiconductors**	**Experiment number:** **HS-801**
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Names and affiliations of applicants (* indicates experimentalists):

* G. Faraci Dipartimento di Fisica, Universita' di Catania

* A. R. Pennisi " " " "

* F. D'Acapito ESRF - beamline Gilda

* F. Zontone ESRF - beamline Troika

Aims of the experiment and scientific background

In this paper, we report on a successful investigation of Xe as-implanted Si: we obtained the X-ray structural characterization of Xe clusters (including nn distance and coordination) showing evidence of the formation of a Xe compressed fluid phase. Also the Si matrix was contemporarily checked and verified to be in an amorphous phase; here too, nn distance and coordination were detected.

The simultaneous measurements both on the matrix and on the Xe inclusions were possible using a new X-ray diffraction method which combines the grazing incidence geometry, highly collimated, very intense Synchrotron Radiation beams and the two-dimensional detection. This method permits to maximize the implanted thin layer contribution. The compressed fluid phase will be simply explained in the frame of the Hard Sphere (HS) model.

Here, we compare an X-ray diffraction study of two Si samples, one implanted at high dose (10^{17} at/cm^2 at 350 keV, sample "HIGH") and the other implanted at a lower dose (1.5×10^{16} at/cm^2 at 200 keV, sample "LOW"). In this conditions the implantation profile has a gaussian shape centered at about 1000 Å below the matrix surface. In order to maximize the scattering from the buried layer rich in Xe we used the grazing incidence geometry near the critical angle for total external reflection (~0.1°). The experiments were performed at the European Synchrotron Radiation Facility on the beamline ID09. The samples were mounted vertically on the high precision goniometer used for High Pressure experiments. The setup can produce a very intense 10x25 μm^2 beam well suited for the typical sample lengths (~10 mm) [[i]]. The working X-ray wavelength was 0.4618 Å. Due to the expected weak cluster signal we used FUJI-A3 flat imaging plates (IP) as detectors. A sketch of the experimental arrangement can be found in Ref.12,13. The reflectivity measurements were done by scanning the grazing angle while collecting the reflected beam with a large area photodiode. The presence of the rare gas-rich layer is revealed by a step above the critical angle for the air-Si interface. Once the working points were chosen the diffraction patterns were collected on to the IP placed at normal incidence at about 344 mm with an integration time of about two minutes.

The quantity of implanted material corresponds to few monolayers in equivalent thickness therefore the diffraction patterns consist of the weak scattering signal from the Xe-rich layer superimposed to the scattering from the single crystal matrix, i.e. Bragg spots, Thermal Diffuse Scattering (TDS) and Compton radiation. This highly patterned background is subtracted directly in the image by taking a reference background pattern of the non-implanted part of the sample in the same grazing incidence condition. In the background image the main feature is the hexagonal distribution of diffuse radiation coming from the (acoustic) phonon scattering. Since the intensity of the TDS has a linear dependence on the volume we have scaled the background pattern in order to obtain a difference image with the minimum TDS contribution. The extra features still visible in the difference image are mainly coming from saturated regions of the IP. The difference pattern shows diffuse rings which are produced by the amorphous phases formed in the buried layer damaged by the ion bombardment. An azimuthal integration is then performed in order to obtain the intensity functions I(q) after having masked the residual extra features in the difference image. The intensity functions are remarkably different in the two samples, in particular the first peak is much higher than the second one in the sample HIGH. This fact suggests either a strong structural change in the amorphized Si phase when implanted at higher doses or more likely the appearance of a disordered phase related to the implanted Xe. We have firstly applied the model to X-ray data for liquid Xe near the triple point. With spheres of D=4.094 Å which occupy a volume fraction η=0.478 we can reproduce the main features in the S(q) and account for the pair correlation function having ~10 first neighbors at a distance ~4.4 Å. We attribute the apparent sharper modulations of the model mainly to the lack of angular resolution in the literature data.

The same model applied to our structure function. The HS model reproduces the experimental S(q) even better than in the previous example with significantly smaller values for the sphere diameter (D=3.837 Å) and the volume fraction (□=0.377). The residual discrepancies are very likely coming from the multiple subtraction procedures. A smaller sphere diameter and a reduced volume fraction account for a G(r) having a broader first peak at the shorter distance of ~4.2 Å. A reduced first neighbor distance implies an overpressurized state related to nanometer sized particles. The effect of compression in small particles due to the surface stress is a very well known effect. The small size of the particle could also explain the 20% reduction in volume fraction, i.e. the high surface-to-volume ratio could be responsible for the reduced average coordination number with respect to the usual liquid state. We therefore think to have evidence of a disordered condensed phase for implanted Xe formed by small fluid bubbles in a overpressurized state. Using the average coordination, the volume and surface density of solid Xe we can now estimate the average radius of these Xe bubbles in about 20 Å, with a pressure acting on the bubbles in the order of 1 Gpa.