EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

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Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: Grazing Incidence Diffuse X-ray Scattering Investigation of Radiation Damage in Ion Implanted Silicon at Ultra-Low Energies**	**Experiment number:** SI 649
Beamline: ID01	**Date of experiment**: 27/06/01 – 02/07/01 from: 7.00 am to: 7.00 am	**Date of report**: 17/08/01
Shifts: 15	**Local contact(s)**: A. Mazuelas and T. H. Metzger	*Received at ESRF:*

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Report

Since the experiment SI649 ended at the beginning of July 2001, the quantitative data treatment was not started yet. Therefore, this report will only describe the implant and the annealing conditions adopted to prepare the Si samples, the set-up used for the measurements and some preliminary qualitative data interpretation.

One set of Si [001] samples was implanted at room temperature with B ions at doses from 1 to 15×10^{14} cm^{-2} and 1 keV energy, another set with As ions at doses from 1 to 10×10^{14} cm^{-2} and energies of 2.5 and 5 keV. The samples were measured before and after rapid thermal processing (RTP) at temperatures from 700 to 1050 °C for 10 s and spike annealing (SA) at 1050 °C.

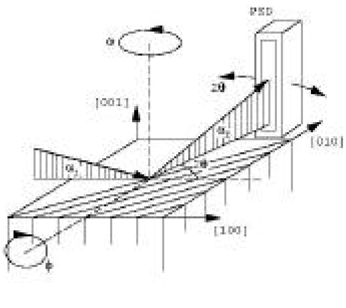


Fig. 1 – Experimental set-up

To study ultra-thin Si surface layers (from about 10 to about 40 nm), such as those resulting from the low energy ion implantation, diffuse scattering and diffraction measurements were performed under conditions of grazing incidence end exit (grazing incidence diffraction, GID). Fig. 1 shows the geometry, where α_i and α_f are the small incidence and exit angles with respect to the sample surface. Both angles were chosen near the critical angle (α_c) for total external reflection. For the X-ray energy of 7.71 keV, $\alpha_c = 0.23°$ for virgin Si.

(220) lattice planes perpendicular to the surface were aligned to diffract the incident monochromatic beam, and the diffracted beam was collected by a linear position sensitive detector (PSD) oriented normal to the sample surface. Different scans were made by (i) rotating sample and PSD in $\omega/2\theta$ mode (radial scan), (ii) rotating the sample with the PSD fixed at the Bragg angle (transverse scan) and (iii) rotating the sample fixed at the Bragg angle around the φ axis (α_i scan). α_f "scans" were carried out by means of the PSD with fixed sample and detector. The defect induced diffuse scattered intensities were obtained by subtracting the intensity scattered by a virgin silicon wafer. From these different scans, information on amorphised surface layer thickness (if any), on defect type, size, density and depth distribution will be obtained by intensity

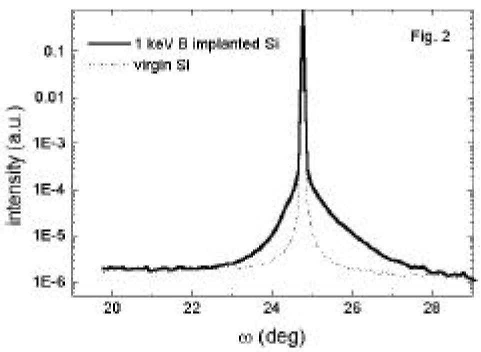
profile simulation with theoretical models based on the theory of GID and diffuse scattering from point defects in the vicinity of Bragg reflections.

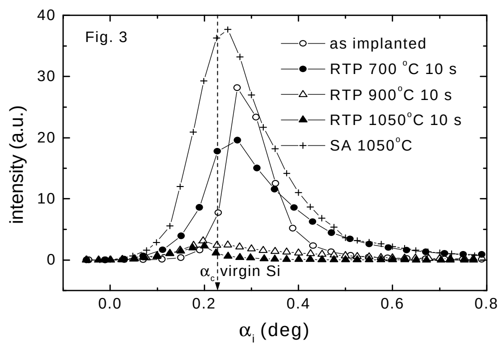
Fig. 2 shows an example of diffuse scattering data obtained by ω/2θ 220 scan at fixed α_i = 0.1° from a sample implanted with 1.5×10^{15} B^+/cm^2 at 1 keV energy. The angular distribution of the diffuse scattering is well above the tail intensities of the virgin silicon and shows a marked asymmetry with respect to the Bragg angle (θ_B=24.75°). The higher intensity scattered in the range $\theta > \theta_B$ indicates that the implantation induced defects are interstitial in nature. From the analysis of the symmetrical component of the diffuse scattering distribution [1], the average size of the defect clusters turned out to be about 2 nm. Considering that the thickness probed by X-rays at α_i = 0.1° is about 7 nm, we conclude that a high density of interstitial clusters is present immediately below the surface.

In Fig. 3 a series of φ (or α_i) scans obtained from As-implanted samples before and after RTP and SA is shown. During these scans, sample and PSD were kept fixed at θ_B and $2\theta_B$, respectively. An inspection of the intensity profiles in Fig. 3 suggests that (i) an amorphous layer of about 6 nm thickness is present at the surface after the implant (in fact, the peak of diffracted intensity occurs at an incidence angle greater than those at which the diffraction peaks appear after annealing), (ii) epitaxial regrowth of the amorphous layer takes place after RTP at 700 °C (see the peak shift to the α_c of the virgin Si), (iii) the mass density of the surface Si decreases after RTP at 900 and 1050 °C (as can be seen from the diffraction peaks at $\alpha_i < \alpha_c$ of the crystalline Si) and (iv) Si lattice recovery occurs after SA at 1050 °C (see the angular shift of the diffraction peak to α_c of the virgin Si and the strong increase in the peak intensity). Item (iv) points out that SA is more efficient than RTP in defect removal, very likely due to its much faster heating ramp.

The mass density decrease could be the consequence of void formation close to the surface, where strong excess vacancies are produced during implant by nuclear collisions of heavy ions (like As) with the target atoms. Mass density reduction of a few 10% were already observed in phosphorus ion implanted Si [2]. Reflectivity measurements will be done with laboratory instrumentation to investigate this phenomenon in detail.

The remarkable intensity drop observed after RTP at 900 and 1050 °C could be ascribed to geometrical effects (the diffracting planes could be misoriented from the normal to the surface in such a way that the





diffraction vector is directed into the sample) or to a lowering in the structure factor of the 220 reflection. This lowering could be the result of the strong displacement field (high static Debye-Waller factor) associated with extended defects which commonly form during high temperature annealing of layers implanted at high dose. A further explanation could be a very rough surface produced by diffusion to the surface of point defects released by thermal defect evolution. A careful analysis of all the collected data and theoretical curve simulation will quantify the role of these different parameters.

The experiment SI649 proved very interesting for different reasons. First of all, the results obtained are essential to understand the physical phenomena governing defect production and thermal evolution in an implant energy regime never explored before. Moreover, this investigation offers the possibility to compare the experimental results with the predictions of the theory of atomic collisions in solids inside ultra-thin surface layers. Finally, what is expected is the optimisation of the technological processes leading to the fabrication of electronic devices of future generation.

[1] P.H. Dederichs, *J. Phys F: Metal Phys.* **3** (1973) 471.
[2] A. L. Golovin, R. M. Imamov and E. A. Kondrashkina, *Phys. Stat. Sol.* (a) **88** (1985) 505.