ESRF	Experiment title: Depth-profiling of relevant dopants implanted at low energies in Si and Ge by using synchrotron radiation based high-resolution micro-GEXRF	Experiment number: MI-1059
Beamline:	Date of experiment: from: 03.11.2010 to: 09.11.2010	Date of report : 31.08.2011
Shifts:	Local contact(s): Dr. FAYARD Barbara	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists): Dr. KAYSER Yves*, Département de Physique, Université de Fribourg, CH-1700 Fribourg, Suisse Prof Dr DOUSSE Jean-Claude*, Département de Physique, Université de Fribourg, CH-1700 Fribourg, Suisse		

Dr. HOSZOWSKA Joanna*, Département de Physique, Université de Fribourg, CH-1700 Fribourg, Suisse

Dipl. Phys. CAO Wei*, Département de Physique, Université de Fribourg, CH-1700 Fribourg, Suisse

Dipl. Phys. NOWAK Stanislaw*, Département de Physique, Université de Fribourg, CH-1700 Fribourg, Suisse

Prof. Dr. PAJEK Marek*, Institute of Physics, Jan Kochanowski University, 25-406 Kielce, Poland

Dr. BANAS Dariusz*, Institute of Physics, Jan Kochanowski University, 25-406 Kielce, Poland

Dr. KUBALA-KUKUS Aldona*, Institute of Physics, Jan Kochanowski University, 25-406 Kielce, Poland

Dipl. Phys. JAGODZINSKI Pavel*, Laboratory Kielce University of Technology, 25-314 Kielce, Poland

Report:

The synchrotron radiation based high-resolution grazing emission (GEXRF) method was already applied successfully to the depth-profiling of Al implantations in Si wafers (see report MI-928) [1-2]. The aim of the present experiment was to further develop and improve this technique for non-destructive depth-profiling of low-energy dopant implantations. In this perspective the setup was expanded to include a focusing polycapillary optics mounted on a five-axis positioning stage in order to allow for a laterally localized characterization of the ion-implanted Si and Ge wafers with an increased gain.

GEXRF was introduced in 1983 [3] and is nowadays considered as an alternative to the well-established grazing incidence x-ray fluorescence (GIXRF) method which has emanated from the total reflection x-ray fluorescence (TXRF) technique. The basic principle of GEXRF is to monitor the dependence of x-ray fluorescence intensity on the emission angle. This is realized in a point-by-point scan where the shallow emission angle, defined relatively to the target surface, is gradually changed. Due to the shallow emission angles, GEXRF is characterized by a considerable enhancement of the surface-to-bulk signal ratio in comparison to standard XRF due to the refraction of the x-rays at the sharp vacuum-sample interface. Since the refractive index of solids is smaller than unity in the x-ray domain, the fluorescence x-rays sustain a larger angle relatively to the critical angle for the emission angles, only the first few nanometers in the depth direction of the sample are probed. The critical angle depends on the sample and the x-ray energy. Consequently GEXRF can be used for non-destructive surface analysis. Increasing emission angle above the critical angle allows thus to analyze the sample in the in-depth direction and to perform depth-profiling measurements.

In the present experiment the surface sensitive character of GEXRF was made profit of to reconstruct the depth distribution of dopant atoms introduced at low implantation energies into the wafers. Low implantation energies were considered because of the reduction in size of semiconductor devices. As a consequence, the implanted depth region should also be shallower in order to keep a constant aspect ratio and to reduce short channel effects. In this perspective P-implanted Si and Ge wafers were considered, the implantation energies being 1, 2, 4, 6, and 8 keV and the implantation flux was 5×10^{15} atoms/cm². In addition In and Sb implantations into Si at energies of 1, 2, and 4 keV and a flux 5×10^{14} atoms/cm² were considered.

The GEXRF measurements were realized at the ESRF ID21 beamline. The primary x-ray beam was delivered by the two undulators of the ID21 beamline and the size of the $3 \times 3 \text{ mm}^2$ beam was defined by two pairs of slits. The full beam was thus intercepted by the polycapillary entrance aperture of 4.4 mm in diameter. The simultaneous use of two undulators resulted in fluxes as high as 3.5×10^{13} and 2.4×10^{13} ph/s for the two selected primary beam energies. Such high photon fluxes were necessary because of the low concentration levels of the dopant implantations. The experimental measurements with the P-, In- and Sb-implanted Si wafers were performed at an energy of 4240 eV (just above the L3 absorption edge of Sb), whereas an energy of 3190 eV was selected for the measurements with the P-implanted Ge wafer. The monochromatization of the primary beam was realized with two Ni/B₄C multilayers, while the higher order harmonics were rejected by Ni mirrors tilted to 7.25 mrad with respect to the incident primary beam. The K- respectively L-absorption edges of the implanted P. In and Sb dopants being all above the K absorption edge of Si, the background contribution of the strong Si Ka fluorescence lines was thus unavoidable. The high-resolution detection with the von Hamos spectrometer permits, however, to separate accurately the different fluorescence signals. This was important for the P-implanted Si wafers, the P Kα-line (2013.7 eV) being separated by only 274 eV from the Si K α -line (1739.9 eV). The high energy resolution of the detection setup was thus a prime advantage for successfully recording the angular intensity profile of the P K α fluorescence line.

The P K α , In L α , Sb L α and Si K α fluorescence x-ray lines of the P-, In- respectively Sb-implanted Si wafers were diffracted by means of a cylindrically curved ADP (101) crystal (2d = 10.642 Å, curvature radius R = 25.4 cm) in first order for the P K α and Si K α -lines, and in second order for the In L α and Sb L α -lines. For the P-implanted Ge wafers a cylindrically curved TlAP (2d = 25.772 Å, curvature radius R = 25.4 cm) was employed in second order for the Ge L α -line and the P K α -line. The diffracted x-rays were detected by means of a back-illuminated position-sensitive CCD (Coupled Charge Device) camera (1340×400 pixels of 20×20 µm2, read-out speed of 1 MHZ). The emission angles were calibrated for each sample by means of the critical angles Si or Ge for the Si K α -line emitted by the bulk Si wafer or the Ge L α -line of the bulk Ge wafer, respectively. It was assumed that the implanted dopants did not affect noticeably the refractive index of the implanted wafers with respect to a pure bulk sample. The angular intensity profiles were measured at 100 different exit angles for 50 seconds per angle for the P K α -line. The points in the angular x-ray fluorescence intensity scans were separated by 0.0225° for the dopants (P K α , In L α and Sb L α -lines), respectively by 0.0450° for the signal from the bulk (Si K α and Ge L α -lines).

The extraction of the depth distribution of the implanted dopant atoms from the angular intensity scan was realized by fitting the experimental intensity curve with the equation for the angular intensity profile from [4] for ion-implanted samples. Indeed the analytical shape of the implanted ion distribution (Gaussian, Pearson IV, joined half-Gaussian, ...) [5] is more or less known from calculations of the dopant distribution for different implantation energies. Thus a given analytical function, in occurrence a joined half-Gaussian distribution, was selected to fit the experimental data. The center and the standard deviation on the left and right side of the center were the free parameters in the fit. The choice of a joined half-Gaussian distribution was supported by SRIM (Stopping and Range of Ions in Matter) [6] calculations for the considered dopant-wafer combinations.

This approach allowed to well fit the experimental data (see Fig. 1, left panel) and extract numerical results describing the depth distribution of the implanted dopant atoms (see Fig. 1, right panel). For the ion-implanted Si wafers, a comparison with the numerical values returned by the SRIM calculations reveals that the experimentally retrieved depth distributions are located further away from the surface (by about 1-2 nm,

depending on the sample) and the overall distribution are also broader. In agreement with the SRIM calculations, the standard deviation for the second half-Gaussian describing the dopant distribution for the depth region extending from the center with the maximum concentration value into the bulk is larger than the one corresponding to the first half-Gaussian. For the P-implanted Ge wafers the interpretation of the numerical values is ambiguous since the trend of the SRIM calculations cannot be reproduced. For instance the samples implanted at 2 and 4 keV present the maximum concentration values at similar depths and differ mainly by the width of the distribution. Considering the SRIM calculations, it would be expected that not only the width of the distribution is larger but also the depth of the concentration peak for the sample implanted at 4 keV. However, the angular intensity curve given by the fitting procedure reproduces the experimental data better than the one calculated with the SRIM depth distribution. The interpretation of the data is still on-going and the diffusion of P in the Ge matrix [7] is suspected to be responsible for the observed differences with the calculations.



Fig. 1: Examples of P K α angular profiles from P-implanted Ge wafers, respectively In L α angular profiles from Inimplanted Si wafers, fitted with joined half Gaussian depth distribution functions. Especially the regions below the respective critical angles are better reproduced by the fits than by the calculated angular intensity profiles. On the right side the retrieved depth distributions are compared to calculated ones (SRIM): the concentration profiles returned by the fitting procedure are broader and the peak concentration is located slightly deeper in the sample.

- [1] Y. Kayser et al., Spectrochim. Acta Part B: Atomic Spectroscopy 65, 445, 2010.
- [2] Y. Kayser et al., Submitted to X-Ray Spectrom.
- [3] R. S. Becker, J. A. Golovchenko, and J. R. Patel, Phys. Rev. Lett., 50, 153, 1983.
- [4] H. P. Urbach and P. K. de Bokx, Phys. Rev. B, 63, 085408, 2001.
- [5] K. Suzuki and R. Sudo, Solid-State Electronics, 44, 2253, 2000
- [6] J. F. Ziegler, SRIM–2003, NIMB, 219 220, 1027, 2004.
- [7] C. O. Chui, et al., Applied Physics Letters, 83, 3275, 2003.