

X-Ray and Neutron Capillary Optics II

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Investigation of ion - implanted layers by X-ray reflectometry method

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ABSTRACT

Investigation of ion-implanted structures by means of X-ray reflectometry was carried out. For this purpose the new methods of reflectivity measurements and the experimental data treatment were developed: double beam method of X-ray reflectometry and the subtraction of trend. The depths or widths of implanted layers, its average density were determined.

Keywords: ion implantation, X-ray reflectometry, semiconductor

The ion - implanted structures were seldom studied earlier by X-ray reflectometry method for good reasons: small changes of sample X-ray optical constants and the boundary smoothness of implanted layers result in small reflectivity oscillations and small absolute shifts of the reflectivity dependence. Indeed, angle repeatability errors are comparable to angle shift of experimental curves. Therefore the new approaches to reflectivity measurements and the experimental data treatment were developed: double beam method of X-ray reflectometry (Fig. 1) and the subtraction of trend. The trend of curve is monotonic curve close to the experimental dependence.

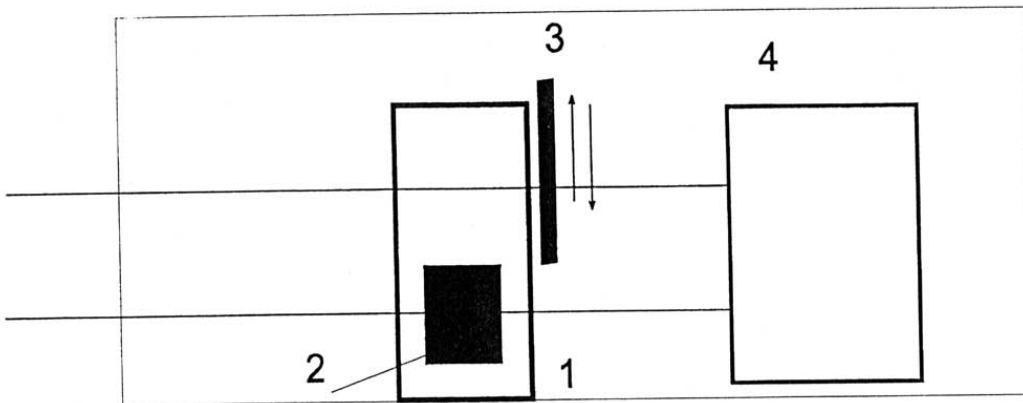


Fig.1. The measurement scheme for the double beam method of X-ray reflectometry. 1 - sample, 2 - irradiation lot, 3 - screen bar, 4 - detector

Measurements were carried out by means of multi-wave reflectometer at wave lengths 1,54 Å and 1,39 Å (Fig. 2) and on two-crystal topographical spectrometer.

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At the double beam method of X-ray reflectometry the reflectivities of etalon and of studied sample are measured in each angular point.

The multi-wave reflectometry method consists of measurements of the reflectivities at several wave lengths simultaneously for one scan.¹ Especially this method is necessary at investigation of small samples in millimeter size region. In this case at small grazing angles the irradiated area is more comparing the material under investigation. The determination of the ratio of the reflectivities on different wave lengths enables to eliminate this geometrical factor.

The measurements by multi-wave X-ray optical scheme were carried out by means of the X-ray tube with the copper anode and power 300 W. The width of a X-ray beam equals 40 microns, the divergence of a bunch is about 0.005° .

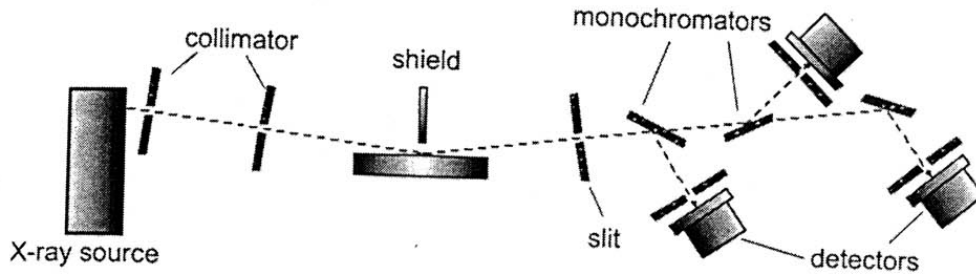


Fig. 2. The X-ray optical scheme of multi-wave reflectometer.

In Fig. 3 the reflectivities of the untreated silicon and silicon, implanted by argon ions with energies 20 keV and 30 keV at the identical dose $1 \times 10^{14} \text{ cm}^{-2}$ are represented. Curves were measured by the double beam method of X-ray reflectometry. For implanted samples the decrease of the critical angle Θ of the total external reflection comparing with that for the initial material is observed. The angle 2Θ is decreased by $4 \times 10^{-3} \text{ deg}$ for ions with energy 30 keV and by $5,7 \times 10^{-3} \text{ deg}$ at 20 keV. Angular shift was determined at half-height of reflectivity angular dependence. Change of average density of implanted samples was determined by means of a relation $\Theta = (2\delta)$. Here δ - the real part of refraction coefficient which is proportional to the material density. Relative changes of average density ρ/ρ are 2 % and 3 % for the samples implanted by ions of argon with energy 30 keV and 20 keV accordingly.

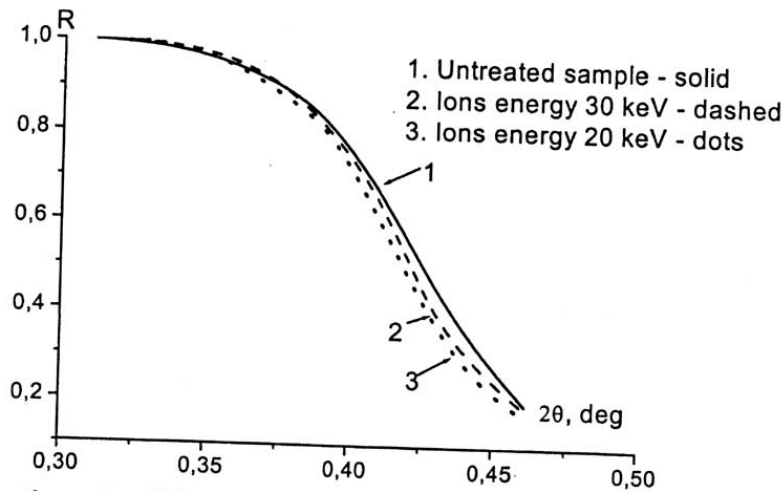


Fig. 3. The reflectivities of an untreated silicon and silicon, implanted by argon ions with energies 20 keV and 30 keV with the identical dose $1 \times 10^{14} \text{ cm}^{-2}$. Curves were measured by the double beam method of X-ray reflectometry

In Fig. 4 the reflectivities of untreated fused silica and fused silica, implanted by argon ions with energy 20 keV and doses $1 \times 10^{15} \text{ cm}^{-2}$ and $3 \times 10^{15} \text{ cm}^{-2}$ are presented. Curves were measured by the double beam method of X-ray reflectometry. For the sample irradiated with a dose $1 \times 10^{15} \text{ cm}^{-2}$ the increase of 2Θ angle equals 0.005° . At dose $3 \times 10^{15} \text{ cm}^{-2}$ the change of 2Θ was $(-0,01^\circ)$. It corresponds to density variations 2,3 % and (-5,7 %) accordingly.

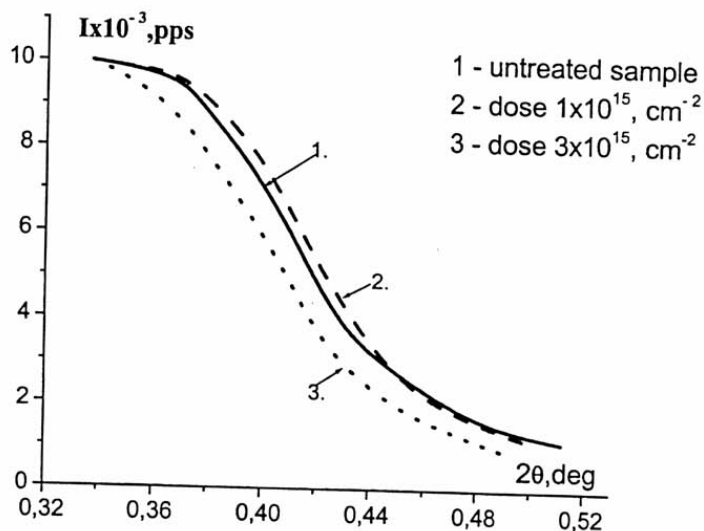


Fig. 4. Reflectivities of untreated fused silica and quartz, implanted by argon ions with energy 20 keV and the doses $1 \times 10^{15} \text{ cm}^{-2}$ and $3 \times 10^{15} \text{ cm}^{-2}$. Curves were measured by the double beam method of X-ray reflectometry.

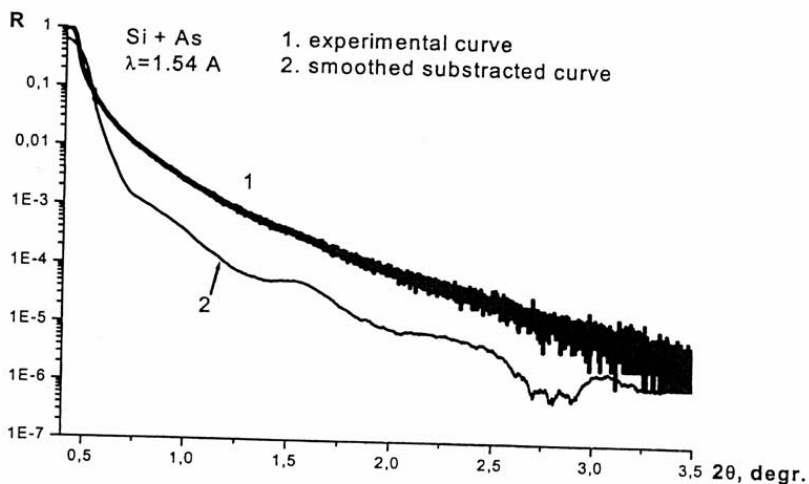


Fig. 5. 1 - reflectivity of silicon implanted by As ions with energy 60 keV and dose $4,4 \times 10^{15} \text{ cm}^{-2}$ at $\lambda = 1,54 \text{ \AA}$, 2 - smoothed result of background subtraction

The reflectivities of silicon, implanted by As ions with energy 60 keV and dose $4,4 \times 10^{15} \text{ cm}^{-2}$ were measured by multi-wave reflectometer at wave lengths 1,54 Å and 1,39 Å. On Fig. 5 the reflectivity at $\lambda = 1,54 \text{ Å}$ (curve 1) and smoothed result of background subtraction (curve 2) are presented. On fig. 6 the experimental measured at $\lambda = 1,39 \text{ Å}$ reflectivity of the same silicon and the reflectivity calculated for Si with taking into account the surface roughness are shown. The depth of implanted layer determined from intensity oscillations (Fig. 5, curve 2) equals $\approx 100 \text{ Å}$. The surface roughness equals $\sigma \approx 7 \text{ Å}$ from data of measurements at wave lengths both 1,54 Å and 1,39 Å.

As example of studies by the two-wave reflectometry method of small samples the reflectivity of implanted diamond at wave lengths 1,39 Å and 1,54 Å was investigated. Approximate crystal sizes were $7 \times 5 \text{ mm}^2$ and the half of sample was bombarded (dark part in insert of Fig. 8). The implanted layer has been manufactured by He ion bombardment with the distributed doze in energy region from 50 keV up to 350 keV and by Ar ion bombardment with energy 30 keV. Integral dose equals $1 \times 10^{16} \text{ cm}^{-2}$. Photo of investigated diamond sample is at Fig. 7.

Experimental (1) and calculated (2) angular dependences of the reflectivity ratio $(R_\beta - r_\beta) / (R_\alpha - r_\alpha)$. Here R_α , r_α , R_β , r_β are reflectivities of implanted and not implanted surfaces of diamond for CuK_α and CuK_β X-ray lines. The distribution of density in diamond, implanted by He and Ar ions, received by means of mathematical simulation is at insert on Fig. 8.

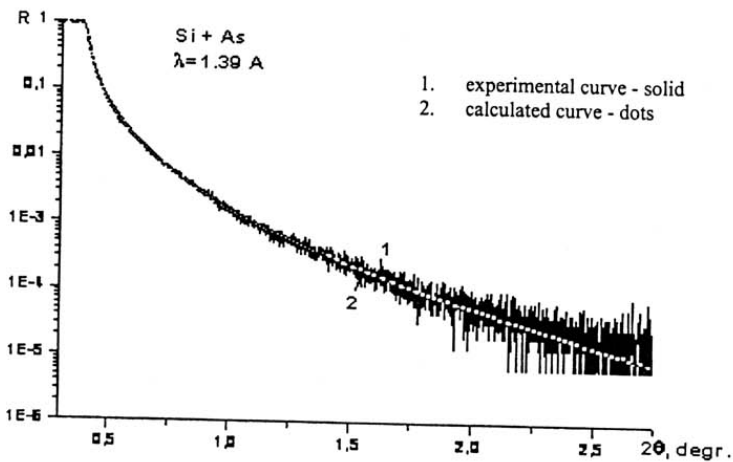


Fig. 6. Reflectivity of the same silicon at $\lambda = 1,39 \text{ Å}$. 1- experimental data, 2 – calculated reflectivity for Si with surface roughness $\sigma = 7 \text{ Å}$

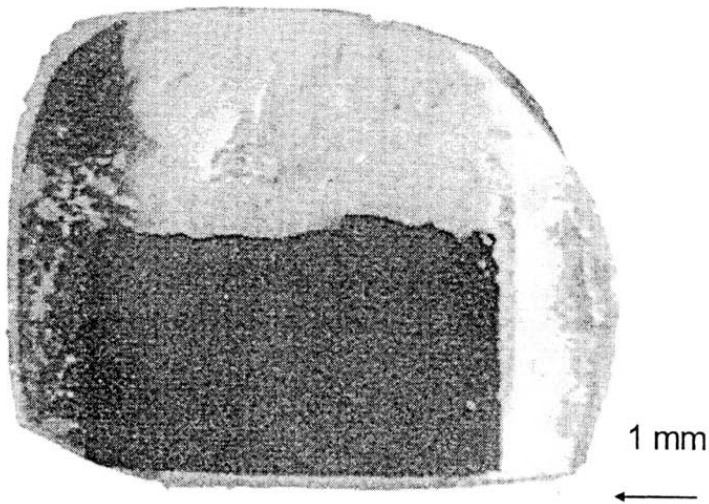


Fig. 7. A photo of diamond. Half of this sample is implanted by ions He and Ar.

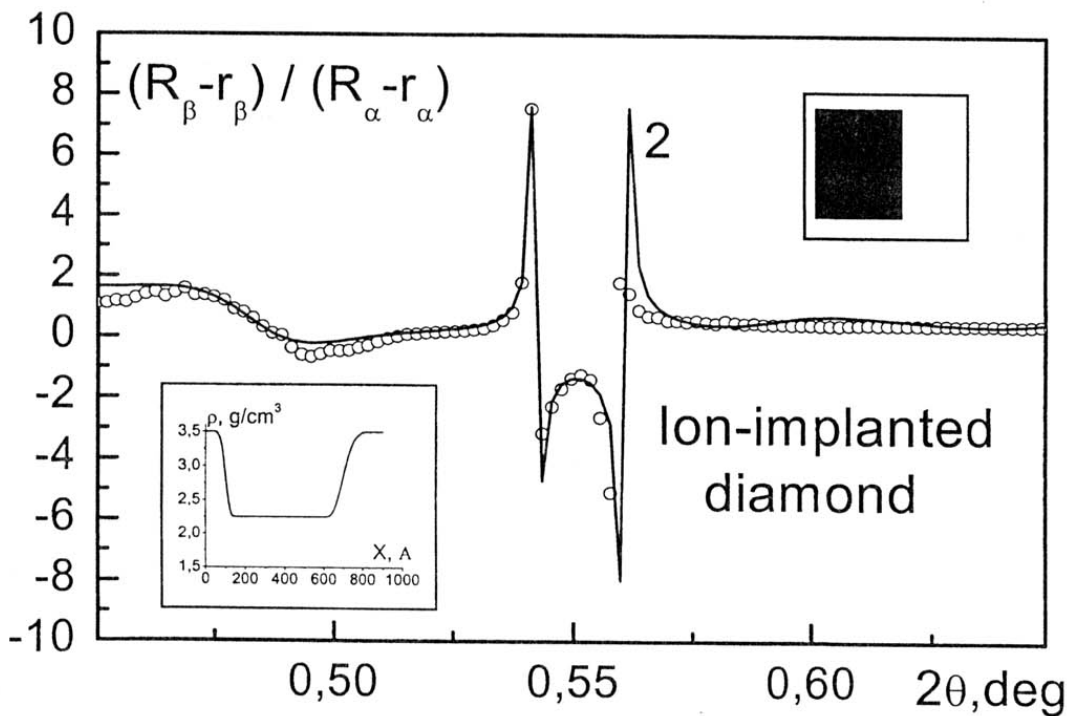


Fig. 8. Experimental (solid) and calculated (circles) angular dependences of the reflectivity ratio at wave lengths of 1,39 Å and 1,54 Å. R_{α} , r_{α} , R_{β} , r_{β} -- reflectivities of implanted and not implanted surfaces of diamond for $\text{CuK}\alpha$ and $\text{CuK}\beta$ X-ray lines. In inserts the density profile of implanted layer (at the low, left) and the sample geometry (at the top, right)

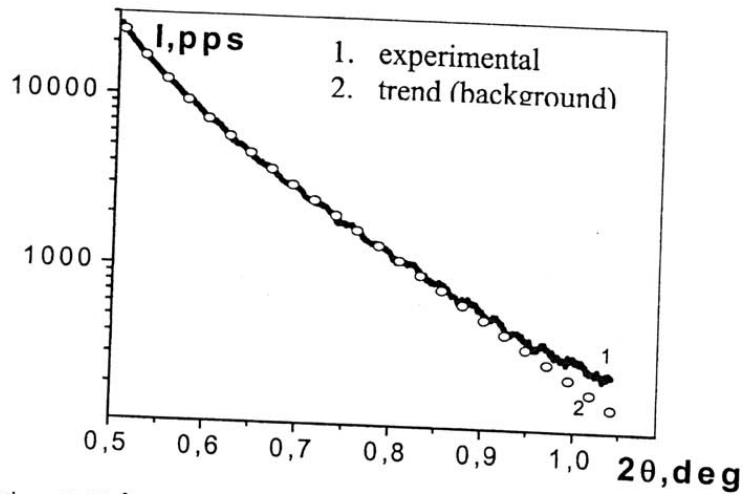


Fig. 9. The intensity of reflection at 1.54 \AA of structures « silicon on isolator » (1) and a background line (2).

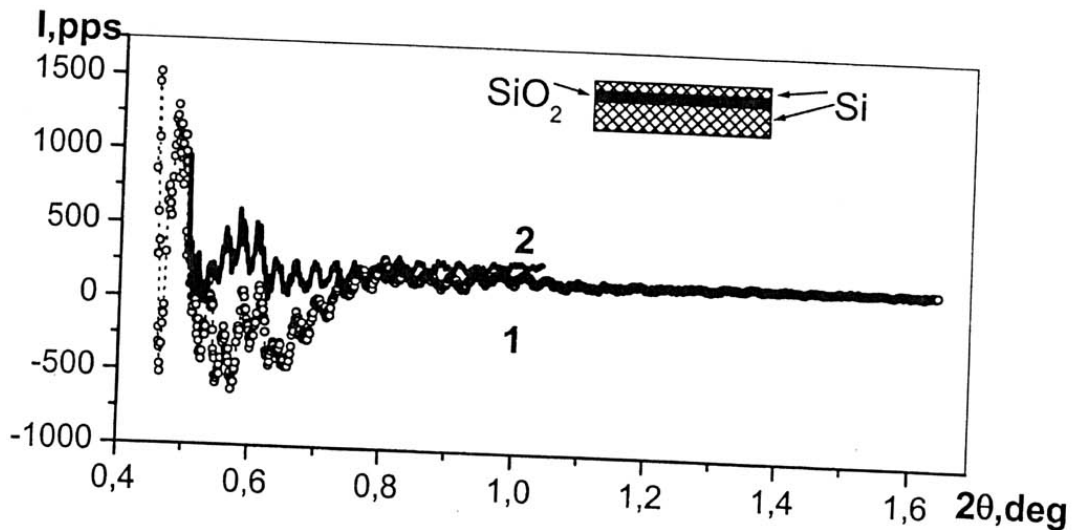


Fig. 10. The reflectivities after subtraction of a background for structures « silicon on isolator »: 1 - initial structure, 2 - the structure irradiated by γ - quanta with energy $1,33 \text{ MeV}$ (source Co^{60}) and the doze 1MR .

Another example of investigation of implanted material by two-wave reflectometry method is the study of structure «silicon on insulator». The sample was manufactured by oxygen ion bombardment of silicon and the subsequent annealing. Thus on a surface an eclectically isolated layer of silicon is formed. The experimental measured reflectivity of structure «silicon on isolator» is presented at fig. 9 (curve 1). Curve 2 at fig. 9 is the background line (trend) witch is in this case the sum of two decreasing exponents.

At Fig. 10 the reflectivity for two structures « silicon on isolator » after subtraction of a background curves are presented. The

thickness of a silicon layers in an initial sample and in the irradiated sample appeared equal 2400 Å and 3400 Å accordingly.

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REFERENCES

1. A.G. Touryanski, A.V. Vinogradov, I.V. Pirshin, *Two-channel X-ray reflectometer*, Nucl Instr & Methods in Physics Research, A 448 (2000), 184 - 187.