GAMMA-RAY DIFFRACTION STUDY OF SURFACE REGIONS IN Si SINGLE CRYSTALS AND SUBSTRATES OF CaF_2/Si MBE HETEROSTRUCTURES AND $(Ti-Ni)/Si$ MONO- AND BILAYERS SYSTEMS

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Bragg diffraction of highly-monochromatic ($\Delta\lambda/\lambda \approx 10^{-6}$), short-wavelength ($\lambda = 0.003$ nm) γ-radiation was used to study the defect structure changes arising during different treatments of Si single crystals, creation of heterostructures and neutron supermirrors based on Si substrates. The integral reflectivities (R_i^{hkl}) for several orders of reflection (hkl) as well as rocking curves were measured by ω-step scanning in transmission geometry.

A decreased Darwin's width of a reflection curve of an ideal crystal (ω_D) for the γ-ray radiation should increase the sensitivity of the diffraction method permitting measurement of weak distortions of the crystal lattice in comparison with classical experiments on X-ray and neutron diffraction.

Although the γ-ray diffraction is usually used to investigate the bulk of large crystals, nevertheless we could study narrow near-surface regions of the samples due to the fact that the incident γ-beam is practically parallel to the surface and so uses a system of crystallographic planes, which is parallel to the surface because of the small magnitude of Bragg angles. The investigated subsurface region thickness of single crystals or substrates is equal to that of the γ-beam (0.1 mm). In our particular geometry, the rocking curves were measured with the wide-open detector, that is, the integrated diffuse intensity was registered.

$CaF_2/Si(111)$ Heterostructures

We have done the investigation of CaF_2/Si heterostructures grown by molecular beam epitaxy (MBE) method with deposition rate $\approx$ 0.3 nm/min on single crystal Si substrates 0.5 mm thick [1]. The technique of R_i^{hkl} renormalization has been developed and successfully applied for the separation of the micro-defect and macro-distortion influences upon R_i^{hkl} increase during γ-radiation diffraction process in real imperfect crystals. Instead of the values of the dynamical integral reflectivity, the value R_i^{hkl}, measured at the initial specimen without CaF_2 film, was used in calculation of the strain gradient B in a framework of the general dynamical theory of scattering in deformed crystals [2] with the purpose of the renormalization of the extinction length of an ideal crystal to the extinction length of a real investigated crystal.

The strain gradient modulus ($\mid B \mid$), the bending radius (ρ) and the average meanings of longitudinal stresses at the film-substrate interface ($\bar{\sigma}$) are shown in Fig.1. as functions of thickness.

The CaF_2 layer influence results in the elastic bending of the Si substrate. The large bending radii ($\simeq$ 1 km) of rather small ($\sim$ 1 cm^2) crystals have been measured owing to a

high sensitivity of the γ-ray diffractometry method only, and the critical layer thickness was found to exist [1]. With the CaF_2 layers the thicknesses of which is more than the critical, the defect formation process in the substrate with the dislocation generation begins due to a difference between the thermal expansion coefficients and a misfit of the lattice constants for the film and the substrate.

$(Ti–Ni)/Si$ Mono- and Bilayer Systems for Neutron Supermirrors

Investigations of $(Ti–Ni)/Si$ systems for neutron supermirrors with a γ-ray diffractometer are similar to the investigations of $CaF_2/Si(111)$ heterostructures and are in progress at present.

The first results indicate that the γ-ray diffraction is sensitive to the influence of even thin layers on a thick substrate.

Integrated reflectivities R_i^{hkl} and FWHMs of the rocking curves show a different character of influence of 1×400 Åand 4×100 Å, both for Ti and Ni deposition process. The measurement of R_i^{hkl} also allows to choose and optimize the parameters of deposition.

Deposition of Ti and/or Ni layers results in the appearance of the diffuse tails on the rocking curves. Distribution of the diffuse intensity is well described in terms of the Huang and the Stokes-Wilson scattering [3] and consists of not only symmetrical but also antisymmetrical part (vacancy-type for Ti layers and interstitial-type for Ni).

Si Single Crystals

The influence of the dislocation-free doping, float-zone grown (FZ) n-type Si on γ-ray diffraction parameters has been studied during phosphorus diffusion. The diffusion was carried out in two stages: deposition on the sample surface from a PCl_3 source at a temperature $T = 1180°C$, during a time $t = 40$ min and diffusion into the sample volume at $T = 1250°C$, $t = 20$ h; the phosphorus concentration on the surface was 5×10^{20} cm^{-3}.

Phosphorus deposition did not practically influence the values of R_i^{111} and FWHM of the curves (Fig.2a). Dramatic changes were observed in the curve shape after phosphorus diffusion. Tails of the diffused γ-ray scattering appeared at angles larger than the ideal Bragg angle (Fig.2b). Appearance of the diffuse scattering can be explained by formation of new structural defects: the clusters of self-interstitials or misfit dislocations formed during diffusion of phosphorus at concentrations higher than 10^{19} cm^{-3}.

The γ-ray diffraction investigations of near-surface regions of neutron-irradiated and annealed Si single crystals have shown that the diffuse scattering was observed under some conditions of heat treatment.

For a sample annealed at $T = 850°C$, $t = 4$ h in argon flow, the diffuse intensity is higher at angles larger than the Bragg angle (Fig.3a). This shows that compressive stresses arise in near-surface layers due to formation of interstitial-type defects. In this sample a diffuse scattering has been registered at depths up to 0.3 mm.

An opposite situation was observed in the sample annealed in chlorine-containing atmosphere (concentrations of CCl_4 and O_2 were 1 and 2 mol.%, respectively) at $T = 1050°C$, $t = 4$ h. The diffuse intensity is higher at angles smaller than the Bragg angle (Fig.3b). It shows that the tensile stresses arise due to formation of vacancy defects. Thus, data on dis-

tribution of the diffuse scattering intensity permit to study formation of defects of different types in near-surface layers.

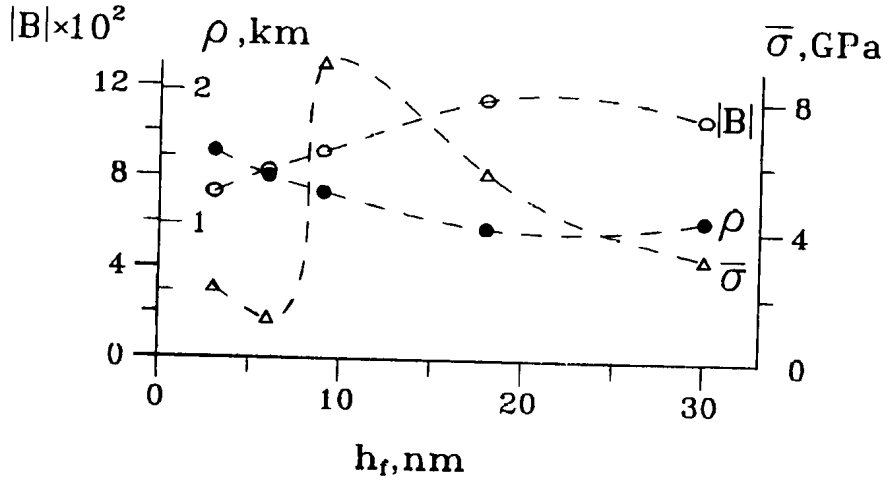

Fig.1. Strain gradient modulus (| B |), bending radius (ρ) and average meanings of longitudinal stresses at film-substrate interface ($\bar{\sigma}$) as function of film thickness

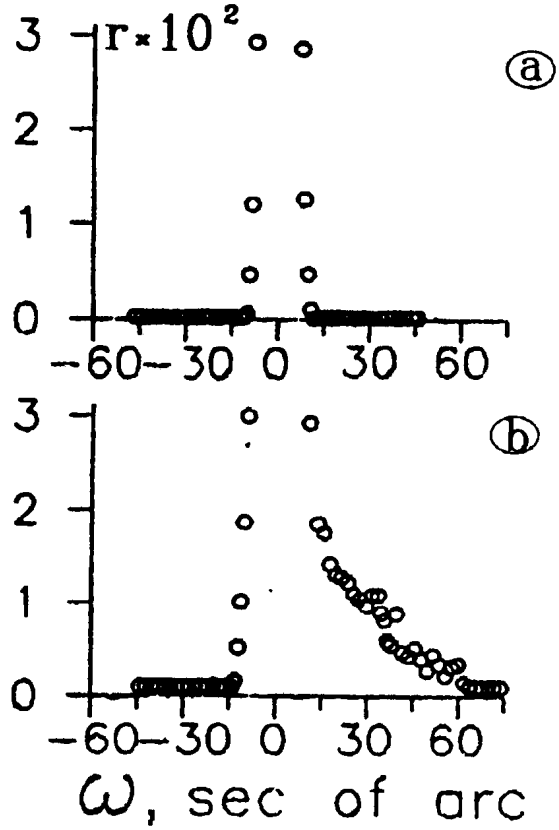

Fig.2. Rocking curves near tails of Bragg peak for (111) reflection of initial Si crystal (a) (similar curve was obtained after phosphorus deposition) and crystal after phosphorus diffusion in sample volume (b)

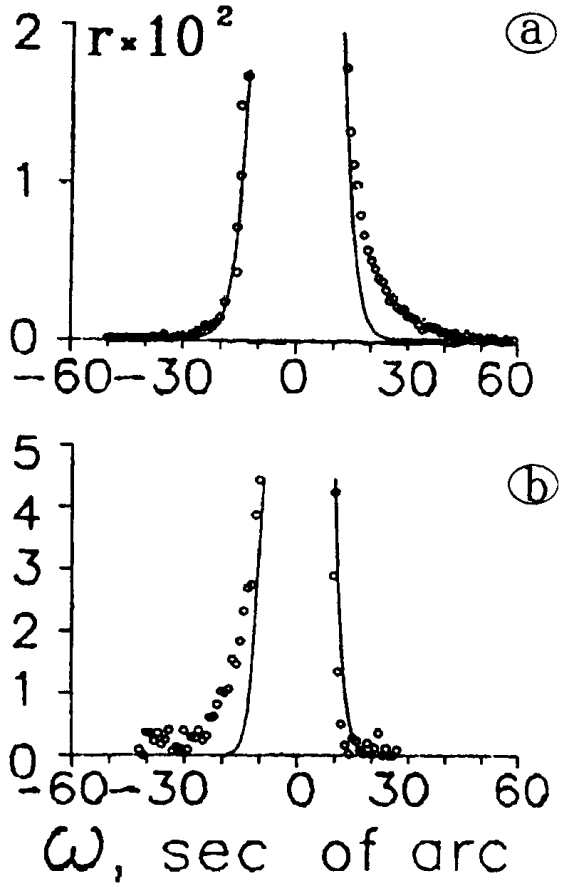

Fig.3. Rocking curves for (111) reflection of *Si* samples irradiated by thermal neutrons and annealed in argon flow (a) and in chlorine-containing atmosphere (b). Solid lines correspond to fitting by Gauss function

REFERENCES

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