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X-RAY STRUCTURAL ANALYSIS OF n-Si<Cr>, IRRADIATED WITH ALPHA PARTICLES

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Abstract. In this work, the effect of alpha particles on the crystal structure and structural characteristics of n-type silicon (n-Si) single crystals was studied using X-ray diffraction. Samples of n-Si were first doped with chromium and then irradiated with alpha particles at a dose of 6×10^{14} см$^{-2}$. It has been established that the irradiation dose used does not lead to the formation of a near-surface amorphous silicon layer. However, the obtained X-ray diffraction patterns indicate a slight deterioration in the crystallinity of silicon samples after their irradiation.

Keywords: *Silicon, chromium, doping, irradiation, alpha particles, X-ray diffraction.*

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1. Introduction

Modern technology for the production of semiconductor microelectronic devices, which is based on ion doping of the surface layer of silicon substrates, has reached the level of industrial production. An essential factor of this technology is the formation of defects under the influence of irradiation with alpha particles and, as a result, their influence on the physicochemical characteristics of silicon substrates. The study of the structural perfection of irradiated silicon samples is an important factor in the selection of the technological mode of implantation in order to obtain controlled properties of the damaged layer for solving certain practical problems.

The main field of practical use of doping silicon with radiation defects during irradiation with protons and alpha particles is to reduce the lifetime of nonequilibrium charge carriers in the structures of power semiconductor devices such as: diodes, transistors and thyristors (Kozlov & Kozlovskiy, 2001).

In this work, X-ray diffraction was used to study the effect of irradiation with alpha particles on silicon single crystals. As is known (Osadchii *et al.*, 2019), X-ray

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analysis is based on the Wulf–Bragg equation, which relates the interplanar distance (d), the angle of incidence of X-rays (θ), and the wavelength (λ):

$$\lambda = 2d sin\theta \tag{1}$$

In X-ray diffractometers, tubes with a copper anode are most often used, and for X-ray diffraction analysis, the CuKα characteristic line with a photon energy of 8 keV and a wavelength of λ = 1.54 Å is used. Monochromator crystals and β filters are used for monochromatization and attenuation of the radiation intensity of the CuKβ characteristic line (Osadchii *et al.*, 2019; Jenkins, 2000; Fults & Hay, 2011). The use of monochromator crystals and β-filters leads to additional absorption of X-ray tube radiation and reduces the efficiency of the diffractometer, to compensate for which it is necessary to increase the power of the tube power source to several kilowatts. The use of silicon semiconductor detectors with high energy resolution made it possible to abandon the stringent requirements for monochromators and filters, to simplify the optical scheme of the diffractometer, to increase the peak/background ratio, and to reduce the power of the X-ray source.

The purpose of this work is to study the effect of irradiation with α-particles on the change in the crystal structure and structural characteristics of chromium-doped n-type silicon single crystals (n-Si<Cr>) using X-ray diffraction.

2. Experimental part

For the experiments, we used n-type silicon grown by the Czochralski method with a resistivity of 40 Ω cm. The concentration of the phosphorus dopant in the initial n-Si single crystals was $7,3\times10^{13}\div7,1\times10^{15}$ cm^{-3}. Doping of silicon with chromium was carried out by the diffusion method with deposition of chromium atoms on the silicon surface in evacuated quartz ampoules at temperatures T = 900÷1250 °C for 2÷10 hours. Subsequent cooling of the samples occurred at different rates (Utamuradova *et al.*, 2019).

Doped n-Si<Cr> samples were first polished with diamond paste (grit 320-4000) and then irradiated with alpha particles with an energy of 800 keV, a current of 1÷1.5 µA, and a dose of $6,0\times10^{14}$ cm^{-2}. The samples were irradiated at the EG-5 electrostatic accelerator in the Laboratory of Neutron Physics of the Joint Institute for Nuclear Research (JINR).

The studies of n-Si samples before and after doping and irradiation were carried out on an X-ray spectrometer with a Miniflex 300/600 goniometer and a D/teX Ultra2 detector. We used CuK$_{\alpha1}$ radiation, wavelength λ = 1.541 Å, at an accelerating voltage of 40 keV and a current of 15 mA on an X-ray tube. Diffraction measurements were carried out in the Bragg-Brentano beam geometry in the 2θ range from 5° to 60° continuously at a scan rate of 10 deg/min and an angular step of 0.02°.

3. Results and their discussion

Fig. 1 shows experimental X-ray diffraction patterns of n-type single-crystal Si before and after doping with Cr and irradiation with α-particles. As can be seen in all cases, the X-ray diffraction patterns show an intense peak in the range 2θ = 28–30° with a full width at half maximum (FWHM) of 0.037–0.091°, which indicates good crystallinity of the synthesized samples.

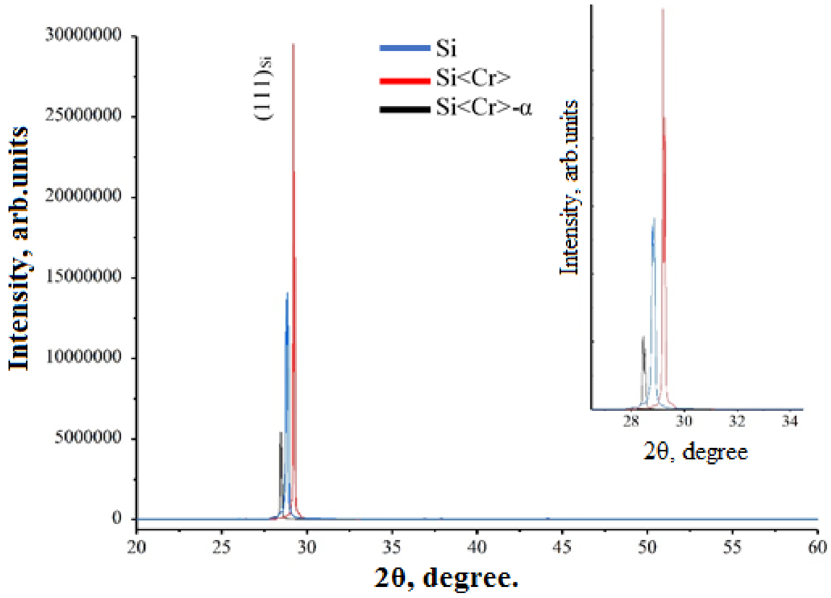

Fig.1. X-ray diffraction patterns of an n-type Si single crystal before and after doping with Cr and irradiation with α-particles

The reflection at 2θ ≈ 28.8° on the X-ray diffraction pattern of the original Si according to the Crystallography Open Database (COD) corresponds to the (111) peak of cubic silicon of space group F-43m (COD#00-080-0018). Further doping of Si samples with chromium (Si<Cr>) leads to a shift (111) of the peak in X-ray patterns towards larger angles (from 28.8° to 29.2°) and a decrease in the interplanar distance (from 3.098 to 3.055 Å), which indicates to a change in the parameters of the crystal lattice.

The unit cell parameter *a* and volume *V* calculated for the initial Si single crystal using the Material Analysis Using Diffraction (MAUD) program, which is based on the full-profile analysis of X-ray patterns by the Rietveld method, are 5.360 Å and 153.991 Å^3, respectively, which are slightly less than the theoretical values: a = 5.392 Å, V = 156.770 Å^3 (COD#00-080-0018). The subsequent introduction of chromium atoms into silicon leads to a decrease in its unit cell constant a to 5.308 Å and its volume to 149.552 Å^3. When silicon is doped with chromium, its unit cell volume is expected to increase due to their different ionic radii. However, volumetric compression occurs. This may be due to the discrepancy between the crystal lattices of silicon and the introduced impurity, which, as a result, causes lattice compression deformation. The calculated coherent scattering region (CSR, D) of Si using the MAUD program increases from 288 to 793 nm after doping, which is consistent with a decrease in the crystal lattice constant. In this case, microvoltage (ε) increases from 5×10^{-9} to 2×10^{-6}. In addition to the shift of the position of the diffraction peak from silicon for the Si<Cr> samples, its intensity doubles and the FWHM value decreases from 0.091° to 0.034°, which is in good agreement with the increase in CSR.

Diffraction peaks from other phases, for example, Cr, $CrSi_2$, etc., are not observed in the obtained X-ray patterns of Si<Cr>, although the presence of chromium in single crystals is confirmed by X-ray spectral analysis, according to which the chromium content in the samples is 19.62 at.% or 31.12 wt%. The energy dispersive spectra presented in figure 2 indicate the presence of only chromium in the composition of the samples under study, no other impurity elements were found. It should be noted that the samples after the alloying process were subjected to polishing, as a result of which elemental chromium or its compounds formed with silicon could be removed from the surface of the samples. Therefore, they cannot be detected by X-ray diffraction. In addition, the work (Utamuradova *et al.*, 2021) shows a very low solubility of Cr in Si at its content less than 6×10^{-6} by radiochemical and electrical methods from 900 to 1280 °C. Nevertheless, the results obtained in this work indicate that the cubic structure of a silicon single crystal is not modified by the introduction of Cr, and Cr ions probably form point defects in the silicon lattice (Utamuradova *et al.*, 2022). The formation of vacancies in the crystal lattice of silicon could also lead to a decrease in the volume of the unit cell, as noted above.

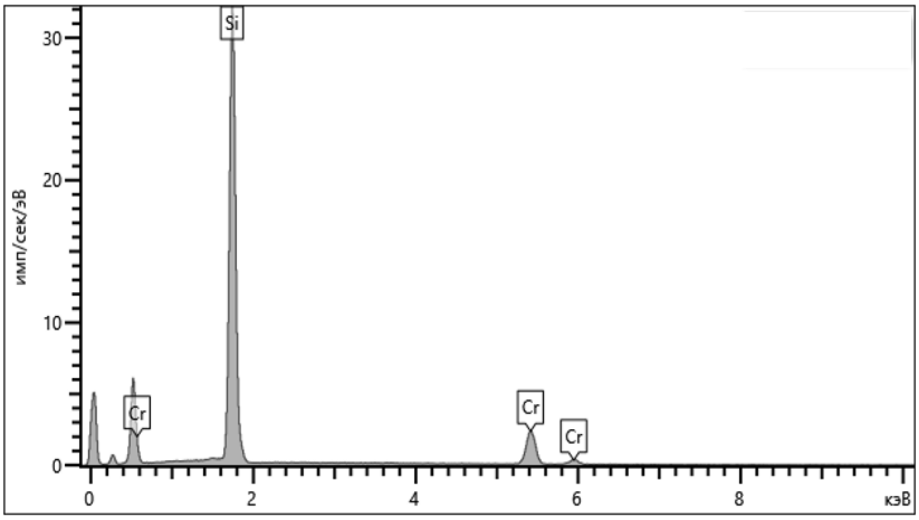

Fig. 2. Energy-dispersive spectra of silicon single crystal doped with chromium

When a Si<Cr> single crystal is irradiated with α-particles, the (111) peak in X-ray patterns shifts towards smaller angles (up to 28.4°) and the interplanar distance increases (up to 3.135 Å), as well as a decrease in its intensity by five times and broadening (from 0.034° to 0.051°). In this case, this peak is also shifted towards smaller angles compared to the initial Si single crystal, and its intensity is more than two times lower.

In Würker et al. (1974), the results of studying the effect of α-radiation on the X-ray spectra of Si single crystals are presented. It has been established that the effect of irradiation with α-particles of silicon at a dose of 20.1×10^{15} cm^{-2} at room temperature is a decrease in the intensity and broadening of its main peak on X-ray patterns, as well as the appearance of a secondary peak with a half-width and intensity comparable to the main peak for Si. When silicon crystals are irradiated with a lower dose (11.5×10^{15} cm^{-2}) and at a temperature of 77 K, only a decrease in intensity and a broadening of the main Si peak are observed. Based on the results obtained, the authors assumed that irradiation with α-particles leads to an increase in the strain field in the crystal. In addition, the damaged near-surface region of the crystal as a result of irradiation with changed crystal lattice parameters gives a secondary peak, while the main peak coming from the bulk is not noticeably affected. In reality, the crystal diffracts into two parts: from the upper damaged layers with varying lattice parameters and from the lower layers with lattice parameters almost the same as from the unirradiated sample. As is known, as a result of irradiation of silicon crystals with protons or α-particles, primary point radiation defects are formed - vacancies and related interstitial silicon atoms (Frenkel pairs) (Fahey *et al.*, 1989). When irradiated at room temperature, most of the formed Frenkel pairs disappear as a result of mutual annihilation, and the separated components of the pairs interact with each other and with impurity atoms of the crystal during their migration, creating more complex and stable secondary radiation defects. In Burgeat & Colella (1969), when studying the irradiation of silicon with α-particles in the dose range of 10^{10}–10^{13} cm^{-2} at an energy of 5.4 MeV, the presence of thin shock-absorbed layers near the sample surface was found. With the help of Raman scattering of light, it was found that the amorphization of silicon was not complete, because Along with the amorphous phase, the spectra also contained a band of vibrations of crystalline silicon. Based on the results obtained in Burgeat & Colella (1969); Skalyauh, (2005), it can be assumed that irradiation of Si<Cr> samples synthesized in this work with α-particles at a dose of 6.0×10^{14} cm^{-2} with an energy of 800 keV leads to disruption of the long-range ordering of the crystal structure Si crystal, which is expressed in a decrease in the intensity and broadening of its main peak (111). The used radiation dose of α-particles does not lead to the formation of an amorphous Si layer, since there are no additional reflections in the obtained experimental X-ray diffraction patterns.

The calculated parameter a and unit cell volume of the irradiated Si<Cr> single crystal using the MAUD program are 5.431 Å and 160.191 Å^3, respectively, which is greater than for the initial sample. Irradiation leads to the expansion of the crystal lattice, probably due to the formation, as noted above, of primary point and more complex stable secondary radiation defects. The calculated values of D and ε for Si<Cr> samples irradiated with α-particles are 113 nm and 8×10^{-5}, respectively. The dislocation density, defined as the length of dislocation lines per unit volume of the crystal, for the Si<Cr> sample and the irradiated sample, calculated by formula (2), is 0.071×10^{-6} nm^{-2} and 1.960×10^{-6} nm^{-2}, respectively.

$$\rho = 15\varepsilon / a\,D \tag{2}$$

An increase in the dislocation density indicates a decrease in the grain size and a deterioration in crystallinity, which is consistent with the results of the calculated CSR value and the decrease in the intensity of the peak in X-ray diffraction patterns.

4. Conclusion

This paper presents the results of studying the effect of α-irradiation on the X-ray spectra of an n-type Si single crystal doped with chromium. Si crystals were obtained by the Czochralski method and doped with phosphorus, the concentration of which was $7.3\times10^{13} \div 7.1\times10^{15}$ cm^{-3}. Prior to irradiation of silicon samples, they were alloyed with chromium by the diffusion method in vacuum. It has been found that the X-ray diffraction patterns of a Si single crystal before and after doping with Cr and irradiation with α-particles contain an intense peak at 2θ = 28–30°, which corresponds to cubic silicon of space group F-43m. There are no reflections associated with Cr, $CrSi_2$, etc. on

X-ray patterns of Si<Cr>, which is due to the polishing of the samples before X-ray examination, the low solubility of chromium in silicon, or an insignificant concentration of the introduced impurity. It has been established that irradiation of Si<Cr> samples with α-particles at a dose of 6.0×10^{14} cm^{-2} does not lead to the formation of a near-surface amorphous silicon layer, since X-ray spectra do not reveal the presence of additional peaks near the main (111) peak from crystalline silicon.

However, in all cases, the (111) peak shifts in the obtained X-ray patterns, so after doping with chromium, this peak shifts towards larger angles, while after further irradiation, it shifts toward smaller angles, which is associated with a change in the unit cell parameter of silicon. The unit cell constant calculated for the initial Si single crystal using the MAUD program is 5.360 Å, and after the introduction of chromium and irradiation with α-particles, it decreases to 5.308 Å, and then increases to 5.431 Å. The detected change in the intensity and broadening of the (111) peak may indicate a deterioration in the degree of crystallinity of the n-Si<Cr> single crystal as a result of irradiation with α-particles.

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