EAST EUROPEAN JOURNAL OF PHYSICS. **2.** 201-205 (2023)
DOI:10.26565/2312-4334-2023-2-21 ISSN 2312-4334

X-RAY STRUCTURAL INVESTIGATIONS OF n-Si<Pt> IRRADIATED WITH PROTONS[†]

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Received April 5, 2023; revised April 25, 2023; accepted April 26, 2023

In this work, the effect of proton irradiation on the change in the structure of silicon samples doped with platinum was studied. The samples were irradiated with protons at a dose of 9×10^{14} cm^{-2} with an energy of 600 keV and a current of 1÷1.5 μA. To determine the change in the structure after irradiation, the methods of X-ray diffraction and atomic force microscopy were used. The obtained results indicate that doping with platinum does not lead to a modification of the cubic crystal structure of silicon, but only to minor changes in the structural characteristics and surface morphology. In this case, proton irradiation of a silicon single crystal with a dose of 9.0×10^{14} cm^{-2} with an energy of 600 keV leads to the formation of defects without the formation of an amorphous near-surface layer.
Keywords: *Silicon; platinum; diffusion; doping; irradiation; proton; X-ray diffraction*
PACS: 78.30.Am

INTRODUCTION

Today in the world, much attention is paid to the development of microelectronics and semiconductor materials science. The influence of various types of radiation on semiconductor materials is being intensively studied, the defects created in semiconductors under the action of radiation, as well as their effect on the electrical conductivity of semiconductor materials and structures based on them, are being studied, ways are being sought to eliminate the influence of radiation-induced defects caused by radiation. All this is considered one of the urgent tasks of our time.

The formation of radiation defects affects the physical processes in semiconductor materials and changes the parameters of devices based on them. Irradiation of semiconductor structures with low-energy protons changes their physical properties in the near-surface region, the depth of which can reach from 0.1 μm to 1 mm. The study of the formation of radiation defects upon irradiation with low-energy protons has both scientific and practical significance [1-3].

The interest in protons is due to the wide range of processed material depths and the absence of complex radiation complexes of defects with a high annealing temperature after proton irradiation. The main factors affecting the change in the properties of semiconductors after proton irradiation are the formation of new impurities as a result of nuclear reactions, radiation defect formation, and the accumulation of hydrogen atoms [4].

The aim of this work is to study the changes in the crystal structure and morphology of the near-surface region of n-Si silicon single crystals after doping with platinum and irradiation with protons using X-ray diffraction and atomic force microscopy.

MATERIALS AND METHODS

For the experiments, we used n-type silicon grown by the Czochralski method with a resistivity of 40 Ω×cm. The phosphorus dopant concentration in the initial n-Si single crystals was $7.3\times10^{13}\div7.1\times10^{15}$ cm^{-3}. Doping of silicon with platinum was carried out by the diffusion method with deposition of platinum 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 [5–7].

Samples of n-Si were polished and then irradiated with protons with an energy of 600 keV at a current of 1-1.5 μA and a dose of 9×10^{14} cm^{-2}. The samples were irradiated at the SOKOL EG-2 electrostatic accelerator at the Research Institute of Semiconductor Physics and Microelectronics.

Doped and irradiated silicon samples were studied on an X-ray spectrometer with a Miniflex 300/600 goniometer and a D/teX Ultra2 detector. Cukα1 radiation (λ= 1.541 Å) was used at an accelerating voltage of 40 keV and a current of 15 mA on an X-ray tube. The measurements were carried out in the Bragg–Brentano beam geometry in the range 2θ = from 5° to 60° continuously at a scan rate of 10 deg/min and an angular step of 0.02°.

To study the surface morphology of silicon single crystals, an NT-MDT atomic force microscope was used.

RESULTS AND DISCUSSION

Figure 1 shows experimental X-ray diffraction patterns of single-crystal n-type Si before and after doping with Pt and irradiation with protons. As can be seen, in all cases, there is an intense peak in the X-ray patterns in the range

[†] ***Cite as***: Sh.B. Utamuradova, A.V. Stanchik, and D.A. Rakhmanov, East Eur. J. Phys. **2**, 201 (2023). https://doi.org/10.26565/2312-4334-2023-2-21

2θ ≈ 28.5–29.5°. According to the Crystallography Open Database (COD), this diffraction peak corresponds to the (111) peak of cubic silicon space group F-43m (COD#00-080-0018).

In the case of irradiation of samples with protons, the (111) peak shifts from silicon in X-ray patterns towards larger angles (from 28.80° to 29.25°), as well as an increase in its intensity by almost one and a half times and a decrease in its full width at half maximum (FWHM) (from 0.091° to 0.052°). Whereas doping with platinum leads to only minor changes in this peak. In this case, the increase in the intensity of the diffraction peak is probably associated with an improvement in the degree of crystallinity of the samples due to recrystallization of the near-surface region during the doping process at high temperature [6] or with a change in the atomic scattering coefficient due to the presence of Pt [8, 9]. Diffraction peaks from other phases in the obtained X-ray patterns of Si<Pt> samples, for example, Pt, Pt-Si, are not observed. The results obtained indicate that the cubic structure of the silicon single crystal is not modified by doping with Pt, and irradiation does not lead to the formation of an amorphous silicon layer.

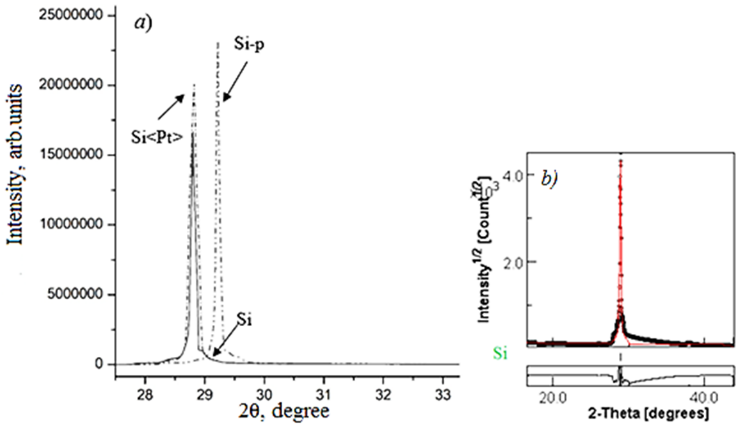

Figure 1. (a) - X-ray diffraction patterns of an n-type Si single crystal before and after doping with Pt and irradiation with protons (b) - Typical X-ray diffraction pattern Si refined by Rietveld using the MAUD program

Table 1 shows the calculated values of the structural characteristics for the samples under study using the Material Analysis Using Diffraction (MAUD) program, which is based on the full-profile analysis of X-ray diffraction patterns using the Rietveld method. The table shows that the unit cell constant a for initial Si is less than the theoretical value (a = 5.392 Å, V = 156.770 Å^3; COD#00-080-0018). The refined X-ray diffraction pattern of silicon by the Rietveld method presented in Figure 1b clearly shows a good fit of the curve to the experimental line, which confirms the data obtained. Doping of silicon with Pt leads to a further slight decrease in the constant a and, accordingly, to volume compression. In this case, the size of crystallites and microstress increase.

Table 1. Parameter (a) and unit cell volume (V), crystallite size (D), microstress (ε) for n-type Si single crystal before and after Pt doping and proton irradiation

Samples	a, Å	V, Å^3	D, нм	ε,
Si	5,360	153,991	288	5×10^{-9}
Si<Pt>	5,342	152,444	619	3×10^{-5}
Si irradiated with proton	5,486	165,108	147	5×10^{-5}

Irradiation of a single crystal of Si with protons leads to an increase in the parameter a of the unit cell, while the main peak from silicon, as noted earlier, undergoes a shift towards larger angles (up to 29.3°) and, accordingly, there is a decrease in the interplanar distance (up to 3.056 Å). The size of the crystallites decreases by half, and the microstress increases and is almost the same as for doped silicon. The dislocation density calculated by Equation (1) for a silicon single crystal increase from 0.49×10^{-9} nm^{-2} to 0.99×10^{-5} nm^{-2} as a result of irradiation. An increase in the dislocation density is associated with a decrease in the crystallite size.

$$\rho = 15\varepsilon / a\,D. \qquad (1)$$

In [10], the transformation of radiation defects in proton-irradiated n-type silicon crystals were studied using high-resolution X-ray diffraction analysis. It was shown that successive implantation of protons into silicon with an energy of 100, 200, or 300 keV at a dose of 2×10^{16} cm^{-2} causes the formation of a damaged layer 2.4 µm thick with a large crystal lattice parameter. The layer is formed simultaneously with the accumulation of intrinsic radiation defects, such as vacancies and interstitials. It was noted in [9,10] that when silicon crystals are irradiated with protons at room temperature, most of the formed Frenkel pairs disappear as a result of mutual annihilation, and the separated pair components create more complex and stable secondary radiation defects. The results obtained in this work are consistent with the results of [10-12], and it can be assumed that proton irradiation of a silicon single crystal at a dose of 9.0×10^{14} cm^{-2} with an energy of 600 keV leads to the formation of defects.

Figures 2 and 3 show AFM images with a scanning area of 10×10 μm^2 of the initial silicon single crystal and after its doping with platinum, respectively. Silicon single crystal samples (Fig. 2a) are characterized by a smooth and uniform surface in different scanning areas. On the surface of the samples, there are depressions up to 1 μm wide in the form of grooves, the appearance of which is due to mechanical processing. Small rounded protrusions are observed on the surface of the single crystal, which is confirmed by the cross-section profile (Fig. 2b), the height of which varies in the range from 2 to 7 nm in diameter. The difference in height of the silicon surface relief is 39.3 nm.

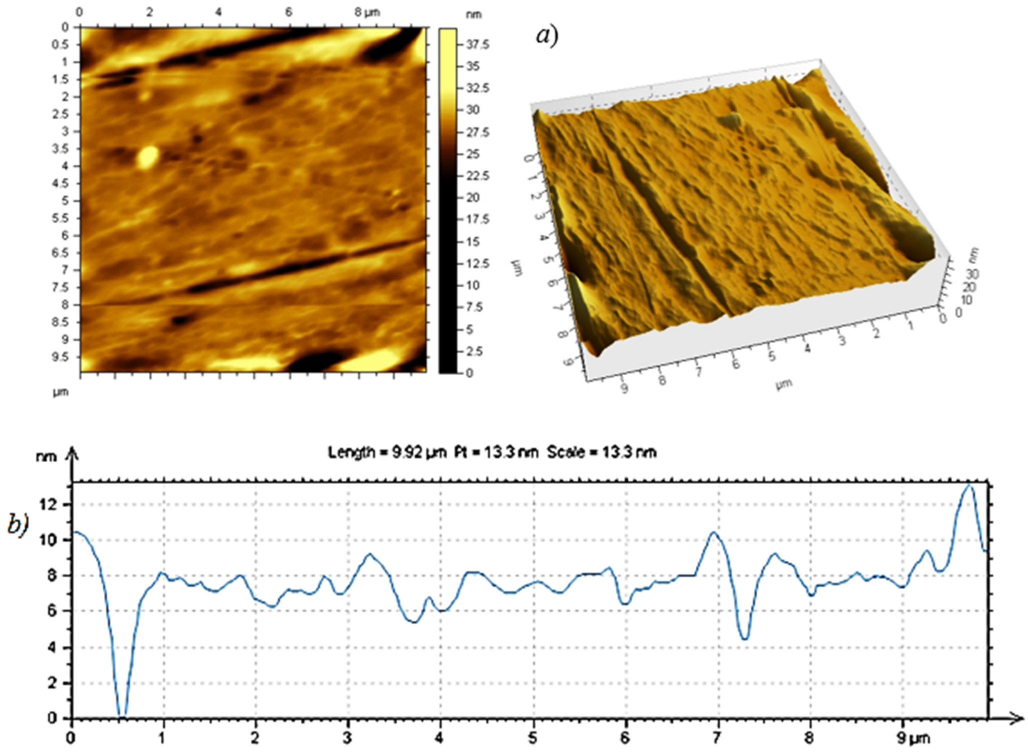

Figure 2. Typical 2D and 3D AFM images of the surface (a), section profile along the main irregularities (b) of an n-type silicon single crystal.

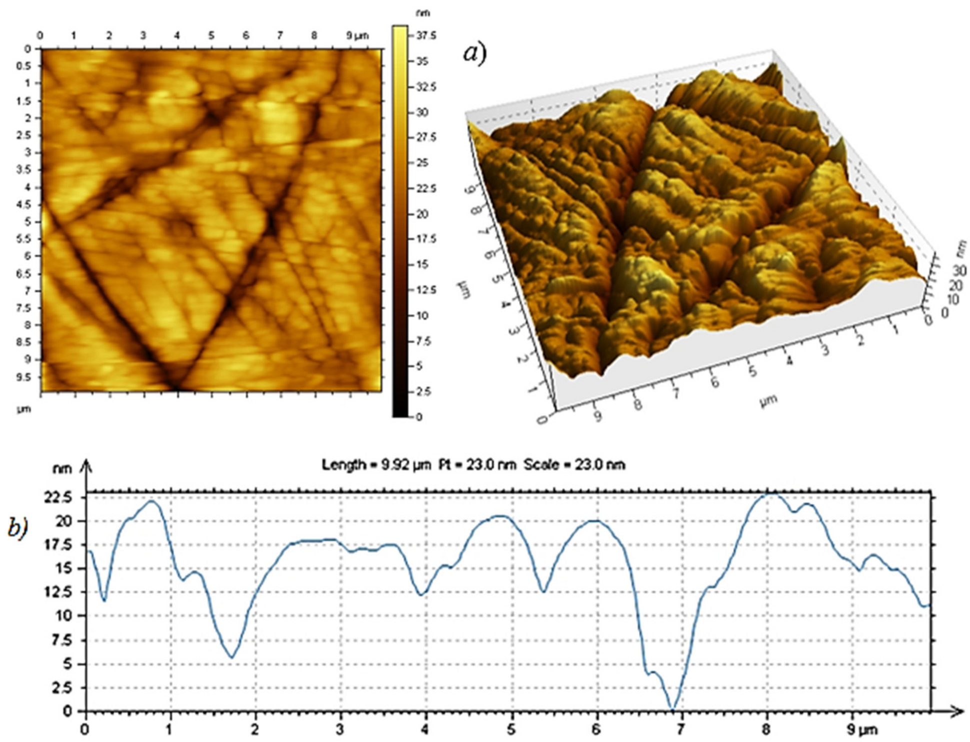

Figure 3. Typical 2D and 3D AFM images of the surface (a), cross-sectional profile (b) of an n-type silicon single crystal doped with platinum

Doping silicon single crystals with platinum leads to significant changes in their surface morphology (Fig. 3a). But at the same time, the height difference of the relief is 38.5 nm, which practically does not change as a result of the diffusion process. Similarly, to the original silicon sample, scratches are observed on the surface of doped samples, which remain after polishing. Large structural elements of different sizes are formed on the surface. According to the profile curve shown in Figure 3b, their sizes range from several hundreds of nanometers to several microns in diameter. Analysis of the 3D image showed that large structural elements are composed of smaller ones. However, this is not visible on the cross-sectional profile of the AFM image of the surface (Fig. 3b), which may be due to the difficulties in visualizing the interfaces due to the nanosize and shape of these structural elements. The observed change in the silicon surface morphology as a result of diffusion by platinum is probably due to the fact that during diffusion at 1200 °C, the collision of platinum ions with the crystal surface leads to structural surface defects of the material, which in turn contributes to the growth of material islands on the surface, their growth and agglomeration [13-15].

CONCLUSION

This paper presents the results of studying the effect of platinum doping and proton irradiation on the crystal structure and surface morphology of an n-type Si single crystal obtained by the Czochralski method. It has been found that the X-ray diffraction patterns of a Si single crystal before and after doping with Pt and irradiation with protons contain an intense peak at $2\theta = 28.5–29.5°$, corresponding to cubic silicon of space group F-43m. It was found that irradiation of samples with protons leads to a shift of the (111) peak from silicon towards larger angles, as well as an increase in its intensity and a decrease in its full width at half maximum, while doping does not lead to significant changes in this peak. The presence of phases associated with Pt, Pt-Si, etc. was not found in the Si<Pt> samples. The unit cell constant calculated for the initial Si single crystal using the MAUD program is 5.360 Å, which is slightly less than the theoretical value. Subsequent doping and irradiation lead to a decrease to 5.342 Å and an increase to 5.486 Å, respectively, of the unit cell constant, which is associated with a change in the size of crystallites and microstress. The revealed change in the morphology of the silicon surface after doping is probably due to the fact that during diffusion at a high temperature, the collision of platinum ions with the crystal surface leads to the formation of structural surface defects, which contributes to the growth of material islands on the surface, their growth and agglomeration. Thus, the obtained results indicate that doping with platinum does not lead to modification of the cubic crystal structure of silicon, but only to minor changes in the structural characteristics and surface morphology. In this case, proton irradiation of a silicon single crystal with a dose of 9.0×10^{14} cm^{-2} with an energy of 600 keV leads to the formation of defects without the formation of an amorphous near-surface layer.

Acknowledgement

The authors are grateful to the staff of the SOKOL EG-2 group at the Research Institute of Physics of Semiconductors and Microelectronics for their practical assistance with proton irradiation in this study.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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РЕНТГЕНО-СТРУКТУРНІ ДОСЛІДЖЕННЯ n-Si<Pt>, ОПРОМІНЕНОГО ПРОТОНАМИ

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У даній роботі досліджено вплив протонного опромінення на зміну структури зразків кремнію, легованого платиною. Зразки опромінювали протонами в дозі 9×10^{14} см$^{-2}$ з енергією 600 кеВ і силою струму 1÷1,5 мкА. Для визначення зміни структури після опромінення використовували методи рентгенівської дифракції та атомно-силової мікроскопії. Отримані результати свідчать про те, що легування платиною не призводить до модифікації кубічної кристалічної структури кремнію, а лише до незначних змін структурних характеристик і морфології поверхні. У цьому випадку протонне опромінення монокристала кремнію дозою $9{,}0\times10^{14}$ см$^{-2}$ з енергією 600 кеВ призводить до утворення дефектів без утворення аморфного приповерхневого шару.

Ключові слова: *кремній; платина; дифузія; допінг; опромінення; протон; рентгенівська дифракція*