OPTICAL AND SENSITIVE PROPERTIES OF NANOSTRUCTURED SILICON IRRADIATED WITH HIGH-ENERGY PARTICLES (PROTONS, α-PARTICLES, AND HEAVY IONS)

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For gas sensorics needs, an attempt has been made to modify a silicon surface by accelerated charged particles which form tracks. The influence of irradiation with 6.8-MeV protons, 27.2-MeV α-particles, and heavy ions (^{40}Ar, ^{131}Xe, and ^{209}Bi) on the optical and adsorption properties of n-Si and SiO_2/Si structures with nanopores has been studied. Scanning electron microscopy and atomic-force microscopy were used to analyze the surface morphology. The optical constants n and k of specimens before and after irradiation were determined making use of multiangle of incidence (MAI) ellipsometry. The modification of optical constants of n-Si specimens subjected to the p^+ or α-particle irradiation was found to be caused by the destruction of a near-surface layer of the material and to be accompanied by an enhancement of the surface roughness. The irradiated structures revealed a higher sensitivity to the adsorption of ammonia and acetone molecules. The optical constants of SiO_2/Si structures were shown to depend on the material porosity. The fill factor of a SiO_2 layer irradiated with ^{131}Xe and ^{209}Bi ions was calculated. The most developed pore surface was found after the irradiation of silicon with ^{209}Bi ions. Accordingly, the largest changes of optical constants were observed in specimens irradiated with bismuth ions.

1. Introduction

Semiconducting micro- and nanoporous materials find application in gas-sensitive optical sensors due to their large surface-to-volume ratio. In particular, the problem of creation of a sensor on the basis of silicon technologies stimulated an intensive study of porous silicon (por-Si). Owing to a developed pore system in por-Si/Si structures, their optical [1, 2], electrical [3], and luminescent [4] properties change, when molecules are absorbed by the surface which can be used at the sensor design. However, por-Si layers produced by electrochemical etching are characterized by a low reproducibility of their properties, and the geometrical parameters of nano- and micropores in such a porous material are widely spread. The variations of pore shapes and sizes result in changing the optical, electrical, and mechanical characteristics of the material. Therefore, when designing a sensor on the basis of por-Si, a problem to obtain devices with prescribed parameters arises. In addition, pores created by electrochemical etching often turn out to be filled with products of chemical reactions.

In this connection, a challenging problem is a creation of silicon-based sensor taking advantage of new methods of controllable nanopore formation. There are a number of techniques aimed at the fabrication of nano-structured Si: dc electrochemical etching, pulse anodizing, photochemical etching, and others. The method of ionic bombardment has a considerable advantage over the other techniques, because it allows not only nanopores of fixed dimensions and a definite geometry to be obtained on a silicon surface, but their distribution and concentration to be monitored as well [5, 6]. When irradiation is carried out with the use of accelerated high-energy particles (protons, α-particles, or heavy ions) [7], local structural variations take place near ion trajectories, and tracks are formed. This results in a modification of the Si and/or SiO_2 surface. The following etching in an buffer HF solution allows a near-surface porous layer to be created, with nanopore dimensions ranging from fractions to hundreds of microns. Hence, one can create a composite material, the physical properties of which would be governed by silicon, air, and a medium that fills the pores.

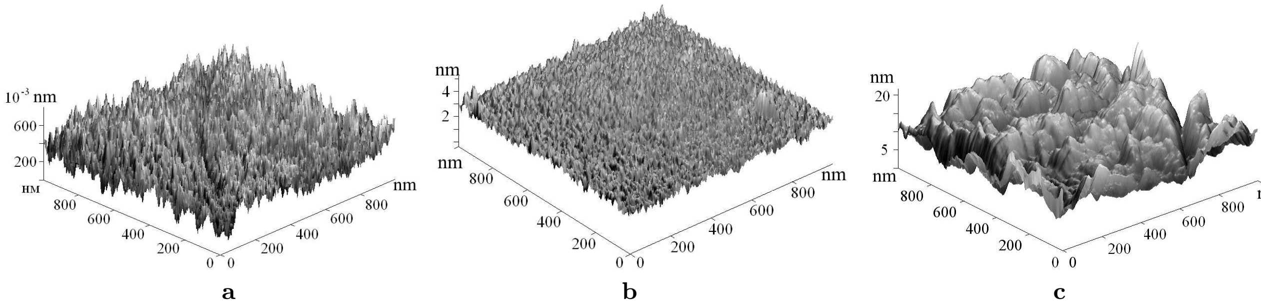

Fig. 1. AFM images of the silicon surface (a) before irradiation, (b) irradiated with protons to an exposure dose of 10^{16} cm^{-2}, and (c) irradiated with α-particles to a dose of 10^{17} cm^{-2}

This work aims at studying the optical and sensitive properties of silicon wafers irradiated with protons and helium ions, as well as SiO_2/Si structures with a nanoporous SiO_2 layer which was formed owing to the irradiation of silicon with high-energy ions.

2. Fabrication of Specimens and Methods of Their Research

The objects of our researches in this work were specimens of two types: monocrystalline Si(100) wafers of the KEF-1 type with an oxide layer about 15 nm in thickness and SiO_2/Si structures with a system of nano-sized pores in a thick layer of silicon dioxide ($d \approx 500$ nm). The specimens of the first type were irradiated with protons accelerated to 6.8 MeV or 27.2-MeV α-particles. Irradiation was carried out on an U-120 cyclotron at the Institute for Nuclear Research of the NASU. The path lengths of those particles in a Si crystal are approximately identical and amount to about 360 μm.

In order to create a system of nano-sized pores in silicon dioxide (SiO_2/Si structures), a SiO_2 layer 500 nm in thickness was grown by the thermal oxidation of the silicon substrate. For the formation of latent ionic tracks, SiO_2/Si structures were irradiated with the following fast ions: ^{40}Ar with an energy of 290 MeV to an exposure dose of 2×10^{10} cm^{-2}, ^{131}Xe with an energy of 372 MeV to a dose of 5×10^{10} cm^{-2}, and ^{209}Bi with an energy of 710 MeV to a dose of 5×10^{10} cm^{-2}. The irradiated specimens were chemically treated in a 2-% solution of hydrofluoric acid (30 g of NH_4F + 10 ml of HF + 45 ml of H_2O), which gave rise to the etching of nano-sized pores in the regions of latent ion tracks. The chemical treatment time was 6 min.

Optical and sensitive properties of specimens modified by irradiation were studied using the ellipsometry method on an LEF-3M zero ellipsometer ($\lambda = 632.8$ nm). Multiangular monochromatic measurements of the polarization angles Ψ and Δ were carried out in a vicinity of the principal angle of incidence of the structure and following the two-zone technique [8]. The measurement accuracy for polarization angles was 0.09° for Δ and 0.03° for Ψ.

At ellipsometric measurements, a specimen was located in a specially designed chamber, in which the composition of a gas environment could be changed. For studying the sensitive properties, ethyl alcohol, acetone, and ammonia were used as adsorption liquids.

The optical constants – the complex refractive index $N = n - ik$, i.e. the refractive index n and the extinction coefficient k – were determined, before and after the irradiation with high-energy particles, by solving the inverse ellipsometric equation. In this case, we used the method of minimization of the special form of the quadratic criterion function [9].

The surface morphology was analyzed with the help of an S-806 scanning electron microscope (SEM) ("Hitachi") and an atomic-force microscope (AFM) of the Dimensional^{TM} 300 type. AFM measurements were carried out in air at room temperature with the use of a silicon nitride needle probe which was attached to an elastic cantilever with a coefficient of rigidity of 0.01 – –0.6 N/m and operating in the tapping mode. The curvature radius of the cantilever tip was approximately equal to 10 nm. Such researches allow the surface microrelief to be studied and the degree of porosity in the near-surface layer of a SiO_2/Si structure to be estimated.

3. Results of Researches and Their Discussion

3.1. Irradiation with protons and α-particles

In Fig. 1, three-dimensional AFM images of silicon specimens irradiated with protons to an exposure dose of

10^{16} cm^{-2} and with α-particles to a dose of 10^{17} cm^{-2} are exhibited. The root-mean square surface roughness of silicon specimens before irradiation was about 0.5 nm. After irradiation with protons, this quantity grew to 5 nm, and after irradiation with α-particles to 20 nm. One can see that the silicon surface irradiated with protons became rougher in comparison with the surface of the initial material, but the surface roughness after irradiation with α-particles increased even more. This testifies to a considerable destruction of the near-surface Si layer.

Hard radiation invokes two processes in an irradiated solid, namely, displacements of atoms from lattice points and the atomic ionization. Primary defects are formed as a result of atomic displacements, and their number is proportional to the integral particle flux. Then the defects are transformed into more complicated radiation-induced formations. At high particle energies, cascades can develop in the crystal. The processes of formation, accumulation, and reorganization of radiation-induced defects depend on the kind of radiation, particle energy, temperature, and crystal state [10, 11]. In addition, the atomic ionization near the surface can also bring about the emergence of half-reversible variations of crystal parameters stimulated by the accumulation and the migration of charges in the insulator.

The influence of high-energy protons on solids differs essentially from that of other forms of radiation. Though point defects are the dominating type of defects generated by electrons and gamma-quanta, and complicated defects of the disordered-region (DR) type are typical of neutrons, a variety of defect types are formed at the proton slowing-down in elastic and inelastic collisions with substance atoms owing to a wide range of the recoil energy. A characteristic feature of the high-energy proton irradiation is the fact that, besides small and relatively large damaged regions (complex formations like disordered regions) in a solid, large smeared regions with a low density of defects in them are formed as well [12].

The case of irradiation with α-particles (helium ions) is somewhat different. The particles of both kinds possess comparatively equal path lengths in silicon at such radiation energies. Nevertheless, the energy release per unit volume is much higher for high-energy helium ions, because their mass and energy are larger.

There is a difference between the results of irradiation with protons and helium ions, which originates from the fact that the studied layer is enriched with vacancy complexes in the former case and is predominantly saturated with interstitial atoms in the latter one. To some extent, this is confirmed by researches of the specimen surface using the AFM method. The substantial absorption of energy in this case leads to a strong perturbation of the surface (Fig. 1) similar to the action exerted by a laser beam [13].

The optical parameters of silicon irradiated with protons to a dose of 10^{16} cm^{-2} and α-particles to doses of 10^{14}, 10^{16}, and 10^{17} cm^{-2} were determined by the method of multiangle ellipsometry. The following known optical constants for single-crystalline Si were used at calculations: $N = 3.882 - 0.019i$ at the light wavelength $\lambda = 632.8$ nm [14]. The values of n and k obtained by us for silicon before irradiation and taking errors into account agreed with the results reported in work [14]. For the structure irradiated with protons to a dose of 10^{16} cm^{-2}, we obtained $N = 3.842 - 0.019i$. A reduction of the refractive index can evidence for the loosening of the near-surface silicon layer and its destruction. We suppose that this phenomenon is associated with the aggregation of predominantly vacancy defects.

After the irradiation with α-particles to exposure doses of 10^{14} and 10^{16} cm^{-2}, the optical parameters of the specimens under investigation did not change in comparison with classical data [14], but for a specimen irradiated to a dose of 10^{17} cm^{-2}. This fact agrees with the data of work [15], in which the minimal doses necessary for the formation of pores to occur in silicon after the irradiation with α-particles was found to be 5×10^{15} and 10^{16} cm^{-2} for the implantation energy ranging from 20 to 300 keV, respectively.

The optical constants of Si irradiated to a dose of 10^{17} cm^{-2} are $N = 4.102 - 0.020i$, being close to those of amorphous ($n = 4.560$) and polycrystalline ($n = 3.987$) silicon [16, 17]. A substantial growth of the refractive index in this case is evidently originated from a high concentration of defects in the near-surface layer of silicon which are probably of the interstitial type. Since the effect manifests itself only at high exposure doses of α-particles, this testifies to the considerable disordering of a matrix structure.

The optical parameters of the rough defective Si surface formed by irradiation turned out sensitive to the molecular adsorption. In Fig. 2, the angular dependences $\Psi(\varphi)$, with φ varying in a vicinity of the Brewster angle, are presented for silicon specimens which have been subjected to the action of ethanol, acetone, and ammonia vapors for 3 h after having attained the saturation. After holding the specimens in air, they restored their angular dependences $\Psi(\varphi)$ to initial values (desorption).

The optical constants of n-Si before and after the irradiation with protons to an exposure dose of 10^{16} cm^{-2}

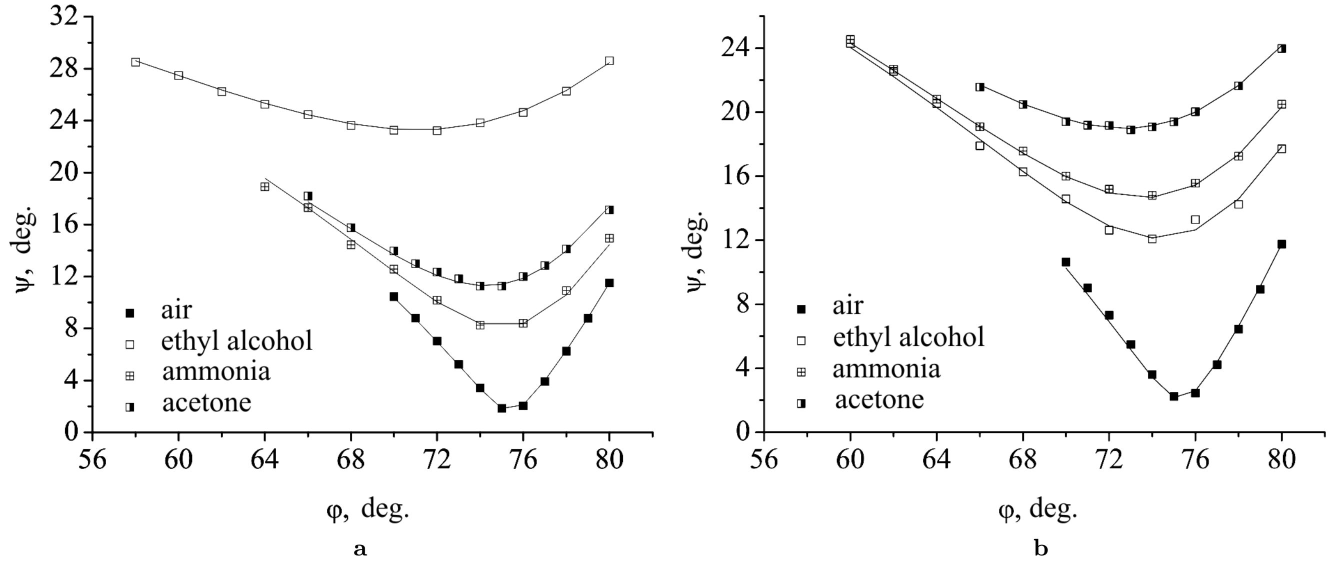

Fig. 2. Dependences $\Psi(\varphi)$ for n-Si before (a) and after the irradiation with protons to a dose of 10^{16} cm^{-2} (b) measured in air and after the adsorption of saturated liquid vapors at room temperature. Symbols correspond to experimental data, curves are fitted theoretical dependences calculated by minimizing the criterion function

T a b l e 1. Sensitivity parameters with respect to the angle Ψ for n-Si before and after the irradiation with protons to a dose of 10^{16} cm^{-2} concerning the adsorption of vapors of various substances

	n-Si		n-Si, 10^{16} cm^{-2}	
	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})
ethyl alcohol	21.40	20.0×10^4	9.86	9.21×10^4
ammonia	6.45	2.30×10^4	12.60	4.50×10^4
acetone	9.45	1.72×10^4	16.70	3.00×10^4

were calculated in the framework of the model "an oxide SiO_2 film on silicon". The optical constants, which were obtained for Si ($N = 3.842 - 0.020i$), agree with the data of work [14] obtained for the light wavelength $\lambda = 632.8$ nm.

The sensitivity of ellipsometric parameters of the structures concerned to the adsorption of vapors of various substances can be estimated numerically by calculating the parameter

$$S_\Psi = \frac{\Delta\Psi}{\rho_{\rm sat}} = \frac{\Psi_{\rm sat}^{\rm min} - \Psi_0^{\rm min}}{\rho_{\rm sat}}, \qquad (1)$$

where $\rho_{\rm sat}$ is the saturated vapor density, and $\Psi_{\rm sat}^{\rm min}$ and $\Psi_0^{\rm min}$ are the depths of minima in the angular dependences $\Psi(\varphi)$ measured, when the vapors are saturated or not, respectively. The calculated values of the sensitivity parameter with respect to the angle Ψ for n- Si specimens before and after the irradiation with protons to a dose of 10^{16} cm^{-2} are quoted in Table 1.

The researches of specimens carried out in various gas environments revealed different irradiation effects on the sensitivity S_Ψ of ellipsometric parameters, provided that the molecules of ethyl alcohol, ammonia, or acetone are available in the air. For instance, after irradiating the structures with protons, the growth of their sensitivity to ammonia and acetone molecules, and its reduction to ethanol ones were observed. This can be resulted from several reasons: an enhancement of the surface roughness; the emergence of dangling bonds, when the proton bombardment "looses" the surface; and the features of the physico-chemical interaction between protons and molecules of saturated vapors.

3.2. Irradiation with fast ions

It is known that structures obtained on the basis of porous SiO_2/Si structures, in which the developed surface is created by nano-sized pores, the diameter of which

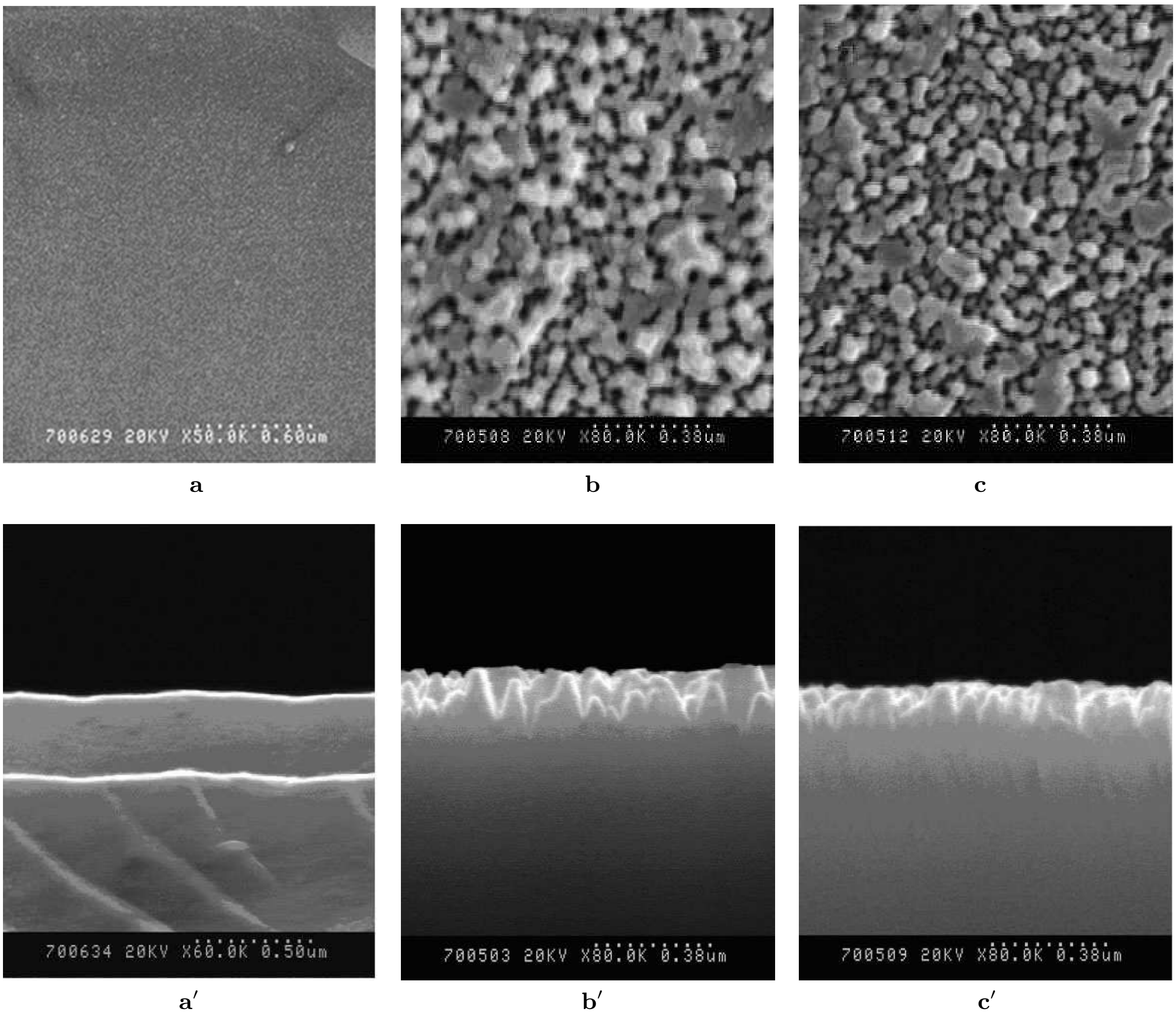

Fig. 3. SiO_2/Si specimens irradiated with fast Ar (a, a'), Xe (b, b'), and Bi (c, c') ions, and treated in a 2% of HF acid. The upper row corresponds to the surfaces, the lower to the transverse cross-sections

is larger than the characteristic size of molecules, can also be effective optical sensor controls.

In Fig. 3, the photographs of the surface and the transverse cross-section of SiO_2/Si specimens irradiated with ^{40}Ar, ^{131}Xe, and ^{209}Bi fast ions and treated in a 2-% solution of HF acid are depicted. As one can see, the irradiation of SiO_2/Si structures with ^{40}Ar ions brings about an insignificant etching of tracks in the SiO_2 layer. The authors of work [18] showed that, when ^{40}Ar ions with an energy of 290 MeV are used for irradiation of SiO_2, the matrix temperature in the regions along ion trajectories is insufficient for the formation of a melted phase and, accordingly, for the formation of latent tracks. Even after the specimens had been treated in a HF-based buffer etchant for 10 min, no etching of ion tracks was observed. It is associated with the fact that the formation of ion tracks in SiO_2/Si structures is strongly influenced by the ionic mass. The irradiation of specimens with fast ^{209}Bi heavy ions turned out the most effective for the formation of nanopores.

The analysis of electron diffraction patterns of specimens with a porous structure testifies that the structures have non-uniform degree of volume filling with pores (Fig. 3). The calculation of the effective filling of a porous substance in the surface phase showed that the filling factor f of a SiO_2 layer amounts to 0.6 ± 0.2 in ^{131}Xe-irradiated structures and to 0.52 ± 0.2 in ^{209}Bi-irradiated ones. Additionally, we have calculated the statistical distribution of f over the surface of specimens (Fig. 4). The obtained results reveal a non-uniformity

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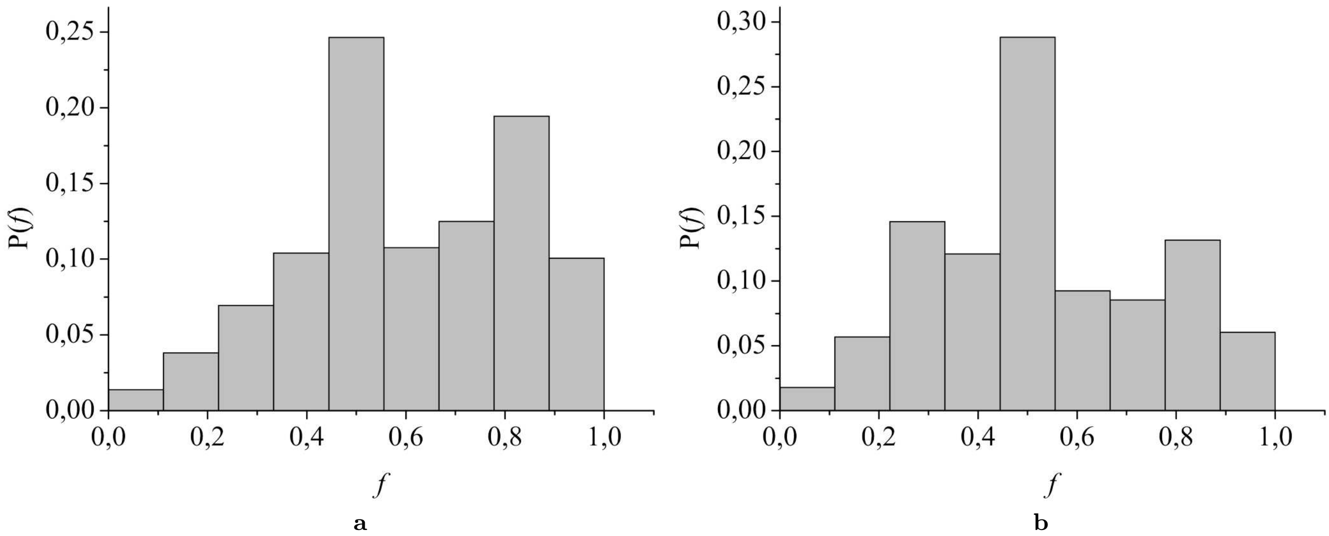

Fig. 4. Statistical distributions of the filling factor for SiO_2/Si structures irradiated with ^{131}Xe (*a*) and ^{209}Bi (*b*)

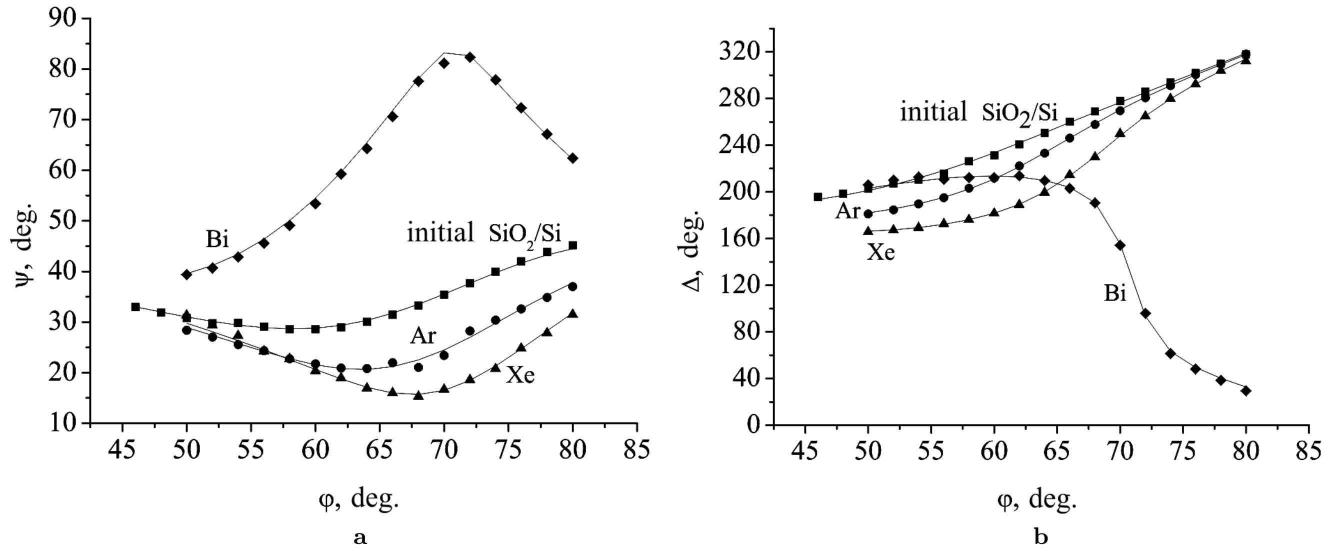

Fig. 5. Angular dependences $\Psi(\varphi)$ (*a*) and $\Delta(\varphi)$ (*b*) for SiO_2/Si structures irradiated with fast ^{40}Ar, ^{131}Xe, and ^{209}Bi ions and treated in a 2-% solution of HF acid. Symbols correspond to experimental data, and curves are theoretical dependences calculated by minimizing the criterion function

of distributions, which testifies to the presence of several types of porosity differing from one another in the pore density. For instance, two maxima were observed for a specimen irradiated with ^{131}Xe (Fig. 4,*a*), and three maxima for a specimen irradiated with ^{209}Bi (Fig. 4,*b*). This means that the most developed pore surface was formed after the irradiation with ^{209}Bi.

As was done in the previous experiments, we used the ellipsometry method to calculate the optical parameters (n, k) and the thicknesses d of SiO_2 layers in the SiO_2/Si structures. It is known that the accuracy of the results of ellipsometric researches (Fig. 5) is affected, first of all, by the adequacy of the selected model with respect to an object under investigation. When choosing a model, we used the results of auxiliary SEM researches of structure surfaces (Fig. 3).

For the calculation of optical parameters of the structures irradiated with ^{40}Ar ions, we used a single-layer model, namely, a porous SiO_2 layer on the semiinfinite Si substrate. For the structures irra-

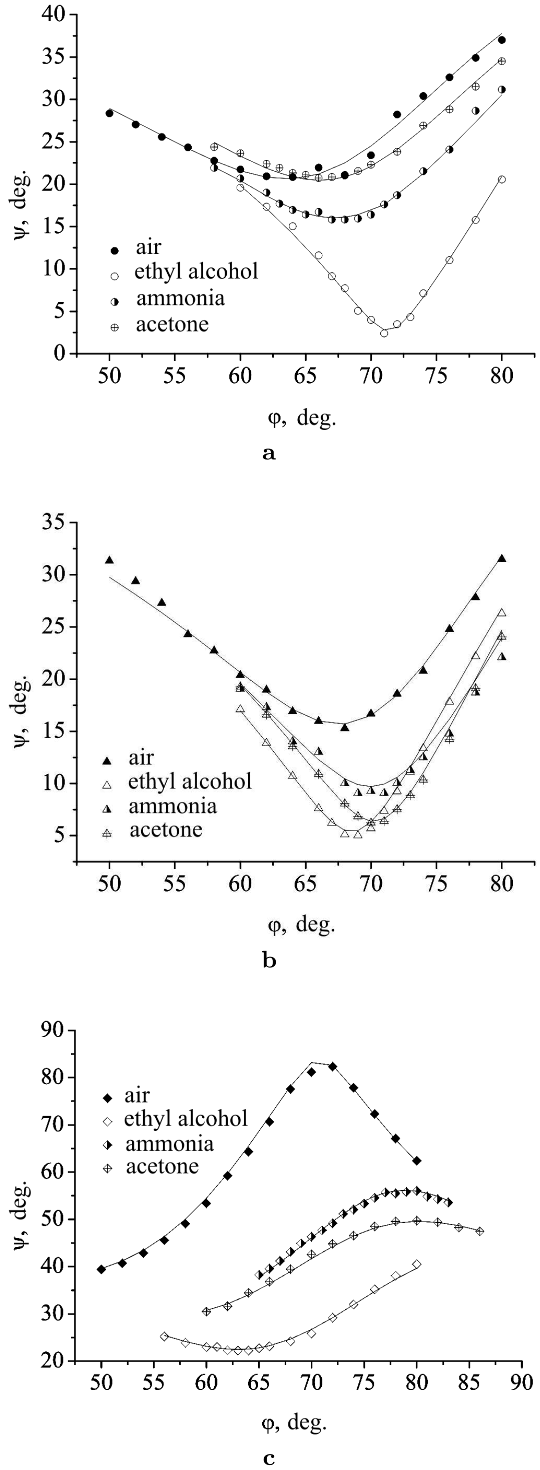

Fig. 6. Angular dependences $\Psi(\varphi)$ for SiO_2/Si structures irradiated with ^{40}Ar (*a*), ^{131}Xe (*b*), and ^{209}Bi (*c*) ions. Symbols correspond to experimental data, curves are theoretical dependences calculated by minimizing the criterion function

diated with ^{131}Xe and ^{209}Bi ions and, afterward, treated in an etchant, the non-uniform etching of nanopores in SiO_2 layer was observed. The upper SiO_2 layer (columns (1) in Table 2) was characterized by a higher porosity than the lower sublayer (columns (2)). Therefore, a two-layer model – porous $SiO_2/SiO_2/Si$ – was selected in such cases (Table 2).

In the framework of the selected model for every SiO_2/Si structure and using the corresponding experimental dependences $\Psi(\varphi)$ and $\Delta(\varphi)$ for polarization angles, the optical parameters and the oxide layer thickness were calculated (Table 2). The optical parameters of the near-surface oxide layer were found to be less than the relevant parameters of raw SiO_2, which is connected with the porosity of this composite material.

The ellipsometry technique was also used to study the influence of the adsorption from gas-like environments on the optical parameters of porous structures. In Fig. 6, the angular dependences of the ellipsometric angle $\Psi(\varphi)$ for irradiated SiO_2/Si structures are exhibited. They were measured in air, at room temperature, and after the molecules of ethyl alcohol, acetone, or ammonia had been adsorbed. The results of calculations of the sensitivity parameter S_Ψ are quoted in Table 3. The results testify that the largest variation of optical parameters at the adsorption is observed in structures with SiO_2 oxide irradiated with ^{209}Bi ions. This can be explained by the formation of a more developed surface in comparison with the case of specimens irradiated with ^{40}Ar and ^{131}Xe ions. The values obtained for the sensitivity agree with the porosity data obtained by the electronic microscopy method (Figs. 3 and 4).

4. Conclusions

Irradiation of silicon with high-energy particles was found to result in its nano-structuring, a modification of its optical constants, and an enhancement of its surface roughness. It was shown that, after the proton irradiation to a dose of 10^{16} cm^{-2}, a reduction of the silicon refractive index is observed, which is explained by

T a b l e 2. Optical parameters of specimens determined by the method of multiangle ellipsometry

	$n_{SiO_2\text{-por}}$ (1)	$k_{SiO_2\text{-por}}$ nm (1)	$d_{SiO_2\text{-por}}$, (1)	n_{SiO_2} (2)	k_{SiO_2} (2)	d_{SiO_2}, nm (2)
initial	1.436	0.000	500.00	–	–	–
Ar	1.202	0.034	340.00	–	–	–
Xe	1.166	0.028	250.00	1.391	0.018	140.00
Bi	1.119	0.014	120.00	1.477	0.041	360.00

T a b l e 3. Sensitivity parameters with respect to the angle Ψ for SiO_2/Si structures irradiated with ^{40}Ar, ^{131}Xe, and ^{209}Bi ions to a dose of 10^{16} cm^{-2} concerning the adsorption of vapors of various substances

	Ar		Xe		Bi		SiO_2/Si initial	
	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})	$\Delta\Psi$, deg.	S_Ψ, deg./(g · cm^{-3})
ethyl alcohol	18.03	16.85×10^4	3.8	3.55×10^4	60.7	56.7×10^4	2.8	2.62×10^4
ammonia	4.83	1.73×10^4	5.6	2.0×10^4	27.2	9.71×10^4	0.4	0.14×10^4
acetone	0.33	0.06×10^4	8.9	1.61×10^4	33.6	6.09×10^4	3.5	0.63×10^4

the destruction of the near-surface Si layer due to, most likely, the accumulation of vacancy defects. On the contrary, the irradiation of the Si surface with α-particles gives rise to an increase of n, which testifies to a compaction of the near-surface layer. The largest growth of the sensitivity parameter S_Ψ due to the adsorption of ammonia or acetone was found for proton-irradiated specimens.

It was demonstrated that the etching of SiO_2/Si structures irradiated with high-energy ^{40}Ar, ^{131}Xe, and ^{209}Bi ions in a 2-% solution of HF acid leads to the formation of pores, the size and the depth of which depend on the kind and the energy of ions. For example, when irradiating with 290-MeV ions of ^{40}Ar to a dose of 2×10^{10} cm^{-2}, only the insignificant etching of tracks in silicon dioxide was observed. For structures irradiated with ^{131}Xe (372 MeV, a dose of 5×10^{10} cm^{-2}) and ^{209}Bi (710 MeV, a dose of 5×10^{10} cm^{-2}) ions, the nonuniform etching of nano-pores took place, with the upper layer appearing more porous than the lower sublayer of silicon dioxide. It was shown that, in ^{209}Bi-irradiated structures, the upper porous SiO_2 layer was the most developed, with the pore diameter ranging from 20 to 25 nm.

The best sensitivity to gases was revealed for the structures irradiated with ^{209}Bi ions, the surface of which turned out to be the most developed. The advantages of a Si surface modification by charged nuclear particles for gas sensor devices based on nano-silicon structures has been demonstrated.

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ОПТИЧНІ ТА СЕНСОРНІ ВЛАСТИВОСТІ НАНОСТРУКТУРОВАНОГО КРЕМНІЮ, ОПРОМІНЕНОГО ВИСОКОЕНЕРГЕТИЧНИМИ ЧАСТИНКАМИ (ПРОТОНИ, α-ЧАСТИНКИ, ВАЖКІ ІОНИ)

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Р е з ю м е

Для потреб газової сенсорики зроблено спробу модифікувати поверхню кремнію прискореними зарядженими частинками, які утворюють треки. Досліджено вплив опромінення 6,8 МеВ протонами, 27,2 МеВ α-частинками та важкими іонами (^{40}Ar, ^{131}Xe, ^{209}Bi) на оптичні та адсорбційні властивості n-Si та SiO_2/Si структур з нанопорами. Морфологію поверхні досліджено методами скануючої електронної мікроскопії та мікроскопії атомних сил. Оптичні константи (n, k) зразків до та після опромінення визначено методом багатокутової монохроматичної еліпсометрії. Встановлено, що зміна оптичних констант зразків n-Si при опроміненні p^+ та α-частинками супроводжується збільшенням шорсткості поверхні та зумовлена деструкцією приповерхневого шару матеріалу. Опромінені структури виявили більшу чутливість щодо адсорбції молекул аміаку та ацетону. Показано, що оптичні константи SiO_2/Si структур залежать від величини пористості матеріалу. Розраховано фактор заповнення матеріалу в шарі SiO_2 в опромінених ^{131}Xe та ^{209}Bi структурах. Встановлено, що найбільш розвинена поверхня пор утворилась після опромінення іонами ^{209}Bi. Відповідно найбільші зміни оптичних констант спостерігаються у зразках, опромінених вісмутом.