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Change in the surface level of germanium with the formation of a thin porous layer during irradiation with bismuth ions

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In this work, the nature of changes in the surface level of a single-crystal c-Ge substrate irradiated by $^{209}\mathrm{Bi}^{++}$ ions with an energy of $E = 36\,\mathrm{keV}$ during the formation of thin surface nanoporous Ge layers with increasing ion dose was analyzed. Dose values varied from $2.0\cdot10^{14}$ to $4.0\cdot10^{16}\,\mathrm{ion/cm^2}$. Observation of the surface sample morphology was carried out using high-resolution scanning electron and probe microscopy. It was found that at low doses of up to $1.0\cdot10^{15}\,\mathrm{ion/cm^2}$, ion sputtering of the sample surface occurs, forming a layer consisting of open surface pores in the form of pits. With increasing dose, this layer is replaced by swelling of the surface with the formation of a porous spongy nanowires structure.

Keywords: nanoporous germanium, ion-enhanced deposition, ion implantation.

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Classical ion sputtering and etching [1], focused ion-beam processing [2], and ion implantation [3] methods based on the effects of interaction of accelerated ions with matter are the cornerstone of modern ion-beam technology. Ion sputtering, which leads to destruction and removal of a part of the irradiated surface, is normally carried out at low energies E on the order of $0.5-30\,\mathrm{keV}$ [4]. In the case of low-energy ion implantation ($E \leqslant 300\,\mathrm{keV}$), which involves deep penetration of impurity ions into the matrix, a surface modification similar to that induced by ion sputtering is to be expected. However, the processes characterizing ion sputtering and ion implantation are often discussed independently in scientific literature. At the same time, the governing physical effects manifested in collisions of primary particles with matrix atoms and their deceleration are fundamentally similar. The existing differences between these processes are specified by the conditions and parameters of ion irradiation, such as energy (E) and type of ions, dose (D), current density (J), temperature of the irradiated substrate (T_{subst}), its structure, chemical composition, etc.

It can be noted, e.g., that the number of published papers focused on the specifics of ion irradiation at low E close to $\sim 30\,\mathrm{keV}$ (i.e., at the boundary between the effects of ion sputtering and ion implantation) for semiconductor Ge [5], which is a material that is used widely in micro- and optoelectronics, is very limited. This is the reason why the effects of surface modification of single-crystal c-Ge in experiments on low-energy irradiation at room temperature are examined in the present study as functions of D. The Ge matrix is specific in that both its sputtering and the formation of a swelling layer of nanoporous germanium (NPGe) may occur under certain conditions in the process of interaction with ions [6]. The results of examination of possible surface modification scenarios with c-Ge substrates used as an example are relevant, since the processes of sputtering and pore formation under ion irradiation were also observed for several other semiconductors, such as GaAs [7], GaN [8], GaSb [9], and Si [10].

Heavy $^{209}\mathrm{Bi}^{++}$ particles with $E = 36\,\mathrm{keV}$, which are characterized by a small penetration depth and a significant level of destruction of the irradiated target, are used as primary ions for c-Ge irradiation in the present study. In practice, irradiation with these ions is traditionally used to investigate various radiation-induced effects at low E both in Ge [4,11,12] and in other semiconductors, such as Si, GaP, GaAs, GaSb, InAs, InSb, InP [1,4].

Polished GDG-45 c-Ge substrates with a thickness of $0.5\,\mathrm{mm}$ and crystallographic orientation (111) were used in experiments. Irradiation with $^{209}\mathrm{Bi}^{++}$ ions with $E = 36\,\mathrm{keV}$, D from $2.0\cdot10^{14}$ to $4.0\cdot10^{16}\,\mathrm{ion/cm^2}$, and $J = 5\,\mu\mathrm{A/cm^2}$ was carried out using the ILU-3 ion accelerator. The c-Ge substrate was kept initially at room temperature, and ions were incident normally on the sample through a surface mask (copper grid with $40\,\mu\mathrm{m}$ square cells). An original design of a heating element for the accelerator vacuum chamber was used to monitor the surface temperature of the irradiated sample [13]. The local morphology and surface structure of implanted c-Ge were examined using a Merlin (Carl Zeiss) scanning electron microscope (SEM) and a Dimension FastScan (Bruker) scanning probe microscope (SPM). The SEM was fitted with an HKL NordLys (Oxford Instruments) electron backscatter diffraction detector.

The distribution of implanted impurity in the volume of Ge irradiated with $^{209}\mathrm{Bi}^{++}$ ions with $E = 36\,\mathrm{keV}$ was modeled using DYNA. The physical principles of calculation implemented in this software were detailed in [14]; it takes into account dynamic variations of the chemical composition of the surface layer of the irradiated matrix and its ion

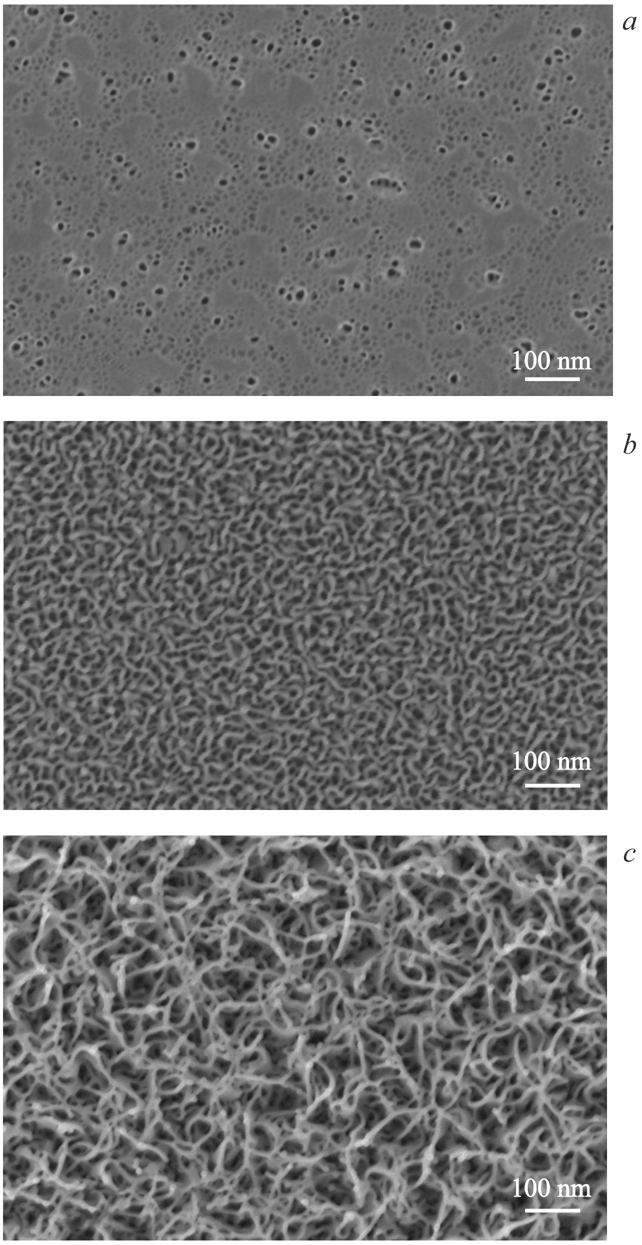

Figure 1. SEM images of the surface of *c*-Ge implanted with $^{209}\mathrm{Bi}^{++}$ ions with $E = 36\,\mathrm{keV}$ at $J = 5\,\mu\mathrm{A/cm}^2$ and $D = 1.3 \cdot 10^{15}$ (*a*), $6.0 \cdot 10^{15}$ (*b*), and $4.0 \cdot 10^{16}\,\mathrm{ion/cm}^2$ (*c*).

sputtering. Calculations revealed that the distribution of impurity $^{209}\mathrm{Bi}^{++}$ atoms within the volume of Ge follows a Gaussian curve with a maximum at depth $R_p \sim 14.4\,\mathrm{nm}$ and an ion range spread relative to R_p of $\Delta R_p \sim 4.9\,\mathrm{nm}$. Thickness $h = R_p + 2\Delta R_p$ of the doped layer is close to 24.2 nm.

It follows from the example SEM images that the irradiated Ge surface ceases to be smooth at dose D slightly higher than $1.0 \cdot 10^{15}\,\mathrm{ion/cm}^2$ and is characterized by a Bi:NPGe structure with numerous pits (pores) distributed uniformly over the sample (Fig. 1, *a*). As D increases, the pitted Bi:NPGe structure is transformed into a spongy structure ($D = 6.0 \cdot 10^{15}\,\mathrm{ion/cm}^2$) consisting of thin intertwined nanowires (Fig. 1, *b*). With a further increase in D to $4.0 \cdot 10^{16}\,\mathrm{ion/cm}^2$ (Fig. 1, *c*), the morphology of the irradiated Bi:NPGe layer remains qualitatively unchanged, although the distance between nanowires and the free volume in the porous material increase noticeably.

SPM measurements were carried out in order to estimate the change in surface level (h) relative to the non-irradiated *c*-Ge substrate as a result of sputtering (h_{sputt}) or swelling (h_{swell}) of the Bi:NPGe sample. An example SPM image of a fragment of the surface in the mask cross-wire region formed at $D = 4.0 \cdot 10^{15}\,\mathrm{ion/cm}^2$ is shown in Fig. 2, *a*. The dark cross-wire region corresponds to the surface of the original *c*-Ge substrate that is covered by the mask during implantation, and the light area represents the Bi:NPGe structure. It follows from the figure that noticeable surface swelling (an increase in volume of the irradiated Bi:NPGe material relative to the initial Ge) occurs under the given conditions of ion irradiation; the h_{swell} value increases, and a step is formed. Figure 2, *b* shows experimental

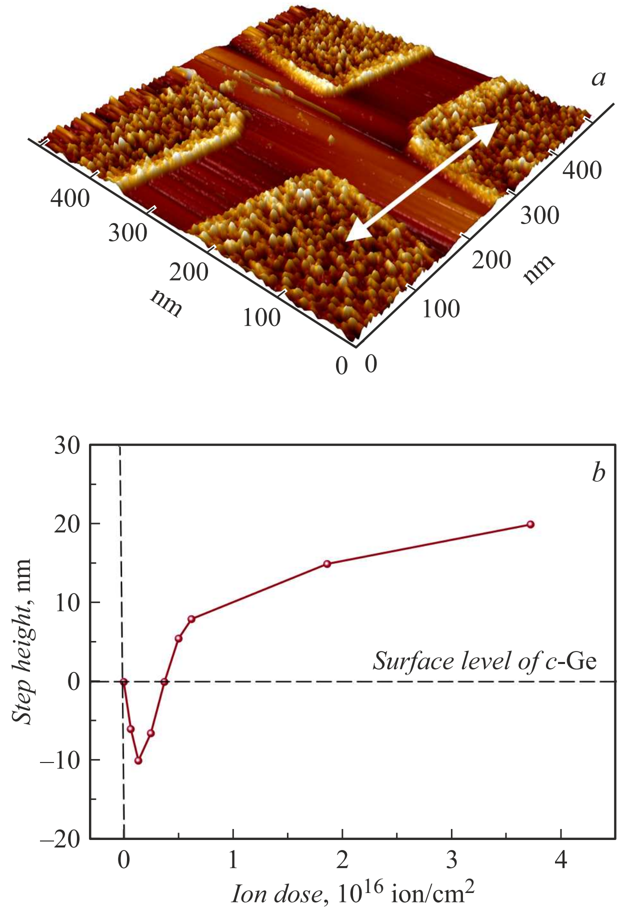

Figure 2. *a* — SPM image of a fragment of the *c*-Ge surface irradiated with $^{209}\mathrm{Bi}^{++}$ ions at $E = 36\,\mathrm{keV}$, $J = 5\,\mu\mathrm{A/cm}^2$, and $D = 4.0 \cdot 10^{15}\,\mathrm{ion/cm}^2$ through a mask. *b* — Dependence of the surface level height measured along the profile (see the light line with arrows in the SPM image) on D. The measured values are indicated by dots.

dependence $h(D)$ plotted for the measured profiles along the light line with arrows (see the example in Fig. 2, a). The variation of h is specific in that ion sputtering of the sample surface proceeds with a monotonic h_{sputt} reduction at the initial stage of irradiation (with an increase in D to $D_{sputt} = 1.3 \cdot 10^{15}\,\text{ion/cm}^2$). When D_{sputt} is exceeded and the Bi:NPGe layer structure is developed, the pattern changes to the opposite: sputtering stops, and h_{swell} starts increasing. It is evident that the change in h_{swell} is associated with the process of swelling of the irradiated layer.

It was reported in several published papers [14,15] that h_{swell} increases monotonically with D when c-Ge is implanted with $^{73}\text{Ge}^+$ and $^{119}\text{Sn}^+$ ions ($E = 150-300\,\text{keV}$, $D_{\max}$ up to $2.0 \cdot 10^{16}\,\text{ion/cm}^2$). Another recently discovered scenario involves irradiation with $^{108}\text{Ag}^+$ ions with $E = 30\,\text{keV}$: as D increases, the surface first swells to h_{swell} values on the order of several nanometers upon reaching $D = 1.25 \cdot 10^{16}\,\text{ion/cm}^2$, which is followed by efficient ion sputtering of the Ag:NPGe layer with a nearly linear h_{sputt} increase observed through to $D = 1.5 \cdot 10^{17}\,\text{ion/cm}^2$ [16]. The scenario of $h(D)$ variation under ion irradiation of c-Ge with initial sputtering followed by effective swelling, which was identified in the present study, was not observed in earlier experiments.

The possible contribution of T_{subst} to the processes of pore formation on the c-Ge surface irradiated with $^{73}\text{Ge}^+$ ions at high E (up to 1 MeV) was discussed in [17,18]. It was concluded that pore formation on the c-Ge surface may occur only at T_{subst} within the range from -50 to $200\,^\circ\text{C}$. To assess the possible influence of sample heating on the identified $h(D)$ scenario, T_{subst} throughout the entire sample irradiation process was measured. The experimental $T_{subst}(D)$ dependence is shown in Fig. 3. It follows from this figure that T_{subst} at the end of irradiation does not exceed $100\,^\circ\text{C}$. This factor negates the possible dominant contribution of T_{subst} to determining the nature of the $h(D)$ conceptual scenario. In addition, it was demonstrated in the works[13,19] that implantation of heated c-Ge substrates with $^{108}\text{Ag}^+$ ions with $E = 30\,\text{keV}$ or their thermal annealing did not lead to the emergence of pores only at substrate temperatures exceeding $T_{subst} = 350\,^\circ\text{C}$.

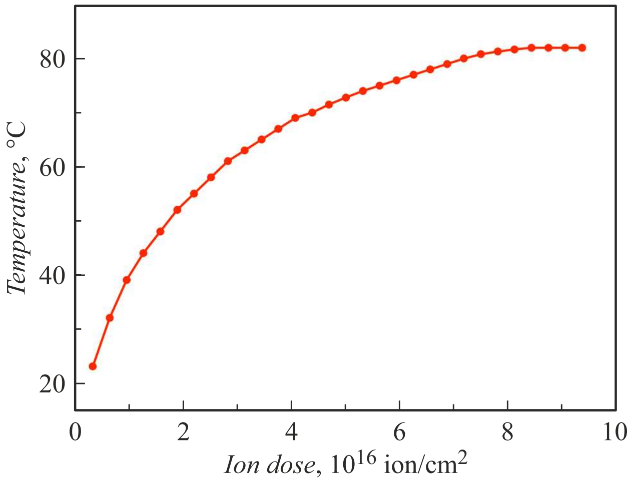

Figure 3. Dependence of the Bi:NPGe sample surface temperature on D.

Thus, structural and topological changes in c-Ge irradiated with $^{209}\text{Bi}^{++}$ at low E were demonstrated. Semiconductor Ge was used as an example to identify experimentally a new conceptual scenario of interaction of accelerated ions with an irradiated matrix, which specifies the nature of transformation of its morphology and the irradiated surface level with an increase in D and consists of successive alternating processes of sputtering and swelling.

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Conflict of interest

The authors declare that they have no conflict of interest.

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