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Study of the effect of neon ion energy on the surface roughness of the main cuts of monocrystalline silicon during ion etching

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The paper presents the results of studying the energy dependences of the sputtering yield and the effective surface roughness of single-crystal silicon irradiated with neon ions with an energy of 100−1000 eV. As a result of the work, the parameters of ion-beam etching with accelerated Ne ions were determined, providing a high sputtering coefficient (etching rate) and an effective roughness value in the spatial frequency range $4.9 \cdot 10^{-2} - 6.3 \cdot 10^{1}\,\mu\text{m}^{-1}$ less than 0.3 nm for the main cuts of single-crystal silicon ($\langle 100\rangle$, $\langle 110\rangle$ and $\langle 111\rangle$).

Keywords: surface, roughness, sputtering, ion etching.

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Introduction

The development of powerful X-ray sources, in particular, free electron lasers and synchrotrons of the 3rd+ and 4th generations, involves the use of single-crystal silicon [1,2] as substrates of optical elements. The requirements for these substrates are only being developed, but it is already clear that the surfaces must be smooth ($\sigma < 0.2$ nm) in the high spatial frequency region ($1 - 10^3\,\mu\text{m}^{-1}$) for providing high reflectances, as well as in the region of medium spatial frequencies ($10^{-3} - 1\,\mu\text{m}^{-1}$) to ensure focusing and collimation of radiation at sliding angles of incidence [3–7]. Thus, the integral roughness value over the entire spatial frequency range — $4.9 \cdot 10^{-2} - 6.3 \cdot 10^{1}\,\mu\text{m}^{-1}$ — should be less than 0.3 nm.

The shape of the surface of the optical elements is a plane or a shape close to the plane for the radiation sources indicated above, and therefore one of the main tasks of this study is to develop experimental data to create, on the basis of ion-beam processing methods, a technique for shaping optical elements from the main sections of single-crystal silicon for X-ray optics applications.

One of the main criteria for creating this kind of optics is the minimum radiation exposure to the near-surface layer of the substrate. Thus, it is necessary to use low-energy ions to correct the shape, however, as shown in the work [8], in the case of argon at energies below 550 eV, the development of surface roughness is observed, and with increasing etching depth, the amplitude of inhomogeneities on the surface increases linearly.

Maintaining the normal angle of incidence of the ion beam to the treated surface [9,10] is the optimal task from the point of view of mathematical modeling and the process itself in problems of shape correction by ion-beam etching, in connection with which the effect of bombardment with Ne ions of various energies (angle of incidence – normal) by the value of the RMS surface roughness of the main sections $\langle 100\rangle$, $\langle 110\rangle$ and $\langle 111\rangle$ of monocrystalline silicon was studied in this paper.

The review shows that the work on ion etching with accelerated neon ions was carried out by other groups of researchers, such as, for example, [11,12], but in these works no attention was paid to the study of the behavior of surface roughness for X-ray optics applications. Also, no such work has been carried out for the three main orientations of monocrystalline silicon, in particular (100), (110) and (111). Most of the work is devoted to ion energies of more than 1.5 keV, which corresponds more to the mode of ion implantation than physical sputtering.

1. Experiment description

The studies were carried out on an ion beam etching installation described in detail in [13]. Standard silicon substrates for the microelectronic industry were used as experimental samples [14] (initial surface roughness ∼0.3 nm). The plate was cut into pieces with size of 15×15 mm. To conduct the experiment, the sample was placed on a slide table under the normal line to the ion beam. To control the etching depth, a „witness“ was used, part of the surface of which was covered with a mask. Next, the working gas pressure of $1.3 \cdot 10^{-2}$ Pa, was created in the chamber, the necessary ion current density of (j) and accelerating voltage (U_{Accel}) the value of which determines the ion energy were set. The sample was subjected to ion bombardment, after which the etching depth and surface roughness were measured. The etching depth was measured using a Talysurf CCI 2000 white light interference microscope (the height of the step formed at the mask boundary was measured). In all experiments, etching was carried out under normal

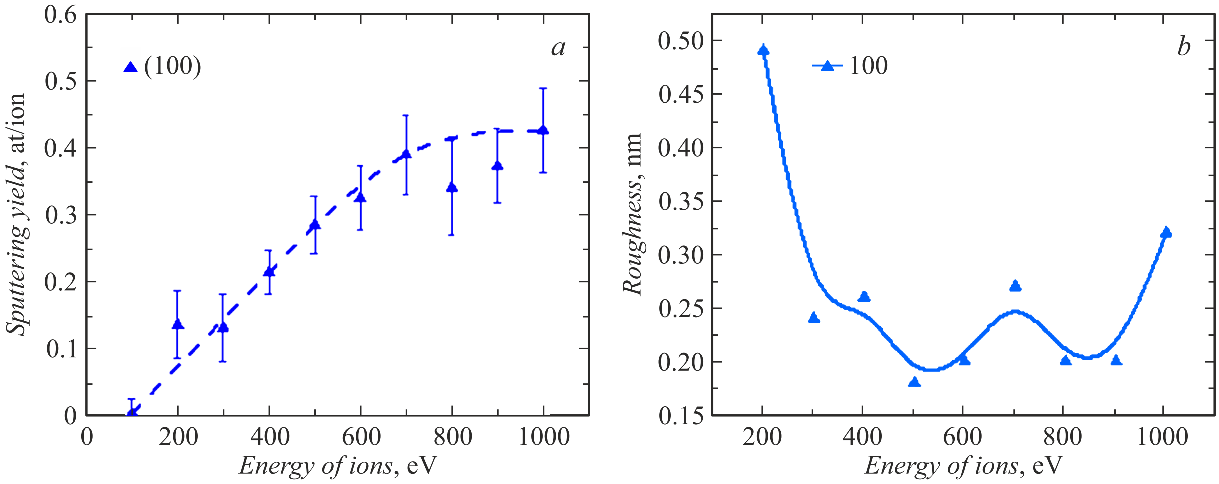

Figure 1. The energy dependences of the sputtering yield (a) and the values of the effective roughness (b) of monocrystalline silicon (100).

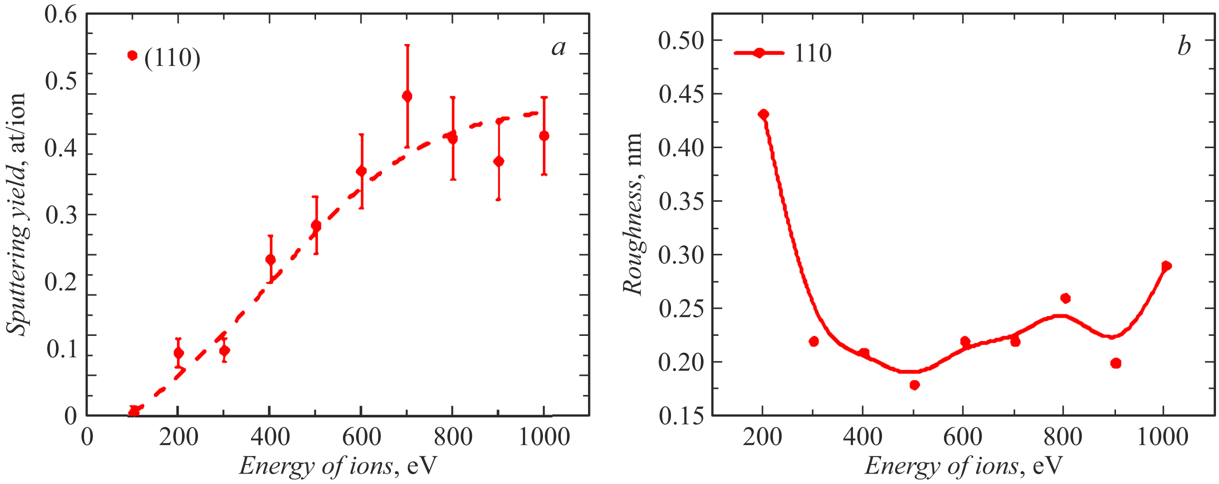

Figure 2. The energy dependences of the sputtering yield (a) and the values of the effective roughness (b) of monocrystalline silicon (110).

(only the energy and time of irradiation changed) to a depth exceeding $1\,\mu$m. This depth is attributable to two circumstances: the first is the fact that the value of the effective roughness reaches saturation and practically does not change when etching more than $1\,\mu$m, at least for amorphous bodies [15]; the second is that typical amount of material removal during the shape correction procedure is about $1\,\mu$m.

The etching rate value V_{Etch} was calculated based on the measured values of the etching depth, and based on the known etching time. This value is proportional to the ion sputtering yield (Y). The expression for Y was obtained taking as a basis the definition of the sputtering yield by small transformations, where the input data are the parameters of the experiment:

$$Y = \frac{\rho e V_{\mathrm{Etch}} N_{\mathrm{A}}}{\cos\Theta_{\mathrm{Inc}}\, j M_2}, \qquad (1)$$

where ρ — target density, N_{A} — Avogadro number, Θ_{Inc} — angle of incidence of ions on the surface, j — ion current density, M_2 — molar mass of the target and $V_{\mathrm{Etch}} = d/t$ — etching rate, d — etching depth, t — exposure time.

The RMS roughness (σ) was measured using Ntegra probe microscope (NT-MDT) in the range of spatial frequencies (q) from $4.9 \cdot 10^{-2} - 6.3 \cdot 10^{1}\,\mu\mathrm{m}^{-1}$ (atomic-force microscope (AFM) frame sizes from 2×2 to $40 \times 40\,\mu$m). The value of the effective roughness is found from the area under the curve of the PSD function, more information about the method can be found in [16].

2. Results and discussion

The energy dependences of the sputtering yields and the values of the effective surface roughness were obtained as a result of a series of experiments on sputtering targets from

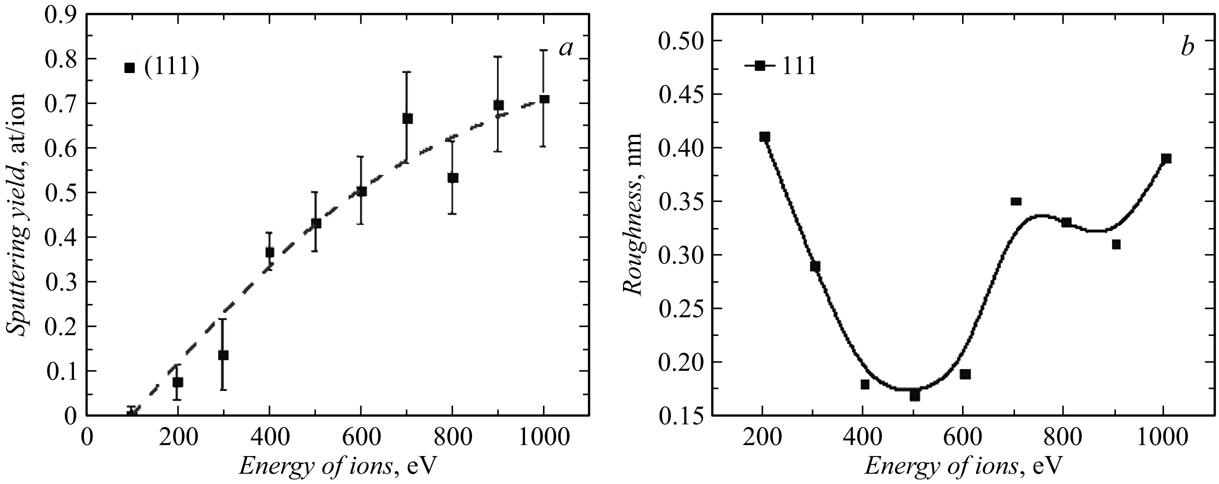

Figure 3. The energy dependences of the sputtering yield (a) and the values of the effective roughness (b) of monocrystalline silicon (111).

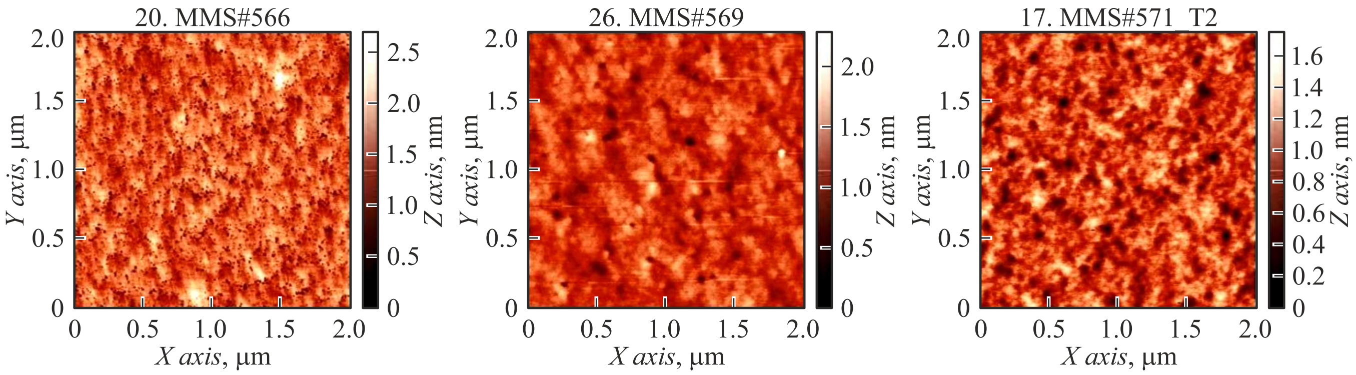

Figure 4. AFM frames $2 \times 2\,\mu$m surfaces of the main sections of monocrystalline silicon after irradiation with Ne ions with an energy of 100 eV.

the main sections of single-crystal silicon with accelerated neon ions (Ne) in the energy range of 100–1000 eV (Fig. 1–3). The energy dependence of the sputtering yield in this energy range has this form, since with an increase in the energy of the incoming ions, the mechanism of collisions moves from single collisions to cascades. Thus, a higher-energy ion generates more knocked-on atoms, whose momentum reaches the atoms in the near-surface layer, whose binding energies on the surface are less than in the volume, which leads to the removal of the atom. With a further increase in the energy of the ion beam, the depth of its penetration also increases, which, in turn, leads to the fact that the energy transferred by the ions to the target atoms is dispersed at a great depth and does not reach the surface atoms.

It can be seen from the obtained dependences that silicon (100) has the lowest sputtering yield and, consequently, the lowest etching rate when irradiated with accelerated Ne ions under normal to the surface. A similar behavior is observed on the energy dependences of the effective surface roughness value for all three orientations, namely, the development of the surface relief at low energies in the region of 200 eV. This behavior was also observed in the paper [8], where attention was paid to the peculiarities of sputtering the main sections of monocrystalline silicon with accelerated argon (Ar) ions. An abrupt change in the behavior of the energy dependence of the effective roughness was observed at energies in the region of 450 eV for Si (100) and at an energy of 550 eV in case Si (110) and Si (111). The development of the relief was observed before the „threshold“ energy with an increase of the value of the effective roughness, and when the „threshold“ energy was exceeded, the roughness was smoothed. A sharp surge of the values of the effective roughness was not observed in this study because the value of the sputtering coefficient at the ion energy of Ne 100 eV is 0.005 at./ion, which in our geometry and experimental parameters corresponds to the etching rate of 0.25 nm/min. It is possible to remove a layer of material no more than 60 nm thick from the surface in 4 h at this rate, which is not enough to characterize the evolution of the surface. However, it is possible to observe the beginning of the formation of craters on the surface on

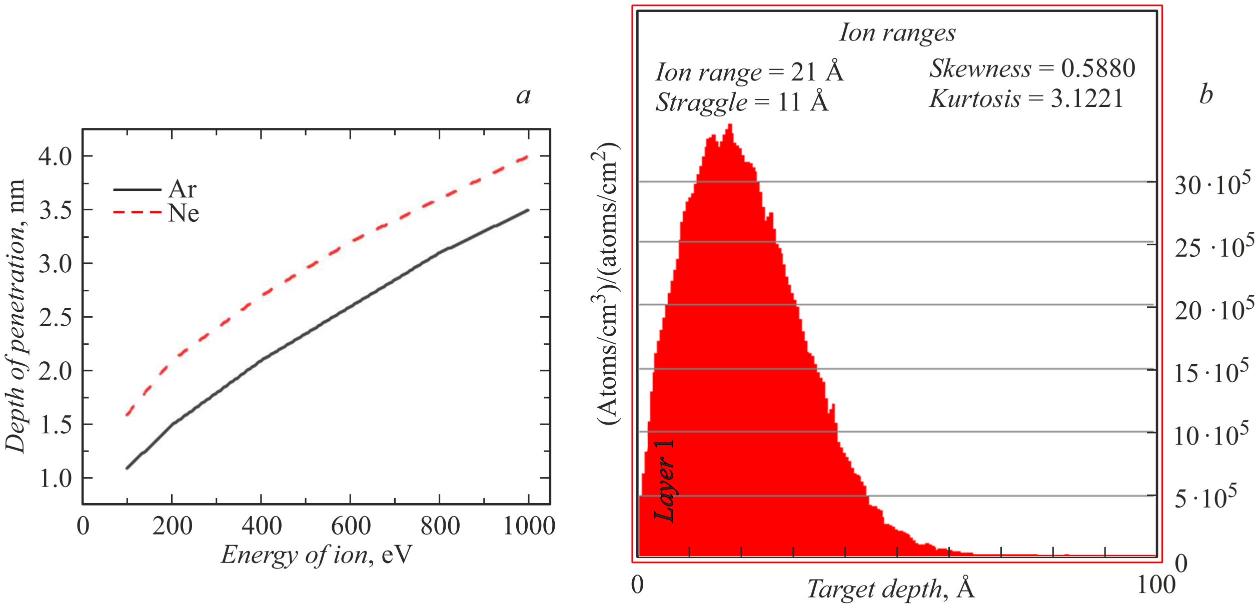

Figure 5. *a* — dependence of the penetration depth of Ar and Ne ions on energy; *b* — calculated profile of implanted ions Ne with an energy of 200 eV in the volume of silicon [16].

the AFM frames $2 \times 2\,\mu$m, shown in Fig. 4, which is a prerequisite for the development of surface roughness.

As mentioned earlier, the minimum disturbed layer is an important circumstance for the procedure of shape correction by accelerated ion beams. Calculations in the SRIM [17] package show that the penetration depth of neon ions is greater than penetration depth of argon ions in the energy range of 100–1000 eV (Fig. 5) and is up to 4 nm inclusive. A layer of more than $1\,\mu$m is usually removed during the shape correction procedure, i.e. a layer saturated with defects after the chemical-mechanical polishing procedure. Thus, the depth of the disturbed layer is units of nanometers after the procedure of ion correction of the surface shape.

Conclusion

The dependences of the sputtering yields and the values of the effective surface roughness for sections (100), (110) and (111) of monocrystalline silicon from the Ne ion energy at normal incidence were obtained in the result of the study. Silicon (100) has the lowest sputtering yield and, consequently, the lowest etching rate when exposed to accelerated ions Ne. A similar behavior of the surface was found like in case of exposure to the accelerated ions Ar, but due to the smaller mass of neon ions, the threshold energy of amorphization is below 200 eV. This fact means that accelerated neon ions can remove the disturbed layer (which is $\sim 1\,\mu$m) during the shape correction procedure and at the same time create a disturbed layer that will be units of nanometers.

Optimal parameters for local correction of shape errors and polishing by beams of accelerated neon ions were determined. A roughness smoothing is observed in the spatial frequency range of $4.9 \cdot 10^{-2} - 6.3 \cdot 10^{1}\,\mu\mathrm{m}^{-1}$ for silicon (100) and (110) in the energy range 300–900 eV. For orientation (111), the best roughness result was obtained for accelerated neon ions in the energy range 300–600 eV.

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Conflict of interest

The authors declare that they have no conflict of interest.

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