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Study of the influence of the energy of argon ions on the surface roughness of the main sections of single-crystal silicon

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Received April 6, 2022
Revised April 6, 2022
Accepted April 6, 2022

The paper presents the results of studying the energy dependences of the sputtering yields and the value of the effective surface roughness of single-crystal silicon upon irradiation with argon ions with an energy of 200−1000 eV. As a result of the work, the parameters of ion-beam etching with accelerated Ar ions were determined, providing a high sputtering yield (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 monocrystalline silicon $\langle 100\rangle$, $\langle 110\rangle$ and $\langle 111\rangle$.

Keywords: sputtering yield, roughness, single-crystalline, monocrystalline, accelerated ions.

DOI: 10.21883/TP.2022.08.54569.70-22

Introduction

Due to the modernization of synchrotrons of the 3rd generation and the appearance of synchrotrons of the 4th generation, as well as free electron lasers, the problems of smoothness and accuracy of the shape of reflective surfaces and their radiation resistance have become even more urgent. The optical elements for the sources indicated above are flat or close to the plane elements, and currently the requirements for their shape accuracy in the standard deviation parameter (RMS) are several nanometers and for roughness — less than 0.3 nm [1]. The problem is complicated by thermally induced shape distortions of optical elements due to large radiation, up to several kW, thermal loads. Both theoretical calculations and practice have shown that only monocrystalline silicon [2] can be considered as a substrate material for mirrors operating under such powerful radiation beams. In this regard, recently there have been papers on finishing single-crystal silicon in order to ensure minimal shape errors and/or surface roughness [3,4]. One of the most promising methods of finishing the surfaces of optical elements is the correction of local shape errors with a small-sized ion beam — IBF (Ion Beam Figuring) [5]. This method allows to obtain high-precision surfaces with minimal roughness [6,7]. However, the use of IBF for processing single-crystal materials faces serious problems.

Ion etching of crystalline materials is a rather complex and not fully understood process. The mutual orientation of the crystallographic planes, the crystal slice, the ion energy and the angle of its incidence on the sample surface can introduce significant ambiguity in the ion-beam etching procedure. There are practically no papers on the study of the shape and surface roughness of the main sections of monocrystalline silicon during ion-beam etching in the literature, with the exception of a large number of works with reactive ion-beam etching [8,9].

In shape correction tasks, the optimal both from the point of view of mathematical modeling and the manufacturability of the IBF process is to maintain the normal incidence of the ion beam on the treated surface [10,11]. In this connection, as part of this study, the effect of bombardment with Ar ions of various energies (angle of incidence−normal line) on the value of the RMS surface roughness of the main sections ($\langle 100\rangle$, $\langle 110\rangle$ and $\langle 111\rangle$) of single-crystal silicon was investigated.

1. Experiment description

The studies were carried out on an ion beam etching installation described in detail in [6]. Standard silicon substrates for the microelectronic industry were used as experimental samples [12] (initial surface roughness ~ 0.25 nm). The plate was cut into pieces 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. Further, the working gas pressure $1.3 \cdot 10^{-2}$ Pa was created in the chamber; the necessary ion current density (j) and accelerating voltage (U_{accel}) were set, the value of which determines the ion energy. 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).

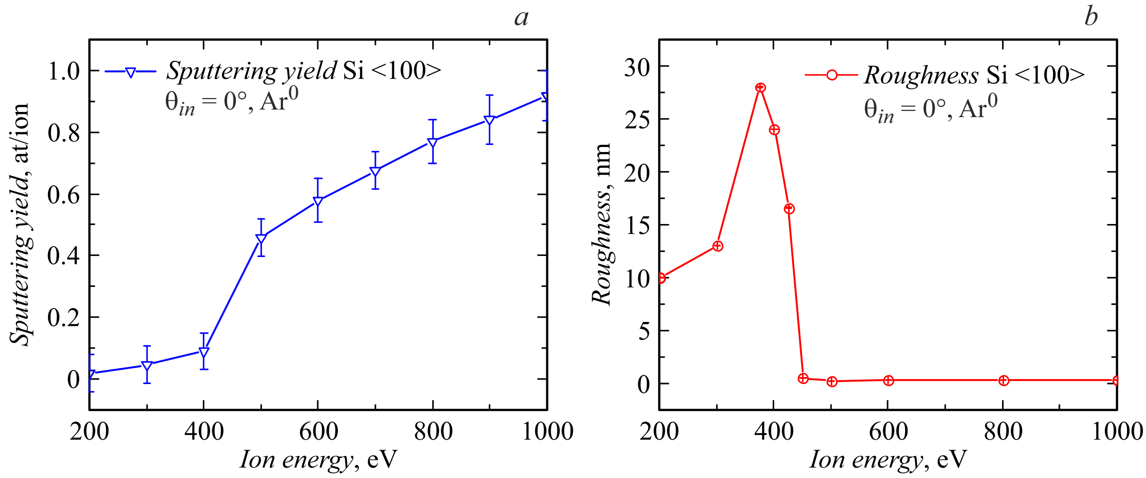

Figure 1. Dependences of the sputtering coefficient (left) and the value of the effective surface roughness Si ⟨100⟩ on the energy of argon ions. Experiment parameters: $j = 0.7\,\mathrm{mA/cm^2}$; $\theta = 90^\circ$.

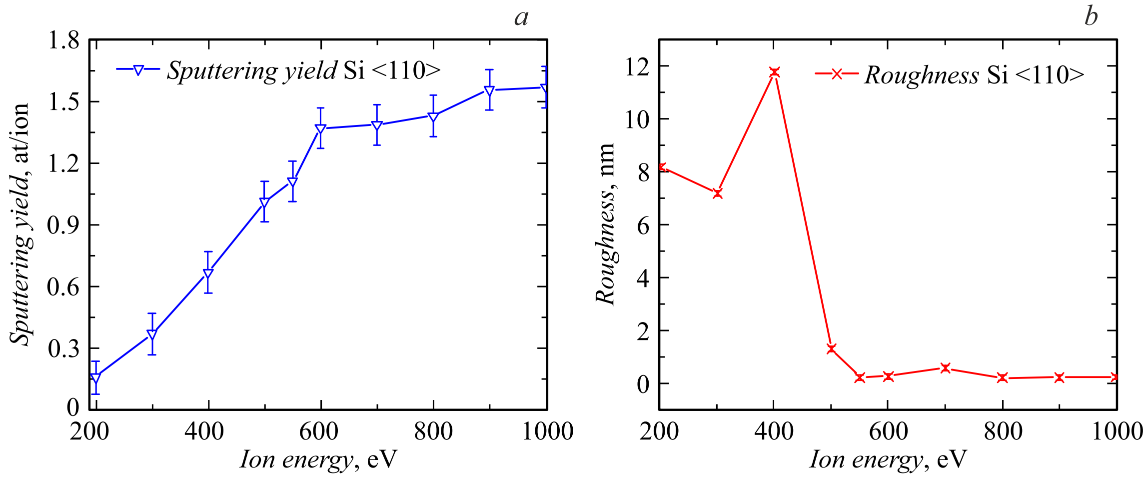

Figure 2. Dependences of the sputtering coefficient (words) and the value of the effective surface roughness Si ⟨110⟩ on the energy of argon ions. Experiment parameters: $j = 0.7\,\mathrm{mA/cm^2}$; $\theta = 90^\circ$.

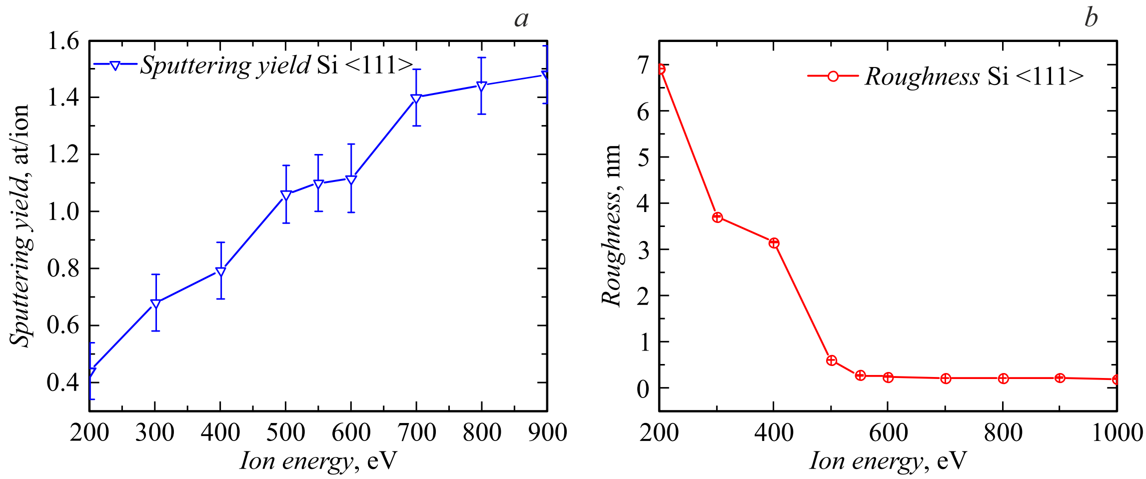

Figure 3. Dependences of the sputtering coefficient (left) and the value of the effective surface roughness Si ⟨111⟩ on the energy of argon ions. Experiment parameters: $j = 0.7\,\mathrm{mA/cm^2}$; $\theta = 90^\circ$.

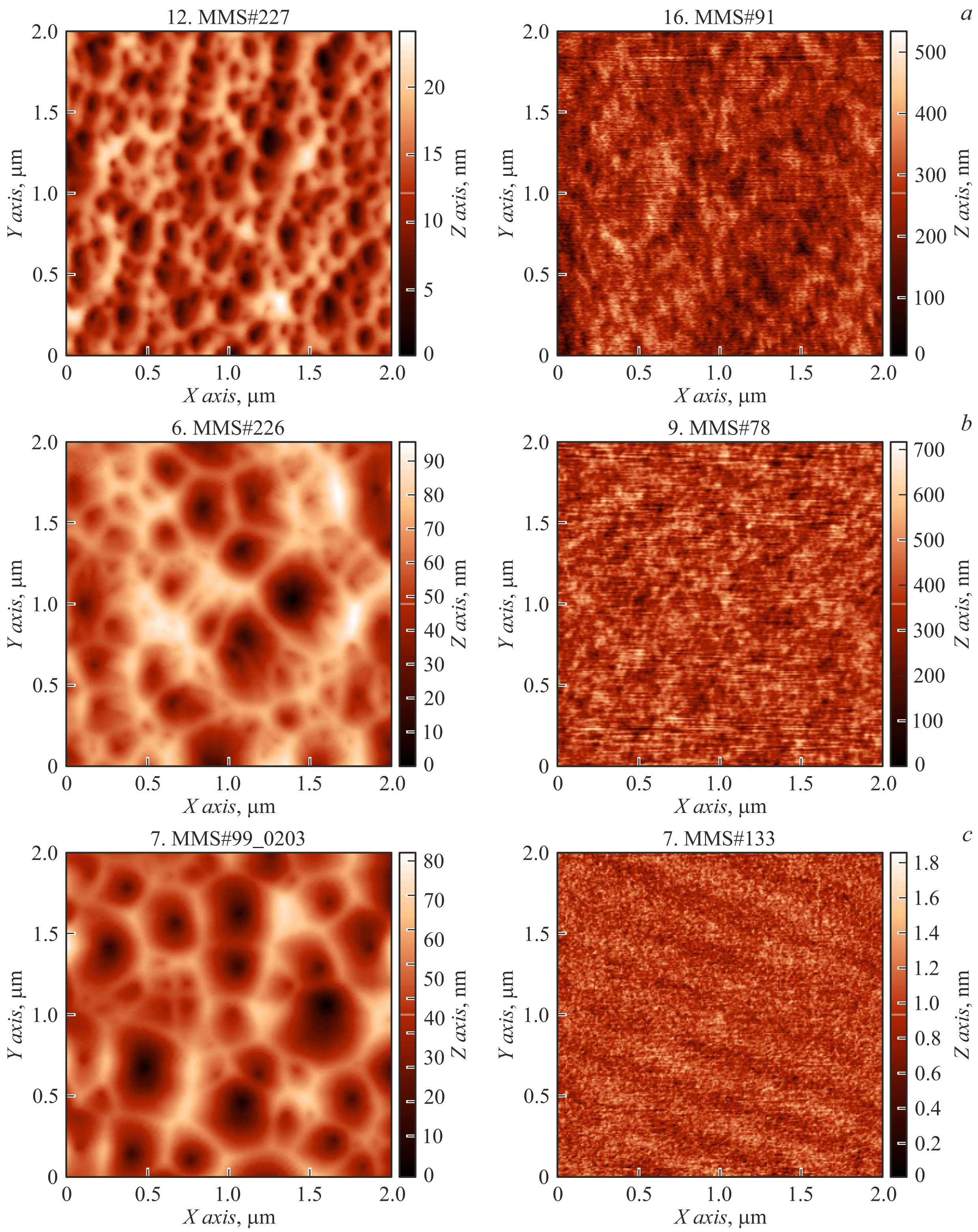

Figure 4. AFM frames of surfaces of monocrystalline silicon etched with argon ions with energy 400 eV (left) and 800 eV (right): *a* — Si⟨111⟩; *b* — Si⟨110⟩; *c* — Si⟨100⟩ — after irradiation under normal line accelerated argon ions with an energy of 400 eV.

From the measured values of the etching depth, knowing the time, the value for the etching rate V_{etching} was calculated. Since V_{etching} is proportional to the ion sputtering coefficient, then by determining this proportionality, we can calculate the values for the sputtering coefficient Y. Taking as a basis the definition of the sputtering coefficient, by small transformations we obtained an expression for Y, where the input data — are the parameters of the experiment:

$$Y = \frac{\rho e V_{\text{etching}} N_A}{\cos\theta_{\text{incid}} j M_2}), \qquad (1)$$

where ρ — target density, N_A — Avogadro number, θ_{incid} — angle of incidence of ions on the surface, j — ion current density, M_2 — the molar mass of the target and $V_{\text{etching}} = d/t$ — etching rate, d — etching depth, t — exposure time.

The RMS roughness is measured on a probe microscope N_{tegra} (NT-MDT) in the range of spatial frequencies $(q)4.9 \cdot 10^{-2}$–$6.3 \cdot 10^{1}\,\mu\text{m}^{-1}$ (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 [13].

2. Results and discussion

Fig. 1 shows the obtained dependences of the sputtering coefficient and the value of the effective surface roughness of monocrystalline silicon $\langle 100 \rangle$ on the energy of argon ions.

It is worth noting that at an ion energy of about 400 eV (425 eV), there is a sharp change in the behavior of the dependence of the sputtering coefficient and effective roughness on energy. This behavior is explained by the fact that at ion energies less than 425 eV, the near-surface layer is weakly amorphized, which is why roughness develops as a consequence of crystal faceting. At ion energies above 425 eV due to ion-induced destruction of the crystal lattice near the surface, this layer begins to behave as amorphous, which leads to a significant decrease in surface roughness. For example, in the study [14] on the study of the effect of ion etching by Ar ions on the roughness of amorphous silicon, it is shown that the surface is smoothed. Thus, it is shown that the IBF technique can be applied to the formation of precision X-ray optical elements from monocrystalline silicon $\langle 100 \rangle$ with effective roughness in the spatial frequency range $4.9 \cdot 10^{-2}$–$6.3 \cdot 10^{1}\,\mu\text{m}^{-1}$ less than 0.3 nm.

Similar studies were carried out for samples with a slice orientation of $\langle 110 \rangle$ and $\langle 111 \rangle$ (Fig. 2, 3). Based on the obtained dependences of the sputtering coefficient and the value of the effective roughness on energy, a similar behavior can be detected, however, unlike the orientation of $\langle 100 \rangle$„ the jump “ occurs at a higher ion energy ~ 550 eV.

It is also worth noting that at energies below „critical “, the surfaces of these slices behave in a similar way, namely, this is manifested in the formation of deep pits (Fig. 4).

As can be seen from the above AFM frames, when etching under normal line with low-energy argon ions, a similar pattern is observed for all three orientations, namely, the formation of a large number of pits with a transverse size up to 500 nm. At the same time, an increase in energy, for example, to the values of 800 eV, allows smoothing even an initially smooth surface (the original $\sigma \sim 0.25$ nm) and maintaining the value of effective roughness at an acceptable level ($\sigma < 0.3$ nm) when shooting several micrometers. In all the experiments described above, the take was 2–$3\,\mu$m.

Conclusion

As a result of the study, the parameters of ion-beam etching with accelerated Ar ions were determined, providing a high sputtering coefficient (etching rate) and the value of effective roughness 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 sections of monocrystalline silicon $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$. In particular, it is shown that the smoothing effect for Si $\langle 100 \rangle$ is achieved at an ion energy of more than 425 eV, and for $\langle 110 \rangle$ and $\langle 111 \rangle$ at an energy of more than 550 eV. At lower ion energies, a significant degradation of the surface roughness is observed. This behavior can be explained by the amorphization of the near-surface layer, which, when a certain ratio of the amorphous phase to the crystalline phase is reached, begins to behave like an amorphous one. Thus, it can be argued that when the argon ion energy is higher than the critical one for each orientation of monocrystalline silicon, the IBF technique, which has already become traditional, can be used to aspherize the surface or finish correcting local shape errors, while maintaining or even smoothing the roughness in the spatial frequency range $\nu = 4.9 \cdot 10^{-2}$–$6.3 \cdot 10^{1}\,\mu\text{m}^{-1}$.

Funding

The study was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation (agreement № 075-15-2021-1362).

Conflict of interest

The authors declare that they have no conflict of interest.

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