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# Method for Obtaining Atomically Smooth Substrates from Single-Crystal Silicon by Mechanical Lapping

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The paper reports on the developed technique for polishing single-crystal silicon substrates using a mechanical lap. The effective substrate roughness was obtained in the spatial frequency range of $0.025-65\,\mu\text{m}^{-1}$ at the level of 0.37 nm and 0.18 nm at a frame size on the surface of $2\times 2\,\mu\text{m}^2$. The result obtained is comparable with the results of chemical-mechanical and dynamic polishing of single-crystal silicon wafers for microelectronics.

**Keywords:** Surface, roughness, synchrotron radiation, polishing.

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## Introduction

According to its thermophysical characteristics: high thermal conductivity, especially at low temperatures; moderate (at the level of $10^{-6}$) temperature coefficient of linear expansion and high microhardness, providing good polishability of the material, monocrystalline silicon is one of the most promising materials for the manufacture of crystal monochromators and mirror substrates for applications in strong radiation loads. In particular, mirrors for synchrotrons of the 3rd and 4th generations, as well as X-ray free electron lasers, are made of this material [1,2].

One of the most difficult requirements for polishing silicon for these tasks is the need to simultaneously ensure low micro-roughness and high accuracy of the surface shape. To ensure high reflection coefficients of multilayer X-ray mirrors that will be applied to these substrates and the diffraction quality of images, the surface micro-roughness should be at the level of no more than 0.2 nm, and the shape accuracy $\leq 1-2$ nm, depending on the period of the multilayer coating used [3]. The dimensions of the mirrors can range from tens of millimeters, up to 1 m and more. The traditional technology of chemical-mechanical polishing, although it allows to obtain a surface roughness at the level of several Å [4], nevertheless does not fully meet the requirements for shape accuracy.

Currently, the best silicon substrates for X-ray optics are manufactured using the dynamic polishing method, when the polishing compound is fed between the workpiece and the polishing tool at a high (up to 1000 atm) pressure [5]. This technology is extremely complex and has actually been mastered by only one company, JTEC, Japan.

An alternative technology for obtaining high-precision and smooth substrates, including aspherical ones, is being developed at the IFM RAS. The approach is based on a two-stage process of surface shaping. At the first stage, a flat or spherical surface [6] is made by the classical method of mechanical lapping. Then correction of local errors and aspherization of surfaces is performed by ion beam etching [7]. The advantages of this approach compared to using a small-sized tool at the shaping stage, for example [8,9], is that lapping provides the best surface parameters both in accuracy and roughness, especially in the area of medium spatial frequencies, which most degrade the spatial resolution [10].

This approach has proven itself well in the processing of quartz substrates [11]. In [3], this approach was first applied to the additional polishing of monocrystalline silicon using suspensions of $CeO_2$ micro-powders. The effective roughness in the spatial frequency range $0.025-65\,\mu\text{m}^{-1}$ was improved from 4 nm after polishing using standard technology using diamond micro-powders and at the finish of the polyrite, to 1 nm. Despite the improvement in the quality of processing, however, the roughness remained at an unacceptable level for X-ray optical applications.

This paper describes an improved technological process that has significantly improved the quality of single-crystal silicon processing.

## 1. Methodology and experiment

Grinding and polishing were carried out on a 3-spindle 3PD350 machine, which allows processing flat and spherical parts with a diameter up to 350 mm. The machine provides the rotation of the polisher and the pendulum movement of the block with the machined parts in a wide range of speeds, as well as pneumatic clamping to the polisher. A photo of the processed block with a fragment of the machine is shown in Fig. 1.

**Figure 1.** Photo of a block of substrates at the workplace.

At the first stage, a block of 7 single-crystal silicon blanks with a diameter of 35 mm was formed. The blanks were cut out of the boule with a diamond saw. Further, manual grinding was carried out on a flat grinder on one side, on which the blanks were glued to a flat duralumin washer in the form of a flower (dense packaging), and filled with pitch (CH-1 — adhesive resin).

Further, the block was subjected to a deep grinding-polishing procedure, when at each subsequent processing stage the material was removed to a depth exceeding twice the grain size of the abrasive used at the previous stage.

At the second stage, the entire flower block was ground using a brass grinder on the machine with silicon carbide grinding powders KZ-40, KZ-28 and KZ-10, the size of the abrasive grain was 40, 28 and 10 $\mu$m, respectively.

At the third stage, polishing was performed. Polishing was carried out with a resin polisher (resin SP-5) at a temperature and humidity controlled directly in the vicinity of the workpiece 25°C and 20% humidity, respectively.

Initially, polishing was performed with aqueous solutions of Opaline $0.5-1.0\,\mu$m and $0.3-0.5$ *mu*m. Then the final polishing was carried out with suspensions of $CeO_2$ micropowders with an average size of polishing particles 300, 100 and 50 nm produced by the Moscow Institute of Physics and Technology (MIPT). A description of the characteristics of the suspensions, as well as the results of their use for polishing fused quartz, can be found in [3,10].

At the final stage, KOH alkali of different concentrations was added to the suspension.

The measurement of micro-roughness was carried out on a stand based on an atomic force microscope (AFM), which allows to examine large-sized parts [12]. The technique uses AFM images to construct one-dimensional spectral dependences of the roughness power density on the spatial frequency $\nu$, PSD $(\nu)$. As an integral characteristic of the surface roughness, effective roughnesses in the corresponding range of spatial frequencies obtained by integrating the PSD function in this frequency range are used. In more detail, the method of measuring roughness, as well as the lateral limitations of AFM microscopy in the direction of long-wave roughness are analyzed in detail in the works [13,14]. The effectiveness of this approach is due to the fact that the roughness of different ranges affects the resolution of mirrors in different ways. Thus, high-frequency roughness with frequencies above $1\,\mu\text{m}^{-1}$ has a greater effect on the intensity of the image without loss of resolution. Alternatively, mid-frequency roughness, frequency range $10^{-3}-1$ *mu*m$^{-1}$, most affect the resolution. Low-frequency roughness, often called shape errors, leads to distortion of the image as a whole [10]. Therefore, the analysis of PSD functions allows us to reliably predict the imaging properties of mirrors.

Since the main purpose of this work was to develop a technique for obtaining atomically smooth substrates from monocrystalline silicon using mechanical lapping, the main controlled parameter was roughness. The surface shape of the already polished substrates was measured using a Zygo Verifier interferometer with a flat reference. According to the Device Certificate, the measurement error of flat parts did not exceed 4 nm.

## 2. Results

The effect of the composition of polishing suspensions, as well as the content of KOH alkali in the suspension on the roughness and morphology of the surface is illustrated in Fig. 2–4. These figures and the following show the measurement results for the central part of the block. However, the measurement results for other parts coincided with each other and with the central part with good accuracy. Fig. 2 shows AFM images of the surface, frames $2 \times 2$ and $40 \times 40\,\mu\text{m}^2$ (Fig. 2, $a$) and the power spectral density function constructed from AFM data the roughness of PSD (Fig. 2, $b$) of the substrate after polishing with an aqueous solution of Apoline with a grain size up to $0.3\,\mu$m. As can be seen from the figure, the effective roughness in the spatial frequency range $0.025-65\,\mu\text{m}^{-1}$ was 0.76 nm.

Polishing with a suspension of $CeO_2$ micropowders with a size up to $0.1\,\mu$m produced by FTI, as can be seen from Fig. 3, led to a decrease in effective roughness by 0.12 nm. The use of a suspension with a grain size of 0.05 nm reduced the polishing speed, but did not affect the roughness, so this suspension was excluded from further work.

To improve the roughness, the effect of KOH alkali additives was studied. The effect of KOH concentration in

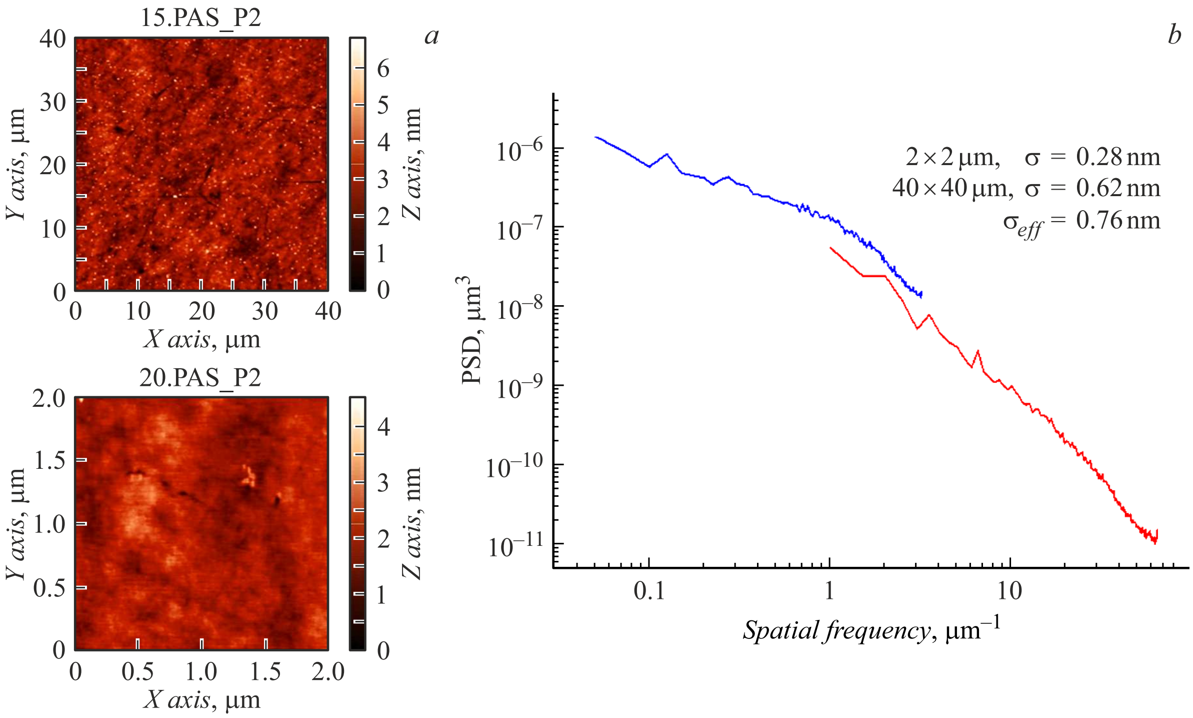

**Figure 2.** Atomic force images of the surface, frames $2 \times 2$ and $40 \times 40\,\mu\text{m}^2$ (*a*) and PSD roughness function (*b*) of the substrate after polishing with an aqueous solution of Apoline with a grain size up to $0.3\,\mu$m.

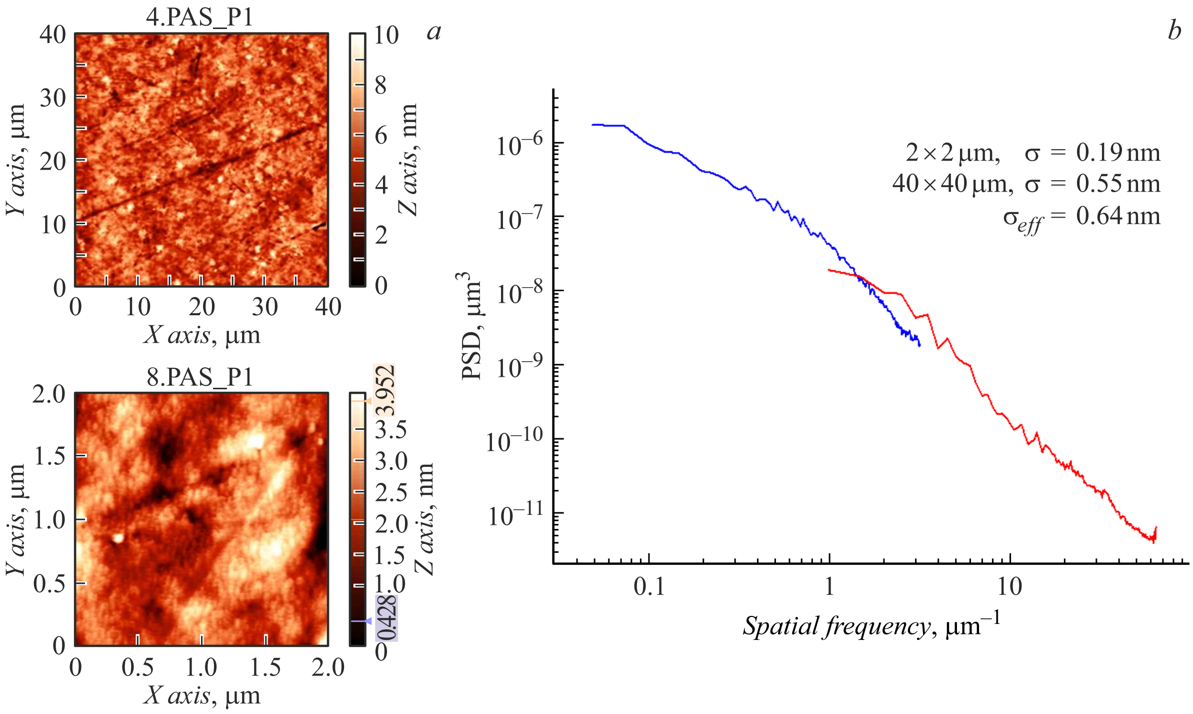

**Figure 3.** Atomic force images of the surface, frames $2 \times 2$ and $40 \times 40\,\mu\text{m}^2$ (*a*) and PSD roughness function (*b*) of the substrate after polishing with $CeO_2$ suspension of micro-powders with grain size up to $0.1\,\mu$m. On the height scale, blue and red (in the online version) indicate the height levels within which the frame was processed.

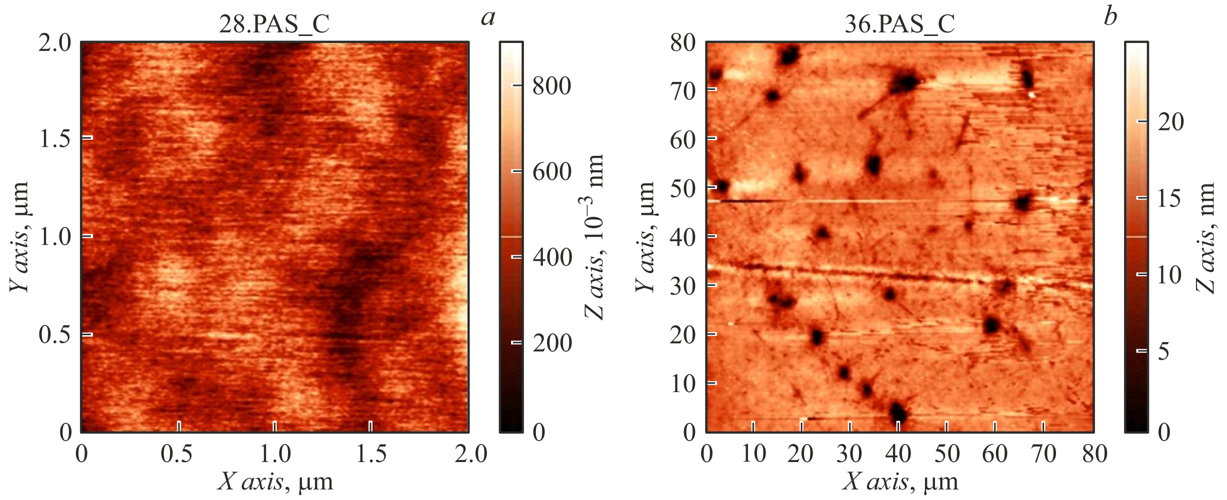

**Figure 4.** AFM frames $2 \times 2$ (*a*) and $40 \times 40\,\mu m^2$ (*b*) single crystal silicon substrate surfaces after polishing with CeO suspension$_2$, grain size $0.1\,\mu$m. The KOH concentration was about 0.5%.

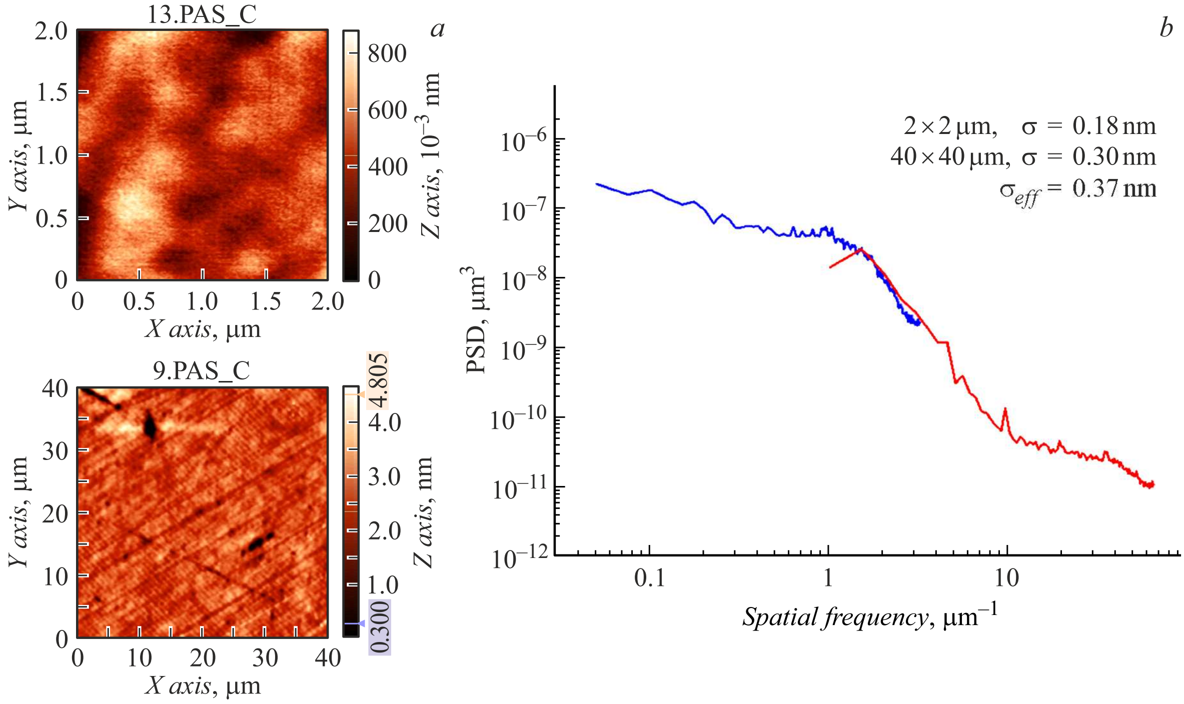

**Figure 5.** Atomic force surface images, frames $2 \times 2$ and $40 \times 40\,\mu m^2$ — ( it a) and PSD roughness function (*b*) substrates after polishing with a suspension of $CeO_2$ micro-powders with a grain size up to $0.1\,\mu$m and a content of 0.1% KOH in solution. On the height scale, blue and red (in the online version) indicate the height levels within which the frame was processed.

polishing suspension on surface roughness and morphology was investigated. An example of excessive concentration is shown in Fig. 4, which shows AFM frames $2 \times 2$ and $80 \times 80\,\mu m^2$. The KOH concentration was about 0.5%. If you look at the frame $2 \times 2\,\mu m^2$, then the roughness value turned out to be small, at the level of 0.1 nm. However, deep pits are clearly visible on the frame $80 \times 80\,\mu m^2$.

During the study, the optimal concentration of KOH was found in the polishing suspension $CeO_2$, grain size $0.1\,\mu$m, which was about 0.1%. The results of measurements of surface roughness and morphology are shown in Fig. 5. As can be seen from the figure, single „mordants“are observed on the surface, and such images were found specifically. In most cases, these „mordants “ are absent. The effective roughness was 0.37 nm. We expect that, according to the work of [15], after ion-beam polishing, the roughness will decrease to 0.2–0.25 nm, which is quite sufficient for X-ray optical applications.

The results of measurements of the shape of the surface of the part using an interferometer are illustrated

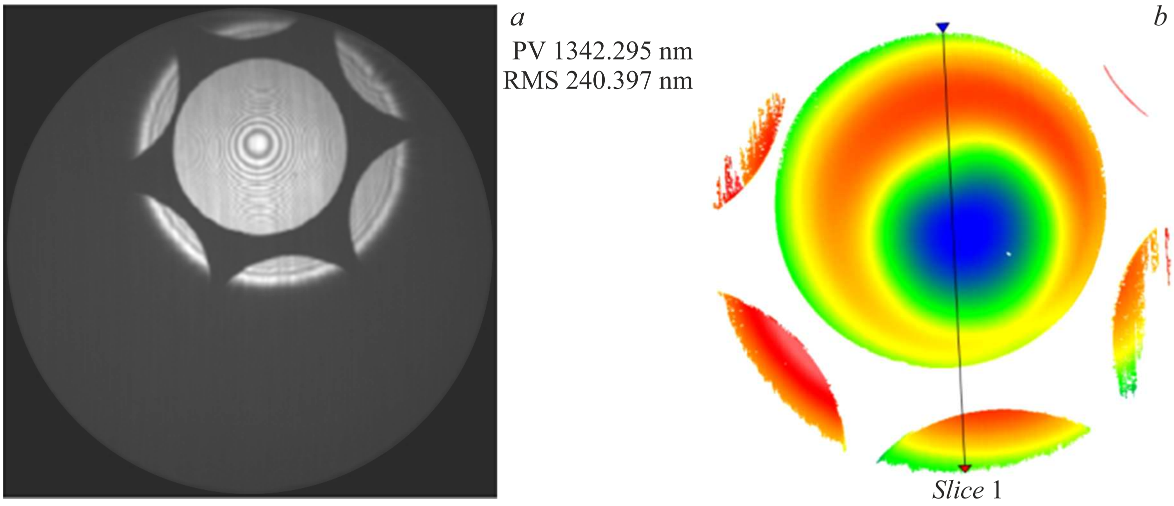

**Figure 6.** The interferogram (*a*) and a map of the deviation of the substrate surface from the plane (*b*), measured on the Zygo VeriFire interferometer.

in Fig. 6. Fig. 6, *a* shows the measured interferogram, and Fig. 6, *b* — shows interferogram-reconstructed map of surface deviations from the plane. The surface parameters were about according to the maximum height difference parameter 1.3 $\mu$m, and by standard deviation 0.24 $\mu$m.

## Conclusion

The main result of the research was the development of a technological process for polishing monocrystalline silicon, which uses a classical mechanical lapping on a solid polisher. This approach will allow to simultaneously obtain low roughness and high quality of the surface shape, unlike the classical method of chemical-mechanical and dynamic polishing of single-crystal silicon for microelectronics.

The effective roughness value in the spatial frequency range $0.025-65\,\mu\text{m}^{-1}$ at the level of 0.37 nm is obtained. This value is comparable to the roughness of classical substrates for the microelectronic industry [16,17]. After ion beam polishing, as shown in [15], one can expect a decrease in roughness to 0.25–0.2 nm, which satisfies the requirements for X-ray optical applications.

The shape accuracy obtained during the experiment, the standard deviation of the surface from the plane 0.24 $\mu$m, does not meet the requirements for X-ray optics. However, it should be noted that the purpose of this work was to develop a technological process that ensures low roughness, and the shape was measured only after polishing, in other words, control and correction of technological parameters for its improvement were not carried out. In addition, the error has an axisymmetric character, which makes it easy to correct it with an ion beam [7].

Currently, using the polishing process developed in this paper, we have started to create experimental samples of single-crystal silicon substrates for synchrotron applications with nanometer accuracy of the surface shape.

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### Conflict of interest

The authors declare that they have no conflict of interest.

## References

[1] D.H. Bilderback, A.K. Freund, G.S. Knapp, D.M. Mills. J. Synchrotron Rad., **7**, 53 (2000). DOI: 10.1107/S0909049500000650

[2] H. Thiess, H. Lasser, F. Siewert. Nucl. Instrum. Meth. A, **616**, 157 (2010). DOI: 10.1016/j.nima.2009.10.077

[3] N.I. Chkhalo, A.A. Akhsakhalyan, Yu.A. Weiner, M.V. Zorina, A.E. Pestov, M.V. Svechnikov, M.N. Toropov, N. Kumar, Yu.M. Tokunov. ZhTF, **91** (10), 1588 (2021). (in Russian). DOI: 10.21883/JTF.2021.10.51375.95-21

[4] Siltronix. Silicon wafer specifications [Electronic resource] Available at: https://www.sitronics-st.com/en/silicon-wafer/silicon-wafer-specifications, free. (date of request: 26.05.2022).

[5] K. Yamauchi, H. Mimura, K. Inagaki, Y. Mori. Rev. Sci. Instrum., **73**, 4028 (2002). DOI: 10.1063/1.1510573

[6] N.I. Chkhalo, I.V. Malyshev, A.E. Pestov, V.N. Polkovnikov, N.N. Salashchenko, M.N. Toropov. UFN, **190** (1), 74 (2020) (in Russian). DOI: 10.3367/UFNr.2019.05.038601

[7] A.D. Akhsakhalyan, E.B. Klyuenkov, A.Ya. Lopatin, V.I. Luchin, A.N. Nechai, A.E. Pestov, V.N. Polkovnikov, N.N. Salashchenko, M.V. Svechnikov, M.N. Toropov, N.N. Tsybin, N.I. Chkhalo, A.V. Shcherbakov. Poverkhnost'. Rentgenovskie, sinkhrotronnye i nejtronnye issledovaniya, **1**, 5 (2017). (in Russian). DOI: 10.7868/S0207352817010048

[8] Y. Huang, B. Fan, Y. Wan, S. Li. Optik, **172**, 540 (2018). https://doi.org/10.1016/j.ijleo.2018.07.039

[9] K.N. Khatri, R. Sharma, V. Mishra, H. Garg, V. Karar. Adv. Mater. Proc., **2** (7), 425 (2017). DOI: 10.5185/amp.2017/704

[10] U. Dinger, F. Eisert, H. Lasser, M. Mayer, A. Seifert, G. Seitz, S. Stacklies, F.-J. Stickel, M. Weiser. Proc. SPIE, **4146**, 35 (2000). DOI:10.1117/12.406674

[11] M.N. Toropov, A.A. Akhsakhalyan, M.V. Zorina, N.N. Salashchenko, N.I. Chkhalo, Yu.M. Tokunov. ZhTF., **90** (11), 1958 (2020). (in Russian). DOI: 10.21883/JTF.2020.11.49990.127-20

[12] N.I. Chkhalo, N.N. Salashchenko, M.V. Zorina. Rev. Sci. Instrum., **86**, 016102 (2015). https://doi.org/10.1063/1.4905336

[13] M.M. Barysheva, Yu.A. Vainer, B.A. Gribkov, M.V. Zorina, A.E. Pestov, N.N. Salashchenko, R.A. Khramkov, N.I. Chkhalo. Bull. Rus. Acad. Sci. Phys., **76** (2), 163−167 (2012). DOI: 10.3103/S1062873812020037

[14] N.I. Chkhalo, S.A. Churin, A.E. Pestov, N.N. Salashchenko, Yu.A. Vainer, M.V. Zorina. Opt. Express, **22** (17), 20094 (2014). DOI: 10.1364/OE.22.020094

[15] M.S. Mikhailenko, A.E. Pestov, N.I. Chkhalo, M.V. Zorina, A.K. Chernyshev, N.N. Salashchenko, I.I. Kuznetsov. Appl. Opt., **61** (10), 2825 (2022). DOI: 10.1364/AO.455096

[16] L.N. Abdulkadir, K. Abou-El-Hossein, A.I. Jumare, P.B. Odedeyi, M.M. Liman, T.A. Olaniyan. Int. J. Adv. Manuf. Technol., **96**, 173−208 (2018). DOI: 10.1007/s00170-017-1529-x

[17] A.R. Belure, A.K. Biswas, D. Raghunathan, Rishipal, S. Bhartiya, R. Singh, S.K. Raid, R.S. Pawade, M.P. Kamath, N.S. Benerji. Mater. Today Proc., **26**, 2260 (2020). DOI: 10.1016/j.matpr.2020.02.490/