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A new technology of thermoplastic glass bending for the manufacture of cylindrical surfaces of hard X-ray range mirrors

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The paper presents a new technique for thermoplastic bending of glass. The technique makes it possible to produce cylindrical surfaces with a guide in the form of a parabola, ellipse, etc. for mirrors in the hard X-ray wavelength range ($\lambda \sim 0.1$ nm). Three samples of the surface of an elliptical cylinder were prepared using this technique. The production time for each sample was two days. The deviation of the guide and its local angle from the calculated values for all samples does not exceed $\Delta y = 0.5\,\mu$m and $\Delta\alpha = 7 \cdot 10^{-5}$ rad, respectively. It is shown that when such surfaces are etched for two hours, their accuracy can be improved by more than two orders of magnitude ($\Delta y = 2$ nm, $\Delta\alpha = 5 \cdot 10^{-7}$ rad).

Keywords: focusing X-ray mirrors, cylindrical mirrors, multilayer structures

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Introduction

Starting from the Goebel's studies [1,2], multilayer cylindrical mirrors for hard X-ray radiation with a guide in the form of an ellipse or parabola are actively used to focus or collimate the radiation of linear X-ray sources. They are most widely used in modern diffractometers, allowing to increase the efficiency of X-ray radiation selection in $10-15$ times [3,4]. In order to exclude the aberrations introduced by the mirror, the deviation $\Delta\alpha = \alpha(x) - \alpha_{clc}(x)$ of the local angle of the guiding surface $\alpha(x)$ from the calculated value $\alpha_{clc}(x)$ must be a lot less than the divergence of the radiation incident on the mirror $\Delta\Omega = s/L$, where s — the size of the source, L — the distance from the source to the mirror. The condition $\Delta\alpha = \Delta\Omega/3$ is usually sufficient. In diffractometers, the apparent width of the anode of the X-ray tube is $s = 25-50\,\mu$m, and the distance of the mirror−anode is about 100 mm. Therefore, the requirement for angular accuracy has the form $\Delta\alpha \leq 7 \cdot 10^{-5}$ rad. Here and further, we take advantage of the fact that for the hard X-ray range ($\lambda \sim 0.1$ nm), the grazing incidence of radiation on the mirror should not exceed $\vartheta = 0.05$ rad. This requirement follows from the fact that multilayer structures reflect radiation with a reflection coefficient R close to unity for periods $d > 2$ nm, and for smaller periods R quickly drops to zero. From Wolfe's law−Bragg follows $\sin\vartheta = \lambda/2d < 0.05$. Therefore , the equality is fulfilled with good accuracy: $\mathrm{tg}\,\vartheta = \vartheta = y'(x)$, where $y(x)$ — is the guide of the cylindrical surface.

Previously, we have developed a number of methods for manufacturing cylindrical mirrors with a given surface shape: methods of thermoplastic and elastic bending of glass, as well as the replica method, especially effective in the manufacture of large batches of mirrors [5–9]. To implement the replica method, a template is required that defines the shape of the manufactured surface. In the manufacture of the template, we use the methods of thermoplastic and elastic bending of glass bars. The previously used method of thermoplastic bending provides accuracy at the level of $\Delta\alpha \leq 7 \cdot 10^{-5}$ rad, however, the method is extremely time-consuming and time-consuming (on the order of several weeks). This is due to the fact that part of the measuring equipment is located in the hot zone at a temperature of $550-600^\circ$C. The elastic bending method allows you to achieve a fairly high accuracy $\Delta\alpha = (2-5) \cdot 10^{-5}$ rad, however, the sample is in a stressed state and may change its shape over time due to the aging of the glue. In addition, the technique is also very time-consuming and time-consuming.

In this paper, we propose a much simpler and more effective technique of thermoplastic bending, which allows for $2-3$ a day to make a stress-free sample with an accuracy of $\Delta\alpha = 7 \cdot 10^{-5}$ rad. This accuracy is quite sufficient for working with laboratory sources (in particular, for the manufacture of mirrors for diffractometers). For those cases where higher accuracy is required, such surfaces can serve as starting points for further correction of their shape by ion or ion-plasma etching methods. We have developed such methods and repeatedly tested them in practice. Their main advantage is that they do not spoil, and in some cases even improve the initial micro-roughness of the surface [10–12]. By etching, it is possible to bring the accuracy of the surface shape to any predetermined value in the presence of appropriate methods of measuring the shape.

1. Bending technique

The methodology is based on the following fact, established by us experimentally. An elastically curved glass bar retains its shape after cooling when heated slowly to a temperature of $T = 550-600°\mathrm{C}$ in a homogeneous temperature field. The essence of the technique is to replace the calculated distribution of curvature $K^*(x)$ with a polyline inscribed in $K^*(x)$.

The method comprises: A glass sample in the form of a parallelepiped is cantilevered in a bending device (Fig. 1). With its elastic bending under the action of a force F applied at the point x_0, the curvature $K(x)$ and the guide $y(x)$ of the resulting cylindrical surface (at $y' \ll 1$) have the form [13]:

$$K(x) \approx y'' = A(x_0 - x), \tag{1}$$

$$y = (A/2)x^2(x_0 - x/3), \tag{2}$$

where $A = F/EI$, $I = ab^3/12$, F — force, E — Young's modulus, a and b — the width and thickness of the bar.

An area with coordinates $x_{\text{big}}, x_{\text{fin}}$ is selected on the sample, inside which it is required to obtain the calculated distribution of curvature $K^*(x)$. This interval is divided by n intervals (in this paper $n = 3$). Bending is carried out in n stages.

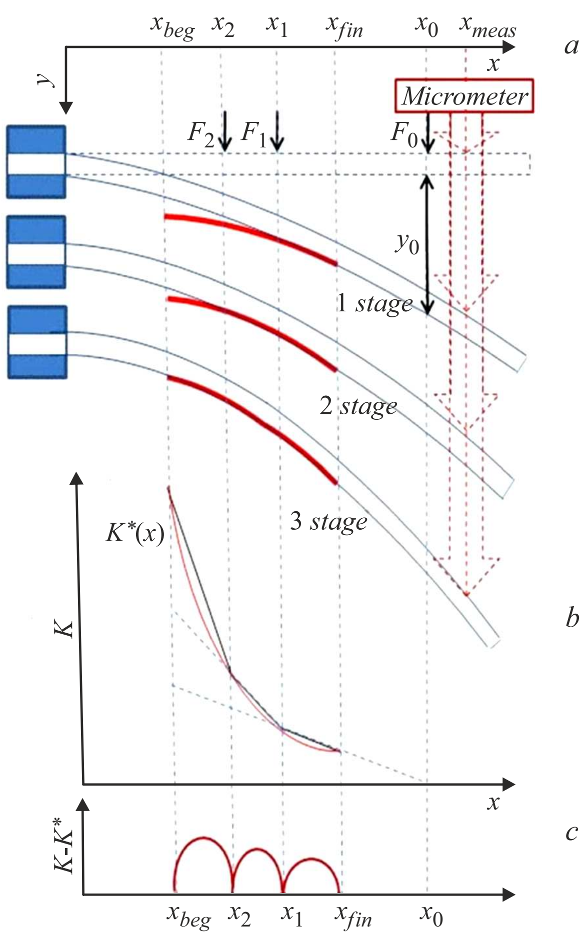

Figure 1. Diagram of the bending technique.

At the first stage, we bend the glass so that the curvature at the points x_{fin} and x_1 is equal to the calculated $K^*(x_{\text{in}})$ and $K^*(x_1)$. Substituting these values into (1), we obtain a system of equations:

$$\begin{cases} K^*(x_{\text{fin}}) = A_0(x_0 - x_{\text{fin}}), \\ K^*(x_1) = A_0(x_0 - x_1). \end{cases} \tag{3}$$

Solving this system of equations, we find A_0, x_0. Substituting them into (2), we find the value of the shift $y_0 = (A_0x_0^3)/3$ at the point x_0, to which we need to move the glass along the y axis to fulfill the condition (3). Further, with a screw installed at the point x_0, under the control of a micrometer, we shift the sample by y_0 and conduct a cycle: heating to $T = 550°\mathrm{C}$ — cooling to room temperature.

At the second stage, the point of application of force is $x = x_1$. It is necessary to shift the glass along the y axis by the amount of y_1 so that the curvature at the point x_2 becomes calculated:

$$K^*(x_2) = A_0(x_0 - x_2) + A_1(x_1 - x_2). \tag{4}$$

The first term on the right in the expression (4) — is the curvature at the point x_2 formed after the first stage. From the equation (4) find A_1, substitute in (2) and find $y_1 = (A_1x_1^3)/3$. Next, with a screw installed at the point x_1, under the control of a micrometer, we shift the sample by y_1 and conduct a cycle: heating to $T = 550°\mathrm{C}$ — cooling.

At the third stage, the point of application of force is $x = x_2$. It is necessary to shift the glass along the y axis by the amount of y_2 so that the curvature at the point x_{bag} becomes calculated:

$$K^*(x_{\text{beg}}) = A_0(x_0 - x_{\text{beg}}) + A_1(x_1 - x_{\text{beg}}) + A_2(x_2 - x_{\text{beg}}). \tag{5}$$

The first two terms on the right in the expression (5) — are the curvature at the point x_{big} formed after the first and second stages. From the equation (5) find A_2, substitute in (2) and find $y_2 = (A_2x_2^3)/3$. We shift the glass by y_2 along the y axis and conduct a heating– cooling cycle. This completes the process.

2. Making an elliptical cylinder

According to this method, the problem of manufacturing a sample with a surface in the form of an elliptical cylinder was solved. Calculation guide — ellipse section $y = (b/a)(a^2 - x^2)^{0.5}$, $a = 240\,\mathrm{mm}$, $b = 4.5\,\mathrm{mm}$, $-170 < x < -110\,\mathrm{mm}$. In the laboratory system (Fig. 1), the coordinates of the points $x_{\text{big}}, x_{\text{fin}}, x_{\text{mas}}, x_1, x_2$ are chosen equal, respectively 10, 85, 195, 50, 28 mm. The calculated value of x_0 is 180 mm. The calculated values of the offsets y_0, y_1, y_2 at the points x_0, x_1, x_2 are equal to 2260, 49 and 16 μm. To increase the accuracy of measurements, it is advisable to install the micrometer not at the points x_0, x_1, x_2, but near the edge of the glass at the maximum possible value of $x = x_{\text{meas}}$ (Fig. 1). In this case, the measured values are $y_i^* = (x_{meas}/x_i)y_i$ noticeably exceed y_i and are equal to $y_0^* = 2448$, $y_1^* = 191$ and $y_2^* = 111\,\mu$m.

According to the formulas (1)–(5) the expected deviation of the final curvature K (polyline curve Fig. 1, *b*) from the calculated $K-K^*$ (Fig. 2, *a*). Integrating it, we obtain the expected deviation of the local angle and the guide from the calculated values (Fig. 2, *b* and *c*). It can be seen from the figure that these values do not exceed 0.025 mrad and 0.15 μm, respectively.

In accordance with the above calculations, experiments on glass bending were carried out. The glass produced by the Bor Glass Factory was used, which is manufactured using the technology of casting on liquid tin („float glass“), having a roughness at the level of $\sigma \sim 0.5$ nm.

A glass bar cut from such glass, having a thickness, width and length equal to 5, 40 and 220 mm, respectively, was cantilevered in the bending device (Fig. 3) with a bar *1*. Then at the point x_0 with the help of a bar and screws *2* shifted by the amount of y_0^* under the control of a micrometer sensor *3* with a division price of 1 μm (set at the point x_{meas}). After that, the sample was placed in a muffle furnace, where the heating – cooling cycle was carried out. At the second stage, the bar *2* moved to the position x_1 and shifted by the amount of y_1^*. At the third stage, the bar *2* moved to the position x_2 and shifted by the amount y_2^*.

The bending was carried out on three samples. On the first sample, after each stage, the dependence of the local derivative on the coordinate was measured on the

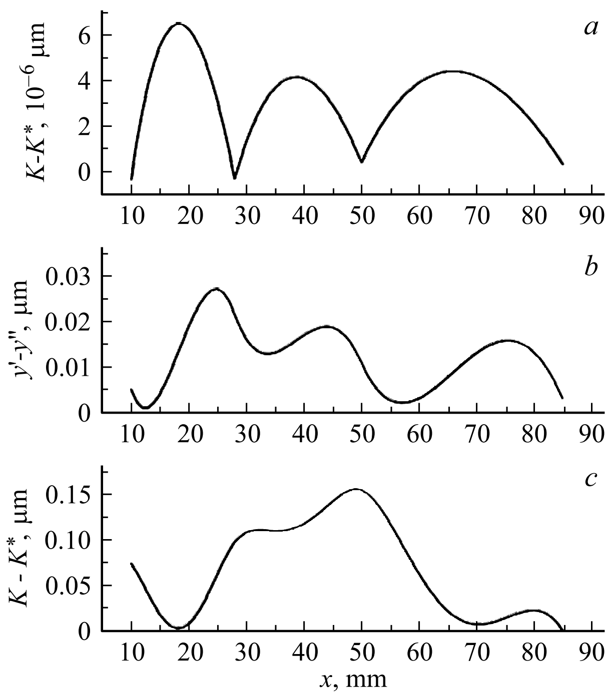

Figure 2. The expected deviation of the curvature (*a*), the local angle (*b*) and the guide (*c*) of the manufactured surface from the calculated values.

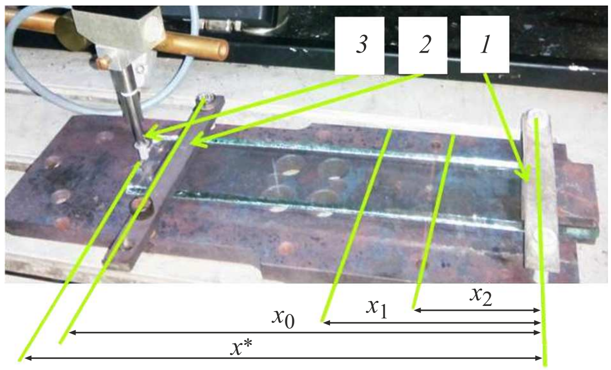

Figure 3. Photo of the bending device: *1, 2* — metal bars, *3* — micrometer.

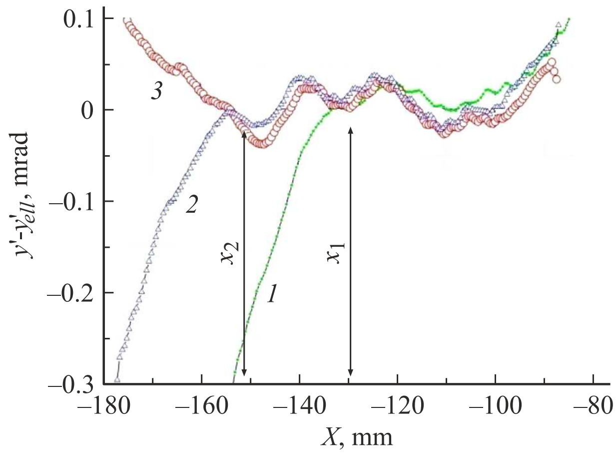

Figure 4. Deviation of the local angle from the calculation for the ellipse after 1, 2 and 3 stages of bending (curves *1*–*3*); x_1, x_2 — points of application of force at 2 and 3 stage.

optical stand [14] (Fig. 4). The measurement accuracy $\Delta\alpha = 2 \cdot 10^{-5}$ rad. The figure shows a step-by-step approximation of the sample shape to the calculation. Two other samples were bent with the same parameters, but without intermediate measurements.

It can be seen from Fig. 5 that on all samples the deviation of the measured profile from the calculation (curves *1*–*3*) does not exceed $\Delta y^* = 0.5\,\mu$m, and the angular accuracy is $\Delta\alpha^* = 0.07$ mrad, which is 2–3 times worse than expected (Fig. 2). At the same time, this accuracy fully satisfies the requirements for the accuracy of the shape of mirrors for laboratory X-ray sources and, in particular, mirrors for diffractometers.

Fig. 5, *b*, draws attention to the fact that in the region $x = -147$ mm, a dip is observed on all samples, which may indicate the presence of the same dip on the original surface of the glass. To test this hypothesis, a glass sample was cut out, located next to (at a distance of 20 mm) with the sample number *1*, and its profile was measured (curves *4*, Fig. 5). If we assume that the initial profile on all three samples is the same and subtract it from the curves *1*–*3*,

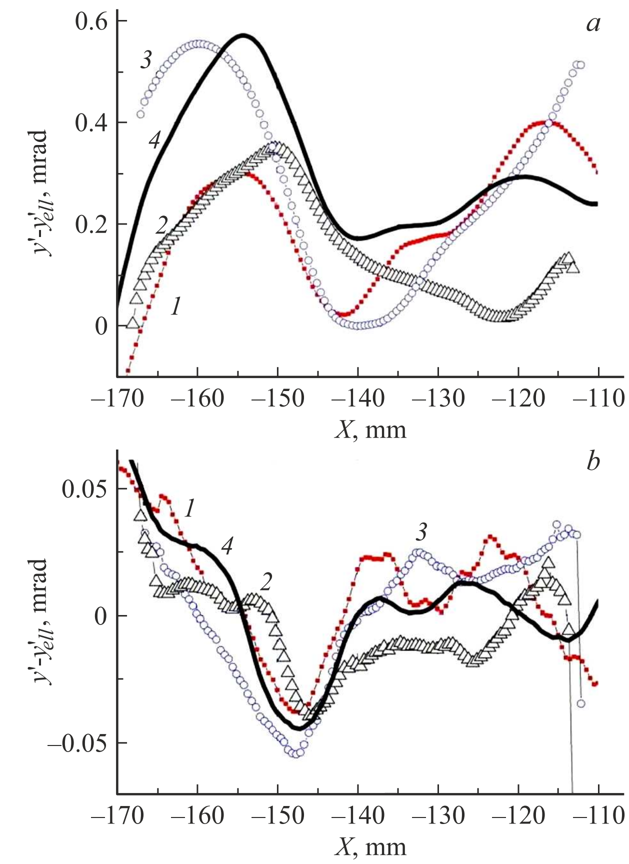

Figure 5. Deviation of the guide (a) and the local angle (b) of the manufactured surface from the calculated values for three samples (curves *1–3*). Relief (a) and its derivative (b) for the original surface (curves *4*), taken on the same glass next to the samples *1–3*.

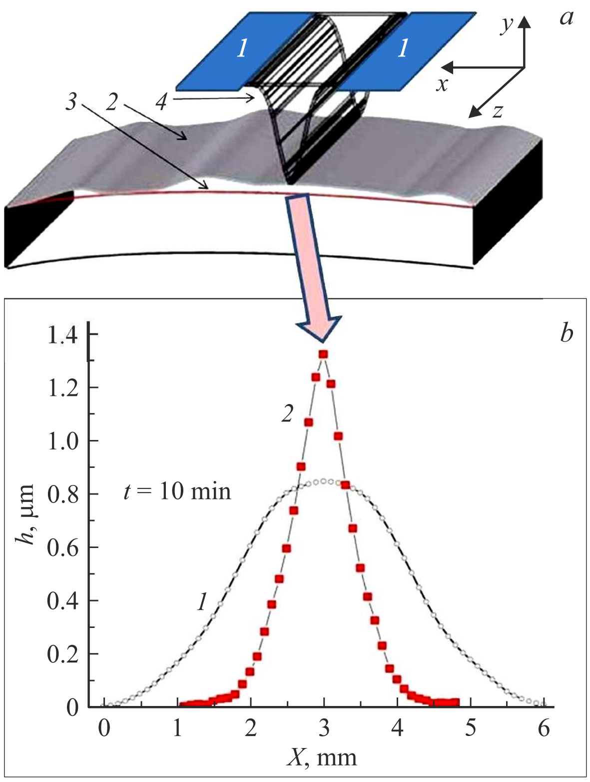

Figure 6. a — RIBE etching technique with focusing slit: *1* — metal slit, *2* — initial surface, *3* — design guide, *4* — etching profile; b — etching profile across the slit width 8 (curve *1*) and 4 mm (curve *2*) in 10 min.

then the deviation of the guide and the local angle of the manufactured surface from the calculated values will be $\Delta y = 0.25\,\mu$m and $\Delta\alpha = 0.02-0.03$ mrad, respectively. This practically coincides with the expected values of Δ and $\Delta y'$ (Fig. 2, b, c). This leads to an important conclusion: before applying this technique, it is necessary to measure the curvature of the initial surface $K_{\mathrm{init}}(x)$ and take it into account in calculations.

Thus, the proposed technique allows you to create cylindrical surfaces with a deviation from the calculated guide and the local angle $\Delta y^* = 0.5\,\mu$m and $\Delta\alpha^* = 7 \cdot 10^{-5}$ rad, respectively. However, such manufacturing accuracy is by no means the ultimate. In the present study, the working area of the sample was divided into 3 intervals ($n = 3$). If we increase the number of intervals, then the deviation of the obtained curvature, and hence the values $\Delta\alpha^*$ and Δy^* from the calculation, will decrease inversely proportional to n^2. By doubling the number of intervals ($n = 6$) and taking into account the initial profile $K_{\mathrm{init}}(x)$, it is possible, within the same equipment, to obtain the following values: $\Delta y^* = 40$ nm, $\Delta\alpha^* = 1 \cdot 10^{-5}$ rad.

3. Modeling of the etching process of the manufactured surface

The bending technique is fully described above. Sec. 3 we decided to include in order to assess how much it is possible to reduce the deviation of the curved surface from the calculation by the reactive ion-beam etching (RIBE) technique with a focusing slit. The reactive ion-beam etching (RIBE) technique is described in detail in [11,12]. For further consideration, it is essential that after the focusing slit, an etching zone is formed on the sample in the form of a rectangle, the long side of which is equal to the length of the slit (60−80 mm). The etching velocity distribution $v(x)$ across the slit does not change along the y axis (Fig. 6), and the width of the distribution $v(x)$ decreases with decreasing width of the slit.

We have created a program that allows us to simulate the etching process in such a (one-dimensional) geometry [15]. The sample moves relative to the slit in the direction of the x axis at a speed of $w(x)$ (Fig. 6). At the input to the program, the initial distribution $\Delta y(x)$ and the experimentally measured distribution $v(x)$ are set. At the output, we get a new distribution after etching $\Delta y^*(x)$, the law of motion of the sample relative to the slit $w(x)$ and the total etching time.

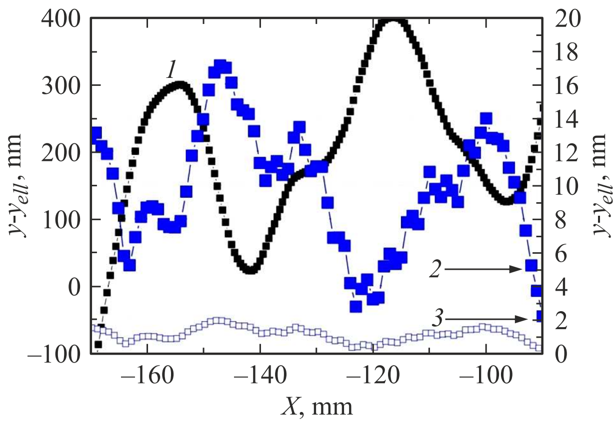

Figure 7. Deflection of the guide surface after thermoplastic bending (curve *1*, left scale) after etching for 80 min with a focusing slit 8 mm wide (curve *2*, right scale) and subsequent etching for 6 min with a focusing slit width 4 mm (curve *3*, right scale) from the calculated guide.

Fig. 7 shows the results of model etching. The guide obtained after bending the sample number *1* (curve *1*, Fig. 5, *a*) was taken as the initial guide surface. At the first stage, etching was carried out using a slit with a width of 8 mm for 80 min. At the second stage, the resulting profile was etched for 6 min using a slit with a width of 4 mm. The corresponding etching profiles measured on the profilometer of the model 130 [16] are shown in Fig. 6, *b*.

The deviation of the profile obtained after etching from the calculation of $\Delta y^*(x)$ is shown in Fig. 7. After the first etching stage, the value of $\Delta y^*(x)$ decreased by about 20 times compared to the original ($\Delta y^*_{\text{init}}(x) = 500$ nm, curve *1*) and was 20 nm (curve *2*, right scale). After subsequent etching with a gap of 4 mm, the value of $\Delta y^*(x)$ decreased by about 20 times and amounted to 1−2 nm (curve *3*, right scale). We note that the glass surface after etching cannot be used as a mirror surface, since micro-roughness develops to the values of 2−5 nm during etching. However, such surfaces can be used as a template for the replica method.

Conclusion

1. The paper presents a new technique of thermoplastic bending of glass. The technique makes it possible to produce cylindrical surfaces with a guide in the form of a parabola, ellipse, etc. for mirrors of the hard X-ray wavelength range ($\lambda \sim 0.1$ nm).

2. According to this technique, three samples of the surface of an elliptical cylinder were made. The manufacturing time of each sample was two days. The deviation of the guide and its local angle from the calculated values for all samples does not exceed $\Delta y = 0.5\,\mu$m and $\Delta\alpha = 7 \cdot 10^{-5}$ rad, respectively.

3. It is shown that when etching such surfaces for 2 h, their accuracy can be improved by more than two orders of magnitude to the values $\Delta y^* = 2$ nm, $\Delta\alpha^* = 5 \cdot 10^{-7}$ rad.

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Conflict of interest

The authors declare that they have no conflict of interest.

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