REGULARITIES OF ELECTRON-BEAM TECHNOLOGY INFLUENCE ON OPERATING CHARACTERISTICS OF OPTICAL ELEMENTS WITH NANO-DIMENSIONAL OXIDE COATINGS

ЗАКОНОМЕРНОСТИ ВЛИЯНИЯ ЭЛЕКТРОННО-ЛУЧЕВОЙ ТЕХНОЛОГИИ НА ЭКСПЛУАТАЦИОННЫЕ ХАРАКТЕРИСТИКИ ОПТИЧЕСКИХ ЭЛЕМЕНТОВ С НАНОРАЗМЕРНЫМИ ОКСИДНЫМИ ПОКРЫТИЯМИ

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Summary: *It is determined, that electron-beam processing of elements from optical glasses with nanosized coatings from metal oxides leads to improvement of their physical and mechanical properties, that influence operational characteristics of elements: negative microdefects on the surface are excluded, its microporosity decreases by 5... 10%, as well as microroughness from 30... 35 nm to 9... 15 nm; the microhardness of the surface increases from 2,3…3,5 GPa to 23,7…24,9 GPa, thus the influence of the coating thickness on its value decreases by 30...40%; wear resistance increases by 7... 12% and service life of optical elements increases by 20... 30%. Mathematical model has been developed, allowing to pre-define critical modes of electron-beam processing of elements, control of which allows to prevent their possible destructions and increase the probability of nonfailure work at operation.*

KEY WORDS: OPTO-ELECTRONIC INSTRUMENT MAKING, OPTICAL ELEMENTS, ELECTRON BEAM

1. *Introduction*

In order to increase durability, reduction of radiation and convective components of thermal losses, nanosized oxide coatings representing compositions of oxides Bi_2O_3, TiO_2, ZnO, SiO_2, Al_2O_3 are applied onto the optical elements of precision instrumentation [1 – 6].

One of the widely used methods of obtaining such coatings is the method of thermal vacuum deposition of materials, which allows to obtain nanosized (< 100 nm) coatings in the form of separate layers. In this case, the obtained coatings are heterogeneous, contain hidden microdefects (cracks, chips, etc.), the surface contains significant microunevenesses and low microhardness, etc. All this leads to a decrease in the functional characteristics of these coatings: their wear resistance decreases; reflection coefficient decreases in the low infrared area and in the area of visible radiation; dispersion coefficient increases, etc.

The wide use of traditional methods of preparation and processing of surfaces of optical elements with oxide coatings (mechanical, chemical, chemical-mechanical) [2 – 4] has shown the impossibility of obtaining simultaneously clean and flawless surface, as well as flawless layers, that leads to deterioration of technical and operational characteristics of optic-electronic devices.

Fundamental researches conducted in the field of development of new high-intensity technologies for processing various materials [7 – 10], including optical materials, showed that the most promising sources of energy for such technologies are the focused streams of charged particles (electrons, ions), laser radiation, etc. However, the use of, for example, ion and laser surface processing of elements has revealed a number of obstacles that limit the possibility of their wide use in precision instrumentation: the disturbance of the surface microgeometry; formation of local high-temperature zones with large temperature gradients, leading to the emergence of critical termotensions in materials and their destruction, etc. As practice has shown [3, 7 – 17], the most convenient, environmentally friendly and the steerable way of elements processing is the electron-beam method, allowing to receive surfaces of high purity with minimal roughness, and also with the increased microhardness and thickness of hardened layers in tens of microns.

However, the wide use of electron-beam technology in precision instrumentation is hampered by the lack of data as to the regularity of the effects of electron-beam treatment regimes, the control of which allows to improve the performance characteristics of optical elements with oxide coatings (roughness and microhardness of the surface, the degree of its purity and porosity of surface layers, wear resistance, lifetime etc.).

Therefore, the purpose of this work is experimental and theoretical study of the influence regularities of electron-beam processing modes of optical elements with nanosized oxide coatings on their operational characteristics.

2. *Results and discussion*

As a result of carried out researches on samples from optical glass K8 (the plates $6\cdot10^{-2}$ m were used, they were $3\cdot10^{-2}$ m wide and $4\cdot10^{-3}$ m thick) it was found that after electron-beam processing of oxide coatings there were no negative microdefects, and the microroughness decreases from 30... 35 nm to 9... 15 nm (Fig. 1, 2).

The studies of surface microhardness of optical elements with oxide coatings showed its increase after electron-beam treatment: from 17,5...21,5 GPa to 23,7...24,9 GPa for coating Al_2O_3; from 9,3...13,1 GPa to 14,7...15,9 GPa for coating ZrO_2; from 2,3...3,5 GPa to 6,3...7,1 GPa for coating TiO_2 (Fig. 3). Thus, for coatings processed by an electron beam, influence of their thickness on the size of surface microhardness weakens by 30... 40%.

It is also determined that after electron-beam processing of oxide coatings on optical elements, their lifetime is increased by 20... 30%. Thus, the porosity of the surface decreases by 5... 10% and wear resistance rises by 7... 12%.

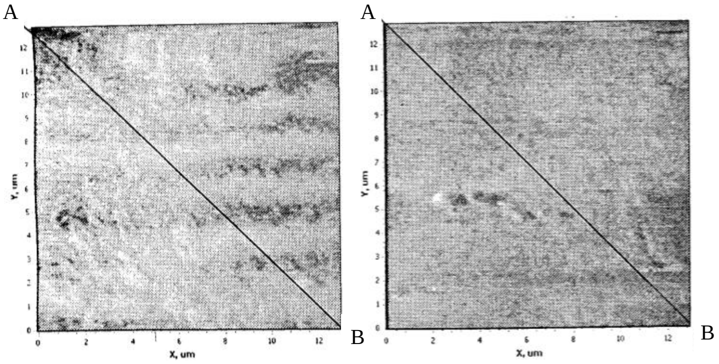
As a result of conducted experimental researches it was also determined, that at critical values of technological parameters of electron-beam installation (electron beam current $I_л$ (mA), accelerating voltage V_y (kV) and distance to processed coating surface l (m)) destructions occur both along the thickness of the optical plate and on the border of oxide coating – the main material of the plate (optical glass K8, BK10, etc.).

Therefore, the preliminary definition and control of the ranges of the critical values of these parameters allows to prevent possible destructions of optical elements and to increase the likelihood of non-failure performance.

Determination of critical modes of electron-beam processing (beam current $I_л^*$, accelerating voltage V_y^* and distance to the processed surface l^*) is carried out for double-layered plate based on the maximum density of thermal exposure of the beam (F_n, Wt/m^2), that allows to find the maximum change ranges of processing modes, within which the plate is not destroyed. The specified F_n values are based on the mathematical processing of experimental data on the sensing of the band electron beam (relative error did not exceed 5... 8%) [3, 15]:

$$F_n(x)=\sqrt{\frac{k_0(I_л,l)}{\pi}}\cdot\frac{I_л\cdot V_y}{B\cdot erf\left[b(I_л,l)\cdot\sqrt{k_0(I_л,l)}\right]}, \qquad (1)$$

$$k_0(I_л,l) = 1{,}237\cdot10^7\text{-}6{,}587\cdot10^5 l\text{-}3{,}725\cdot10^4\, I_л + 1{,}518\cdot10^2\, I_л\, l, \qquad (2)$$



a) b)

Fig. 1. *Topography of the surface area (13 × 13 microns) of the optical element with coating TiO$_2$ to (a) and after (b) electron-beam processing.*

Thereat, it was determined that for performance change ranges of the determined parameters ($I_л$ = 50…300 mA, l = 0,04...0,08 m, B = $6\cdot10^{-2}…8\cdot10^{-2}$ m) the following change processing ranges are realized: k_0 = $(0{,}5…5)\cdot10^7$ m^{-2}; $2b$ = $(0{,}5…1{,}5)\cdot10^{-3}$ m; F_n = $10^6…10^9$ W/m^2.

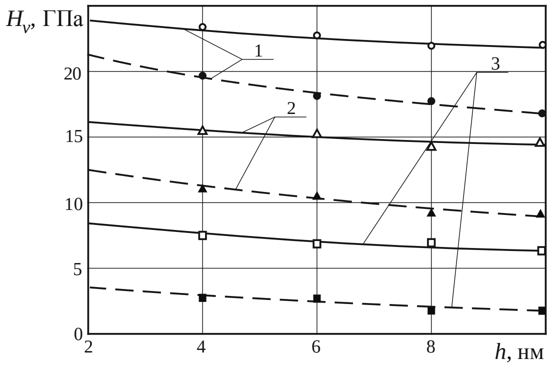


Fig. 3. *Dependences of surface microhardness of optical elements with oxide coatings Al$_2$O$_3$ (1), ZrO$_2$ (2) and TiO$_2$ (3) from their thickness: ——— – after electron beam treatment; – – – – to electron-beam treatment; ∆, ○, □, ▲, ■, ● – experimental data.*

Mathematical model of the heating process of the double-layered plate. The considered plate, which is processed by the scanning electron beam, that realizes the uniform heat effect F_n (fig. 4) on the whole surface. Thus, the plate consists of two layers of heterogeneous materials (one is an optical material, and the other is oxide or other material (aluminum, titanium, etc.)). Between the layers there is an ideal thermal contact, radiation and convective heat losses, in the first approximation, not taken into account, but fulfilled are the conditions: $\delta_1 = 2\cdot\left(a_{01}^2\cdot\tau\right)^{1/2} \sim H_1$ and $\delta_2 = 2\cdot\left(a_{02}^2\cdot\tau\right)^{1/2} << H_2$ [2] (δ_1, δ_2 – penetration depth of the thermal wave inside the coating and plates, m; τ – time of thermal influence of the electron beam, s; a_{01}^2, a_{02}^2 – coefficients of diffusivity of coating and plate, m^2/s), that is, the plate is considered as half boarder environment, on the underside of which the conditions of heat exchange aren't taken into account.

$$b(I_л,l)\frac{1{,}75}{\sqrt{k_0(I_л,l)}}, \qquad (3)$$

where k_0 is concentration coefficient (acuteness of thermal impulse), m^{-2}; $2b$, B is thickness and width of the band electron beam, respectively, m.

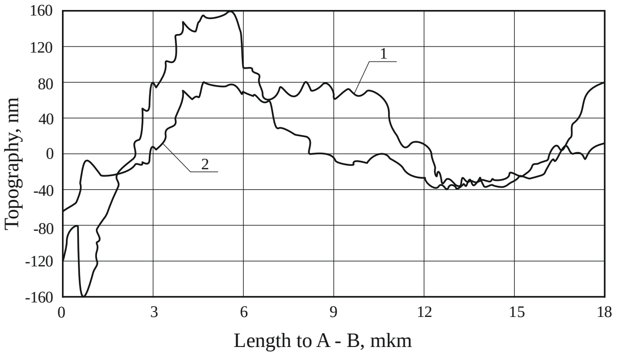


Fig. 2. *Topography along A-B line of the surface area (13 × 13 microns) of the optical element with coating TiO$_2$ to (1) and after (2) electron-beam processing.*

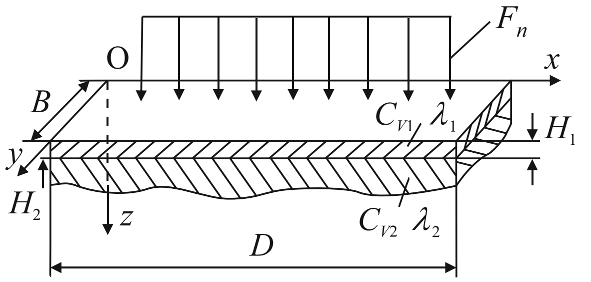


Fig. 4. *Heating scheme of an optical plate with an oxide coating with an electronic beam with thermal impact density F_n: B, D – plate thickness and length, m; C_{V1}, λ_1, H_1, C_{V2}, λ_2, H_2 – volumetric heat capacity (J/m^3·K), thermal conductivity coefficient (Wt/m·K) and length (m) in correspondence with oxide coatings (1) and optical material of the plate (2).*

Equations of the mathematical heating model of the double plate have the form of [18]:

$$C_{V1}(T_1)\cdot\frac{\partial T_1}{\partial t}=\frac{\partial}{\partial z}\left\lfloor \lambda_1(T_1)\cdot\frac{\partial T_1}{\partial z}\right\rfloor,\quad t>0,\ 0<z<H_1, \qquad (4)$$

$$C_{V2}(T_2)\cdot\frac{\partial T_2}{\partial t}=\frac{\partial}{\partial z}\left\lfloor \lambda_2(T_2)\cdot\frac{\partial T_2}{\partial z}\right\rfloor,\quad t>0,\ H_1<z<+\infty, \qquad (5)$$

$$T_1\big|_{t=0}=T_2\big|_{t=0}=T_0, \qquad (6)$$

$$-\lambda_1(T_1)\cdot\left.\frac{\partial T_1}{\partial z}\right|_{z=0}=F_n, \qquad (7)$$

$$T_1\big|_{z=H_1}=T_2\big|_{z=H_1}, \qquad (8)$$

$$\lambda_1(T_1)\cdot\left.\frac{\partial T_1}{\partial z}\right|_{z=H_1}=\lambda_2(T_2)\cdot\left.\frac{\partial T_2}{\partial z}\right|_{z=H_1}, \qquad (9)$$

$$T_2\to T_0,\quad \left(\frac{\partial T_2}{\partial z}\right)\to 0 \ \text{at}\ z\to+\infty. \qquad (10)$$

Taking dependencies into account [2, 3]

$$C_{Vi}=C_{Vi0}\cdot T_i^{\nu},\quad \lambda_i=\lambda_{i0}\cdot T_i^{\nu}\quad (i=1{,}2) \qquad (11)$$

and replacing of variables

$$\theta_i(z,t)=T_i^{\nu+1}-T_0^{\nu+1}, \qquad (12)$$

we get linear system of equations:

$$\frac{\partial\theta_1}{\partial t}=a_{01}^2\cdot\frac{\partial^2\theta_1}{\partial z^2}, \qquad (13)$$

$$\frac{\partial\theta_2}{\partial t} = a_{02}^2 \cdot \frac{\partial^2\theta_2}{\partial z^2}\,, \tag{14}$$

$$\theta_1\big|_{t=0} = \theta_2\big|_{t=0}\,, \tag{15}$$

$$-\frac{\partial\theta_1}{\partial z}\bigg|_{z=0} = \bar{q}_{n0}\,, \tag{16}$$

$$\theta_1\big|_{z=H_1} = \theta_2\big|_{z=H_1}\,, \tag{17}$$

$$\lambda_{01} \cdot \frac{\partial\theta_1}{\partial z}\bigg|_{z=H_1} = \lambda_{02} \cdot \frac{\partial\theta_2}{\partial z}\bigg|_{z=H_1}\,, \tag{18}$$

$$\theta_2 \to 0\,,\quad \frac{\partial\theta_2}{\partial z} \to 0 \ \text{at} \quad z \to +\infty\,, \tag{19}$$

where $$a_{0i}^2 = \frac{\lambda_{0i}}{C_{V0i}}\,, \qquad \bar{q}_{n0} = \frac{\nu+1}{\lambda_{01}} \cdot F_n\,. \tag{20}$$

To solve the obtained linearized problem (13) – (19) we use the direct and inverse Laplace transform by t variable [18] and, considering (12), we get the solution of the initial problem:

$$T_1(z,t) = \left\{ T_0^{\nu+1} + \frac{4(\nu+1)\cdot F_n \cdot a_{10} \cdot \sqrt{t}}{\lambda_{10}} \cdot \right.$$

$$\left. \cdot \left[\sum_{n=0}^{\infty} b^n \cdot i \cdot erfc\left(\frac{2H_1 n + z}{2a_{10}\cdot\sqrt{t}}\right) - \sum_{n=0}^{\infty} b^n \cdot i \cdot erfc\left(\frac{2H_1 n - z}{2a_{10}\cdot\sqrt{t}}\right)\right]\right\}^{\frac{1}{\nu+1}}, \tag{21}$$

$$T_2(z,t) = \left\{ T_0^{\nu+1} + \frac{4(\nu+1)\cdot F_n \cdot a_{10} \cdot \sqrt{t}}{\lambda_{10}\cdot(1+d_0)} \cdot \right.$$

$$\left. \cdot \sum_{n=0}^{\infty} b^n \cdot i \cdot erfc\left(\frac{H_1 n}{a_{10}\cdot\sqrt{t}} + \frac{z}{2a_{20}\cdot\sqrt{t}}\right)\right\}^{\frac{1}{\nu+1}}, \tag{22}$$

where $$i \cdot erfcu = \int_u^{\infty} erfc\xi d\xi = \frac{1}{\sqrt{\pi}} \cdot e^{-u^2} - u \cdot erfcu \tag{23}$$

and $$erfcu = 1 - erfu\,, \tag{24}$$

$$erfu = \frac{2}{\sqrt{\pi}} \int_0^u \cdot e^{-\xi^2} d\xi \quad - \tag{25}$$

special functions [2].

Determination of thermoelastic stresses in the double-layered plate at the electron beam impact. To find the thermoelastic stresses in the considered plate $\sigma_i(z,t)$ (i = 1, 2 – the numbers of coating and plate, respectively) we use the standard ratios for calculating stresses in flat layers [2, 18]:

$$\sigma_i(z,t) = \frac{\alpha_{T_i}\cdot E_i}{1-\nu_i} \cdot \left[-T_i(z,t) + \frac{2}{H_1^2} \cdot (2H_1 - 3z) \cdot \right.$$

$$\left. \cdot \int_0^{H_1} T_i(z,t) dz - \frac{6}{H_1^3} \cdot (H_1 - 2z) \cdot \int_0^{H_1} T_i(z,t) \cdot z dz \right], \tag{26}$$

where α_{Ti}, E_i, ν_i (i = 1, 2) – thermal coefficient of linear expansion (K^{-1}), Jung Module (Pa) and Poisson coefficient respectively.

The results of calculations of thermoelastic stresses $\sigma_1(z,t)$ at the thickness of oxide coating with the use of known physical and mechanical properties of glass K8 and oxide Al_2O_3 [2, 3], as well as standard software are presented on Fig. 5.

From the results of calculations, it follows that the maximum values of thermoelastic stresses can be reached near the surface of the plate, that is, in the oxide coating. It is determined, that they essentially depend on technological parameters of electron-beam installation: increase of current from 200 mA up to 300 mA and accelerating voltage from 6 kV to 8 kV, and also reduction of distance from the processed surface from $8\cdot10^{-2}$ m до $4\cdot10^{-2}$ m lead to growth of thermoelastic stresses in 1,5…2,1 times; at the same time their maximum varies from $0{,}7\cdot10^8$ N/m^2 to $1{,}5\cdot10^8$ N/m^2 and is achieved in the middle of the coating.

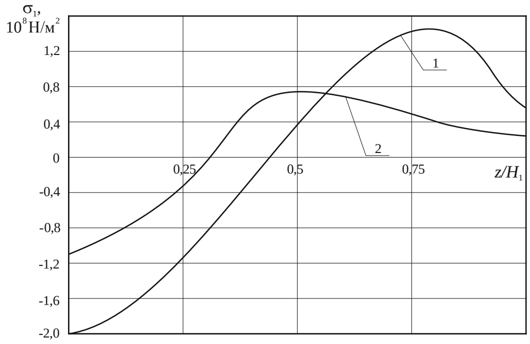


Fig. 5. *Distribution of thermoelastic stresses on the thickness of plates of optical glass K8 with oxide coating Al_2O_3 at their electron beam processing for different times of the beam influence (T_0 = 300 K, H_1 = 10 μm): 1 – $I_л$ = 300 mA, $V_у$ = 8 kV, $l = 4\cdot10^{-2}$ m, t = 0,2 s; 2 – $I_л$ = 200 mA, $V_у$ = 6 kV, $l = 8\cdot10^{-2}$ m, t = 0,2 s.*

In addition, while comparing the obtained values $|\sigma_1|$ with the critical values σ_1^* [3], it is possible to determine the critical values of the controlled parameters $I_л^*$, V_y^* and l^*, the excess of which leads to the destruction of the oxide coatings processed by the electron beam.

In conclusion, it should be noted that the conducted theoretic-experimental researches have allowed to establish the following optimal modes of electron-beam technology, within the boundaries of which there is an improvement of physical and mechanical properties and performance characteristics of optical elements with nanosized coatings from metal oxides: $F_n = 7\cdot10^6 \ldots 8\cdot10^8$ Wt/m^2.

Thus, in light of modern advanced technologies used in optoelectronic instrumentation, electron-beam processing of optical elements with nanosized coatings of metal oxides is potentially applicable for high quality processing of flat and curved elements, obtaining functional microprofiles on their surfaces by means of electronic beams, that can be used as an element base in microoptics, integral and fiber optics, optoelectronics, functional electronics and other areas of precision instrumentation. In addition, the undeniable preferences of electron-beam technology is its ecological purity and the ability to obtain on a common board of optical material in a single technological cycle microelements with improved operational characteristics, the use of which in optical parts of optic-electronic devices contributes to their nonfailure work during operation.

3. Conclusions

1. It is determined, that for optimized modes of electron-beam processing of (density of thermal influence of electron beam $F_n = 7\cdot10^6 \ldots 8\cdot10^8$Wt/м^2) optical elements with wear-resistant and heat-insulating nanosized (< 100 nm) coatings made of metal oxides (SnO_2, Bi_2O_3, TiO_2, ZnO, SiO_2, Al_2O_3, etc.) there is an improvement of their physical and mechanical properties and operational characteristics:

– no negative microdefects are observed on the surface, its porosity decreases by 5... 10%, thereat microroughness decreases from 30... 35 nm to 9... 15 nm;

– the surface microhardness increases from 2,3…3,5 GPa to 23,7…24,9 GPa, and the effect of the coating thickness on the size of the surface microhardness is reduced by 30...40%;

– durability of optical elements increases by 7...12%, and also service life increases by 20... 30%.

2. For the first time mathematical model has been developed for determining the critical modes of electron-beam processing of optical elements with nanosized oxide coatings (beam current $I_л^*$, accelerating voltage V_y^* and distance to the processed surface l^*), that allows to prevent possible destruction of elements and to increase the probability of their nonfailure work during operation by controlling ranges of their changes.

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