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## IMPROVEMENT OF SURFACE LAYERS PROPERTIES OF PRECISION ENGINEERING ELEMENTS OF OPTICAL CERAMICS BY PRELIMINARY ELECTRON-BEAM SURFACING

*I.V. Яценко. Покращення властивостей поверхневих шарів елементів точного приладобудування з оптичних керамік шляхом попередньої електронно-променевої обробки їх поверхонь.* Для запобігання руйнуванням елементів з оптичних керамік практичне значення має попередня електронно-променева обробка поверхонь на стадії виготовлення приладів на їх основі, яка дозволяє покращувати властивості поверхневих шарів елементів. **Мета:** Метою роботи є дослідження впливу параметрів попередньої електронно-променевої обробки елементів з оптичних керамік на попередження їх руйнувань, покращення властивостей поверхневих шарів і підвищення стійкості до зовнішніх термодій. **Матеріали і методи:** Для дослідження впливу параметрів електронного променя на властивості поверхневих шарів елементів з оптичної кераміки використовувались диски діаметром  $3 \cdot 10^{-2} \dots 5 \cdot 10^{-2}$  м і товщиною  $4 \cdot 10^{-3} \dots 6 \cdot 10^{-3}$  м, півсферичні обтічники діаметром  $4 \cdot 10^{-2} \dots 8 \cdot 10^{-2}$  м. Для проведення досліджень термічної дії рухомого електронного променя на елементи з оптичної кераміки було використано електронно-променеве обладнання, що дозволяє реалізувати стрічковий електронний промінь шириною  $5 \cdot 10^{-4} \dots 5 \cdot 10^{-3}$  м, довжиною  $6 \cdot 10^{-2} \dots 8 \cdot 10^{-2}$  м, густиною теплової дії  $F_n = 5 \cdot 10^6 \dots 9 \cdot 10^8$  Вт/м<sup>2</sup> і швидкістю переміщення  $V = 5 \cdot 10^{-3} \dots 10^{-1}$  м/с. **Результати:** В результаті проведених експериментальних досліджень встановлено, що для розглядуваних діапазонів зміни параметрів електронного променя ( $F_n = 10^6 \dots 1,6 \cdot 10^7$  Вт/м<sup>2</sup>,  $V = 10^{-3} \dots 10^{-1}$  м/с) мікротвердість поверхні елементів змінюється від 1.2...2.9 (для необроблених елементів) до 5.7...6.4 ГПа (для оброблених елементів). Покращення вказаних властивостей приводить до підвищення стійкості елементів до зовнішніх термодій. Збільшено у 1.3...1.7 рази критичні значення зовнішніх теплових потоків та час їх дії. Перевищення цих параметрів призводить до руйнування елементів та виходу з ладу приладів для досліджуваного діапазону зміни зовнішнього тиску  $10^5 \dots 10^7$  Па. Підвищено гранично допустимі значення термопружних напружень з 50...140 до 160...370 МПа при температурах нагріву 300...1200 К.

*Ключові слова:* точне приладобудування, оптична кераміка, електронний промінь, мікроструктура, твердість, термопружні напруження.

*I.V. Yatsenko. Improvement of surface layers properties of precision engineering elements of optical ceramics by preliminary electron-beam surfacing.* To prevent destruction of the elements made of optical ceramics the practical importance has the preliminary electron-beam treatment of their surfaces during the manufacturing stage of devices based on them. This allows improving the properties of the surface layers of the elements, making them more resistant to external thermal and mechanical impacts. **Aim:** The aim of this research is to research the impact of parameters of preliminary electron-beam treatment of the elements made of optical ceramics to prevent their destruction, improvement of the surface layers properties and increasing of their resistance to external thermo-influences. **Materials and Methods:** The discs with diameter of  $3 \cdot 10^{-2} \dots 5 \cdot 10^{-2}$  m and thickness of  $4 \cdot 10^{-3} \dots 6 \cdot 10^{-3}$  m and hemispherical cowl with diameter of  $4 \cdot 10^{-2} \dots 8 \cdot 10^{-2}$  m were used to research the impact of electron-beam parameters on surface layers properties of the elements made of optical ceramics. **Results:** After researches it was established that for studied range of electron beam parameters ( $F_n = 10^6 \dots 1.6 \cdot 10^7$  W/m<sup>2</sup>,  $V = 10^{-3} \dots 10^{-1}$  m/s) the microhardness of the elements surface increases from 1.2...2.9 GPa (unprocessed elements) to 5.7...6.4 GPa (processed elements). It was defined, that improvement of these properties leads to improvement of elements resistance to external thermo-influences. The critical values of external heat streams and the time of their actions are increase in 1.3...1.7 times. The excess these parameters leads to the destruction of the elements and failure of the devices for the studied range of external pressure variation  $10^5 \dots 10^7$  Pa. The maximum allowable values of thermal stresses in elements are raised from 50...140 to 160...370 MPa at the heating temperatures of 300...1200 K.

*Keywords:* precision engineering, optical ceramics, electron beam, microstructure, hardness, thermal stresses.

**Introduction.** The successes achieved over the past quarter century in the development of electron beam technology led to the creation of various electron-beam equipment for industrial, scientific, medical and military purposes.

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Modern instruments with elements of optical ceramics, (KO1, KO2, KO3, KO5, KO12 etc) for measurement and thermal testing of different physical objects (plates and discs as substrate filters of infrared devices, input protective window of laser sighting systems for surveillance in IR spectrum, hemispherical fairing of infrared homing devices and monitoring facilities etc. [1...6]) (Fig. 1) are subjected to intense external thermo-influences (high heating temperature and external pressure, shock thermo-influences in terms of shots and flight etc. ).

In these conditions there is a significant change in the properties of the surface layers of the optical elements up to their destruction (cracking, chips and others. defects), leading to a significant deterioration in technical and operational characteristics of devices (reliability, service life, etc.) and their failure.

So important is the prevention of these undesirable effects at the design stage.

Experimental studies [7...14] show that to prevent the destruction of elements with optical ceramics the electron beam methods of pre-treatment of the working surfaces have practical importance. They can significantly improve (more than 2...3 times) properties of the surface layers of the elements (microhardness, thickness reinforced layers, etc.), which in turn affect the stability of materials for external thermo-influences.

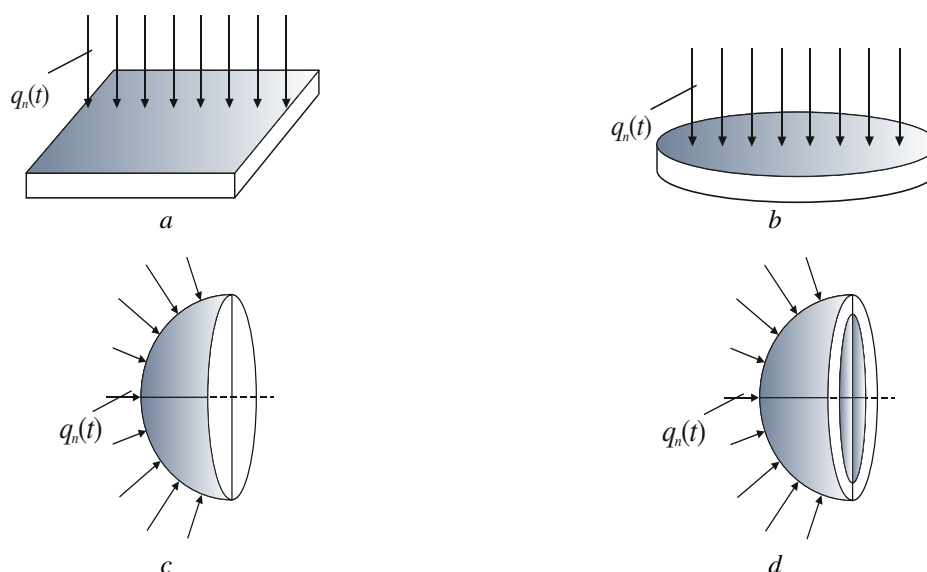


Fig. 1. General view of the optical elements of precision instrumentation which are subjected of external thermo-influences in conditions of operation of devices based on them,  $q_n(t)$  — density of external thermo-influences,  $W/m^2$ : a — plate; b — discs; c, d — hemispherical fairing

Currently, the process of prevention of possible destruction of elements of the precise instrument with optical ceramics under influence of external thermo-influences studied not enough. For example, not paid attention to the calculation the permissible ranges of parameters of the electron beam (density of thermo-influences, velocity), within which there would be a significant improvement of the properties of the surface layers of processed elements and would be missing their local destruction (cracks, bumps, depressions, chips etc).

Further study of the effects of external thermo-influences to elements of precise instrument with optical ceramic will help to improve their resistance to external heat loads and, ultimately, improve the technical and operational characteristics of the devices in their operation.

**The aim** of this research is to research the impact of parameters of preliminary electron-beam treatment of the elements made of optical ceramics to prevent their destruction, improvement of the surface layers properties and increasing of their resistance to external thermo-influences.

**Materials and Methods.** The discs with diameter of  $3 \cdot 10^{-2} \dots 5 \cdot 10^{-2}$  m and thickness of  $4 \cdot 10^{-3} \dots 6 \cdot 10^{-3}$  m and hemispherical cowl with diameter of  $4 \cdot 10^{-2} \dots 8 \cdot 10^{-2}$  m were used to research

the impact of electron-beam parameters on surface layers properties of the elements made of optical ceramics (KO1, KO2, KO3, KO5, KO12) [10, 15].

For research the thermo-influences of moving electron beam to the optical elements with ceramics the specialized electron-beam equipment [10]. The main characteristics of strip electron beam were as follows: width —  $5 \cdot 10^{-4} \dots 5 \cdot 10^{-3}$  m, length —  $6 \cdot 10^{-2} \dots 8 \cdot 10^{-2}$  m, density of thermo-influences —  $F_n = 5 \cdot 10^6 \dots 9 \cdot 10^8$  W/m<sup>2</sup> and velocity —  $V = 5 \cdot 10^{-3} \dots 10^{-1}$  m/s.

*The electron-beam equipment and its main elements.* Equipment was established on the basis of universal vacuum installation UVN-74P3 (Fig. 2) [10]. The vacuum system consists of a vacuum chamber and a vacuum installation post UVN-74P3, oil vapor diffusion pump NP-400, vacuum pump AVZ-20, vacuum meters VIT-3 and VMB-8, vacuum sensors (thermocouple TP-1, ionization IP-1, magnetic blocking M-2) located in the vacuum volume. In a vacuum chamber of the installation the special technological equipment for electronic data processing was placed; there are quartz infrared preheating and final cooling oven, electron gun with Pierce optics to form the strip electron stream, the mechanism of movement of the optical elements. The following external devices provide the special technological equipment operations: high-voltage power source of electron gun based on unit UELI-1, control unit of a quartz oven on the base of thermal sensor — the thermostat RIF-101, an automated processing control system was developed.

For modeling the thermal effects on the studied optical elements under normal conditions ( $T_0 = 293$  K,  $P = 10^5$  Pa) and for finding the critical values of parameters (heat flow  $q_n^*$  and time of action  $t^*$ ) controlled infrared heating has been used quartz lamps of type KNM-220-1000-1 with RIF-101 sensors for temperature control of surfaces elements in the range of 300...1900 K and heat flow that come to them.

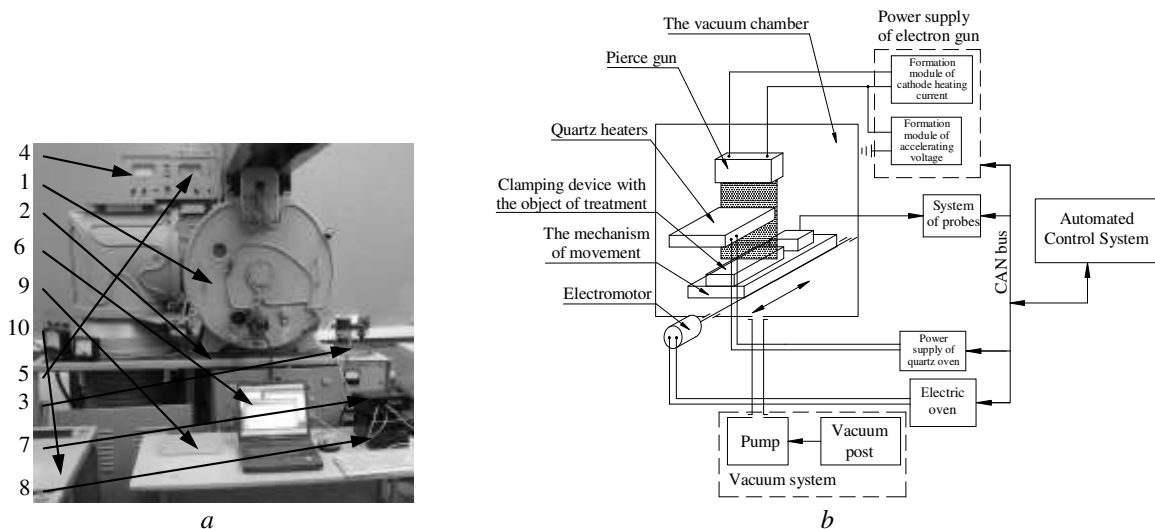
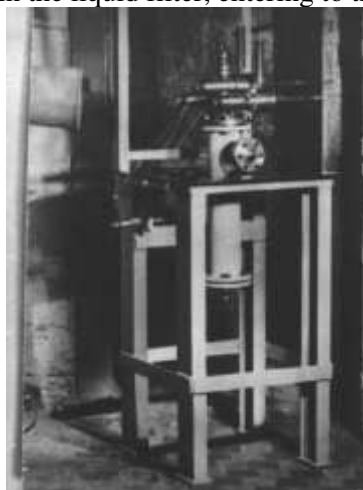


Fig. 2. Appearance (a) and general scheme (b) of equipment for the electronic processing of optical elements: 1 — vacuum chamber; 2 — electric drive mechanism for moving of the optical elements; 3 — temperature control system of optical elements based on thermostat RIF-101; 4 — vacuum meter VIT-3; 5 — vacuum meter VMB-8; 6 — PC for installation control; 7 — central unit of automatic control system; 8 — electric control unit; 9 — modules of temperature measuring in the area of processing and sensing of electron stream; 10 — power supply and control system of Pierce electron gun

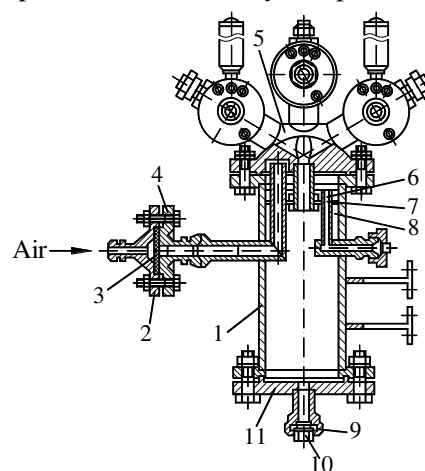
For the modeling of high heating temperatures impact (1500 K) and external pressures ( $10^7$  Pa) the specialized equipment was used, tests on which were held using methodic developed at SDP SE “Arsenal” (Kyiv) and Cherkasy State Technological University.

*Installation for the study of optical elements at high temperatures (1500 K) and external pressures (up to  $3 \cdot 10^7$  Pa).* The installation is shown in Fig. 3 and designed for simultaneous testing of

three elements. Accuracy of working pressure in the installation is  $\pm 5\%$ . The installation consists of a device of permanent pressure and heating system, temperature control and temperature recording. Constant pressure device contains three test chambers which are connected to one unit. Unit of cameras is combined with the body of liquid filter which filling with water before testing. Heating of elements was made directly in chambers. When testing the device of constant pressure filled with an inert gas. All three elements are tested simultaneously. Products that cooled and cleaned of condensed particles in the liquid filter, entering to the valve of constant pressure controlled by compressed gas.



a



b

Fig. 3. Appearance of settings (a) and scheme of constant pressure device (b) 1 — filter housing; 2 — valve cap; 3 — membrane; 4 — valve body; 5 — unit of testing chambers; 6 — drain pipe; 7 — mechanical disk filter; 8 — water; 9 — nut; 10 — cap; 11 — filter cap

To determine the properties of the surface layers of the of optical elements before and after electron beam treatment (microhardness of surface ( $H_v$ , MPa), the quantities of residual thermo stressed ( $\sigma$ , MPa) and thickness of reinforced layers ( $\Delta$ , m) we used known methods of physical and chemical analysis (micro identification by Vickers method, methods of optical microscopy and microprobe analysis, which includes the raster and scanning microscopy (SEM) and transmission electron microscopy (TEM), diffractometers DRON-0.5, DRON-2.0, DRON-3.0 with special consoles for measuring the microstresses in the surface layers, etc. [16...18]). Tensile strength of optical elements  $\sigma^*(T)$  before and after electron-beam processing was found by the central annular bending method [10, 15].

In studies conducted to determine the above mentioned properties of the surface layers of optical elements and critical parameters of external influences the relative error does not exceed 5...10%.

**Results.** Experiments with electronic processing of elements with optical ceramics have shown that they can not be melted in a vacuum because of the high elasticity of vapor [5, 10]. Thus, pre-heating in vacuum of optical elements even up to 1300 K, leads to advanced evaporation of material, and when trying it melting the liquid phase is not formed.

This electronic processing of elements with optical ceramics without heating leads to increase their microhardness, streamline and strengthen the structure by forming the surface layers of compressive stresses and thereby to increase the strength of products to thermal shock effects, which they are exposed to in operation.

Electron microscopic analysis of images of surfaces and transverse thin sections of optical ceramics before and after processing shows that there is a noticeable change in the structure of the material in depth (up to 250...300  $\mu\text{m}$ ), which depending on the electron beam parameters ( $F_n$ ,  $V$ ).

As a result of experimental studies it was established that for studied range of electron beam parameters ( $F_n = 10^6 \dots 1.6 \cdot 10^7 \text{ W/m}^2$ ,  $V = 10^{-3} \dots 10^{-1} \text{ m/s}$ ) the microhardness of the elements surface increases from 1.2...2.9 GPa (unprocessed elements) to 5.7...6.4 GPa (processed elements). The increase in heat density  $F_n$  from  $10^6 \text{ W/m}^2$  to  $1.6 \cdot 10^7 \text{ W/m}^2$  leads to increasing of ceramics surface

microhardness in 1.5...1.7 times, and increase the velocity  $V$  from  $10^{-3}$  to  $10^{-1}$  m/s leads to decrease in microhardness of ceramic surface in 1.3...1.4 times (Fig. 4).

The results of studies of microhardness change on depth of elements with optical ceramics processed by electron beam, shown in Fig. 5. These data suggest that the microhardness of the material of all types of ceramics that were considered sufficiently decreases rapidly heading for its value for uncultivated material. The thickness of the hardened layer ( $\Delta$ ), where there are major structural changes and increased microhardness of the material for the electron beam parameters under consideration ranges from 70...90  $\mu\text{m}$  to 210...230  $\mu\text{m}$  in thickness of processed products  $4...6 \cdot 10^{-3}$  m. The thickness of the hardened layer  $\Delta$  depends greatly on the nature of ceramics as well as the parameters of the electron beam (Fig. 6): increase in heat density  $F_n$  from  $10^6$  W/m<sup>2</sup> to  $2 \cdot 10^7$  W/m<sup>2</sup> leads to increased thickness of the hardened layer in 1.8...2.6 times, and increase the speed of the beam from  $1.5 \cdot 10^{-3}$  m/s to  $2 \cdot 10^{-2}$  m/s leads to decrease in thickness of hardened layer in 1.7...2.5 times.

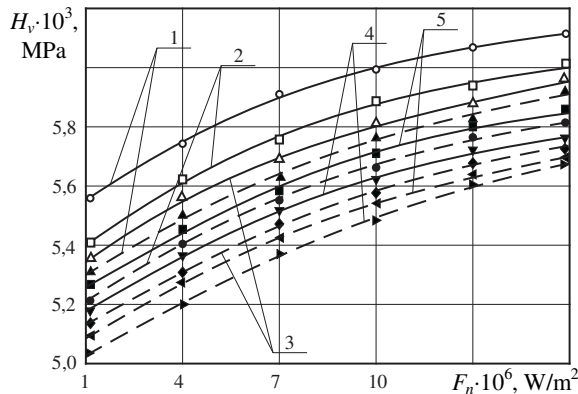


Fig. 4. Dependence of microhardness of the surface with optical ceramics KO12 (1), KO2 (2), KO1 (3), KO5 (4) and KO3 (5), which are processed by electronically beam on its thermal effects density:  $V=7 \cdot 10^{-3}$  m/s (-----);  $V=1,5 \cdot 10^{-2}$  m/s (- - - -);  $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\blacklozenge$ ,  $\blacktriangledown$ ,  $\bullet$ ,  $\blacktriangleright$ ,  $\blacktriangleleft$  (experimental data)

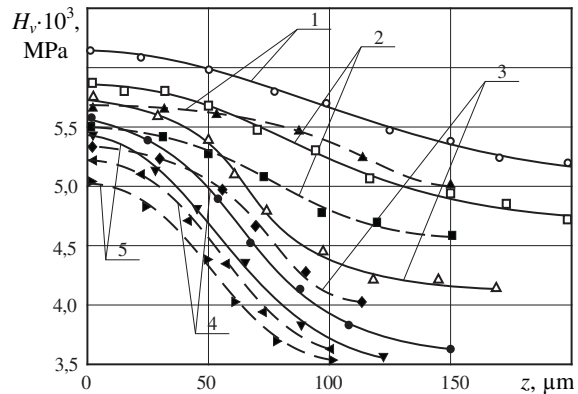


Fig. 5. Change of microhardness on depth of elements with optical ceramics KO12 (1), KO2 (2), KO1 (3) KO3 (4) and KO5 (5), which are processed by electron beam for different speeds of its movement ( $F_n=1,5 \cdot 10^7$  W/m<sup>2</sup>):  $V=7 \cdot 10^{-3}$  m/s (-----);  $V=1,5 \cdot 10^{-2}$  m/s (- - - -);  $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\blacklozenge$ ,  $\blacktriangledown$ ,  $\bullet$ ,  $\blacktriangleright$ ,  $\blacktriangleleft$  (experimental data)

It was established that the influence of the electron beam on the surface of the optical ceramics elements leads to a mosaic block increase and decrease the microstrains of crystal lattice. The value of mosaic blocks for optical elements that electron beam treated, compared to their condition before treatment increased in 3.9 times for items from the KO1, in 5.5 times for items from KO2, in 3.3 times for the elements of KO12, in 4.7 times for items from KO3 and in 7.7 times for items from KO5, and the value of microstrains reduced in 3.7 times for items from the KO1, in 5.4 times for items from KO2, in 4.2 times for items from KO12, in 5.5 times for items from KO3 and in 5.9 times for items from KO5.

It is shown that regardless of technological modes of processing (values  $F_n$  and  $V$  for the examined range of change) of elements with optical ceramics in all cases an increase in the size

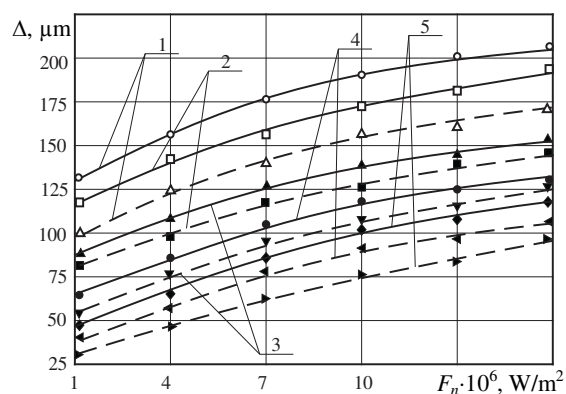


Fig. 6. Dependence the depth strengthening by electron beam the optical elements with ceramics KO12 (1), KO2 (2), KO1 (3) KO3 (4) and KO5 (5) from the values of its thermo-influences density:  $V=7 \cdot 10^{-3}$  m/s (-----);  $V=1,5 \cdot 10^{-2}$  m/s (- - - -);  $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\blacklozenge$ ,  $\blacktriangledown$ ,  $\bullet$ ,  $\blacktriangleright$ ,  $\blacktriangleleft$  (experimental data)

of the mosaics blocks and reduce of microstrains their crystal lattice, that is, as a result of electronic processing we obtain a coarse surface layers with stress in the crystal lattice.

Analysis of the changes parameters of the elements crystal (after processing according to known methods of calculating these radiographs [18], based on the line analytical relationship between residual stresses acting on the surface element and change the period of the crystal lattice of the main components under consideration ceramics, showed the presence of compressive stresses in thin surface layers of the elements of depth 40...60  $\mu\text{m}$  for the central part of the treated areas (plots size  $4 \cdot 10^{-2} \dots 5 \cdot 10^{-2} \text{ m}$ ) in the considerable range of parameters of electron beam, for elements of optical ceramics KO1 — up to 30...40 MPa, for elements of optical ceramics KO2 — up to 60...70 MPa, for elements of optical ceramics KO3 — up to 25...30 MPa, for elements of optical ceramics KO5 — up to 55...65 MPa, for items from optical ceramics KO12 — up to 75...90 MPa.

As a result of studies it was found that after preliminary electron beam processing of optical elements there is increased the critical values of external heat flow  $q_n^*$  and the time of action  $t^*$  in 2...4 times (Fig. 7). The increase in external pressure up to  $10^7 \text{ Pa}$ , which can be implemented, for example, as the shock front at supersonic airflow with fairings infrared device in flight and shot [3, 10], resulting in increased value  $q_n^*$  and  $q_n^*$  only in 1.3...1.7 times (Fig. 8).

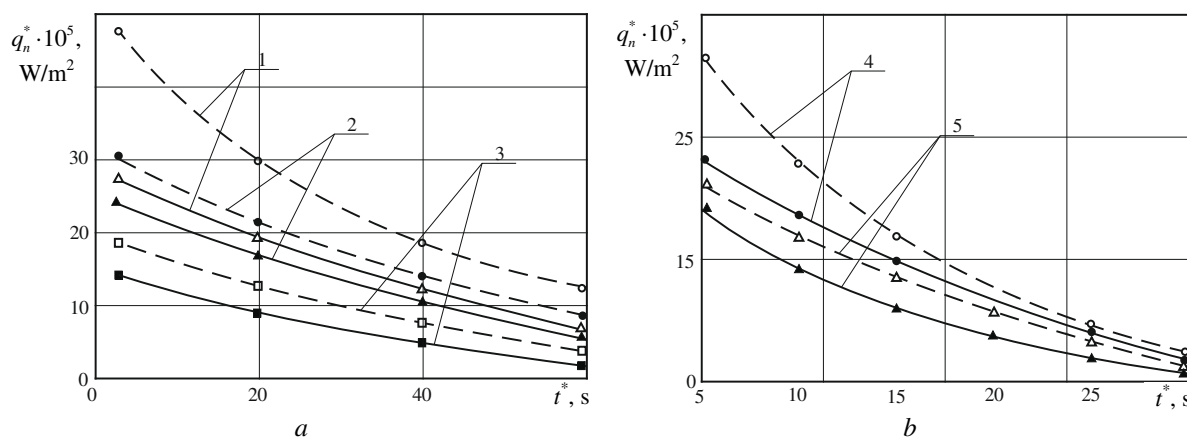


Fig. 7. Dependence of critical values of external heat flows  $q_n^*$  from the time of their effects  $t^*$  on treated and untreated optical elements by the electron beam (element thickness  $H = 4 \cdot 10^{-3} \text{ m}$ ,  $T_0 = 300 \text{ K}$ ,  $P = 10^5 \text{ Pa}$ ):  
untreated elements (—); treated elements ( $F_n = 1,6 \cdot 10^7 \text{ W/m}^2$ ,  $V = 10^{-3} \text{ m/s}$ ) (---);  
a — elements with optical ceramics KO5 (1), KO1 (2) and KO12 (3);  
b — elements with optical ceramics KO3 (4) and KO2 (5);  
 $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\bullet$  (experimental data)

In addition, it was also shown that the maximum allowable value of thermoelastic stresses  $\sigma^*$  at various temperatures of heating  $T$  for the optical elements processed by electronic beam in 1.8...2.7 times higher than for unprocessed elements (Fig. 9).

Using the results obtained in the design and production of new and modernization of serial devices with the examined optical elements for measuring and thermal control objects of different physical nature (IR optical instruments, laser sighting systems, infrared homing devices and surveillance, laser medical devices based on optical fibers, etc.) will increase their basic technical and operational characteristics (reliability, resource and service life, etc.) during operation, taking into account the impact of external thermo actions. For example, during storage and transportation in terms of the emergence of high-temperature centers of fire (warehouse storage, combat zone, etc.), and application of products with infrared homing devices and monitoring in terms of shots and flight (drums exterior thermal and mechanical effects, etc.).

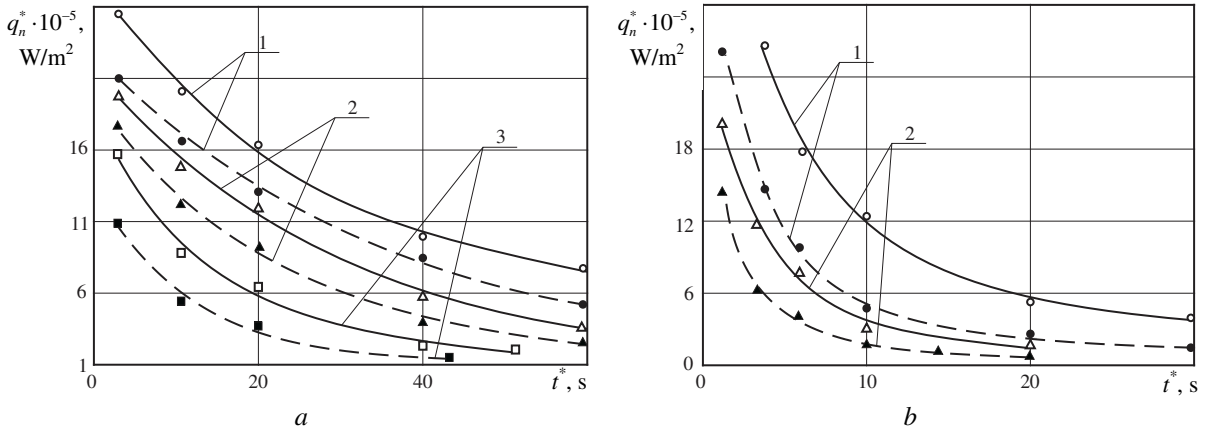


Fig. 8. Dependences of critical values of external heat flows  $q_n^*$  from the  $t^*$  time of their impact on processed by electron beam optical elements (element thickness  $H = 6 \cdot 10^{-3}$  m,  $F_n = 1,6 \cdot 10^7$  W/m<sup>2</sup>,  $V = 10^{-3}$  m/s):  $P = 10^5$  Pa (—);  $P = 10^7$  Pa (- - -); a — elements with optical ceramics KO5 (1), KO1 (2) and KO12 (3); b — elements with optical ceramics KO2 (4) and KO3 (5);  $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\bullet$  (experimental data)

**Conclusions.** The study found that after pre-treatment of working surfaces of optical elements with ceramics (KO1, KO2, KO3, KO5, KO12) by moveable electron beam for studied range of electron beam parameters ( $F_n = 10^6 \dots 1,6 \cdot 10^7$  W/m<sup>2</sup>,  $V = 10^{-3} \dots 10^{-1}$  m/s) the basic properties of the surface layers improve without local damage:

— The surface microhardness is increasing in 1.9...2.3 times;

— Compressive thermostatic stress of 25...90 MPa occur in the surface layers of thickness 40...60  $\mu$ m, which lead to the formation of reinforced layers with thick from 210 to 230  $\mu$ m.

It was defined, that improvement of these properties leads to improvement of elements resistance to external thermo-influences:

— The critical values of external heat flow and the time of their actions are increase in 2...4 times that lead to the destruction of the elements and failure of the devices; increasing in external pressure from  $10^5$  to  $10^7$  Pa reduces critical values in 1.5...1.9 times;

— The maximum allowable values of thermal stresses in elements are raised from 50...140 MPa to 160...370 MPa at the heating temperatures of 300...1200 K.

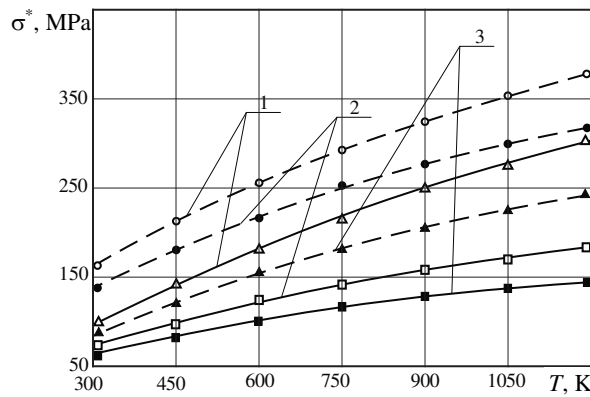


Fig. 9. The dependence of the maximum permissible thermoelastic stresses in the elements from optical ceramics KO1 (1), KO2 (2), KO3 (3) of the heating temperature ( $P = 10^5$  Pa, the thickness of the element  $H = 4 \cdot 10^{-3}$  m,  $F_n = 1,6 \cdot 10^7$  W/m<sup>2</sup>,  $V = 10^{-3}$  m/s): unprocessed element (—); element processed by electron beam (- - -);  $\Delta$ ,  $\circ$ ,  $\square$ ,  $\blacktriangle$ ,  $\blacksquare$ ,  $\bullet$  (experimental data)

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