G.V. Kanashevich

COOLING OF PLATES FROM OPTICAL GLASS AFTER ELECTRONIC MICRO-TREATMENT

Cherkasy State Technological University, Shevchenko street, 460, Cherkasy, 18006, Ukraine

It is shown in the before published works [1–7], that low-power ($E \le 10$ keV) electronic heat treatment of glass plates provides the receipt of chemically clean, optically homogeneous surfaces on these plates, and allows to use electronic streams for microelectronics and integral optics plates production. In works [5; 7] it is reported about the use of electronic method in creation of functional layers in the surface of optical materials. At the same time, low-power electronic treatment ranks to the high temperature processes, due to fact that temperature in the area of stream's local action reaches 1200°C and more, while temperature gradient here amounts to at least 10^2-10^3 °C/mm, that often causes destruction of samples at their treatment and cooling. Today the review of literature confirms that at enough number of works that describes the processes of cooling and annealing of glass after different types of heat treatment, there are no works about glass heat-treatment after its low-power electronic treatment. Taking into account that the technique of the optical materials cooling after their electronic treatment has nothing common with the industrial annealing [8, 9], the issue of determination of the modes of cooling the products from these materials cooling after their electronic treatment, becomes actual.

Determination of the modes of annealing and complete cooling of plates from optical glass after their heat electronic treatment is the purpose of the present work.

Experimental. For electronic treatment round flat-parallel plates from optical glass K8, K108, 5K10, TK21 were used, with the diameter of 20 mm and thickness 4; 6 mm and rectangular flat-parallel plates $70 \times 14 \times 6$ mm, the surfaces of which were formed by the method of deep polishing. Electron-beam apparatus is constructed on the base of the vacuum apparatus "VBH-74", into the chamber of which a thermal stove with the vacuum-tube heaters ("KГM-220-1000-1") was integrated. The stove construction, location of the heaters and plates in space provides the good heat exchange of all points of plates' surfaces with the walls of stove, that in the turn provides isotherm at heating and cooling, fig.1.



Fig. 1. Chart of thermal stove with three screens and reflector, used for heating and cooling at electronic treatment:

1-Source of radiation heating-lamp KΓM-220-1000-1;
2 - Optical glass plate; 3 - Undercup armature;
4 - Vacuum chamber; 5 - To vacuum pump; 6 - Thermal screens; 7 - Reflector

Thermal balance equation for this chart is as follows:

$$\begin{cases} Q_{\Sigma} = Q_{1} - Q_{1}' + Q_{2} - Q_{2}' \\ Q_{O} = Q_{1} - Q_{1}' \end{cases},$$

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where Q_{Σ} – warm content, that is emanated by the reflector; Q_1 – warm content, that is expended on heating of optical item; Q'_1 – warm content, that is emanated by the surface of optical item; Q_2 – warm content, that is expended on heating of undercup armature; Q'_2 – warm content, that is emanated by the surface of undercup armature; Q_3 – warm content, that is expended on heating of thermal screens; Q'_3 – warm content, that is emanated by the surface of thermal screens; Q_{12} – warm content, that is passed from the optical item to undercup armature due to the mechanism of heat exchange; Q_{21} – warm content, that is passed from the undercup armature to optical item due to the mechanism of heat exchange.

Conducted calculations show that a thermal efficiency after the given chart is:

$$\eta = \left(\frac{Q_o}{Q_{\Sigma}}\right) \cdot 100\% = 82.87\%.$$

Cooling was conducted forcedly by means of the device "PH Φ -101" with the error of measuring ±0.5 °C. As an instrument for electronic surfaces plate treatment the Pirs electron beam gun with the band form of electronic stream was used. Parameters of electronic treatment: $P = 10^2 - 5 \cdot 10^3$ W/cm²; $\nu = 1.5 - 4.5$ cm/s; temperature of the preliminary glass heating T = 350 - 450 °C. Remaining thermal tensions in plates were determined by means of the device "IIKC-250".

Results and discussion. The rise of dielectric materials conductivity by the preliminary heating is a physical basis for its electronic treatment. It is a low-power electronic stream of a band form, depending on power and time of action, that can locally warm up, melt, and if necessary to evaporate the thin $(0,5-120 \ \mu\text{m})$ undersurface layer (UL) of optical material plate. Moving along the plate surface such a stream, in the result of thermal and electric action in the conditions of vacuum, forms under itself a new surface and new UL with other composition of chemical elements, that advantageously differs by the optical, mechanical, physical and chemical properties from the original one.

Efficient low-power electronic treatment for optical glass can be realized on two stages of the thermal influencing (STI) of a band low-power electronic stream, namely – without formation of a liquid phase in the surface of material – I STI, and with formation of a liquid phase in the surface of material – II STI.

I STI pursues the purpose of improving of optical properties of the UL of optical wares, namely reducing the component of light dispersion from an optical surface due to homogenization of chemical composition of hydrolysis products, which filling an defected UL. Such a micro-treatment is conducted by an electronic stream to the depth of up to $2,0 \,\mu$ m.

II STI pursues the purpose of improving both optical and mechanical parameters of optical glass items' surfaces by the complete removal of imperfect and crack-type layers and micro-relief reduction of optical surfaces to 2-5 nm. On II STI UL is melted up to the depth of up to $10-120 \mu m$ (fig.2).



Fig. 2. The re-melted electronically superficial layer of 50 μ m in the surface of plate from optical glass TK21 (a) and K108 (b). Zoom×500

The items cooling after their electronic treatment will be efficient and will achieve the technological objective, if to execute it in three stages, namely: time-exposure of products at the maximal fixed temperature, corresponding cooling and rapid (not corresponding) cooling of products (fig. 3).

The product time-exposure is performed at the fixed temperature the value of which can be found within the limits of $T_g - (10-20)^{\circ}$ C for every brand of glass. Satisfactory results are obtained for the products of small size (up to 5 mm) at thermal time-exposure of 40–45 min. For medium-sized products (5–30 mm) and large products (with linear dimensions exceeding than 30 mm) the term of time-exposure increases sev-

eral times accordingly. Taking into account that the interval of cooling includes temperatures of transitional period, which are resulted by an electronic stream in material, time-exposure of products after electronic treatment is to be done as possibly nearer to the top temperature of annealing of material from which the given product is made.



Fig. 3. Chart of cooling and annealing of plates from optical materials after electronic treatment

It allows not only maximally to take off thermal tensions in material, which are carried by the electronic stream, but also to increase chemical homogeneity of the melted layer. Corresponding cooling, as well as high temperature time-exposure, pursues the purpose of minimizing the remaining thermal tensions and forming of transitional layer between the melted surface and basic material in product. Taking into account that the initial optical glass raw item is made preliminary in the conditions of optical manufacturing and tension in it is practically absent, and the thickness of UL, modified by an electronic stream, is much less than general thickness of product, the term of the corresponding cooling can be diminished in relation to standard industrial terms conditions to 4–6 hours.

At cooling of plates in their surfaces expansion tensions appear, and in the middle, accordingly, tension of compression appears. Expansion tensions are more dangerous and can cause the destruction of surface (fig. 4) through its defects (points, scratches and others like that), or to general curvature of product.





Fig. 4. K8 optical glass plate surface destruction as a result of speed-up cooling after electronic treatment, zoom ×3.2

Especially it concerns to the products of small thickness (up to 1.5 mm) with the melted superficial layer of up to 5 μ m. At an incorrectly chosen speed of cooling the product surface collapses (fig. 4), at once after its unloading from a technological volume, or collapses on air after some time (during a few weeks, months).

With the purpose of obtaining high-quality glass plate surfaces a linear law is chosen, and a temperature interval is limited by the values of temperature of annealing (upper temperature) and lower temperature of 300°C, after which products cool down together with a stove. The lower limit temperature (300°C) is chosen on conditions that at this temperature relaxation processes in a structure are finished for silicate glasses.

In the case of presence in the products of the increased remaining thermal tension (exceeding 6.6 MP) after their cooling after a chart (fig. 1), it is necessary, and extremely responsible provide to additionally heat treatment with heating, cooling of products and their ultimate annealing. Advantageous (from merely economic reasons) in this case is annealing on air, as by such a method it is possible to anneal large lots of products simultaneously, what is practically impossible to do in the electron beam apparatus. Annealing on air is conducted after a chart (fig. 5), with the use of industrial stoves for annealing.



Fig. 5. Heat treatment of products from optical materials on air after electronic treatment in vacuum

Table 1. Experimental data from the modes of optical glass heat treatment of different brands at the use of electronic method of treatment

Sizes of blanks	Speed of heating, T °C/min			Temperature of the previous heating, $T_{0\text{max}}$, °C			Time of time- exposure τ, min			Time of the corre- sponding cooling, min		
	K8 K108	BK10	TK21	K8 K108	BK10	TK21	K8 K108	BK10	TK21	K8 K108	BK10	TK21
Shallow blanks (up to 5 mm)	33	33	33	520	550	600	25	25	25	40	40	40
Middle blanks (5–30 mm)	20	20	20	520	550	600	40	40	40	90	90	90
Large blanks (exceeding 30 mm)	18	18	18	520	550	600	120	120	120	120	120	120

Taking into account insignificant sizes and weight blanks and that the Adams and Vilyamson classic formulas and methods [9] do not work correctly for blanks, the surfaces of which are processed by a band electronic stream, the author has experimentally defined for optical glass of brands K8, K108, BK10, TK21 the maximum speeds of heating, maximum temperatures of the previous heating and time of exposition at the maximum temperature for the both stages of the thermal influence at the maximum legitimate values of power of stream and minimum treatment speed. Separate data from the modes of heat treatment of optical glass of different brands after electronic treatment are presented in table 1.

Maximum remaining thermal tensions in details from optical glass after cooling and annealing (after table 1) are found within the limits of 6.1–6.6 MP (fig. 6), that correspond to their reliable operation for not less than 15 years.



Fig. 6. Picture of remaining tensions on the butt end of rectangular plate from the optical glass after electronic treatment of its surface through a mask with next cooling after a chart (fig. 2)

Conclusion. As a result of the conducted work it is possible to do the following conclusions:

1. To prevent destruction of products from optical glass K8, K108, K10, K21 after electronic treatment due to the action of remaining thermal tensions, cooling of products should be done depending on their sizes, and speed of cooling (for blanks with dimensions exceeding 30 mm) must not exceed $2.5^{\circ}C/min$.

2. In case if optical details after the complete cycle of electronic treatment and cooling in vacuum possess thermal tensions which exceed 6.6 MT, their additional annealing in industrial thermal stoves in the conditions of air environment can be the effective tension reduction method.

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Summary

The modes of annealing and complete cooling for plates from optical glass after heat electronic treatment were determined. Recommendations on cooling of optical glass for avoidance of destruction and reduction of remaining thermal tensions in optical products are given.