Improving the mechanical resistance of the glass surface for optoelectronic devices by electron beam processing

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Abstract. The paper considers the issues of hardening the surface of phosphate glasses for optoelectronic devices. The effect of electron-beam processing on the physical-mechanical properties of the glass surface is investigated. It was found that the processing of phosphate glass “SZS23” increases the surface microhardness by 24% owing to removing the damaged layer and changing the elemental composition of the glass surface.

1. Introduction

The state-of-art of the optoelectronic device mainly depends on the quality of its optical appliances. In turn, the quality of the optical appliance is determined by the accuracy of the shape, the stability over time of the optical characteristics and the ability to resist the negative effects of working environment.

The key optical properties transluency, light absorption, dispersion coefficient, refractive index of glass are decisive when choosing the type of optical glass and achieving the best optical characteristics of the device. However, often the unique optical properties of glasses are supplemented with poor technological parameters (low chemical resistance and mechanical strength, etc.). As a rule, this is due to the chemical composition of the glass, the presence of weak bonds between the glass-forming components.

In accordance with the specifics, there are two groups of optical glasses - colorless and colored [1], and the last in most cases refers to non-silicate glasses. The composition of such glasses consists mainly of glass-forming boron, phosphorus, zinc, beryllium and other elements. In our work, we studied zinc phosphate glass, which belongs to the group of metaphosphates.

Colored optical glass “SZS-23” is considered, some of its promising applications are the optical system for spectral filtering of orientation objects radiation in static devices and nanophotonics [2,3]. It is known that the optical sensitivity of the device directly depends on the state of the surface of zinc-phosphate glass, since the glass material is considered soft [2,4]. Electron beam modification of the surface of optical glass can increase the hardness of the glass surface, so in general, the device acquires resistance to aggressive atmosphere. [5].

2. Materials, methods and experimental details

Mechanically ground and polished glass of the metaphosphate group – SZS-23 type were cut into samples of 10×10×5 mm. Before processing, the samples were degreased in a soap solution and distilled water for 15-20 minutes, than were dried by centrifugation.

Electron beam processing (figure 1) was carried out in the high vacuum system described in [6-8]. Electron gun with Pierce optics was used to form a low-energy electron beam with a current density of 50-100 mA/cm². Processing conditions ensured melting of the surface layer to a depth of at least 500 nm. Then samples were slowly cooled (less than 1°C/min) and kept in vacuum (10⁻³ Pa) until room temperature.
Figure 1. The principle of electron beam processing (1 - electron beam gun, 2 - electron beam, 3 - collector, 4 - sample, 5 - secondary electrons, 6 - heater).

The electron beam processing technique is described by the following sequence. Electron beam 2, formed by a gun 1 with a cathode assembly, interacts with sample surface 4. The selection of secondary electrons is carried out by collector 3. The latter is located closed to sample surface. This assembly provides efficient and uniform secondary current extraction throughout sample area. The energy of the primary electron beam is selected in accordance with condition of equal currents $I_1 = I_2$ in the area of stable equilibrium of the secondary emission of the dielectric.

The study of the physical-mechanical properties of initial and electron beam processed [7,8] glass surfaces was carried out by nanoindentation using a nanosclerometric probe module. A cantilever-like bimorph piezoelectric ceramic resonator was applied in accordance with figure 2. A tuning fork type design with two identical consoles is used to increase the sensitivity and noise immunity of the sensor.

Figure 2. Transducer.

The mechanical (elastic) properties were determined by contact techniques of atomic force microscopy and nanoindentation, which are based on monitoring the interaction of the indenter with the surface of the material.

Hardness $H$ and elastic modulus $E$ of the surface were determined by the Oliver-Fahr method.

The value of the hardness $H$ of the sample was determined in accordance with the equation:

$$H = \frac{P_{\text{max}}}{A}$$

here $P_{\text{max}}$ – maximum applied load, $A$ – remaining area at $P_{\text{max}}$.

The contact area at maximum load $A$ is determined by the indenter geometry and the depth of indenter mark or indentation depth $h_{\text{max}}$, and is described by the needle shape function $A = f(h_c)$. 


The contact area at maximum load $A$ is determined by the indenter geometry and the depth of the indenter mark, i.e., the length $L$.

The reduced modulus of elasticity $E^*$ of the sample surface was calculated from inclination angle of the discharge curve using the relations:

$$E^* = \frac{\sqrt{\pi} \cdot S}{2 \sqrt{A}},$$

here $S = \left( \frac{dP}{dh} \right)_{P=P_{\text{max}}}$ – contact stiffness.

The elastic modulus of sample $E_1$ was determined from the equation of the “film-identifier” system:

$$E_i = \frac{3(1-\nu_1^2)}{4 \left( E^* - \frac{3(1-\nu_2^2)}{4E_2} \right)},$$

here $E_2$ – indenter elastic modulus, $\nu_1, \nu_2$ – Poisson’s ratios of contacting materials.

Nanoindentation is the result of the development of techniques and improvement of the design of conventional hardness testers that measure mechanical properties at the macro level. Upon indentation, a solid needle of a known shape is pressed into the surface of the sample at a constant speed. Upon reaching the specified load or indentation depth, the needle is retracted in the opposite direction. During loading, the load values are recorded (figure 3) and the needle is moved.

The resulting characteristic curve, in accordance with figure 4, is called the “load-deformation curve”. It consists of two parts corresponding to the loading and unloading process.

The loading curve characterizes the resistance of the material to the penetrating action of the needle and reflects both the elastic and viscous properties of the material. The part of the curve corresponding to unloading is mainly determined by the elastic recovery of the material. Applying the physical models of hardness testers to the described loading-deformation curve, the hardness and Young’s modulus of the material can be determined.

### 3. Results and discussion

It is known from the authors’ works [2,4] and [5,7] that double aluminophosphate glass, which is close in composition to aluminum metaphosphate, has the highest values of elastic modulus and hardness of 5.7 GPa. Zinc-aluminophosphate glass has the maximum value of structural strength. This assumption is explained by the fact that in zinc-aluminophosphate glass, along with chain structures, three-dimensional sewn structures can exist due to the incorporation of zinc ions into the anion network in tetrahedral coordination.

The surface hardness of the processed glass samples are presented in table 1.
Figure 4. Load and unload curves.

Table 1. Hardness (H) and elasticity (E) values of the electron beam processed surface of zinc-phosphate (SZS-23) glass samples.

|   |   |   |   |   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|---|---|---|---|
| # | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| H, GPa | 7.4 | 7.4 | 7.2 | 7.3 | 7.4 | 7.0 | 7.1 | 7.1 | 7.6 | 7.1 |
| E, GPa | 80.5 | 96 | 98 | 92.3 | 96.8 | 83.1 | 82.9 | 88.0 | 99.9 | 111.3 |

It was experimentally found that owing to electron beam processing, the microhardness of the glass surface increases by 24%. Based on the obtained results, it can be assumed that aluminum metaphosphate or three-dimensional sewn structures appear on the surface, e.g. zinc-potassium polyphosphate KZn$_4$(PO$_4$)$_3$, which will contribute to the strengthening of the surface layer [2,7].

4. Conclusion

Electron beam processing is accompanied by a modification of the surface of the optical details in the form of removing the damaged layer and changing the phase composition of the surface layer. Structural transformations in the surface layer contributed to a change in its mechanical properties. In addition to changing the surface morphology in the modified layer, an increase in its tribomechanical characteristics was observed.

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