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Design of A Thermo-Cell for Studying Optical Characteristics of The Surface Layer During the Heating Process

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Abstract: Study of changes in optical parameters of the surface layer of glass after thermal treatment of optical glasses X-230 and 6Ba-4, as well as changes in optical parameters during storage in air, in a desiccator and for two years in air.

Keywords: Ellipsometry, surface layer thickness, thermocouple, temperature, isothermal vessel, heat treatment, refractive index.

Introduction: The study of the dynamics of optical characteristics of the surface layer (SL) of glasses during heating seems important, since heat treatment greatly increases the processes of aging and corrosion of the glass surface, which occur under natural conditions for a long time. Thus, such an experiment allows us to judge the stability of the surface or possible rearrangements in the SL based on comparatively short experiments.

The purpose of the study was to identify the temperature and storage conditions of the surface layers (SL) and to study the dynamics of their characteristics under conditions simulating operating conditions.

The study of changes in the optical characteristics of the surface layer (SL) of glasses during heating seems important, since heat treatment greatly increases the processes of aging and corrosion of the glass surface, which occur under natural conditions for a long time of storage and operation. Thus, such an experiment allows us to judge the stability of the surface or possible rearrangements in the SL based on comparatively short experiments.

In this work, to study the dynamics of optical characteristics of ПС glasses during heating and simultaneous measurement of ellipsometric parameters (Δ и ψ) ПС of X-230 and 6Ba-4glasses, the research was carried out in a thermal cell in an atmosphere of air Δ and ψ on a device ЛЭФ-2 (radiation wavelength λ=632,8nm at angles of incidence ϕ_0= 〖65〗^(0) и 〖60〗^(0).

The calculation of optical characteristics ПС was carried out within the framework of the model of an inhomogeneous layer with preliminary sets of the optimal profile type [1-3]

In the present work, a thermal cell, schematically shown in Fig. 1, was assembled to study the dynamics of optical characteristics of ПС glasses during heat treatment. The heating element of the cell was made of IXIBHYT stainless steel and had the shape of a glass with a recess for the heating coil and a cavity for the sample. A large ratio of the mass of the heating element to the mass of the sample (~30:I), as well as several layers of thermal insulation made of special materials, ensured good thermal stability of the cell during the test period. A quartz glass in which the cell was placed also contributed to the weakening of heat exchange with the environment. The working space of the cell (the place where the sample was placed) was limited from above by split covers made of stainless steel and quartz, in which there were coaxial holes for a thermocouple, as well as for the passage of incident and reflected rays. The small dimensions of the thermal cell (height ~80 mm; largest diameter ~100 mm) allowed it to be installed on the ellipsometer stage without limiting the possibility of its adjustment in all necessary directions.

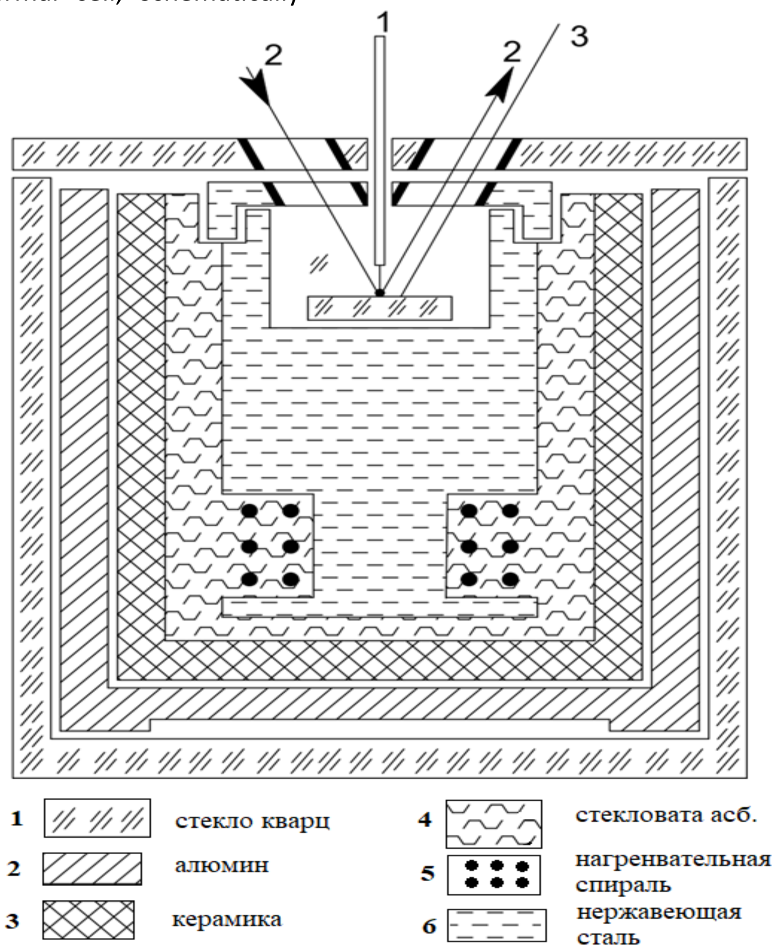

Fig. 1. Construction of a thermal cell for studying the optical characteristics of ПС materials during heating (1-thermocouple, 2-incident and reflected beam, 3 sample)

The cell temperature was controlled by applying a metered voltage to the coil through ЛАТР. The temperature was recorded using a chromel-alumel thermocouple placed in a protective shell and connected to a recorder. In the working state, the thermocouple layer was at a distance of ~ 1 mm from the sample surface, directly above the ellipsometry laser beam spot. In this position, the difference between the sample surface temperature and the recorder readings did not exceed IoC. Taking into account the thermocouple error, the absolute measurement error in the temperature range of 50-3500C did not exceed ± 30 C. A typical temperature-time characteristic of the cell is shown in Fig. 2.

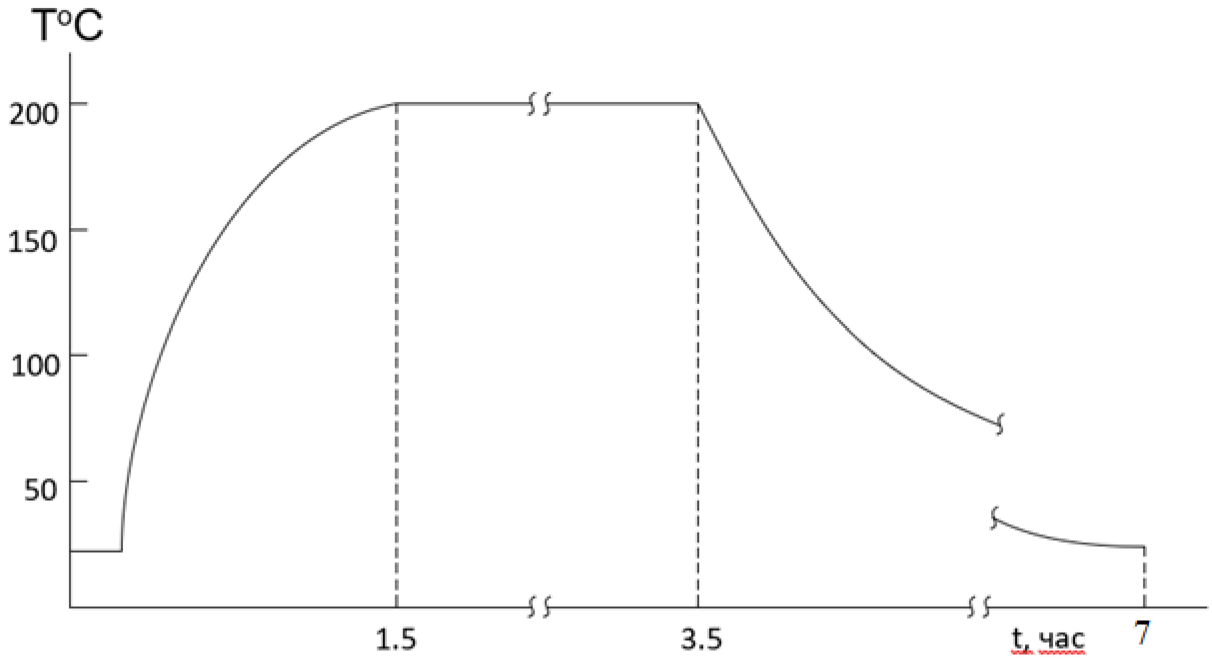

Fig.2 Calibration of the thermocell

The main experimental results are presented in Tables 1-3. Based on these experimental results, on the basis of model representations of the real surface layer, the effective optical characteristics of the layer - the refractive index and thickness - were calculated. ПС [3-7]

Changes in ellipsometric parameters during heat treatment of glass 6Ba-4;

Table -1.

T^0C	φ^0	Δ	φ	n	d	d_0
18^0	60	11,01	2,12	1,6588	1986	2236
	65	2,42	10,03	1,7852	2015	2057
40^0	60	10,46	2,15	1,6447	2058	2301
	65	1,16	10,00	1,6698	2047	2216
60^0	60	10,01	2,14	1,6612	2027	2231
	65	2,37	10,03	1,7718	2026	2068
80^0	60	10,40	2,15	1,6687	2047	2218
	65	2,51	10,00	1,7813	2019	2217

100^0	60	9,43	2,14	1,6600	2037	2080
	65	2,32	10,03	1,7703	2048	2223
120^0	60	10,04	2,15	1,6660	2048	2223
	65	2,34	10,02	1,7222	2090	2160
140^0	60	10,11	2,13	1,6585	2005	2236
	65	2,13	10,04	1,9074	1867	1885
160^0	60	9,55	2,17	1,6817	2079	2197
	65	1,53	10,02	1,6837	2136	2229
180^0	60	8,49	2,17	1,6755	2088	2205
	65	1,54	10,03	1,7168	2114	2268
200^0	60	9,12	2,17	1,6777	1085	2201
	65	2,16	10,01	1,6852	2117	2226

Changes in ellipsometric parameters during heat treatment of glass X-230;

Table -2.

T^0C	φ^0	Δ	φ	n	d	d_0
18^0	60	0,23	4,385	1,5383	1474	2545
	65	0,13	12,19	1,5383	1474	2545
40^0	60	0,35	4,36	1,5383	1414	2487
	65	0,11	12,22	1,5373	1512	2547
60^0	60	0,13	12,20	1,5380	1497	2546
80^0	60	0,36	12,22	1,5373	1354	2548
	65					
100^0	60	0,052	12,20	1,5378	1364	2546
	65					
120^0	60	0,33	4,34	1,5394	1382	2485
	65	0,051	12,20	1,5378	1363	2546
140^0	60	0,34	4,37	1,5383	1425	2488
	65	0,016	12,16	1,5390	1291	2543
160^0	60	0,12	4,36	1,5386	1303	2487

	65	0,12	12,18	1,5386	1444	2544
180^0	60	0,16	4,37	1,5383	1330	2488
	65	0,061	12,21	1,5375	1393	2547
200^0	60	0,27	4,37	1,5384	1388	2488
	65	0,18	12,20	1,5382	1575	2545

In the heat treatment experiment, glass samples X-230 and 6Ba-4 of size 15x 5мм2 were selected. The samples were polished using standard technology (aqueous suspension of polyrite). In the initial state after polishing, large deviations in the refractive indices (Δn=0,05) from the bulk value and a spread of effective thicknesses ПС (Δd=1500A^0) were observed, both from sample to sample and over the surface of the sample. Moreover, the refractive indices in 07 were less than in the bulk. Heating the samples to 2000 C led to the alignment of both the refractive index and the thickness 08. At a temperature of 180-2000C C, the refractive index approached the bulk value. The spread in thickness also decreased to d=1500A0.

Polishing of glass in anhydrous suspensions led to similar results, which confirmed the significant influence of water on the characteristics of the ПС environment.

Table 3 shows the results of ellipsometric measurements on glass 6Ba-4 and borosilicate glass X-230, depending on storage conditions. From the data in Table 3 it follows that during storage in air the refractive index decreases, and the thickness of the PS increases, which also confirms the influence of air

humidity on the optical characteristics of the ПС. The study showed that heat treatment of glass stabilizes the parameters of the ПС regardless of the history of the treatment. Moreover, this state is maintained for glass 6Ba-4 within 5-6 days, and for glass X-230 up to 3 days. This can be used in practice when manufacturing products from this type of glass.

Changes in ellipsometric parameters during heat treatment of glass X-230 и 6Ba-4 during storage.

Table -3.

Glass brand	№ обр.	Original		2 months on air		2 months on a desiccator		2 years in the open air	
		$d(A^0)$	n	$d(A^0)$	n	$d(A^0)$	n	$d(A^0)$	n
X-230	1	87	1,488			1533	1,5312	1985	1,478
n=1,531	2	320	1,533			1518	1,5301	1832	1,489
	3	110	1,513	305	1,4049			972	1,514
	4	117	1,524	326	1,4929			914	1,523
6Ba-4	1	87	1,568	140	1,587			2069	1,587

n=1,639 0	2	126	1,567	124	1,597			2176	1,5480
	3	139	1,567			147	1,586	2112	1,5920
	4	126	1,594			210	1,597	2051	1,6020

The following can be noted:

1. It has been established that temperature treatment stabilizes the optical properties of the surface. The refractive indices approach the volumetric value.

2. Surface layer of glass X-230 и 6Ba-4 consists of a layer of adsorbed water with a thickness of 100 to 300A0 and a diffuse layer of bound water of thickness 1500A0 до 2000A0 (and maybe more, since ellipsometry allows us to determine the thickness of transparent layers with an accuracy of up to half a period, which in this case is about 2000A0);

3. Glass storage X-230 in a desiccator with silica gel leads to the removal of the adsorbed layer and the exposure of the diffuse layer, which is large. Heating up to 2000 C leads to the same results.

4. Determination of optimal heat treatment and chemical etching conditions for the purpose of stabilizing the optical characteristics of ПС in glass X-230

5. A method for determining the water content in glass has been developed X-230.

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