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Research of optical and mechanical properties of lithium aluminosilicate glass-ceramics

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ABSTRACT

An optical method of registration of optical and mechanical coefficients in glassceramic materials is offered. The method is based on the modulation of polarization of laser radiation transmitted through anisotropic area and the definition of its anisotropy parameters by means of this modulation. Residual stresses in aluminosilicate glass-ceramics caused by the temperature gradient were studied by the offered method. The functional relationship of the coordinate dependence of the residual stress and stress optical coefficient in Poisson equation was established. It is shown that in the absence of temperature gradient the birefringence value and dichroism are related by the Kramers-Kronig relations. These parameters are connected with a residual mechanical stress, which has a spatial distribution.

1. Introduction

As it is well known, glass-ceramics are inorganic materials of wide-range technical purpose, which have significant hardness, mechanical strength, thermal and chemical resistance [1]. Glass-ceramics can be obtained by means of bulk crystallization of glass consisting of one or several crystalline phases [2]. In case of their homogeneous distribution in the glass phase, these substances are amorphous by structure and have isotropic mechanical and optical properties. However, one of the important production steps is a cooling process that inevitably causes inhomogeneity of the temperature gradient. In accordance with the thermoelasticity equation, changes of temperature values in time and in volume of materials are accompanied by generation of mechanical stresses [3,4]. The thermoelasticity equation is based on the theory of heat conduction [5] and elasticity [6]. Moreover, such structural transformations as crystallization, surface evaporation and diffusion of components cause residual stress. Getting information about the magnitude and nature of these stresses in the initial state, their evolution after annealing and glass hardening [7] is the most important task. Knowledge about stresses on the surface and inside of the samples is the most valuable information for modeling, accounts and creation of glass-ceramic based products.

Among numerous known methods of detecting thermal stresses [8], optic-polarization method has a leading position based on its historical origin [9] and effectiveness [10]. In order to register mechanical stresses using optical-polarization method, it is necessary to create condition of half-wave plate in the sample. Herewith informational content and detection ability of material dielectric properties to anisotropy is fairly small. Improvement of this method by modulation polarimetry technique allows registering small changes of complex refractive index $N = n - ik$ in materials [11,12]. At the same time detection ability, value range and informational productivity of the method increase several times. Verification of the informational ability of Kramers-Kronig relations for increments of refractive index values Δn and absorption coefficient $\Delta \kappa$ is the first task of this research. The second task of the research is establishment of connection between optical parameters Δn, $\Delta \kappa$ and residual stress function.

2. Experiment and samples

2.1. Physical principle of the method and optical schema of the setup

For registration of residual stresses, the method of photoelasticity is proposed. This method has recently evolved and is related to a category of non-destructives. However, it is not the only version of the method described in classical monographs. There is a newest version, which allows us to find low residual stresses in Zerodur kind materials where these stresses usually considers as absent [13]. In this case, we deal with the technique of the modulation polarimetry (MP) whose detectivity is unsurpassed for registration of the birefringence caused by a directed strain. MP technique is described in detail in [11–13].

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The optical schema of equipment for measuring Δn is shown in Fig. 1a. Its operation principle is the following: the radiation from monochromator “1” ($\lambda = 0.34$–1 μm) is directed on the polarizer “2”, after which the radiation became linearly polarized at angle of 45° to X- and Y-axes. This radiation directed on the sample “3”, which is uniaxial compressed along the Y-axis. Due to anisotropy caused by compression, the linearly polarized radiation is converted into elliptically polarized and is directed on the photodiode “6” through the polarization modulator “4” and polarizer “5”. The elliptic polarization is a mixture of linearly and circularly polarized light. As it was stated above, the intensity of a circular component is proportional to the magnitude of stress $(\sigma_1 - \sigma_2)$. The polarization modulator “4” is a special device that allows registering the intensity of only the circular (or linear) components of the elliptically polarized light. Thus, by scanning over the sample surface it is possible to register the distribution of birefringence Δn and stress in real time. The setup allows to register a signal proportional to the value of $(\sigma_1 - \sigma_2)$ in relative units. However, stress distribution can be obtained in absolute units. In this case, the setup should be calibrated.

The optical schema of equipment for measuring $\Delta\kappa$ is shown in Fig. 1b. The radiation from monochromator 1 ($\lambda = 0.34$–1 μm) is directed on the polarizer 2 and then on the λ/4 phase plate 7, after which the radiation became circularly polarized. Further, the radiation is directed on the polarization modulator 4, which converts circularly polarized radiation in a linearly polarized twice for a period. Azimuths of planes of linearly polarizations are parallel or perpendicular to optical axis of the sample 3, which was uniaxial compressed along the Y-axis. The difference between transmitted radiation intensities registered by photodiode 6.

Analysis of systematic and random errors of the photoelastic effect by using modulation polarimetry technique was performed. The measured signal (photoelastic signal) was induced by non-stationary anisotropy of the refractive index at the point with x – coordinate [14–16]:

$$U(t,x_{\mathrm{i}}) = \frac{I_0}{\pi k} A_i(\ F_0(t,x_{\mathrm{i}}), Bi(t,x_{\mathrm{i}}))\ \sin\left(\frac{2\pi}{\lambda}\left[n_{\perp} - n_{||}\right]\right) \times \\ \times\ \sinh(\sigma_T(t,x_{\mathrm{i}}))e^{-\sigma_T(t,x_{\mathrm{i}})} \tag{1}$$

I_0 – intensity laser, t – time, x_i - sample coordinate, A_i - correction function of heat exchange processes at temperature front, F_0 - thermal Fourier number, Bi – Biot number, σ_T – mechanical stress.

The signal value was measured, and then U value was calculated using the formula (1) in our technique. Herewith the error of the U value is estimated according to the formula:

$$\varepsilon_U = \frac{\Delta U}{U} * 100\% = \sqrt{\sum_{p=1}^{m}\left(\frac{\partial f}{\partial n} * \frac{\Delta \overline{n}}{\overline{n}}\right)} * 100\% = 3,5\% \tag{2}$$

m –number of index refraction arguments n, $\frac{\partial f}{\partial n}$ - partial derivative.

Thus, the error of the U value equals to 3,5%. It meets the conditions of experimental results while measuring photoelastic processes in solids.

Experimental curve errors were evaluated. The value of the errors is comparable to the thickness of the graph line and equals to 1%. This was proven by multiple reproductions of experimental points on the graphs.

2.2. Samples

Samples of lithium aluminosilicate (Li_2O–Al_2O_3–SiO_2) glass ceramics were made as follows. The initial glass ingot had a composition: $SiO_2 = 63.3\%$, $Al_2O_3 = 25\%$, $TiO_2 = 5.5\%$, $Li_2O = 3.9\%$, $ZnO = 1\%$. This ingot was made by a technology described in Ref. [17]. Samples of the size of $x \times y \times z = 30 \times 10 \times 4$ mm were obtained from the ingot by cutting. Further, the samples were annealed at 700°C for 1 h for nucleation. During the heating and cooling, the temperature gradient (grad T) arose in the sample. The temperature gradient was oriented in the heat flow direction. After annealing, all surfaces of the sample were subjected to optical polishing to decrease surface stress and scattering of radiation. Optical anisotropy measurements of samples have been performed in two ways: free sample at a room temperature after the heat treatment; the sample under adjusted uniaxial compression at a room temperature.

3. Results and discussion

3.1. Experimental functions of optical and optical-mechanical coefficients

Graphic model of complex refractive index components is shown in Fig. 2 for a discussion of measurements results. These components are frequency characteristics of amplitude and phase of the classic oscillator and related by Kramers-Kronig relations. The transmission spectrum

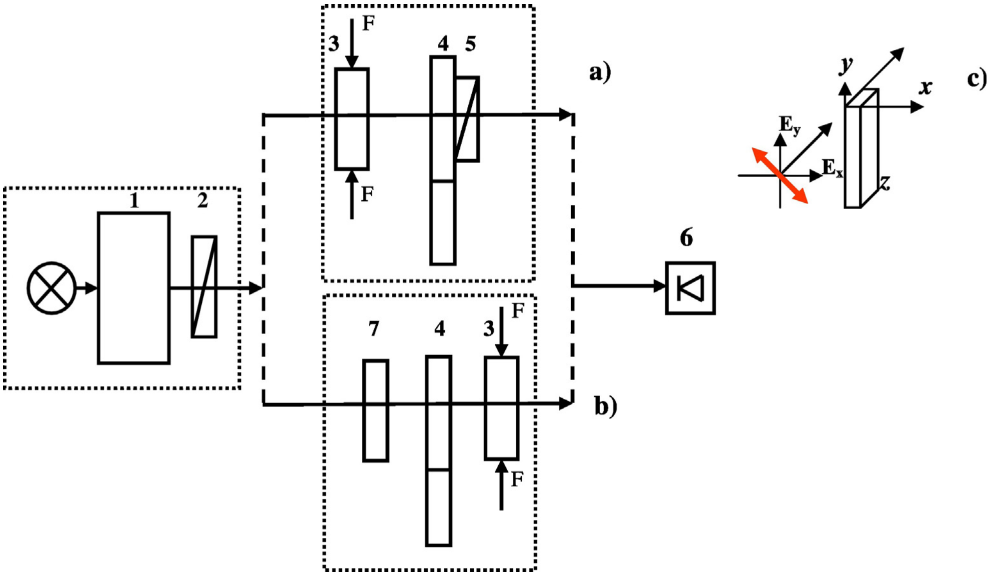

Fig. 1. The optical schema for measurements of birefringence (a) and linear dichroism (b): 1 - light source (halogen lamp, a monochromator); 2, 5 – linear polarizers; 3 - uniaxially compressed sample; 4 - photoelastic polarization modulator; 6 - photodetector; 7 - λ/4-phase plate; c) geometry of the experiment.