MANUFACTURING METHODS FOR OPTICALLY AND IR TRANSPARENT GLASS CERAMICS


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A method for manufacturing optically transparent sitalls from glasses based on brown coal ashes from the Kuybyshev Automobile and Tractor Electrical Equipment and Carburetor Plant (KATEK) was developed in laboratory conditions. The optical, mechanical, and thermal properties of the transparent sitalls were as good as for quartz glass. They are promising for use as the base for optical and magnetooptic information carriers and in fabrication of band-pass filters in the visible and near IR region of the spectrum.

The problem of finding materials capable of replacing traditionally used quartz glass has become pressing due to the intense development of glass fiber optics and optical data reading and recording systems using semiconductor lasers.

One possible alternative version could be the use of transparent glass ceramics — sitalls (US Patent No. 4,707,458). There is a method of clarifying sitalls fabricated from glasses based on metallurgical slags with addition of zinc oxide as the clarifier.* Light tones of sitalls which are not transparent are obtained in this case. Glasses of spodumene or mullite composition appropriately treated with heat are used for manufacturing optically and IR transparent sitalls (US Patents Nos. 4,057,434, 4,575,493, 4,396,720). However, these methods require use of high-melting (melting point of 1500-1700°C) and expensive oxides (Li2O, Ga2O3) and rare-earth elements as the initial raw material.

We have developed a method for manufacturing sitalls which are transparent in the visible and near infrared regions of the spectrum from glasses based on ashes from burning brown coals from KATEK.

Ash of the following composition (here and below, wt. %) was used for conducting the experiments: 13.0-55.1 SiO2, 5.0-13.1 Al2O3, 23.1-54.5 CaO, 6.0-16.1 Fe2O3, 1.2-5.7 FeO, 1.9-6.0 MgO, 0.2-0.7 K2O, 0.1-1.1 Na2O, 0.1-0.7 TiO2, 0.07-0.57 S. A batch consisting of ash of the indicated composition with zirconium and chromium oxides added in the amount of 2.0 and 0.5%, respectively, was used as the starting material.

The ash or batch was melted in a graphite crucible (reducing medium) at 1350-1400°C for 1-1.5 h. The oxide and sulfide forms of iron were reduced to the metallic state and the glass melt was homogenized. On pouring the melt into water, amorphous glass in the form of porous granules of white glass melt and beads of metallic iron, removed manually or with a magnet, was obtained.

The glass granules were crushed to 0-80 μ in size and wet magnetic separation was conducted, after which the concentration of iron in the glass powder decreased to 0.1-0.2%. Samples were molded from the powder and after two-stage heat treatment, they spontaneously cooled to room temperature in an electric furnace.

The angles of rotation of the polarization plane of light passing through the samples in fields of up to 15 kOe (magnetooptic activity), optical absorption spectral curves, and vitrification temperatures were investigated to determine the practical use of samples of transparent sitalls as the basis for creating reversible magnetooptic data carriers for large and superlarge computers.

The tests were conducted on a Specord spectrophotometer using a profilometer (scattered light) on a magnetooptic bench, a URM3-279-050 vacuum technological setup, and a KS-600 high-temperature furnace. The samples of the sitalls were made in the form of disks 16 mm in diameter and 1 mm high whose ends were polished according to class 14. The transmission spectra were investigated in the range of wavelengths from 30·103 to 14·103 cm⁻¹, which corresponds to the visible region of the spectrum, including part of the ultraviolet and near infrared. The results of the tests are reported in Table 1.
Fig. 1. Transmission spectra of the samples: 1) sample based on glass from ash; 2) same, with addition of 2% zirconium oxide; 3) same, with addition of 0.5% chromium oxide.

The most characteristic transmission spectra of three samples of transparent sitalls are shown in Fig. 1. In the sitalls manufactured from ash-based glass with no additives, the edge of the transmission band is located in the 29·10^3 cm\(^{-1}\) region. The intensity of the spectrum attains the maximum (−80%) in the 18·10^3 cm\(^{-1}\) region and drops off sharply in the 15·10^3 cm\(^{-1}\) region. The spectra of the sitalls fabricated from ash-based glass with zirconium oxide additive are of the same type, but the edge of the transmission band is shifted to the ultraviolet region; for this reason, the transmission coefficient for wavelengths of 30·10^3 cm\(^{-1}\) is equal to 7%.

When chromium oxide was added to the ash, the edge of the transmission band was shifted to the long-wave region to 26·10^3 cm\(^{-1}\), and the transparency coefficient decreased to 50%.

Tests were conducted to obtain optical surfaces by ordinary and deep polishing and with spray-coating and a complex study of RZ-PM magnetooptic films and magnetodielectric films. The results of the tests showed that the samples satisfactorily undergo optical polishing, and the films sprayed on them exhibited satisfactory adhesion and the magnetic and magnetooptic properties traditional for these materials.

It should be noted that the use of these sitalls as supports for magnetodielectric films allows obtaining (in contrast to quartz supports) a more finely disperse crystal structure, which decreases crystal noise in magnetooptic reading of data.