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Borosilicate Radiation Shielding Glass

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Abstract—The results of studies of the electrophysical and thermophysical characteristics of borosilicate glass designed to attenuate electromagnetic radiation in the microwave range are presented. The compositions of glass with the minimal tendency to crystallization are determined. The effect of the chemical composition of glass on its thermophysical (temperature coefficient of linear expansion (TCLE) and heat capacity) and electrophysical (attenuation index, standing wave ratio, and dielectric loss tangent) properties is found. The proposed glass compositions can be recommended for the manufacturing of products used as shielding against radiation.

Keywords: radioprotective glass, microwave electromagnetic radiation, attenuation index, standing wave ratio, heat capacity, temperature coefficient of linear expansion, glass structure

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INTRODUCTION

At present, the ecology and environmental protection are priority issues throughout the world. A significant part of industrial production is accompanied by various types of radiation. Their nature is due to the processes occurring in the substance: moving electric charges (electromagnetic waves), the change in the time of the dipole electric moment of the electric system (electric dipole radiation), the transition of the thermal energy of the substance into radiation energy, the change in the excited state of the nuclei of the substance (hard electromagnetic gamma radiation), etc. [1].

The World Health Organization has classified electromagnetic radiation as one of the types of energy pollution of the environment, and electromagnetic smog has been identified as one of the main components of environmental pollution. The biological activity of electromagnetic fields increases with a decrease in the length of the electromagnetic wave and reaches the maximum level in the microwave (UHF) region [2].

One of the main means of protection against radiation is shielding, i.e., reflection, absorption, and multiple reflection. At a sufficient distance from the radiation source, its intensity weakens to a value that is safe for humans due to the interaction of radiation with atmospheric air. Limiting the time spent in the irradiated area and the use of protective screens help to ensure the safety of humans. Depending on the nature of the radiation, the safe distance, time spent in the irradiation zone, and protective shielding are calculated [3].

Currently, engineering and technical methods, as well as the means of protection against microwave radiation, are based on the implementation of the principle of electromagnetic wave reflection using metal shields, grids, and foil and are widely used in practice but do not allow solving the whole complex of problems of protection against microwave radiation [4].

In relation to this, the problem of developing qualitatively new effective methods and means of protection against microwave radiation has become urgent; in particular, glass with a special set of electrophysical characteristics, which is designed for highly efficient absorption or reflection of electromagnetic radiation and is considered radioprotective, is of interest [5].

The interaction of glass with an electromagnetic field predetermines the set of special requirements for them: the required value of the dielectric constant, the tangent of the dielectric loss angle, a certain amount of absorption or reflection of electromagnetic radiation in the radio frequency range, and the presence of electrical conductivity.

The aim of this study is to develop compositions of radioprotective glass that attenuate electromagnetic radiation in the range of 1–3 GHz and study the effect of the chemical composition on the complex of thermal and electrophysical characteristics.

EXPERIMENTAL

The glass compositions are given in Table 1: mol %, $(17.5–27.5)R_2O–(10.0–20.0)B_2O_3–(62.5–72.5)SiO_2$, where R_2O is the sum of K_2O, Na_2O, and Li_2O. The high content of alkaline ions is due to their mobility;

Table 1. Compositions of the studied glass

Composition no.	Oxide content, mol %				
	SiO_2	B_2O_3	R_2O		
			Na_2O	K_2O	Li_2O
1	72.5	10.0	16.5	0.5	0.5
2	70.0	10.0	19.0	0.5	0.5
3	70.0	12.5	16.5	0.5	0.5
4	67.5	10.0	21.5	0.5	0.5
5	67.5	12.5	19.0	0.5	0.5
6	67.5	15.0	16.5	0.5	0.5
7	65.0	10.0	24.0	0.5	0.5
8	65.0	12.5	21.5	0.5	0.5
9	65.0	15.0	19.0	0.5	0.5
10	65.0	17.5	16.5	0.5	0.5
11	62.5	10.0	26.5	0.5	0.5
12	62.5	12.5	24.0	0.5	0.5
13	62.5	15.0	21.5	0.5	0.5
14	62.5	17.5	19.0	0.5	0.5
15	62.5	20.0	16.5	0.5	0.5

however, the upper limit in using alkali metal oxides is limited to 30 wt % content, which is due to the fact that they cause a sharp decrease in the chemical resistance of the glass.

We used SiO_2 and H_3BO_3, as well as carbonates Li_2CO_3, Na_2CO_3, and K_2CO_3 with the basic substance content not less than 99.9% as raw materials. The glass was synthesized in a gas furnace at 1500 ± 20°C with isothermal exposure for 2 h and subsequent annealing at 580 ± 5°C.

The crystallization ability of the synthesized glass was assessed by the method of gradient heat treatment in the temperature range 400–1100°C and isothermal exposure for 1 h in a SP30/13 LAC gradient furnace.

Thermal analysis was performed by differential scanning calorimetry (DSC) on an STA 449 F1 JUPITER calorimeter (Netzsch) in the temperature range of 50 to 1000°C in an argon atmosphere at a purge rate of 20 mL/min and heating rate 10°C/min. Analysis of the DSC spectra and separation of the peaks were performed using the NETZSCH Proteus software package.

A DSC 404 F3 Pegasus calorimeter (Netzsch) was used to determine the specific heat capacity in the temperature range of 50 to 1000°C. The heating rate was 10°C/min. The results of measurements were processed using the NETZSCH Proteus software.

The determination of the temperature coefficient of linear expansion (TCLE) of glass was carried out on a DIL 402 PC electronic dilatometer (Netzsch) in the temperature range of 20 to 300°C, heating rate 5°C/min.

The glass density was determined by hydrostatic weighing at room temperature in accordance with *GOST* (State Standard) *9553-2017*.

The thermal stability of the studied glass was determined by the method described in *GOST 25535-2013*. Defect-free annealed glass samples in the form of plates were used as the test samples.

The attenuation index of electromagnetic waves in the microwave range by the test glass was determined by the waveguide method of measurement on an Agilent E5061B vector network analyzer connected to the measuring stand of a highly stable source of an external reference signal from the LPFRS-01 rubidium reference generator.

When measuring, we used waveguide chambers representing sections of the standard waveguides with a cross section of the given frequency range. The inlet and outlet sections of the measuring waveguide chamber were covered with flat layers of fluoroplastic 0.1 mm thick. The choice of fluoroplastic as a material was due to its low losses (the attenuation coefficient was 0.1–0.2 dB).

The waveguide method makes it possible to measure the standing wave ratio (SWR) and the attenuation index characterizing the glass under study from the point of view of its practical use in the microwave range. The determination error was ±0.5% [6].

The dielectric loss tangent was calculated using the following formulas based on the experimentally obtained SWR data and the attenuation index:

$$\tan\delta = \varepsilon''/\varepsilon', \quad (1)$$

where $\tan\delta$ is the dielectric loss tangent, ε' is the real part of the dielectric permittivity, and ε'' is the imaginary part of the permittivity;

$$\varepsilon' = (\mathrm{SWR})^2, \quad (2)$$

where SWR is the standing wave ratio;

$$\varepsilon'' = \frac{\Delta N \lambda_f \sqrt{\varepsilon'}}{8.7\pi d}, \quad (3)$$

where ΔN is the attenuation index, dB; λ_f is the wavelength at the resonant frequency, nm; and d is the sample plate thickness, mm.

The IR absorption spectra of glass in the range 250–1500 cm^{-1} were recorded on a Specord-IR-75 spectrophotometer.

Raman spectra were recorded using a Confotec MR350 3D scanning laser microscope (SOL Instruments) and a 532-nm laser under the same conditions and room temperature.

RESULTS AND DISCUSSION

Evaluation of the crystallization ability of the studied glass by the method of gradient crystallization showed that compositions containing 65.0–67.5 mol %

SiO_2 and 17.5–20.0 mol % R_2O are characterized by the presence of a crystalline crust in the temperature range 930–1110°C, while for glass with the content of 67.5 to 70.0 mol % SiO_2 and 10.0–12.5 mol % B_2O_3, as well as for the range of glass compositions containing 10.0 mol % B_2O_3, crystallization was not detected. For individual glass compositions of the indicated regions, the results obtained were confirmed by the DSC data (Figs. 1a, 1b), where the absence of exo effects caused by glass crystallization can be clearly seen. The endo effects in the DSC curves at 522–600°C are attributed to the onset of the glass softening process [7].

Knowledge of the temperature of the onset of softening is a rather important indicator, since its value determines the working range of the use of glass in practice in various spheres of the national economy. With an increase in the R_2O content in the experimental glass compositions, a natural decrease in this indicator is observed, which is caused by the depolymerization of the boron–silicon–oxygen frame [8]. An increase in the B_2O_3 fraction leads to an increase in the temperature of the onset of softening. Apparently, this is due to the fact that at an R_2O content of 27.5 mol % (in this case, the amount of oxide B_2O_3 remains constant) some of the alkali metal cations are involved in the formation of structural groups $[BO_4]R$, and the excess of these cations performs the function of depolymerization (the appearance of terminal –Si–O–R bonds). With an increase in the B_2O_3 content in the glass composition, the proportion of alkaline cations involved in depolymerization decreases, which causes an increase in this indicator [9].

The energy of the electromagnetic field moves along with the field itself in space and can be converted into other types of energy, thermal, mechanical, etc. A feature of the electromagnetic field is its ability to affect electrically charged particles of matter [10]. When the energy of an electromagnetic wave propagates in a substance, it is converted into other types of energy, in particular, into electrical and thermal [11]; therefore, the radioprotective material must be heat-resistant. In this regard, the study of the thermophysical properties of glass (TCLE, heat capacity, and heat resistance) is urgent.

Heat resistance characterizes the ability of the test glass to withstand sudden temperature changes without destruction and depends primarily on the TCLE. It has been found that the TCLE of the glass under study varies in the range $(42.0–73.0) \times 10^{-7}$ K^{-1}. In this case, the equimolar substitution of SiO_2 for B_2O_3 at a constant R_2O content in the test glass compositions leads to a significant decrease in this indicator. The thermal stability of the test glass varies from 120 to 130°C, which allows us to conclude that they are resistant to destruction in the case of the partial conversion of the electromagnetic field energy into thermal energy.

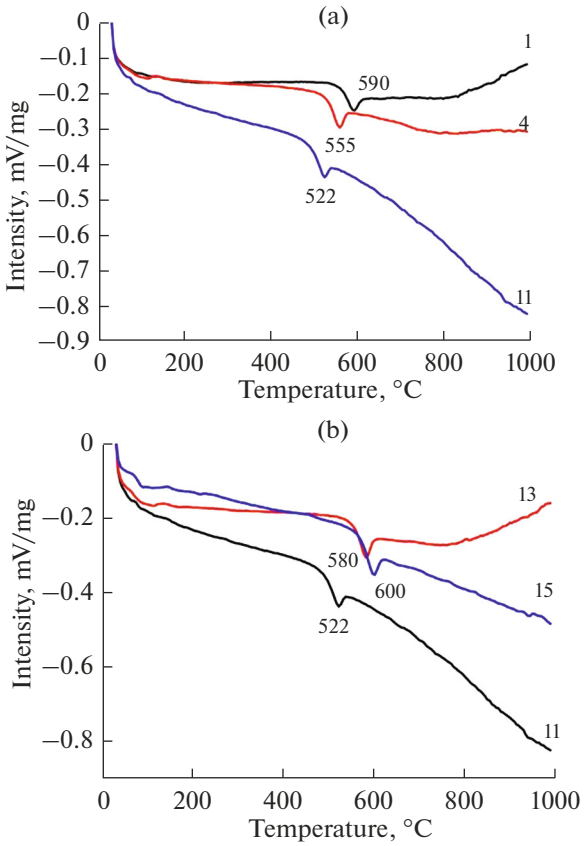

Fig. 1. DSC curves of glass; (a) R_2O (mol %): 17.5 (1), 22.5 (4), 27.5 (11); (b) B_2O_3 (mol %): 10.0 (11), 15.0 (13), 20.0 (15). The number of glass compositions in accordance with Table 1 are given in brackets.

The heat capacity reflects the ability of materials to absorb heat with increasing temperature and determines their thermal inertia. For glass used as radioprotective glass, the value of the heat capacity characterizes the rate of temperature equalization across the thickness of the product and, as a consequence, determines the heat resistance of the finished product.

The heat capacity of glass varies in the range 0.49–0.85 kJ/kg K depending on its chemical composition at a temperature of 50°C (Figs. 2, 3).

With an increase in the content of R_2O from 17.5 to 27.5 mol % at a constant content of B_2O_3, the heat capacity of glass is exponentially increased from 0.74 to 0.85 kJ/kg K (Fig. 2). This is related to the decrease in the degree of polymerization of the boron–silicon–oxygen glass frame due to an increase in the proportion of the modifier oxide. The heat capacity of solid glassy polymers is usually represented as an additive function of two components that are caused by the lattice vibrations of the main glass-forming frame and characteristic vibrations of the terminal bonds and the Si–O–Me type bonds (where Me are the R^+ ions). Lattice vibrations are low-frequency, acoustic, make the main contribution to the heat capacity of solids,

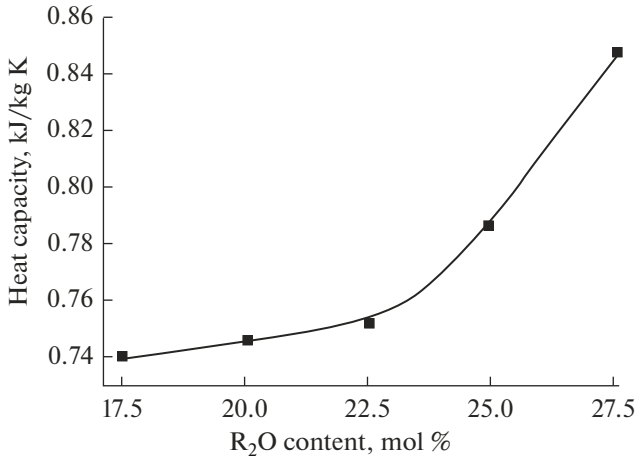

Fig. 2. Effect of R_2O on the heat capacity of experimental glass with a constant B_2O_3 content.

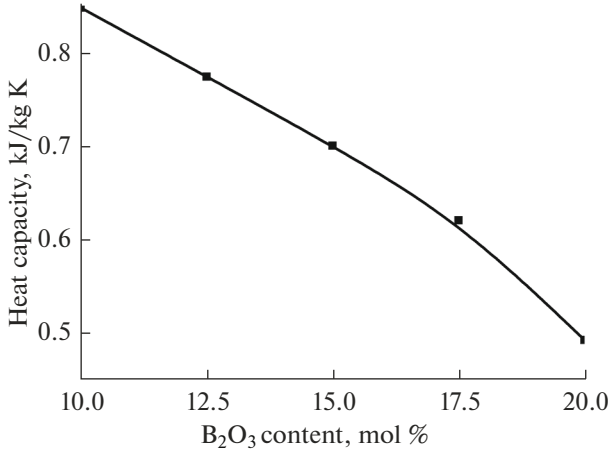

Fig. 3. Effect of B_2O_3 on the heat capacity of experimental glass with a constant SiO_2 content.

and depend mainly on the mass of the glass-forming frame. The characteristic vibrations of the terminal Si–O–Me bonds appear in the region of higher frequencies and, therefore, higher temperatures and are determined by the ratio of the atomic masses of the main glass-forming frame and modifier cations [12].

An increase in the B_2O_3 content from 10.0 to 20.0 mol % (Fig. 3) at a constant SiO_2 content causes a decrease in the heat capacity of glass from 0.85 to 0.49 kJ/kg K and, as a consequence, an increase in the degree of connectivity of the boron–silicon–oxygen glass frame.

The study of the heat capacity makes it possible to assess the degree of polymerization of the silicon–oxygen glass frame and the degree of the structure's development. The transition to a more developed three-dimensional structure is accompanied by a decrease in the heat capacity of the glass. The range of glass compositions containing 25.0–27.5 mol % R_2O and 10.0–15.0 mol % B_2O_3 is optimal in terms of the thermophysical properties from the point of view of obtaining radioprotective glass.

The electrophysical properties of glass (SWR, attenuation index, and dielectric loss tangent) make it possible to evaluate the material under study in terms of its practical use in the microwave range. The SWR characterizes the reflectivity of an electromagnetic wave. The higher its value the more intensely the glass reflects the electromagnetic radiation of the microwave range. It has been found that the SWR of glass varies within 3.21–7.85 (Figs. 4a, 4b).

From Fig. 4 it can be seen that the maximum SWR value is observed at a frequency of 1.84 GHz. In this case, a change in the content of R_2O introduced instead of SiO_2 from 17.5 to 27.5 mol %, generally causes an increase in the studied indicator from 3.51 to 7.65 dB (Fig. 4a). When the R_2O content is about 20.0 mol %, the minimum of this indicator is observed in the presented dependences.

A change in the content of B_2O_3 introduced instead of R_2O from 10.0 to 20.0 mol % (Fig. 4b) causes an increase in SWR from 3.35 to 7.29 dB. At the B_2O_3 content of 15 mol %, the minimum of this indicator is observed for all frequencies.

The data obtained are in a certain way consistent with the results of studying the density of the experimental glass, which makes it possible to indirectly judge the relationship between the values of the glass density and the standing wave ratio. The higher the density of glass the higher the SWR. Knowledge of the glass density, on the one hand, allows us to estimate the mass of the finished product; and on the other hand, the degree of constancy of the density and chemical composition of glass characterizes the glass homogeneity. It has been experimentally found that the density of glass varies from 2442 to 2577 kg/m^3. The content of oxides R_2O and B_2O_3 has a decisive influence on this value. As is known, the density of glass is increased with an increase in the proportion of oxides of alkali metals [9]. This is due to the increase in the packing density of the structural elements. However, an increase in the content of boron oxide in the glass composition from 10.0 to 15.0 mol % at a constant R_2O content equal to 17.5 mol % causes a change in the proportion of three- and four-coordinated boron, which is determined by the R_2O/B_2O_3 ratio. The maximum density is observed at the R_2O/B_2O_3 ratio of 1.16. At the specified amount of alkali metal and boron oxides, the ratio of the fraction of the $[BO_3]$ and $[BO_4]$ groups is 1 : 1. An increase in the B_2O_3 content from 15.0 to 20.0 mol % causes the opposite phenomenon, i.e., a decrease in the glass density due to the increase in the fraction of $[BO_3]$ groups that occupy a larger molar volume in contrast

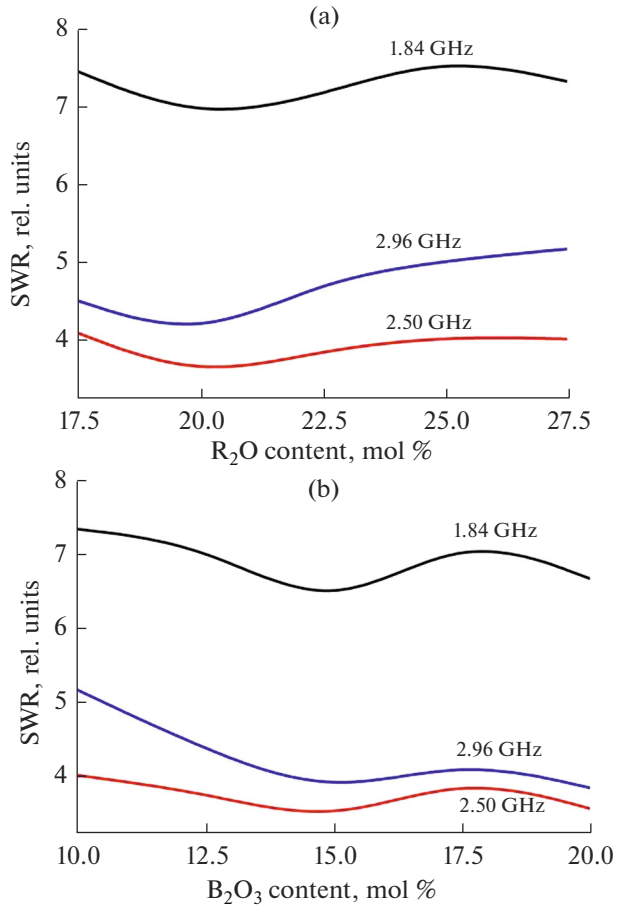

Fig. 4. Dependence of SWR of the studied glass on the content of (a) R_2O and (b) B_2O_3.

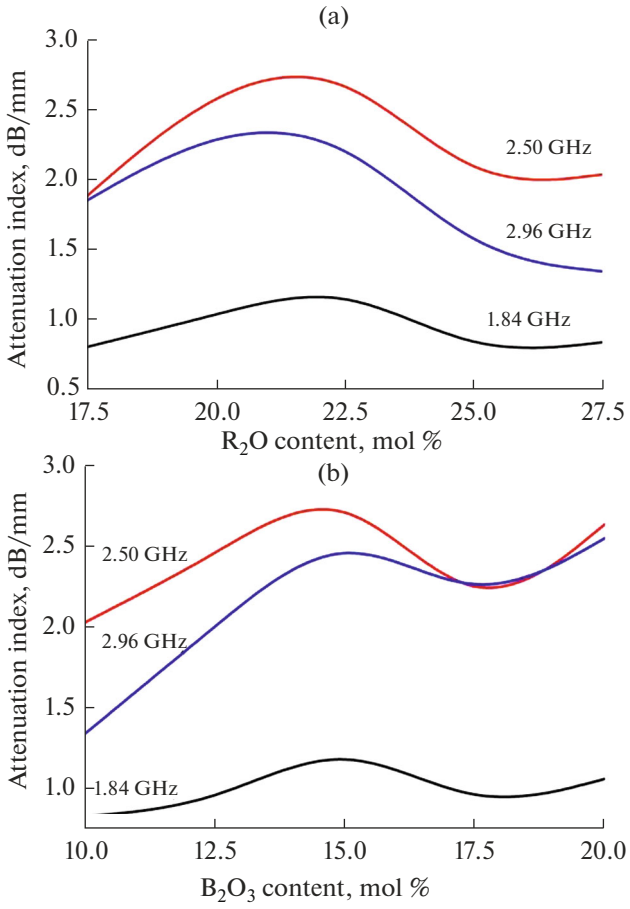

Fig. 5. Dependence of the attenuation index of the studied glass on the content of (a) R_2O and (b) B_2O_3.

to the [BO_4] tetrahedra [9]. With an increase in the B_2O_3 content from 10.0 to 20.0 mol % at a constant R_2O content equal to 17.5 mol %, the maximum density value equal to 2577 kg/m^3 is observed.

For the glass compositions under study, the attenuation index of the electromagnetic wave in the microwave range varies from 0.69 to 3.21 dB/mm. In this case, the maximum value of the attenuation index is observed at a frequency of 2.50 GHz (Figs. 5a, 5b). The increase in the content of R_2O introduced instead of SiO_2 from 17.5 to 27.5 mol % causes a change in the studied indicator from 0.84 to 2.84 dB/mm (Fig. 5a). The rate of attenuation of electromagnetic radiation by glass in the microwave region depends on the magnitude of dielectric losses (conduction, relaxation, and deformation losses) [13] that are mainly determined by the chemical composition of the glass and its structure. The effect of the chemical composition of glass on the magnitude of dielectric losses is similar to its effect on the electrical conductivity: components that increase the electrical conductivity also increase the dielectric losses in glass. Therefore, glass containing alkali metal oxides is characterized by increased dielectric losses. The dielectric losses are mainly related to the processes of establishing polarization, which occurs in a dielectric when exposed to an electric field. Intensive polarization processes cause the absorption of the energy of the electric field when the frequencies of the natural oscillations of ions and electrons coincide with the frequency of the electric field. Various types of polarization related to the thermal motion of ions and electrons are established much more slowly and in most cases are the main source of dielectric losses in a wide range of radio frequencies [14, 15].

At an R_2O content of 20.0 to 22.5 mol % (Fig. 5a), the maximum value of the studied indicator is observed in the presented dependences; and at 25.0–27.5 mol %, the minimum. This is due to the fact that weakly bound alkali metal ions are one of the sources of relaxation losses in inorganic semiconductors. The application of an electric field to the test material causes an asymmetry in the distribution of charges, as a result of which an electric moment arises, which affects the growth of the attenuation index.

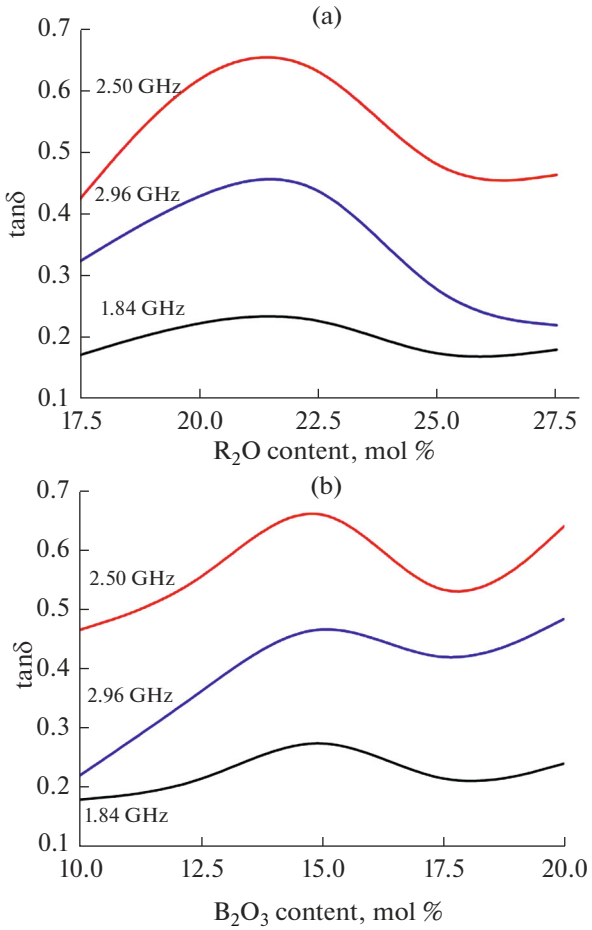

Fig. 6. Dependence of the dielectric loss tangent of glass on the content of (a) R_2O and (b) B_2O_3 at different frequencies.

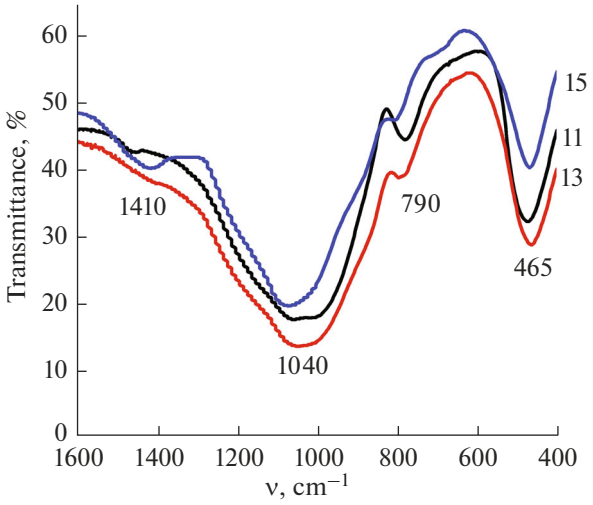

Fig. 7. IR spectra of glass at a B_2O_3 content (mol %): 10.0 (11), 15.0 (13), and 20.0 (15). The number of glass compositions in accordance with Table 1 are given in brackets.

As seen from Fig. 5b, when the content of B_2O_3 introduced instead of R_2O is changed from 10.0 to 20.0 mol %, there is an increase in the investigated indicator from 0.74 to 2.84 dB/mm. At the B_2O_3 content slightly exceeding 17.5 mol %, the minimum appears in the presented dependences, which is consistent with changes in the dielectric losses of glass (Fig. 6b).

The tangent of the dielectric loss angle of the test glass varies from 0.1718 to 0.744. Figures 6a and 6b show the dependences of the dielectric loss tangent on the change in the R_2O and B_2O_3 contents at different frequencies.

When the R_2O content is changed from 17.5 to 22.5 mol % (Fig. 6a), the dielectric loss tangent is increased from 0.156 to 0.679, passing through the maximum at an R_2O content of about 20.0–22.5 mol %. The maximum values of this indicator are observed at a frequency of 2.50 GHz. It should be noted that with an increase in the frequency of the applied field, the extremums in the given dependences become more pronounced.

With an increase in the B_2O_3 content from 10.0 to 20.0 mol % (Fig. 6b), the dielectric loss tangent is changed from 0.181 to 0.744. In the case of an increase in the frequency of the electromagnetic field from 1.84 to 2.50 GHz, an increase in tanδ is observed. A further increase in the frequency to 2.96 GHz causes a decrease in the studied parameter. With an increase in the B_2O_3 content from 10.0 to 15.0 mol %, an increase in tanδ is observed, and a change in the content of this oxide from 15.0 to 17.5 mol % leads to a decrease. The subsequent increase in the content of boron oxide to 20.0 mol % causes an increase in this indicator. As in the previously given dependence, an increase in the frequency of the applied field makes the extremums on these dependences more pronounced.

The magnitude of the electrophysical characteristics of glass is determined not only by its chemical composition but also by the frequency of the applied electromagnetic field. The frequency of the applied electromagnetic field has the opposite effect on the attenuation and SWR of the test glass. Thus, the maximum values of the attenuation index can be achieved at an electromagnetic field frequency of 2.50 GHz, while the SWR value for a similar frequency is minimal.

The results of studying the attenuation index of electromagnetic radiation by the experimental glass are confirmed in the context of studying their structure by the methods of infrared (Fig. 7) and Raman spectroscopy (Fig. 8).

The broad intense absorption bands in the region 900–1100 cm^{-1} in the IR spectra (Fig. 7) are due to the stretching vibrations of the $[SiO_4]$ and $[BO_4]$ tetrahedra, which can overlap each other [8, 16]. The position of the absorption band's maximums depends on the degree of polymerization of these tetrahedra. The higher the

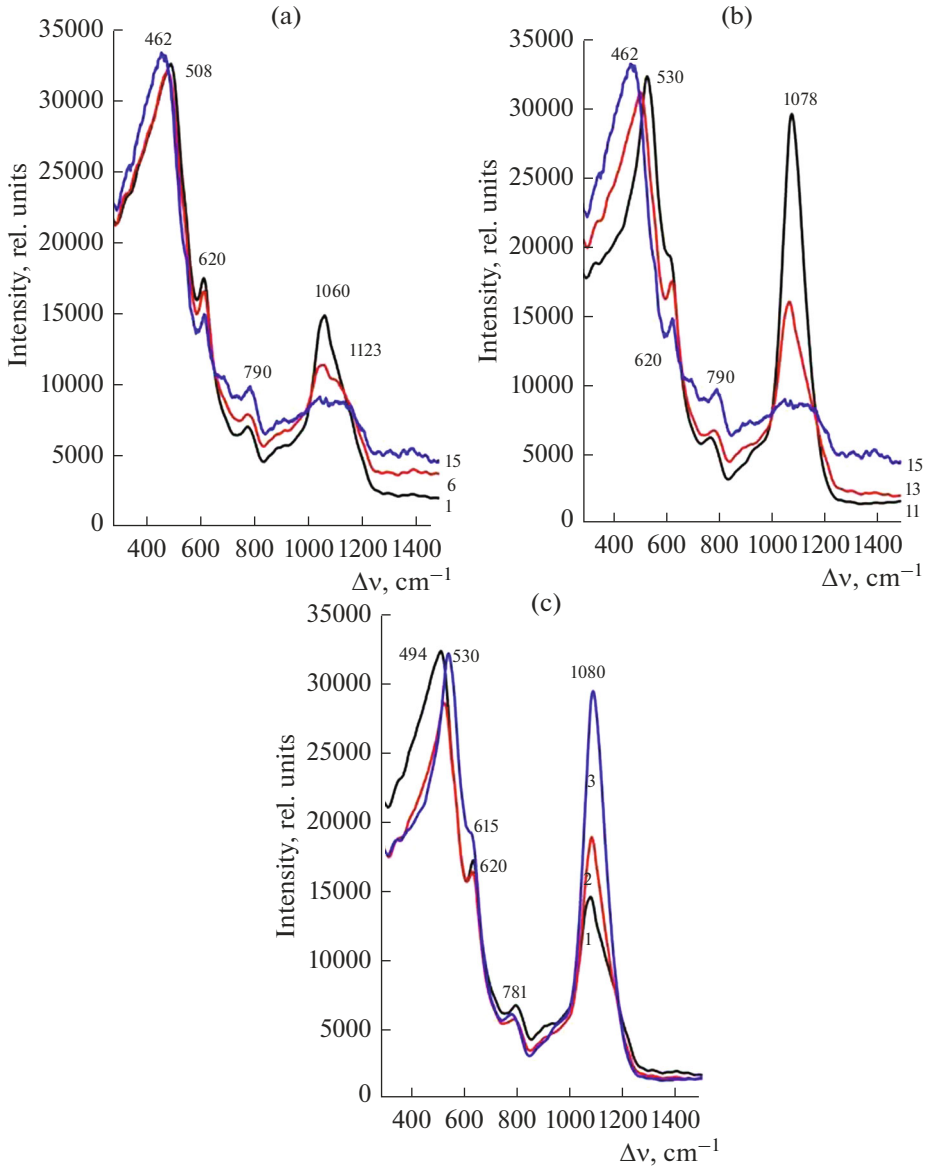

Fig. 8. Raman spectra of glass; (a) SiO_2 (mol %): 62.5 (1), 67.5 (6), 72.5 (15); (b) B_2O_3 (mol %): 10.0 (11), 15.0 (13), 20.0 (15); (c) R_2O (mol %): 17.5 (1), 22.5 (4), 27.5 (11). The number of glass compositions in accordance with Table 1 are given in brackets.

degree of their connectivity with each other the higher the frequency region in which the main absorption maximum will be located. An increase in the content of modifiers in the composition of the test glass determines the proportion of the nonbridging bonds of the Si–O type, which leads to depolymerization of the structural network of the glass.

The absorption band in the range of 740 to 800 cm^{-1} is attributed to the formation of ring structures of $[SiO_4]$ tetrahedra in the glass lattice [8, 17]. The band in the region of 400 to 500 cm^{-1} belongs to the bending vibrations of the $[SiO_4]$ tetrahedra, and the appearance of the absorption band at 1400 cm^{-1} indicates the appearance of three-coordinated boron, which is natural.

In the case of Raman spectroscopy (Figs. 8a–8c), the scattering bands in the high-frequency region (1000–1200 cm^{-1}) reflect the degree of connectivity of the silicon–oxygen component of the structural framework of the test glass. The structure of alkaline borosilicate glass is represented by the silicon–oxygen and alkaline borate components.

The band at 1123 cm^{-1} characterizes the presence of the silicon–oxygen tetrahedra with 4 bridging oxygen atoms (Q4); and in the range of 1060–1080 cm^{-1}, $[SiO_4]$ tetrahedra, in which there are 3 bridging oxygen atoms (Q3). This indicates that the silicon–oxygen component is characterized by an insignificant num-

ber of structural breaks caused by the presence of alkali metal oxides in the glass composition [18, 19].

In all the presented dependences (Figs. 8a–8c), in the region of 790 cm^{-1}, there is a band reflecting the presence of bridges of the $B^{IV}–O–B^{III}$ type in the glass structure; and its insignificant intensity characterizes their small fraction.

Apparently, the band at 630 cm^{-1} indicates the presence of danburite-like ring structures consisting of two silicates Q4 and two boron–oxygen tetrahedral units $[BO_{4/2}]R^+$ [19]. The band at 480 cm^{-1} corresponds to the vibrations of B–O–B bridges in the plane perpendicular to the plane of the boroxol ring [20].

Studying the Raman spectra of alkaline borate glass has shown that the most significant changes in the spectra are observed in the region of low (460–530 cm^{-1}) and high frequencies (1000–1200 cm^{-1}), which indicates a change in the coordination state of boron in the glass structure and the degree of connectivity of the $[SiO_4]$ tetrahedra [20].

CONCLUSIONS

Based on the studies carried out, it has been found that glass containing (mol %) R_2O 20.0–22.5 and B_2O_3 10.0–12.5 can be used as glass that significantly attenuates electromagnetic radiation and best meets the requirements for it. The studies of the physicochemical and electrophysical properties of $R_2O–B_2O_3–SiO_2$ glass have made it possible to determine the range of glass compositions that can be used as radiation shielding glass.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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