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GLASS FORMATION AND PROPERTIES OF GLASSES IN THE SYSTEM $SrO-B_2O_3-SiO_2-xAl_2O_3$ (x=0; 10 MOL.%)

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The paper shows the prospects of the system $SrO-Al_2O_3-B_2O_3-SiO_2$ as a basis for the synthesis of new vitreous and glass-ceramic materials, which are widely used as electrical insulated and high-temperature coatings, for sealing of solid oxide fuel cells, and in the production of heat resistant materials. We experimentally established the conditions of glass formation, regions of glass-forming melts and properties of glasses, the chemical composition of which is limited by the following content of components (mol.%): SrO 30–80, B_2O_3 10–60, SiO_2 10–60, and Al_2O_3 0–10. It is shown that during the synthesis of glasses in the corundum crucible at the temperature of 1350^{0}C the region of glass formation in the system $SrO-B_2O_3-SiO_2$ is limited by the following content of components (mol.%): SrO 30–60, B_2O_3 10–60, and SiO_2 10–50. It is found that the introduction of Al_2O_3 to the composition of these glasses expands the region of glass formation towards increase of the SiO_2 content in the glass up to 60 mol.%. Experimentally determined values of glass properties are within the following limits: coefficient of linear thermal expansion $(67-118)\cdot10^{-7}$ K^{-1}; glass transition temperature 570–660^{0}C; dilatometric softening point 580–700^{0}C; and density 2.62–3.71 g cm^{-3}. The established patterns of influence of the components and conditions of glass formation on the physical and chemical characteristics of glasses may serve as an experimental basis for designing of new materials with a complex of specified properties, which allows solving the problems of their practical use.

Keywords: glass, glass formation, thermal expansion, glass transition temperature, volume resistivity.

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Introduction

One of the urgent and highly demanded, from a practical standpoint, issues of modern materials science is the development of scientific and technological bases for the creation of new glass-ceramic and vitreous materials based on alkali-free borosilicate systems with a complex of specified properties.

The system $SrO-Al_2O_3-B_2O_3-SiO_2$ is one of the promising glass-forming systems for the synthesis of various technologically demanded materials, from nonlinear optical and laser media [1,2] to sealants [3] and bioactive materials [4,5]. These materials have a number of valuable properties, e.g. chemical stability, heat resistance, biocompatibility, impermeability to gases and water and protection against ionizing radiation [4–6]. Aluminum oxide plays an important role in this system. At moderate concentrations from 0 to 10 mol.%, the addition of Al_2O_3 counteracts crystallization of glass, reducing the liquidus temperature of the system and rate of the crystal growth [7]. At low concentrations, aluminum oxide, obviously, serves as a glass-forming component [8,9], and at higher concentrations it acts as a glass network modifier [7,10]. Performing this dual role, Al_2O_3 can both inhibit and enhance the crystallization of glass [10]. Aluminum oxide also has a positive effect on the electrical insulation properties of alkali-free borosilicate systems. With an increase in the content of Al_2O_3 due to SiO_2, the electrical resistance of glasses grows, while dielectric losses decrease [7–9]. However, there is no sufficient information in the literature on the conditions of glass formation and physicochemical properties of the abovementioned glasses, which could serve as an experimental base for designing of new materials with a complex of specified properties.

In view of this, the purpose of this study is to

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establish the conditions of glass formation and relationship between the properties of oxide glasses and their chemical composition for the systems that are limited by the following content of components (mol.%): SrO 30–80, B_2O_3 10–60, SiO_2 10–60, and Al_2O_3 0–10.

Materials and methods

Finely ground quartz sand and chemical reagents of the laboratory reagent and analytical reagent grade (boric acid, strontium carbonate, alumina) were used to prepare the mixtures of experimental glasses. Melting of glasses was carried out in the corundum crucibles with the volume of 100 ml in the electric furnace with silicon carbide heaters at the temperature of 1350^{0}C during 60 minutes. Samples of glasses were made by the method of glass melt casting into steel molds followed by annealing in the muffle furnace at the temperature of 500^{0}C. Then the properties of the prepared glasses were determined.

Melting characteristics of the experimental glasses were studied in the corundum crucibles with the volume of 10 ml. Crucibles with the mixture were placed in the furnace heated to 1350^{0}C and held for 60 minutes. At the end of the holding time, the crucibles were removed from the furnace and cooled in the air.

The properties of glasses were determined by standard methods: the density (d) of glasses was determined by hydrostatic weighing in accordance with GOST 9553-74; dilatometric studies of the coefficient of linear thermal expansion (CTE) were performed in the temperature range of 20–400^{0}C; dilatometric softening point (t_d) and glass transition temperature (t_g) were found in accordance with GOST 10978-2014.

Volume resistivity of glasses was measured by E6-13A teraohmmeter with the use of the electrode thermocell (graphite electrodes).

Crystallization ability of glass powders was examined by the method of differential thermal analysis on the derivatograph Q-1500D in the temperature range of 20 to 1000^{0}C at the heating rate of 10^{0}C/min. Alumina oxide fired at the temperature of 1450^{0}C was used as a reference.

X-ray phase analysis of glasses was carried out on a diffractometer DRON-3M in Co-K_α radiation. To identify crystalline phases, X-ray card index of ASTM was used.

Results and discussion

When studying glasses in the system $SrO-Al_2O_3-B_2O_3-SiO_2$, we took into account the results of previous studies of the conditions of glass formation in the system $BaO-Al_2O_3-B_2O_3-SiO_2$ [8]. The introduction of Al_2O_3 (up to 10 mol.%) into the composition of these glasses contributed to the reduction of crystallization ability of glass melts and expansion of the region of glass formation towards an increase of BaO content in the glass to 60 mol.%.

The synthesis of glasses and study of glass formation conditions were carried out in the cross sections of the system $SrO-B_2O_3-SiO_2$ and $SrO-B_2O_3-SiO_2-10Al_2O_3$ with the following content of basic components (mol.%): SrO 30–80, B_2O_3 10–60, and SiO_2 10–60. As a result of the study of the melting properties of experimental glasses, boundaries of glass formation at the temperature of 1350^{0}C and regions of glasses which were visually clear and also those crystallized were plotted on the diagram of the system $SrO-B_2O_3-SiO_2-xAl_2O_3$ (Fig. 1).

Experimental data of glass formation conditions show that the system under consideration is characterized by a wide range of visually transparent glasses as well as glasses resistant to crystallization in the production, including the compositions with a high content of SrO. Under assumed temperature

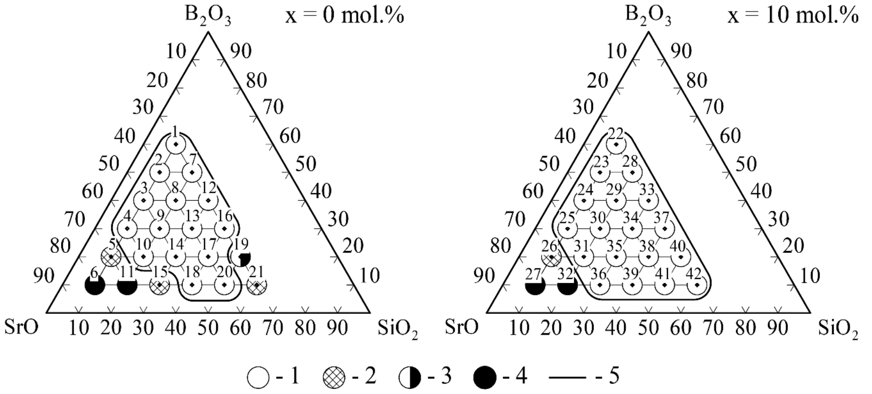

Fig. 1. Chemical compositions of experimental glasses and glass formation in the system $SrO-B_2O_3-SiO_2-xAl_2O_3$: 1 – clear glass; 2 – crystallization; 3 – liquation; 4 – sinter; 5 – border of glass-forming region

and time conditions, the melting of mixture components with the formation of homogeneous glass-forming melts stops at the SrO content of 60 mol.%.

According to data reported by Tyurnina et al. [11], the glass of the system $SrO-B_2O_3-SiO_2$ synthesized in the platinum crucible is formed at the SrO content from 30 to 50 mol.%. At the same time, in case of synthesis in corundum crucibles, we obtained the glass with SrO content of up to 60 mol.%. Expansion of the glass formation region in the ternary system $SrO-B_2O_3-SiO_2$ under study in case of its synthesis in corundum crucibles is caused by their corrosion, resulting in the diffusion of a part of Al_2O_3 from the crucible material to the glass melt. The above contributes to the formation of glass at high content of SrO, where Al_2O_3, apparently, acts as glass-forming component. Aggressiveness of the mixture and glass melt in relation to the material of corundum crucibles has been previously established in the studies of glasses in the system $RO-B_2O_3-SiO_2$ (R=Ba; Ca) [8,12].

When comparing the glass-forming regions in cross sections of the system under consideration, we can draw the conclusion that the addition of Al_2O_3 expands the range of visually clear glasses, as well as glass resistant to crystallization in the production, towards an increase in the SrO and SiO_2 content to 60 mol.%. Additional introduction of 10 mol.% of Al_2O_3 into the compositions of glasses No. 4 and No. 15 (Fig. 2,a and Fig. 2,c) reduces the crystallization ability of the melts of these glasses, as

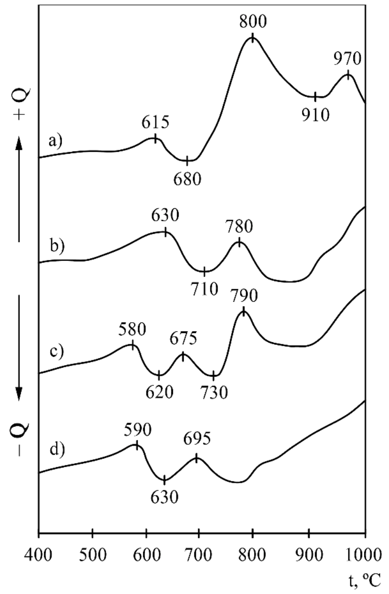

Fig. 2. DTA curves of glass powders: a – No. 15; b – No. 36; c – No. 4; d – No. 25

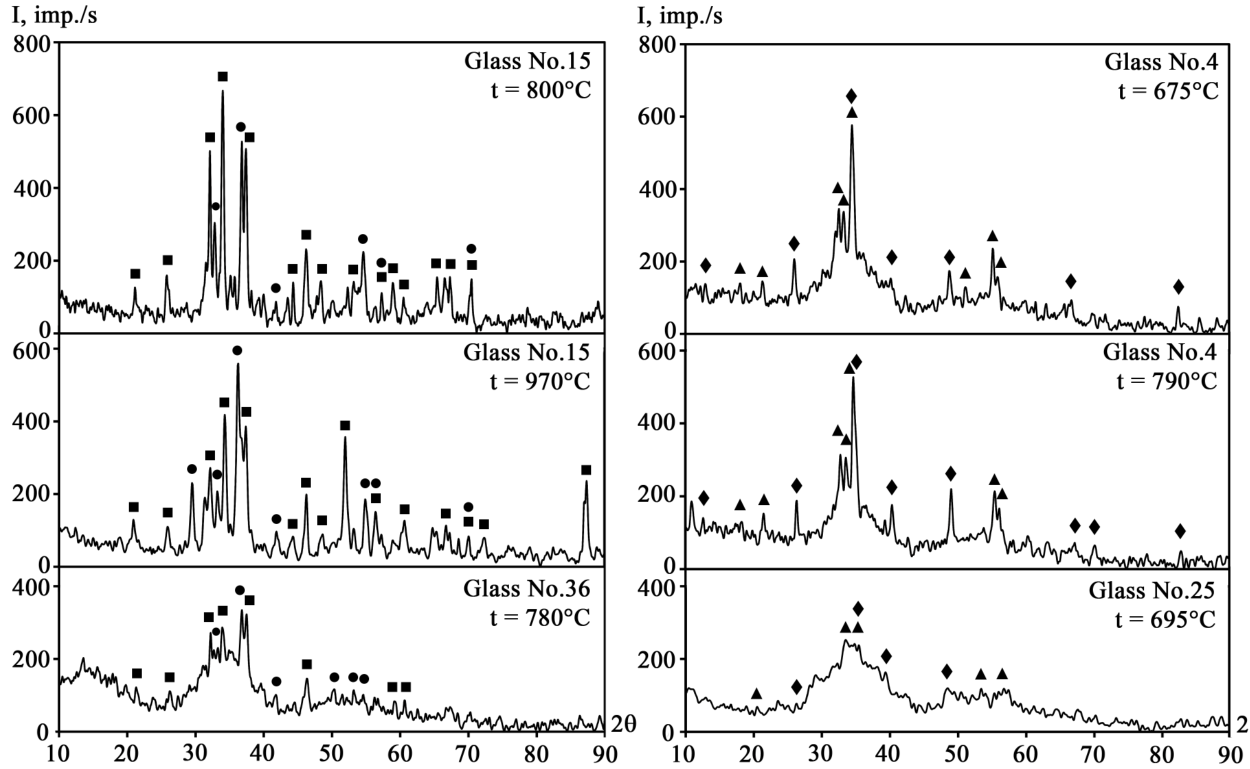

Fig. 3. X-ray patterns of powders of glass in the system $SrO-Al_2O_3-B_2O_3-SiO_2$ after heat treatment during 2 hours at different temperatures of crystallization: ● – $Sr_2Al_2SiO_7$; ■ – Sr_2SiO_4; ▲ – $Sr_2B_2O_5$; ◆ – $SrAl_2Si_2O_8$

evidenced by a decrease in the intensity of the relevant exothermic effects on the DTA curves of powders of glasses No. 25 and No. 36 (Fig. 2,b and Fig. 2,d).

Diffusion of Al_2O_3 from the crucible material into the glass melt owing to their active interaction is confirmed by the results of X-ray phase analysis of glasses No. 4 and No. 15 (Fig. 3), mixtures of which did not contain Al_2O_3 at all. According to the data of X-ray phase analysis, strontium silicate (Sr_2SiO_4) and Sr-gehlenite ($Sr_2Al_2SiO_7$) can be identified as the main crystalline phases during heat treatment of glass No. 15. Position of the main diffraction maximums on X-ray patterns of the glass powder No. 4 indicates that the main crystalline phases formed during heat treatment of this glass are strontium borate ($Sr_2B_2O_5$) and Sr-anortite ($SrAl_2Si_2O_8$). Addition of 10 mol.% of Al_2O_3 to the composition of glasses No. 4 and No. 15 reduces the amount of the crystalline phase, which is confirmed by a decrease in the intensity of diffraction maxima on X-ray patterns of powders of glasses No. 25 and No. 36 (Fig. 3).

Further study of the physical and chemical properties of glasses was carried out on the compositions of visually clear glasses and glasses resistant to crystallization in the production (Fig. 1). Experimentally established vales of properties of experimental glasses are given in Table 1.

Data analysis shows that depending on the chemical composition of experimental glasses their properties vary over the wide range: coefficient of linear thermal expansion of $(67-118)\cdot10^{-7}$ K^{-1}; glass

Table 1

Physical and chemical properties of glasses

Glass No.	CTE, $\alpha\cdot10^7$, K^{-1}	t_g, ^0C	t_d, ^0C	lgρ, Ohm·cm (150^0C)	Density, d, g/cm^3
1	69	570	580	12.21	2.82
2	82	580	600	12.39	3.10
3	92	580	610	12.86	3.30
4	107	590	620	13.54	3.46
7	74	600	610	12.25	3.00
8	86	610	620	12.35	3.19
9	97	610	640	12.85	3.39
10	118	620	650	13.26	3.68
12	75	600	630	12.33	3.05
13	88	620	640	12.57	3.22
14	103	630	660	13.11	3.46
16	75	640	670	12.42	3.09
17	96	650	670	13.49	3.35
18	105	640	680	13.49	3.55
20	94	650	690	13.38	3.40
22	67	570	600	12.58	2.62
23	77	580	610	12.72	3.02
24	87	580	620	13.29	3.24
25	101	590	630	14.49	3.39
28	67	600	630	12.62	2.95
29	78	605	630	13.00	3.10
30	94	610	640	13.52	3.28
31	106	620	655	14.00	3.48
33	68	615	635	12.70	2.93
34	81	620	645	13.18	3.20
35	101	620	650	13.45	3.42
36	109	615	670	14.25	3.71
37	68	620	640	12.75	2.96
38	86	630	650	12.85	3.26
39	101	640	680	14.03	3.50
40	70	640	690	13.00	3.05
41	88	650	690	13.91	3.32
42	74	660	700	13.74	3.06

transition temperature of 570–660^{0}C; dilatometric softening point of 580–700^{0}C; and density of 2.62–3.71 g/cm^3. The volume resistivity of experimental glasses at the temperature of 150^{0}C is within 10^{12}–10^{14} Ohm·cm, indicating their high electrical insulation properties.

The relationship between the properties of multicomponent glasses and their composition in glass chemistry and technology is expressed with the use of the additive formula:

$$V=S(v_i \cdot x_i)/100,$$

where V is the calculated value of glass properties; v_i are the additive coefficients (partial contributions of oxides to the value of glass properties); x_i are the contents of oxides in the glass, mol.%.

This additive formula represents a compact form of the generalized and quantitative description of the patterns of change in glass properties depending on its composition. In this context, additive coefficients in the equations for calculating the values of properties of the experimental glasses (Table 2) were determined in this study by the multiple correlation method. Accuracy of the calculation of these properties was evaluated by the value of the multiple correlation coefficient (R) and by comparison of the residual dispersion S^2_{res} with the dispersion relative to the average value of the experimental properties S^2_y [13,14]. As indicated by the data of Table 2, S^2_{res} is much less than S^2_y, so we can assume that Eq. (1) gives a reasonable approximation for the experimental data of Table 2.

It can be seen that general tendencies in the change of properties of glasses of the system $SrO–Al_2O_3–B_2O_3–SiO_2$ are maintained, as a whole, for the similar properties glasses of the system $BaO–Al_2O_3–B_2O_3–SiO_2$ as well [8]. Silicon dioxide makes the largest partial contribution to the values of glass transition temperature and dilatometric softening point. This fact should be taken into account when choosing the compositions of glasses for the protective coatings, as an increase in SiO_2 content will lead to an increase in the temperature of formation of the protective coatings. Replacement of SiO_2 by B_2O_3 causes no significant changes in the CTE values and at the same time contributes to the sharp decrease in the glass transition temperature and dilatometric softening point of experimental glasses. The latter is due to the fact that B_2O_3 reduces the glass viscosity.

The largest partial contribution to the values of the coefficient of linear thermal expansion and density of glasses is made by SrO. An increase in the SrO content in the glass due to SiO_2 promotes reduction of glass transition temperature and dilatometric softening point of a glass. It is explained by low degree of covalence bond Sr–O and large radius of Sr^{2+} (1.21 Å) [7].

The introduction of Al_2O_3 into the composition of the system $SrO–B_2O_3–SiO_2$ contributes to strengthening of the glass lattice, which in turn assists in reduction of crystallization ability of glass melts, lowering of the coefficient of linear thermal expansion and increasing the volume resistivity of the experimental glasses.

Conclusions

We have experimentally established the conditions of glass formation, regions of glass-forming melts and properties of glasses, the chemical composition of which is limited by the following content of components (mol.%): SrO 30–80, B_2O_3 10–60, SiO_2 10–60, and Al_2O_3 0–10. We showed that during synthesis of glasses in the corundum crucible during 60 minutes at the temperature of 1350^{0}C the region of glass formation in the system $SrO–B_2O_3–SiO_2$ is limited by the following content of components (mol.%): SrO 30–60, B_2O_3 10–60, and SiO_2 10–50. It was found that the introduction of Al_2O_3 into the composition of these glasses expands the region of glass formation towards the increase of SiO_2 content in glass up to 60 mol.%. Experimentally established values of experimental glass properties are within the following limits: coefficient of linear

Table 2

Values of additive coefficients (v_i), their standard deviations (S_v) and results of statistical analysis of calculation formulas

Properties	Values of $v_i \pm S_v$ for respective oxides				R	S^2_{res}	S^2_y
	SrO	B_2O_3	SiO_2	Al_2O_3			
CTE, $\alpha_i \cdot 10^7$, K^{-1}	164±3	22±3	52±3	22±10	0.99	7.23	209
Glass transition temperature, $(t_g)_i$, ^{0}C	601±7	538±7	727±8	571±25	0.99	46	619
Dilatometric softening temperature, $(t_d)_i$, ^{0}C	645±6	531±5	762±6	698±20	0.99	28	896
Volume resistivity, $lg\rho_i$, Ohm·cm	15.2±0.3	10.2±0.3	12.2±0.3	17.9±0.9	0.99	0.08	0.37
Density, d_i, g/cm^3	4.45±0.06	2.01±0.06	2.87±0.07	2.17±0.22	0.99	0.003	0.06

thermal expansion of (67–118)·10^{-7} K^{-1}; glass transition temperature of 570–660^{0}C; dilatometric softening point of 580–700^{0}C; and density of 2.62–3.71 g/cm^3. At the temperature of 150^{0}C, the volume resistivity of glasses is in the range of 10^{12}–10^{14} OhmЧcm. Generalization of the dependences of glass properties on their chemical composition was carried out with the use of additive formula, for which the partial contributions of oxides to the values of the corresponding properties were determined by experimental and statistical methods. The established patterns of influence of the components and conditions of glass formation on the physical and chemical characteristics of glasses in the system SrO–Al_2O_3–B_2O_3–SiO_2 may serve as an experimental base for designing of new materials with a complex of specified properties to solve the problems of their practical use.

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СКЛОУТВОРЕННЯ ТА ВЛАСТИВОСТІ СТЕКОЛ В СИСТЕМІ SrO–B_2O_3–SiO_2–xAl_2O_3 (x=0; 10 МОЛ.%)

Ю.С. Гордєєв, О.А. Амеліна

У роботі показано перспективність застосування системи SrO–Al_2O_3–B_2O_3–SiO_2, як основи для синтезу нових склоподібних і склокерамічних матеріалів, що широко використовують як електроізоляційні та жаростійкі покриття, для герметизації твердооксидних паливних елементів, а також у виробництві термостійких матеріалів. Експериментально встановлено умови склоутворення, ділянки склоутворюючих розплавів і властивості стекол, хімічний склад яких обмежений наступним вмістом компонентів (мол.%): SrO 30–80, B_2O_3 10–60, SiO_2 10–60, Al_2O_3 0–10. Показано, що при синтезі стекол в корундовому тиглі при температурі 1350^{0}С регіон склоутворення в системі SrO–B_2O_3–SiO_2 обмежений вмістом компонентів (мол.%): SrO 30–60, B_2O_3 10–60, SiO_2 10–50. Встановлено, що введення до складу цих стекол Al_2O_3 розширює інтервал склоутворення в напрямі збільшення в склі вмісту SiO_2 до 60 мол.%. Експериментально встановлені значення властивостей дослідних стекол знаходяться в наступних межах: температурний коефіцієнт лінійного розширення (67–118)·10^{-7} K^{-1}; температура склування 570–660^{0}C; дилатометрична температура розм'якшення 580–700^{0}C; щільність 2,62–3,71 г/см^3. Встановлені закономірності впливу компонентів і умов склоутворення на фізико-хімічні характеристики стекол можуть слугувати експериментальною базою для проектування нових матеріалів з комплексом заданих показників властивостей, що вирішує задачі їх практичного використання.

Ключові слова: скло, склоутворення, теплове розширення, температура склування, питомий об'ємний опір.

GLASS FORMATION AND PROPERTIES OF GLASSES IN THE SYSTEM $SrO–B_2O_3–SiO_2–xAl_2O_3$ (x=0; 10 MOL.%)

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The paper shows the prospects of the system $SrO–Al_2O_3–B_2O_3–SiO_2$ as a basis for the synthesis of new vitreous and glass-ceramic materials, which are widely used as electrical insulated and high-temperature coatings, for sealing of solid oxide fuel cells, and in the production of heat resistant materials. We experimentally established the conditions of glass formation, regions of glass-forming melts and properties of glasses, the chemical composition of which is limited by the following content of components (mol.%): SrO 30–80, B_2O_3 10–60, SiO_2 10–60, and Al_2O_3 0–10. It is shown that during the synthesis of glasses in the corundum crucible at the temperature of 1350^{0}C the region of glass formation in the system $SrO–B_2O_3–SiO_2$ is limited by the following content of components (mol.%): SrO 30–60, B_2O_3 10–60, and SiO_2 10–50. It is found that the introduction of Al_2O_3 to the composition of these glasses expands the region of glass formation towards increase of the SiO_2 content in the glass up to 60 mol.%. Experimentally determined values of glass properties are within the following limits: coefficient of linear thermal expansion (67–118)·10^{-7} K^{-1}; glass transition temperature 570–660^{0}C; dilatometric softening point 580–700^{0}C; and density 2.62–3.71 g cm^{-3}. The established patterns of influence of the components and conditions of glass formation on the physical and chemical characteristics of glasses may serve as an experimental basis for designing of new materials with a complex of specified properties, which allows solving the problems of their practical use.

Keywords: glass; glass formation; thermal expansion; glass transition temperature; volume resistivity.

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