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PROPERTIES OF GLASSES IN THE SYSTEM $BaO-B_2O_3-SiO_2-xAl_2O_3$ (x=0; 5; 10 MOL.%)

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This article shows the prospect of the system $BaO-Al_2O_3-B_2O_3-SiO_2$ as the basis of vitreous and glass ceramic materials, which are widely used in rocket production for high-temperature protection of heat resistant alloys, in the power industry for sealing solid oxide fuel cells, and in the production of heat resistant glass ceramic materials. We examined the conditions of glass formation and properties of glasses with the following content of components (mol.%): BaO 30–70, B_2O_3 10–50, SiO_2 20–60, and Al_2O_3 0–10. We established experimentally that the physical and chemical properties of glass, depending on its chemical composition, vary within the following limits: coefficient of linear thermal expansion of $(71-122)\cdot10^{-7}$ K^{-1}; glass transition temperature of 500–650⁰C; dilatometric softening point of 540–670⁰C; and density of 3.20–4.21 g cm^{-3}. The volume resistivity of the studied glasses is within $10^{11}-10^{13}$ Ohm·cm at the temperature of 150⁰C. Generalization of the dependences of glass properties on their chemical composition was carried out with the use of the additive equations, 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 components and conditions of glass formation on the physical and chemical characteristics of glasses allows implementing the process of designing of a wide range of glass compositions with the complex of specified properties in order to solve the tasks of their practical use.

Keywords: glass, borosilicate glass, glass formation, thermal expansion, glass transition temperature.

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Introduction

Alkali-free borosilicate glasses are widely used in the instrument making and rocket production as insulating coatings and heat-resistant coatings [1–3], in the power industry for sealing of solid oxide fuel cells [4–6], and in the production of heat resistant glass ceramic materials [6–8].

Glasses in the oxide system $BaO-Al_2O_3-B_2O_3-SiO_2$ with the BaO content of more than 30 mol.% can be used in the above fields of application. This system serves as basis for the synthesis of new vitreous and glass ceramic materials. These materials have valuable properties, such as corrosion resistance, mechanical strength, heat resistance, gas and water tightness and even protection against ionizing radiation [3–6]. However, there is no sufficient information in the technical literature about the conditions of glass formation and properties of abovementioned glasses, which is required for the substantiation of their production technology and selection of the optimal composition.

Ascertaining the patterns of the influence of components and conditions of glass formation on the glass physical and chemical characteristics allows implementing the process of designing of a wide range of glass compositions with the complex of specified properties for practical use.

In this regard, the purpose of the paper is to establish the conditions of glass formation and relationships between the properties of oxide glasses and their chemical composition, which is limited by the following content of components (mol.%): BaO 30–70, B_2O_3 10–50, SiO_2 20–60, and Al_2O_3 0–10.

Materials and methods

Finely ground quartz sand and chemical reagents (boric acid, barium carbonate, alumina, all of the laboratory reagent and analytical reagent grade) 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 1350⁰C for 60 min. In order to determine the properties of glasses, their samples were fabricated by the method of glass melt casting into steel molds followed by annealing in the muffle furnace at the temperature of 400⁰C.

Melting characteristics of the experimental glasses were studied in the corundum crucibles with the volume of 5 mL. Crucibles with the mixture were placed in the furnace, heated to 1350⁰C and then held for 60 min. At the end of the holding time, the crucibles were removed from the furnace and cooled in the air.

The properties of glass were determined by the standard procedures as follows: density (d) of glasses was measured by hydrostatic weighing as per GOST 9553-74; dilatometric studies of the coefficient of linear thermal expansion (CTE) were conducted in the temperature range of 20–400⁰C, dilatometric softening point (t_d) and glass transition temperature (t_g) were determined 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 properties of glass powders were examined by the method of differential thermal analysis using a derivatograph Q-1500D in the temperature range of 20–1000⁰C at the heating rate of 15⁰C·min^{-1}. Alumina oxide fired at the temperature of 1450⁰C was used as a reference.

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

Results and discussion

The melting properties of experimental glasses were experimentally studied and the boundaries of glass formation at the temperature of 1350⁰C and the regions of glasses, which were visually clear and crystallized, were plotted for the system $BaO-B_2O_3-SiO_2-xAl_2O_3$ (Fig. 1). In cross sections of the experimental system under assumed temperature and time conditions, the melting of mixture components with the formation of homogeneous glass-forming melts ceases at the BaO content of 60 mol.%.

It was previously reported [9,10] that the region of glass formation in the system $BaO-B_2O_3-SiO_2$ in the course of the synthesis of glasses in a platinum crucible is slightly less than in the case of a corundum crucible. It should be noted that the glass of the system $BaO-B_2O_3-SiO_2$ synthesized in the platinum crucible is formed at the BaO content of up to 40–50 mol.%. At the same time, in the course of the synthesis in the corundum crucibles, we obtained the glass with BaO content of up to 60 mol.%. Expansion of glass-forming region in the triple experimental system $BaO-B_2O_3-SiO_2$ at the synthesis in corundum crucibles is probably due to their corrosion, resulting in diffusion of a part of Al_2O_3 from the crucible materials to the glass melt. The above promotes the formation of glass at the increased content of BaO, where Al_2O_3, obviously, acts as a glass-forming component. Aggressiveness of glass melts in relation to the material of corundum crucibles was also established in the study of glasses of the lead-borosilicate system [11,12].

When comparing the glass-forming regions in cross sections of the system $BaO-B_2O_3-SiO_2-xAl_2O_3$, it can be concluded that the addition of Al_2O_3 expands the range of visually clear glasses as well as glasses resistant to crystallization in the production. The addition of Al_2O_3 to the composition of glasses with a high BaO content (No. 5 and No. 6) reduces the crystallization ability of the melts of these glasses, as evidenced by a decrease in the intensity of the

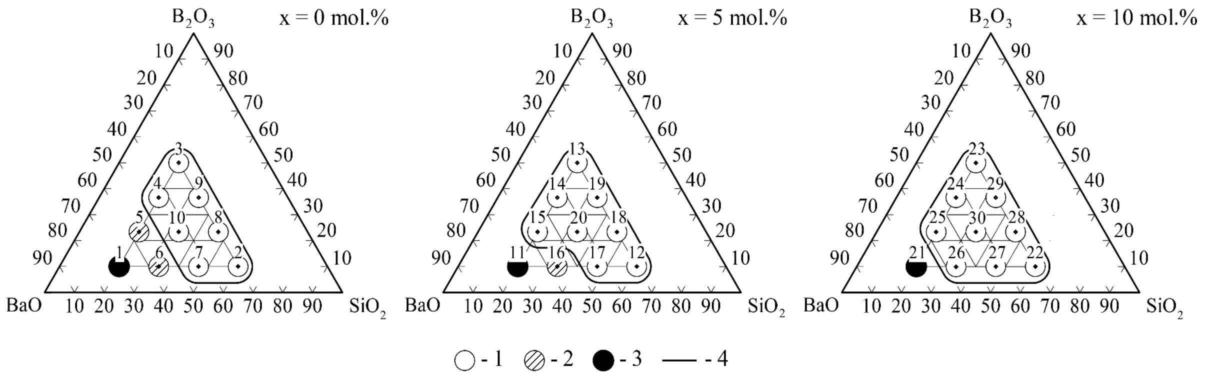

Fig. 1. Chemical compositions of experimental glasses and glass formation in the system $BaO-B_2O_3-SiO_2-xAl_2O_3$: 1 – clear glass; 2 – surface crystallization; 3 – sinter; and 4 – border of glass-forming region

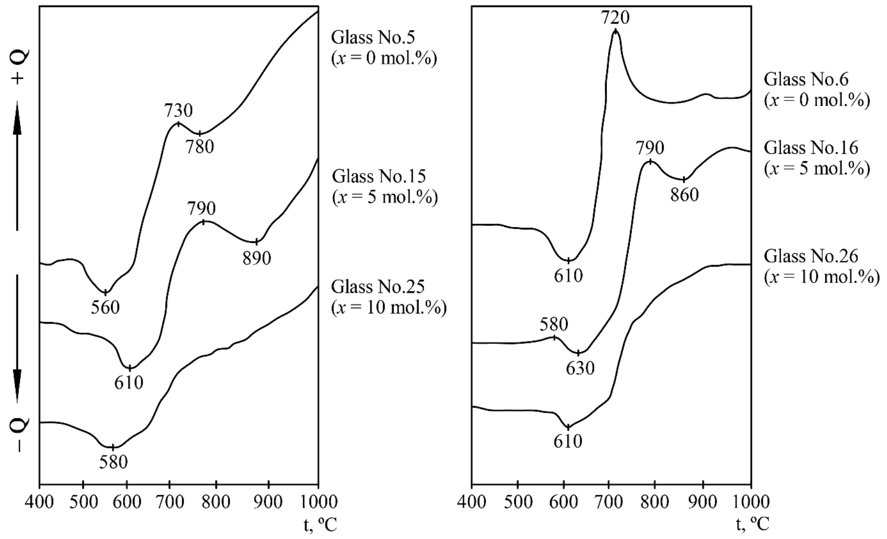

Fig. 2. Thermograms of glass powders of the system $BaO-B_2O_3-SiO_2-xAl_2O_3$

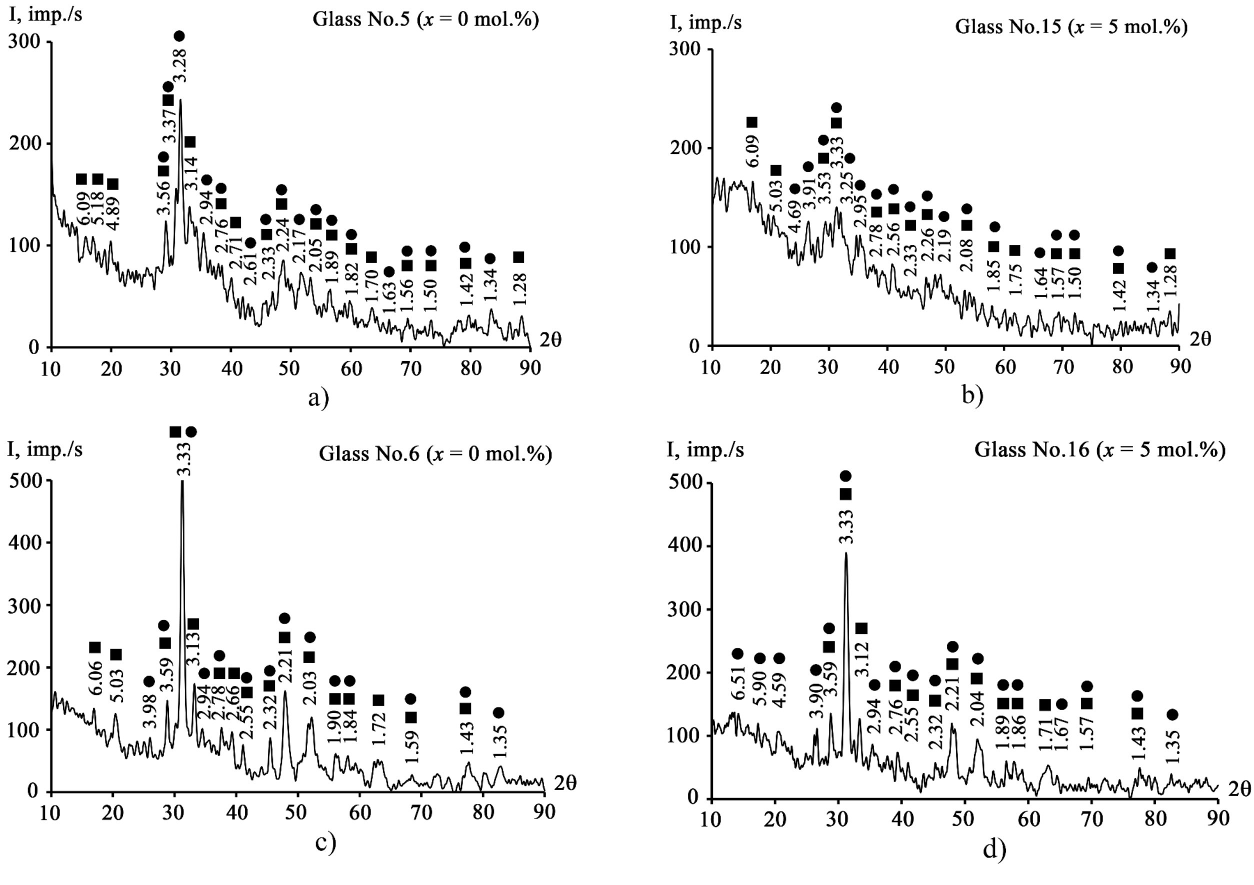

Fig. 3. X-ray patterns of the powders of glasses in the system $BaO-B_2O_3-SiO_2-xAl_2O_3$ after heat treatment during 2 hours at the temperature: a – 730°C; b – 790°C; c – 720°C; d – 790°C ■ – $BaSiO_3$, ● – $BaAl_2Si_2O_8$

relevant exothermic effects on the DTA curves (Fig. 2).

Diffusion of a part 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 the glasses No. 5 and No. 6 (Fig. 3,a and 3,c), the mixtures of which did not contain Al_2O_3 at all. The positions of the main diffraction maximums on X-ray patterns of glass powders No. 5 and No. 6 indicate that the main crystalline phases formed during the heat treatment of these glasses are barium silicate ($BaSiO_3$) and β-celsian ($BaAl_2Si_2O_8$). Additional introduction of 5 mol.% of Al_2O_3 into the composition of these glasses reduces the amount of crystalline phase, as evidenced by a decrease in the intensity of diffraction maximums on the X-ray patterns of glass powders No. 15 and No. 16 (Fig. 3,b and 3,d).

Experimentally established values of some properties of experimental glasses are given in Table 1.

Data analysis shows that depending on the chemical composition of glass, its physical and chemical properties vary over wide ranges as follows: the coefficient of linear thermal expansion of $(71-122)\cdot10^{-7}$ K^{-1}, glass transition temperature of 500–650^{0}C, dilatometric softening point of 540–670^{0}C, and density of 3.2–4.2 g cm^{-3}.

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

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

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); and x_i are the content of oxides in the glass (mol.%).

These additive equations represent a compact form of the generalized and quantitative description of the patterns of changes in glass properties depending on glass composition. In this context, additive coefficients in the equations for calculating the values of properties of the experimental glasses were determined by the multiple correlation method (Table 2). Accuracy of the calculation of these properties was evaluated by the value of the multiple correlation coefficient (R) and by comparison between the residual dispersion S^2_{res} and the dispersion relative to the average value of the experimental properties S^2_y [13–15]. As follows from the data shown in Table 2, S^2_{res} is much less than S^2_y; hence we can assume that the above equation for V gives a reasonable approximation for the experimental data

Table 1

Physical and chemical properties of glass

Glass No.	CTE, $\alpha\cdot10^7$, K^{-1}	t_g, ^{0}C	t_d, ^{0}C	lg ρ, Ohm·cm (150^{0}C)	Density, d, g cm^{-3}
2	85	650	670	11.70	3.63
3	82	540	580	11.69	3.37
4	96	510	550	13.03	3.72
5	121	500	540	12.90	4.06
6	122	550	580	13.43	4.21
7	111	610	630	12.20	4.08
8	83	620	650	12.79	3.61
9	83	580	610	11.96	3.47
10	94	590	630	13.31	3.81
12	75	640	660	11.96	3.45
13	78	570	600	11.75	3.23
14	101	550	590	12.04	3.55
15	107	530	560	12.71	3.95
16	112	560	600	13.12	4.16
17	93	620	640	12.42	3.79
18	76	600	630	12.27	3.39
19	77	580	610	12.03	3.36
20	93	570	600	12.20	3.78
22	76	620	650	11.79	3.32
23	76	570	610	11.96	3.20
24	88	540	580	12.40	3.53
25	102	520	560	12.52	3.74
26	107	580	610	12.82	4.02
27	87	600	630	12.13	3.75
28	71	600	640	12.03	3.32
29	79	580	620	12.32	3.33
30	91	560	600	11.91	3.66

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±S_v for respective oxides				R	S^2_{res}	S^2_y
	BaO	B_2O_3	SiO_2	Al_2O_3			
CTE, $\alpha_i \cdot 10^7$, K^{-1}	174±5	41±5	44±10	–25±19	0.99	15.1	214.9
Glass transition temperature, $(t_g)_i$, ^{0}C	428±18	530±18	772±17	610±74	0.99	222	1302
Dilatometric softening temperature, $(t_d)_i$, ^{0}C	480±13	584±12	773±12	638±52	0.99	107	950
Volume resistivity, $\lg\rho_i$, Ohm·cm	14.6±0.4	11.0±0.4	11.2±0.3	8.7±1.7	0.99	0.12	0.25
Density, d_i, g cm^{-3}	5.23±0.08	2.34±0.08	3.08±0.08	1.21±0.33	0.99	0.005	0.09

given in Table 2.

The established values of additive coefficients for the calculation of CTE, glass transition temperature and density of experimental glasses are consistent with the coefficients that we proposed earlier [15] for borosilicate glass. The calculated values of additive coefficients indicate that the introduction of Al_2O_3 promotes the reduction of CTE and increase in the values of t_g and t_d of glass, i.e. its viscosity.

Barium oxide makes the maximum contribution to the increase of the coefficient of linear thermal expansion, volume resistivity and density of glasses. At the same time, an increase of barium oxide content in glass sharply reduces the glass transition temperature and dilatometric softening point of the glass.

SiO_2 makes the largest partial contribution to the values of glass transition temperature and dilatometric softening point. This fact is to be taken into account, first of all, when choosing the compositions of glasses for protective coatings, as an increase in SiO_2 content will lead to an undesired increase in the temperature of formation of the protective coatings. Replacing SiO_2 with B_2O_3 reduces the glass transition temperature and the dilatometric softening temperature of the glass. This is due to the fact that B_2O_3 in the glass acts as a fluxing agent. It is also found that the replacement of SiO_2 by B_2O_3 within the studied limits of the content of components does not cause significant changes in the values of CTE of the glass.

Conclusions

We have experimentally established the conditions of glass formation, regions of glass-forming melts and properties of glasses with the following chemical composition (mol.%): BaO 30–70, B_2O_3 10–50, SiO_2 20–60, and Al_2O_3 0–10. It is shown that at the synthesis of glasses in the corundum crucible during 60 minutes at the temperature of 1350^{0}C, the glass-forming region in the system BaO–B_2O_3–SiO_2 is limited by the following contents of components (mol.%): BaO 30–50, B_2O_3 10–50, and SiO_2 20–60. It is found that the introduction of Al_2O_3 into the composition of these glasses expands the glass-forming region towards the increase in BaO content in glass to 60 mol.%. Experimentally established values of properties of the experimental glasses are within the following limits: coefficient of linear thermal expansion of (71–122)$\cdot 10^{-7}$ K^{-1}, glass transition temperature of 500–650^{0}C; dilatometric softening point of 540–670^{0}C, and density of 3.20–4.21 g·cm^{-3}. At the temperature of 150^{0}C, the volume resistivity of glasses is within the range of 10^{11}–10^{13} Ohm·cm. Generalization of the dependences of glass properties on their chemical composition was carried out with the use of additive equations, for which the partial contributions of oxides to the values of the corresponding properties were determined by experimental and statistical methods. The patterns of influence of components and conditions of glass formation on the physical and chemical characteristics of glasses of the system BaO–Al_2O_3–B_2O_3–SiO_2 allow implementing the design of a wide range of glass compositions for various functional purposes with the complex of specified properties.

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ВЛАСТИВОСТІ СТЕКОЛ В СИСТЕМІ $BaO-B_2O_3-SiO_2-xAl_2O_3$ (x=0; 5; 10 МОЛ.%)

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У роботі показано перспективність застосування системи $BaO-Al_2O_3-B_2O_3-SiO_2$, як основи склоподібних і склокристалічних матеріалів, які широко використовують в ракетобудуванні для високотемпературного захисту жароміцних сплавів, в енергетиці для герметизації твердооксидних паливних елементів, а також у виробництві термостійких склокристалічних матеріалів. Досліджено умови склоутворення та властивості стекол з наступним вмістом компонентів (мол.%): BaO 30–70, B_2O_3 10–50, SiO_2 20–60, Al_2O_3 0–10. Експериментально встановлено, що в залежності від хімічного складу скла, його фізико-хімічні властивості змінюються в наступних межах: температурний коефіцієнт лінійного розширення $(71-122)\cdot 10^{-7}$ K^{-1}; температура склування 500–650°C; дилатометрична температура розм'якшення 540–670°C; щільність 3,20–4,21 г/см³. Питомий об'ємний опір досліджених стекол при температурі 150°C знаходиться в межах 10^{11}–10^{13} Ом·см. Узагальнення залежності властивостей стекол від їх хімічного складу виконано за допомогою адитивних формул, для яких експериментально-статистичними методами визначено парціальні вклади оксидів у значення відповідних властивостей. Встановлені закономірності впливу компонентів і умов склоутворення на фізико-хімічні характеристики стекол дозволяють здійснювати процес проектування широкого спектра складів стекол із комплексом заданих показників властивостей, що вирішує задачі їх практичного використання.

Ключові слова: скло, боросилікатне скло, склоутворення, теплове розширення, температура склування.

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This article shows the prospect of the system $BaO-Al_2O_3-B_2O_3-SiO_2$ as the basis of vitreous and glass ceramic materials, which are widely used in rocket production for high-temperature protection of heat resistant alloys, in the power industry for sealing solid oxide fuel cells, and in the production of heat resistant glass ceramic materials. We examined the conditions of glass formation and properties of glasses with the following content of components (mol.%): BaO 30–70, B_2O_3 10–50, SiO_2 20–60, and Al_2O_3 0–10. We established experimentally that the physical and chemical properties of glass, depending on its chemical composition, vary within the following limits: coefficient of linear thermal expansion of $(71-122)\cdot 10^{-7}$ K^{-1}; glass transition temperature of 500–650°C; dilatometric softening point of 540–670°C; and density of 3.20–4.21 g cm⁻³. The volume resistivity of the studied glasses is within 10^{11}–10^{13} Ohm·cm at the temperature of 150°C. Generalization of the dependences of glass properties on their chemical composition was carried out with the use of the additive equations, 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 components and conditions of glass formation on the physical and chemical characteristics of glasses allows implementing the process of designing of a wide range of glass compositions with the complex of specified properties in order to solve the tasks of their practical use.

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