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OPTICAL GLASS WITH INCREASED REFRACTIVE INDEX BASED $BaO - B_2O_3 - SiO_2$ SYSTEM

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The aim of the research is to develop lead-free optical glass with increased refractive index based on $BaO - B_2O_3 - SiO_2$ system. A study of technological and physical-chemical properties of glass was conducted. It has been shown that the glass of this system tends to the phase separation of segregation type. The area of glass composition with refractive index of 1.6195 – 1.6650 has been established. The peculiarities of influence of the modifiers – Al_2O_3 and oxides of the RO group – on the technological and optical properties of the glass of examined system have been established. Lead-free optical glass with the refractive index of 1.6620 has been developed, which is dense crown glass.

Keywords: optical glass, dense crown, phase separation, refractive index

ОПТИЧЕСКИЕ СТЕКЛА С ПОВЫШЕННЫМ ПОКАЗАТЕЛЕМ ПРЕЛОМЛЕНИЯ НА ОСНОВЕ СИСТЕМЫ $BaO - B_2O_3 - SiO_2$

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Целью работы является разработка бессвинцового оптического стекла с повышенным показателем преломления на основе системы $BaO - B_2O_3 - SiO_2$. Проведено исследование технологических и физико-химических свойств стекол. Показана склонность стекол данной системы к фазовому разделению ликвационного типа. Определена область составов однородных стекол с показателем преломления 1,6195 – 1,6650. Установлены особенности влияния модификаторов – Al_2O_3 и оксидов группы RO – на технологические и оптические свойства стекол исследуемой системы. Разработано бессвинцовое оптическое стекло с показателем преломления 1,6620, относящееся к типу тяжелых кронов.

Ключевые слова: оптическое стекло, тяжелый крон, фазовое разделение, показатель преломления

$BaO - B_2O_3 - SiO_2$ TIZIMI ASOSIDAGI YUQORI SIRISH INDEKSLI OPTIK SHISHALAR

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Ishning maqsadi $BaO - B_2O_3 - SiO_2$ tizimi asosida yuqori nur sindirish ko'rsatgichiga ega qo'rg'oshinsiz optik shisha tarkibini ishlab chiqishdan iborat. Shishalarning texnologik va fizik-kimyoviy xususiyatlari tadqiq qilindi. Ushbu tizim shishalarining likvatsiya turidagi fazalar bo'linishiga moyilligi ko'rsatib o'tilgan. Nur sindirish indeksi 1,6195 - 1,6650 bo'lgan shisha hosil bo'lish kompozitsiyalari diapazoni aniqlandi. Modifikatorlar - Al_2O_3 va RO guruhi oksidlarining o'rganilayotgan tizim shishalarining texnologik va optik xususiyatlariga ta'sirining o'ziga xos xususiyatlari aniqlandi. Og'ir kronlar turiga mansub bo'lgan, 1,6620 nur sindirish indeksiga ega qo'rg'oshinsiz optik shisha tarkibi yaratildi.

Kalit so'zlar: optik shisha, og'ir toj, fazalarni ajratish, sindirish ko'rsatkichi

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Introduction

$BaO - B_2O_3 - SiO_2$ system serves as the basis for production of glass and glass-ceramic materials for various purposes. Based on this system with the addition of aluminum oxide, radioprotective glass has been produced that is used in medicine and industry for radiation facilities [1]. The study of low-silica part of $BaO - B_2O_3 - SiO_2$ system with a BaO content of 30 and 50 mol.%, and B_2O_3 20 – 60 mol.% has allowed to obtain low melting glass for welding and sealing ceramics and metal [2]. Based on the borosilicate glass, insulation coatings have been obtained for instrument making as well as heat-resistant coatings for parts designed for work under high temperature conditions [3, 4].

One of the promising areas for the use of the alkaline-free borosilicate glass is the creation of materials for welding and sealing solid oxide fuel cells [5–7].

At development of electrical insulating materials, limits of glass formation at 1350 °C in $BaO - B_2O_3 - SiO_2$ system with the content of components, mol. %: BaO 30 – 70, B_2O_3 10 – 50, SiO_2 20 – 60 have been established. It has shown that in the studied composition area homogeneous melt production is limited by 50 mol. % BaO content. Introduction the 5 and 10 mol. % Al_2O_3 widens the area of glass formation and increases glass resistance to crystallization [8].

Optical glass of dense crown type based on $BaO - B_2O_3 - SiO_2$ system is in demand in the instrument making. The refractive index of the dense crown glass is 1.55–1.66 at the dispersion factor of 50–64 [9–12].

Leading manufacturers have developed optical glass with various combinations of the optical constants – refractive index, medium and partial dispersions. The range of optical glass is increased due to development of the new compositions with the optical constants that expand the known areas of compositions on Abbe diagram. Such materials include optical glass types with a high refractive index because the lenses from such glass types have less curvature, which makes it easier to correct spherical aberrations. These glass types include dense and extra dense crown glass [12–16]. A number of optical glass grades of crown type contain lead oxide. The replacement of toxic lead oxide without losing the highly refractive properties of the glass is one of the priority areas of research in optical glass making [17–19].

The aim of the research is to develop lead-free optical glass with increased refractive index based on $BaO – B_2O_3 – SiO_2$ system. In optical glass development the first step is to ensure the established values of the optical properties that are determined by the chemical composition of the glass. The main technological problems to be solved in the development of the optical glass are the achievement of the specified values of optical constants, as well as a high degree of homogeneity of the glass and optical constants. Homogeneity of the optical glass, absorption, bubble class and other normalizable parameters are determined by the production technology: choice of the main and auxiliary raw materials, melting and annealing modes. It is therefore necessary to pay attention to the mentioned technological factors during optical glass development.

Research methods

Experimental glass compositions include, mol. %: SiO_2 40 – 60, B_2O_3 20 – 40, BaO 20 – 40. The experimental planning method was used (Scheffé simplex-lattice plans) [20].

Based on the basic system, glass was synthesized, into which MgO, CaO, ZnO, Al_2O_3 oxides were introduced in amount of 2.5–15 mol.% as modifiers, and Sb_2O_3 as brightening agent. During the selection of the modified compositions of the optical glass, preliminary calculation of the optical constants was performed with the use of the methods developed by A.A. Appen and L.I. Demkina [9, 21].

The raw material to be introduced for SiO_2 was quartz grit, barium oxide was introduced in the form of barium carbonate and nitrate, Al_2O_3 and $Al(OH)_3$ were used as the aluminum containing raw material. Use of barium nitrate with antimony oxide ensures glass clarification and discoloration. Glass was synthesized in a gas furnace with turbulent flame movement at the maximum temperature of 1350–1400 °C for 1 hour in order to stabilize the melting mode. The furnace temperature was increased at the rate of 250 °C/h, the gas environment is oxidizing with air excess factor of up to 1.13. Glass working was performed by molding.

The crystal structure was investigated by X-ray diffraction measurements using diffractometer D8 Advance with CuKα radiation source. The software DIFFRACPLUS from Bruker package was used to identify crystalline phases.

The crystallization ability of glasses was determined from the results of gradient heat treatment. Gradient crystallization was carried out in a gradient furnace SP30/13 nin the temperature range of 700–1000 °C.

Low-temperature viscosity of glass was determined by dilatometry method. The research was performed with use of quartz dilatometer Netzsch DIL 402 PC. The measurement was performed within the temperature range of 20–750 °C at the constant heating rate of 10°C/min. Dilatometric chart in application programme is used to determine a number of characteristic temperatures corresponding to certain low-temperature viscosity values, and linear temperature expansion coefficient parameters.

Glass refractive index was measured with use of analog Abbe refractometer KRÜSS Optronic AR4.

Microhardness of the test glass was determined with use of microhardness tester 401/402-MVD.

Results and Discussion

According to results of $BaO – B_2O_3 – SiO_2$ system glass synthesis it has been established that melts with silicon oxide content of 40 mol.% are uniform and have low glass viscosity at a temperature of 1350 °C. Figure 1 presents results of evaluation of the quality of glass according to its position in the concentration triangle. During the glass

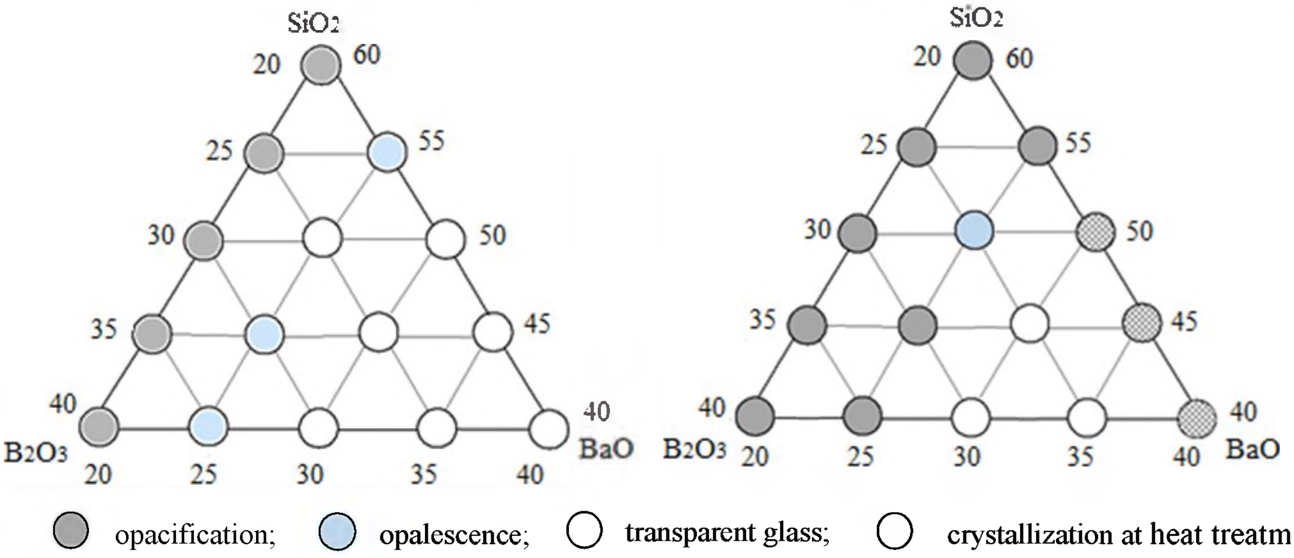


Figure 1. Chemical compositions of experimental glasses in the system BaO–B_2O_3–SiO_2 (mol. %) and the results of heat treatment.

working opacified, opalescent and transparent glass samples were obtained. Opacified effect is typical for test glass with BaO 20–25 mol.% content and is due to the phase separation of liquation type characteristic of borosilicate glass. In RO – B_2O_3 – SiO_2 and R_2O – B_2O_3 – SiO_2 glass forming systems there are areas of metastable liquation, the dimensions of which increase with the increase of the force of the modifier cations area. The cause of liquation is incompatibility of [BO_3] and [SiO_4] structural groups [22–24].

The liquation nature of opacification is confirmed by the X-ray amorphousness of the samples. The opalescence effect is typical for the samples with binodal type of liquation, at which the size of drops is less than 0.1 μm. The compositions of opalescent glass appear to be close to the boundary of the liquation cupola.

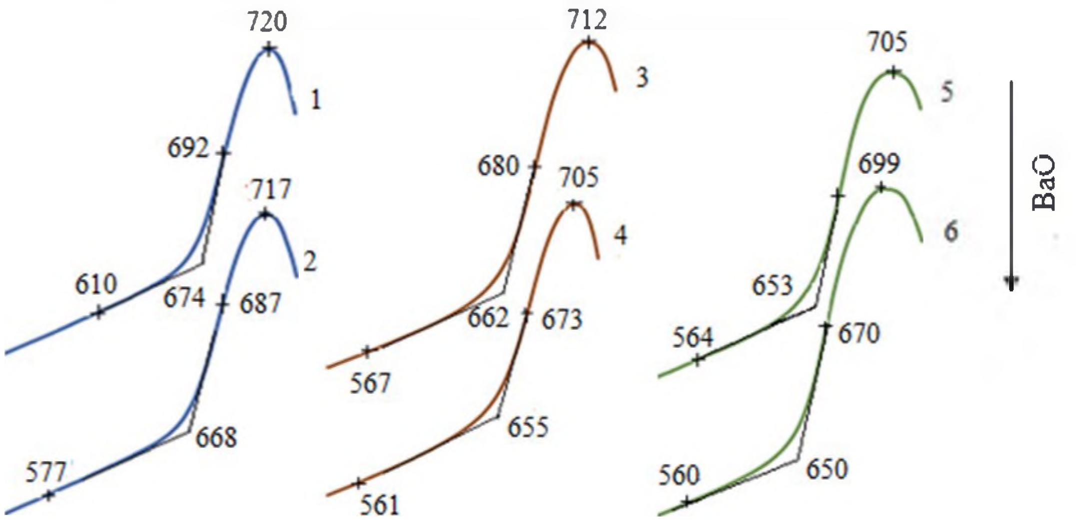
Homogeneous samples of glass with high light transmission are obtained in the area of the compositions including mol.%: 40 SiO_2, 20 – 30 B_2O_3, 25 – 40 BaO. Based on the results of the study of crystallization ability of these types of glass, it is established that surface crystallization in the temperature range of 800–1000 °C is characteristic of glass with the 20 mol.% B_2O_3 (Fig. 1).

Glass refractive index in this area of compositions is 1.5880–1.6750, and it increases naturally with the increase of the barium oxide content. The contribution of boron oxide into the refractive index increases with the increase of BaO/ B_2O_3 molar ratio, which is due to the changes in coordination state of B^{3+} ions and their position in the glass structure. In borosilicate glass structure with BaO content 35–45 mol.% with the gradual substitution of B_2O_3 with SiO_2, transformations among borate structural units prevail [25]. With the boron substitution for silicon, borosilicate ring structures are observed to appear [26, 27].

The microhardness of the test glass is 3950–5230 MPa, the silicon oxide being the determining factor.

With the use of dilatometry method, a number of characteristic temperatures have been defined that allow to assess the influence of glass composition on its low temperature viscosity and to determine temperature interval of critical annealing stage (Fig. 2).

The dilatometric curve of the glass have the following characteristic temperatures: the glass transition temperature (viscosity is $10^{12.3}$ Pa·s); lower annealing temperature (viscosity is $10^{13.5}$ Pa·s); upper annealing temperature (viscosity is 10^{12} Pa·s); glass deformation (viscosity is $10^{10.3}$ Pa·s) [23, 28]. The test glass is characterized by increased glass transition temperatures of 650–674 °C. Initial deformation temperature of the glass is 699–720 °C, the lower annealing temperature is above 560 °C. As seen in Figure 2, when B_2O_3 is replaced by BaO, glass transition temperature decreases. This indicates a more pronounced fluxing effect of barium oxide in comparison with that of boron oxide. Considering that the viscosity of the melts, which is less than 10 Pa·s, is reached at the temperatures of 1350–1400 °C, it can be con



Content SiO_2, mol.%: 1, 2 – 50; 3, 4 – 45; 5, 6 – 40

Figure 2. Dilatometric curves of the glasses.

cluded that the test glass has a higher viscosity gradient in temperature intervals of molding and glass transition.

In order to study the influence of the modifiers on the optical glass properties, glass base composition (mol.%) $30BaO \cdot 25B_2O_3 \cdot 45SiO_2$ was synthesized where boron oxide was substituted with RO (MgO, CaO, ZnO) group oxides and Al_2O_3. During the glass synthesis it was established that the calcium oxide introduction into the glass composition in amounts exceeding 5 mol % increases the corrosive power of the melt, which causes the crucibles to corrode.

Positional heat treatment of charge of modified compositions was performed at the temperatures of 800, 1000 and 1200 °C in order to assess the influence of the modifiers on the glass formation processes. At the heat treatment temperature of 800 °C, grains of the charge sinter in the samples. Active melting of mineral phases and dissolution of refractory components leads to formation of bottom layer of the melt at the heat treatment temperature of 1000 °C. For the basic composition sample, the glass phase quantity is 30%, at RO group modifiers introduction in amount of 10 mol.%, volume content of the vitreous phase increases to 50–75%. When composition is modified with alumina-containing components, activity of glass formation processes depends significantly on the type of the raw material. In particular, use of aluminum hydroxide accelerates glass forming process contrary to aluminum oxide, with which the sample is a vitreous foam mass at 1000 °C.

The increase in heat treatment temperature up to 1200 °C causes formation of the melt, on the surface of which there is batch foam that comprises crystal, glass and gas phases. The minimum volume of the batch foam is typical for the samples modified with CaO and ZnO.

Crystallization capacity of the glass is predictably determined by the content of modifiers. At RO group oxides content of 40 mol.% or more the heat treatment in the gradient electric furnace leads to surface crystallization. The glass with CaO and ZnO as modifiers has sufficiently high resistance to crystallization, the signs of which are not detected during the gradient heat treatment. Resistance to crystallization during glass working allows the use of various molding methods and modes, including prolonged cooling.

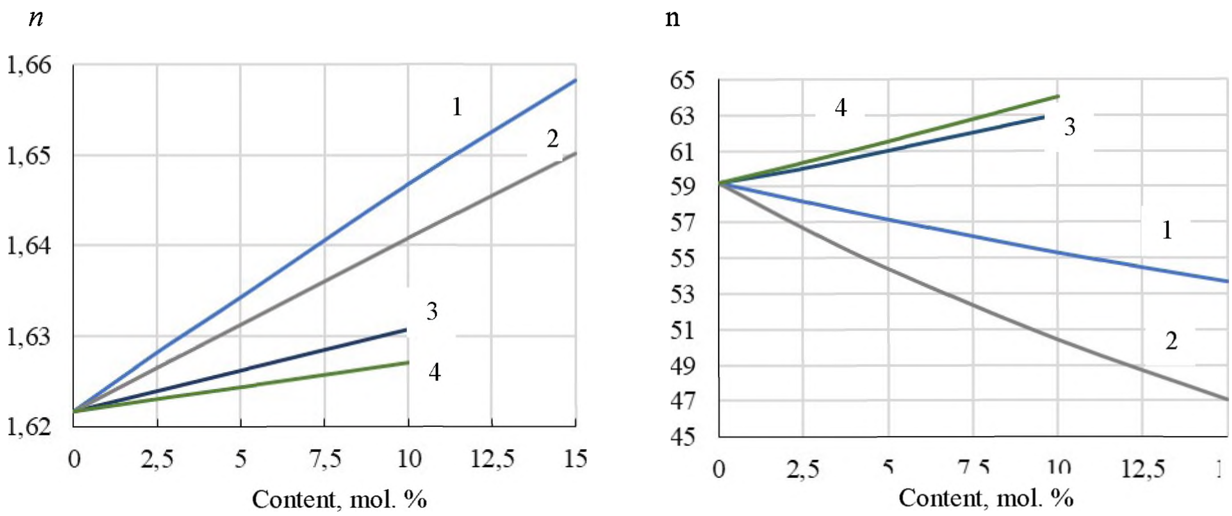
Based on the dilatometric test of the glass containing 10 mol.% of modifiers, characteristic temperatures and linear expansion thermal coefficient indicators have been determined (Table 1). Modification of the glass with magnesium and calcium oxides leads to an increase in viscosity parameters, which is manifested in increase in glass transition temperature and other characteristic temperatures. Introduction of ZnO and Al_2O_3 does not have a pronounced influence on the low-temperature viscosity of the test glass. The influence of modifiers on linear expansion thermal coefficient indicators complies with their partial contribution to this value.

Figure 3 shows the dependence of the refractive index (n) and dispersion (n) of the test

Table 1

Characteristic temperatures and linear expansion thermal coefficient of modified glasses

Modifier	Temperature, °C, corresponding to viscosity, lgh				Coefficient of linear thermal expansion, $a \times 10^7$, K^{-1}
	13.5	12.3	12	10.3	
Basic composition	567.4	662.2	682.2	713.3	63.2
MgO	575.9	666.6	687.7	724.8	70.8
CaO	583.3	667.5	688.2	726.8	79.4
ZnO	576.8	661.4	681.8	718.3	74.5
Al_2O_3	568.8	660.8	682.2	719.0	70.6



Modifiers: 1 – CaO; 2 – ZnO; 3 – MgO; 4 – Al_2O_3

Figure 3. The influence of modifiers on the optical properties of glasses.

glass on the type and content of the modifiers. Introduction of CaO and ZnO modifiers in amount of 10 mol.% causes an increase in the refractive index from 1.6216 to 1.6467.

MgO and Al_2O_3 increases refractive index to a much lesser extent, nevertheless, these components increase dispersion coefficient. Due to the ambiguous influence of MgO and Al_2O_3 on the optical properties, these components can be used in dense crown glass in the form of minor additives in order to adjust the physico-chemical and technological properties.

Based on the results of the study of optical and technological properties of BaO – B_2O_3 – SiO_2 glass system with MgO, CaO, ZnO, Al_2O_3 additives, lead-free optical glass has been developed with the following optical properties: refractive index is 1.6620, dispersion coefficient – 54.2. Technological characteristics are: glass melting temperature is 1400±10 °C, temperature range of critical annealing is 590–688°C.

Conclusion

BaO – B_2O_3 – SiO_2 system is the basis for production of the glassy materials for various technical purposes with wide range of performance requirements. For development of materials in demand in the instrument-making, systematic studies have been conducted on glass of the given system according to indicators of technological and optical properties. Glass with 40 – 60 SiO_2, 20 – 40 B_2O_3, 20 – 40 BaO mol.% composition has been synthesized, and peculiarities of glass formation properties at the temperature of 1400 °C have been established. At the molar ratio BaO/$B_2O_3 \leq 1$, phase separation of liquation type occurs during melt cooling process. Crystallization capacity of homogeneous glass samples increases with the increase of BaO content, and stability of the glass state is ensured when content of this component is less than 35 mol.%. According to the dilatometry data, replacement of B_2O_3 with BaO results in a decrease in low temperature viscosity. The refractive index of the homogeneous glass is 1.6195–1.6650, its microhardness – 3950–5230 MPa. The basic composition of $30BaO\times25B_2O_3\times45SiO_2$ (mol. %) has been selected according to the set of its technological and optical properties. A study of influence of RO (MgO, CaO, ZnO) group oxides and Al_2O_3 introduced in the amount of 2.5–15 mol.%, on the properties of glass base composition has been conducted. Introduction of modifiers accelerates glass formation processes and increases stability of glass state. Introduction of MgO and CaO oxides causes an increase in low temperature viscosity. Modification of CaO and ZnO oxides leads to an increased glass refraction. At optimization of the type and the number of modifiers, lead-free optical glass with increased refractive index of 1.6620 and a dispersion coefficient of 54.2 has been developed. Reduced crystallization capacity of this glass is an important technological factor determining the molding modes for the optical preforms.

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