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*A.V. Zaichuk*<sup>a</sup>, *Y.R. Kalishenko*<sup>a</sup>, *A.A. Amelina*<sup>a</sup>, *Y.S. Hordieiev*<sup>a</sup>, *S.A. Halushka*<sup>b</sup>,  
*O.S. Savchenko*<sup>b</sup>, *M.V. Nahorny*<sup>b</sup>

## FEATURES OF FORMATION OF THE CELSIAN PHASE DURING FIRING OF HEAT-RESISTANT CERAMICS IN THE SYSTEM BaO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub>

<sup>a</sup> Ukrainian State University of Chemical Technology, Dnipro, Ukraine

<sup>b</sup> Sumy National Agrarian University, Sumy, Ukraine

In the synthesis of heat-resistant ceramics in the system BaO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> (BAS), it is important to find ways of intensifying the process of transition of hexagonal celsian to monoclinic celsian without an increase in sintering temperature. Monoclinic form of celsian is characterized by higher thermal, electro-insulating, and mechanical properties. This paper deals with the features of formation of the phase composition of celsian ceramics when using BAS glass of eutectic composition and glass in the system Li<sub>2</sub>O–Al<sub>2</sub>O<sub>3</sub>–B<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> (LABS) of spodumene composition as modifying components. It is shown that monoclinic celsian is the final crystalline phase formed in ceramics synthesized on the basis of barium carbonate and kaolin. Monoclinic celsian is formed stepwise; and the hexagonal celsian appears first. The complete transition hexagonal celsian→monoclinic celsian occurs only in the process of high temperature firing at 1250°C. Notably, the degree of ceramic sintering remains low (water absorption is 11.0%). Introduction of BAS glass contributes to the complete transition of hexagonal celsian to monoclinic celsian at a reduced temperature of 1100°C. Maximum effect in the formation of monoclinic celsian is achieved by the introduction of LABS glass. As a result, the temperature of formation of this modification maximally decreases to 800°C. In this case, complete sintering of celsian ceramics is achieved at the temperature of 1250°C.

**Keywords:** heat-resistant ceramics, monoclinic celsian, hexagonal celsian, crystallization, solid-phase sintering, coefficient of linear thermal expansion.

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### Introduction

Thermal stability is the main characteristic ensuring the long-term operation of ceramics under conditions of high thermo-mechanical loads. Products made of heat-resistant ceramics are capable of withstanding stresses, which arise in the material at sudden temperature drops. At the same time, the material retains the required values of operational properties (mechanical strength, chemical stability, and dielectric indices). Consequently, heat-resistant ceramics is increasingly used for high-temperature equipment in the household sector, machine building, electronics, power engineering, aviation, and aerospace industries [1]. Quartz ceramics [2] and glass-crystalline materials obtained predominantly

based on aluminosilicate systems [3] are used as relatively inexpensive and effective heat-resistant materials.

A significant disadvantage of quartz ceramics is high porosity; therefore, additional hardening and sealing are required for its effective use [4,5].

Lithium- [6,7] and magnesium-containing [8, 9] aluminosilicate ceramics have a set of high functional characteristics, but the limited range of operating temperature (generally up to 1100°C).

Therefore, despite the abundance of existing types of ceramics, the problem of creating new compositions with increased thermal stability is still relevant.

Alkali-free ceramic materials, in particular,

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*A.V. Zaichuk, Y.R. Kalishenko, A.A. Amelina, Y.S. Hordieiev, S.A. Halushka, O.S. Savchenko, M.V. Nahorny*

those based on the system  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$ , are rather promising.

Celsian ceramics features the electronic nature of electrical conductivity. The ionic component of electrical conductivity becomes significant at temperatures above  $600^\circ\text{C}$  only. Along with high electro-insulating properties, celsian ceramics exhibits good thermal and chemical stability, as well as resistance to the impact of extremely high temperatures (up to  $1400^\circ\text{C}$  and above). The complex of physical and technical indices is achieved due to the properties of the main crystalline phase. The major drawback of celsian ceramics is its relatively low mechanical strength. Celsian ( $\text{BaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ) can crystallize in the monoclinic or hexagonal system. The hexagonal form features  $\alpha$  and  $\beta$ -modifications. A reversible transition from one modification to another occurs at  $300^\circ\text{C}$ . As a result, there is a volumetric expansion of more than 0.3%, which can lead to the formation of stresses and cracks in the material. The monoclinic form has no modification transformations and exhibits higher electro-insulating, thermal and mechanical properties [10].

It follows from the above that creating the monoclinic celsian form is highly preferred during the synthesis of heat-resistant BAS ceramics.

The modern literature contains descriptions of various modifying additives, which allow intensifying the transition of the hexagonal celsian to the monoclinic form without increasing the ceramics sintering temperature. In this case, a dense structure of the material is achieved.

Tong et al. [11] proposed a method for preparation of celsian glass-ceramics containing the monoclinic form, where the powder of BAS glass of the eutectic composition was sintered with powders of  $\text{BaAl}_2\text{O}_4$  and  $\text{SrAl}_2\text{O}_4$ . Formation of monoclinic celsian is completed at the temperature of  $1350^\circ\text{C}$  when the content of  $\text{SrAl}_2\text{O}_4$  exceeds 0.2 mol. At the same time, glass ceramics under study is characterized by the high value of the linear coefficient of thermal expansion (linear coefficient of thermal expansion  $\text{LCTE}=49\cdot 10^{-7} \text{ deg}^{-1}$ ), which has a negative effect on thermal stability.

The kinetics of transformation of hexa-celsian into monoclinic celsian was also studied when BAS ceramics was modified by mineralizers with the cations of different ionic radii and degrees of oxidation. The data of X-ray diffraction analysis showed that compounds with the oxidation degree of 3+ were less effective than mono- and divalent cations characterized by ionic radius from 0.68 Å to 1.12 Å [12].

The papers [13,14] described the mineralizing

effect of lithium oxide on celsian ceramics when lithium oxide was introduced using glasses of the system  $\text{Li}_2\text{O}-\text{B}_2\text{O}_3$  (5–7 wt.%) [13] and  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{B}_2\text{O}_3-\text{SiO}_2$  of spodumene composition (20–30 wt.%) [14]. In this case, monoclinic celsian is the main crystalline phase of the synthesized ceramics. Ions of  $\text{Li}^+$  act effectively in the sintering process and modification transition hexagonal celsian  $\rightarrow$  monoclinic celsian in BAS glass-ceramics obtained by sol-gel method [15]. Transition of the hexagonal form of celsian into the monoclinic form and intensive sintering is completed at the temperature of  $1400^\circ\text{C}$ .

The purpose of this was is to study the features of formation of the celsian phase in the system  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  with the use of BAS glass of eutectic composition and LABS glass of spodumene composition as the modifying components.

#### **Experimental**

In order to obtain the celsian ceramics, we used enriched kaolin of zref-1 grade and barium carbonate chemical brand. Glass in the system  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{B}_2\text{O}_3-\text{SiO}_2$  of spodumene composition and glass in the system  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  of eutectic composition were used as the modifying components. To provide the additional high fusibility of BAS glass,  $\text{B}_2\text{O}_3$  (10 parts by weight over 100 wt.%), which did not take part in phase formation reactions, was additionally introduced into the charge. Technical alumina G-0 brand and silicon(IV) oxide A brand were introduced as additional components to achieve the stoichiometric composition of the celsian ceramics based on BAS glass.

For melting of LABS glass and BAS glass developed by us, we used the following raw materials of technical purity: lithium carbonate ( $\text{Li}_2\text{CO}_3\geq 99.9$  wt.%), barium carbonate ( $\text{BaCO}_3\geq 99.0$  wt.%), technical alumina G-0 brand ( $\text{Al}_2\text{O}_3\geq 98.0$  wt.%), silicon(IV) oxide A brand ( $\text{SiO}_2\geq 99.5$  wt.%), and boric acid ( $\text{H}_3\text{BO}_3\geq 99.8$  wt.%). Melting of LABS glass and BAS glass was carried out in corundum crucibles at temperatures of  $1350^\circ\text{C}$  and  $1300^\circ\text{C}$ , respectively, for 1 h.

Ceramic slips were prepared from the initial components by the method of joint wet grinding until complete passing through the sieve No. 0063. Samples of the prepared slips with the moisture content of 25–27% were cast into plaster molds in the form of cylinders ( $d=h=10$  mm) and square section rods ( $5\times 5\times 50$  mm). Samples dried to the residual moisture content of 0.5% were fired in the electric furnace in the air according to specified temperature-time conditions. Temperature of sintering was equal to  $800-1250^\circ\text{C}$  with the

isothermal holding for 1 h.

For the calculation of LCTE of the celsian ceramics in the temperature range of 20–400°C, we measured the relative elongation (DI) of samples of 5×5×50 mm in size at the heating rate of 3°C/min.

The mineralogical composition of crystallized BAS glass, LABS glass, and celsian ceramics was determined on Philips APD-15 diffractometer in  $\text{CoK}_\alpha$  radiation.

### Results and discussion

This paper considers the features of formation of the celsian phase in the system  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  with the use of BAS glass of the eutectic composition (eutectic temperature 1320°C) and LABS glass of spodumene composition as modifying components.

These modifying components were chosen in order to evaluate the effectiveness of the action of  $\text{Li}^+$  ions and  $\text{Ba}^{2+}$  ions contained in glasses on the modification transition hexagonal celsian→monoclinic celsian during firing of celsian ceramics.

LCTE of BAS glass is  $89.1 \cdot 10^{-7} \text{ deg}^{-1}$ . LABS glass was characterized by LCTE equal to  $60.7 \cdot 10^{-7} \text{ deg}^{-1}$  [14].

Glass was introduced into the celsian ceramics in amounts that allowed achieving complete sintering of the material. Our previous studies [14] proved the

optimal content of LABS glass being equal to 30 wt.% (composition C-3). It is advisable to introduce BAS glass in the amount of 50 wt.% (composition C-5).

BAS glass after its crystallization is represented by hexagonal celsian and barium metasilicate  $\text{BaSiO}_3$ . Therefore, during the synthesis of celsian ceramics, barium metasilicate was bound into the celsian phase by introducing missing components ( $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ ). The product of the crystallization of LABS glass is  $\beta$ -spodumene.

For comparison, we also studied a blank sample of celsian ceramics (composition C-0), which did not contain the studied glass. The content of oxides in the composition C-0 corresponded to their stoichiometric ratio in the celsian ( $\text{BaO}:\text{Al}_2\text{O}_3:\text{SiO}_2=1:1:2$ ).

X-ray phase analysis and dilatometric studies were used to evaluate the change in the phase composition of the celsian ceramics. LCTE of the monoclinic form of celsian is  $27 \cdot 10^{-7} \text{ deg}^{-1}$ , which is significantly lower than LCTE for the hexagonal form ( $80 \cdot 10^{-7} \text{ deg}^{-1}$ ) [10].

The dynamics of changes in the phase composition of celsian ceramics was studied on samples fired in the temperature range of 800–1250°C

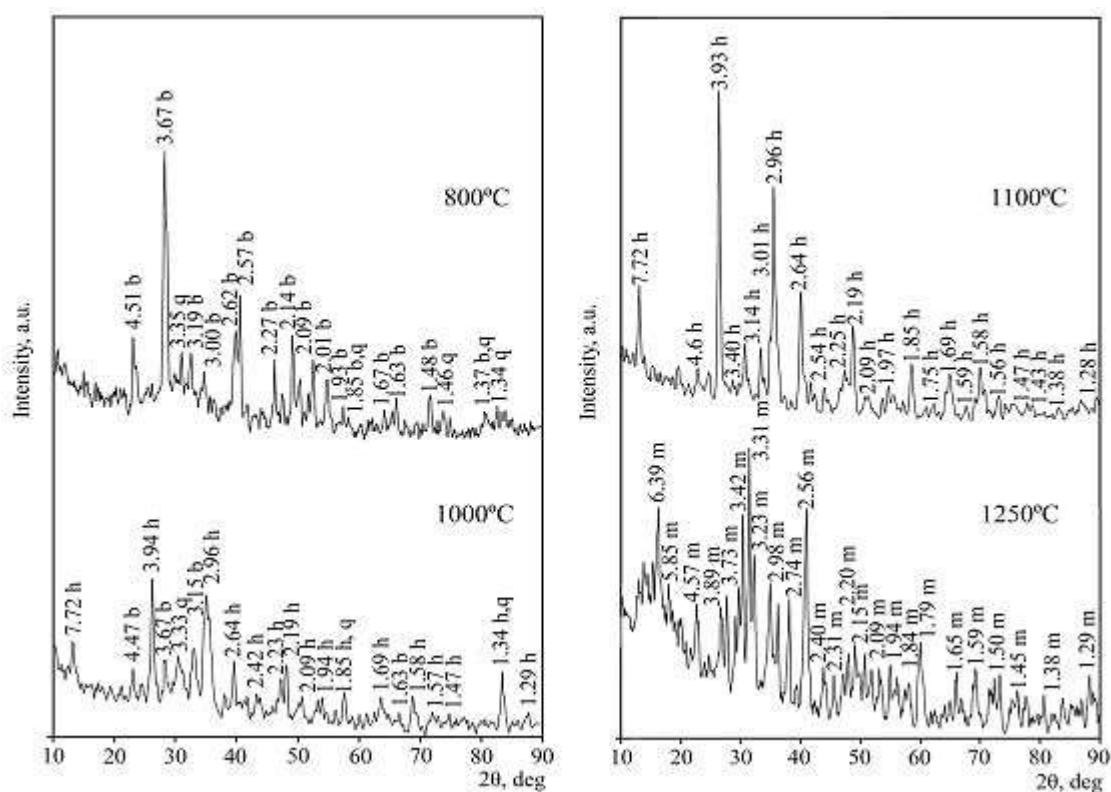


Fig. 1. X-ray patterns of celsian ceramics C-0 fired in the temperature range of 800–1250°C:

b – barium carbonate, q –  $\beta$ -quartz, h – hexagonal celsian, m – monoclinic celsian

with the holding of 1 hour.

The results of X-ray phase analysis showed (Fig. 1) that in the composition C-0 without experimental glasses, the formation of the celsian phase begins at the temperature of 1000°C. Here, the celsian is represented by a hexagonal form ( $d \cdot 10^{10} = 7.72; 3.94; 2.96; 2.64; \text{ and } 2.19 \text{ m}$ ). It should be noted that interaction of the initial raw materials is not completed at the temperature of 1000°C. The above is confirmed by the presence of barium carbonate and  $\beta$ -quartz in the phase composition of the compound. An increase in the firing temperature to 1100°C causes the enhancement of the diffraction pattern of hexagonal celsian; this can be explained by the formation of a more perfect crystalline phase structure and an increase in its amount. This conclusion is consistent with the results of dilatometric measurements (Fig. 2), i.e., there is an increase in the values of LCTE of the blank sample of celsian ceramics to  $91.3 \cdot 10^{-7} \text{ deg}^{-1}$ . Monoclinic celsian is formed only after firing at the temperature of 1250°C. In this case, its main diffraction maxima ( $d \cdot 10^{10} = 6.39; 3.42; 3.31; 2.98; \text{ and } 2.56 \text{ m}$ ) are characterized by low intensity. Formation of the monoclinic form of celsian causes a sharp drop in the LCTE of ceramics C-0 to  $40.8 \cdot 10^{-7} \text{ deg}^{-1}$ .

The process of formation of the celsian phase during the firing of ceramics C-5 includes three

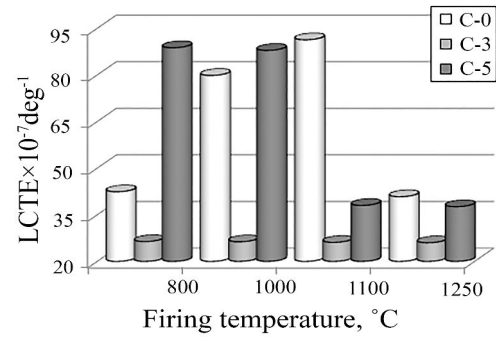
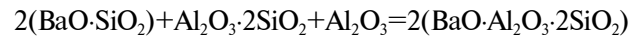


Fig. 2. Dependence of LCTE of celsian ceramics on the firing temperature

components (Fig. 3). Firstly, celsian is formed through solid-phase interaction of raw materials (kaolin and barium carbonate). Secondly, crystallization of the celsian phase from BAS glass takes place. In addition, the binding of the BAS glass components into the celsian phase is recorded. This process can be predominantly described by the following reaction:



The use of LABS glass as a modifying additive in the composition of celsian ceramics (composition C-3) allows achieving the effect of the appearance

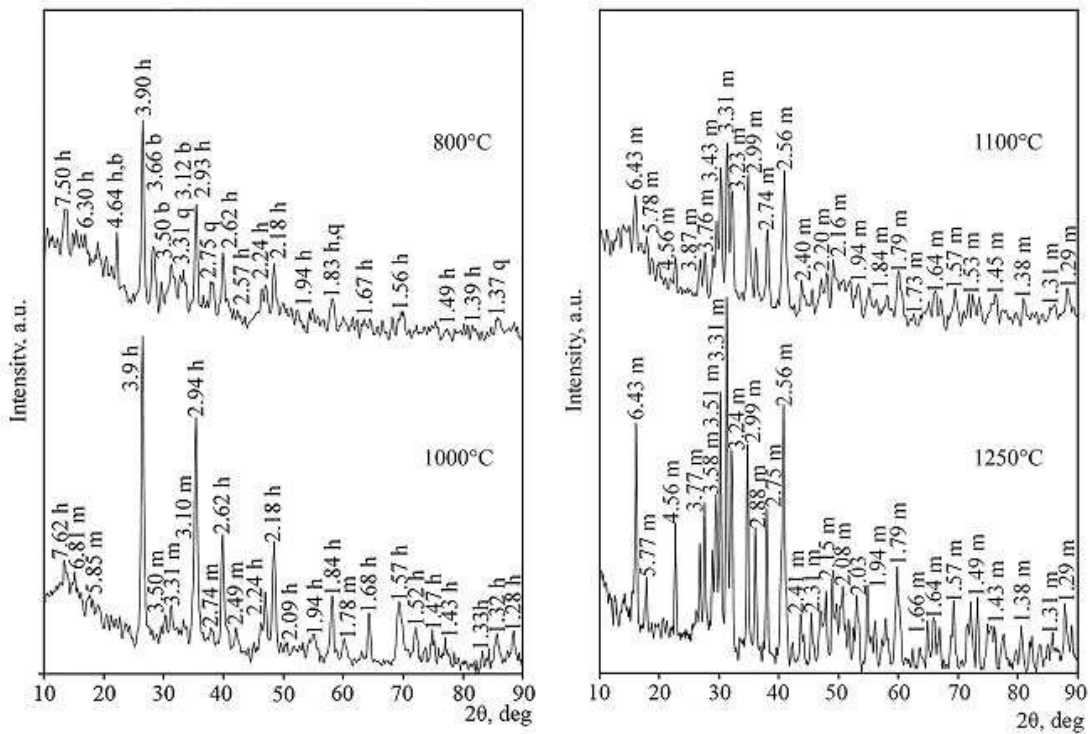


Fig. 3. X-ray patterns of celsian ceramics C-5 fired in the temperature range of 800–1250°C:  
 b – barium carbonate, q –  $\beta$ -quartz, h – hexagonal celsian, m – monoclinic celsian

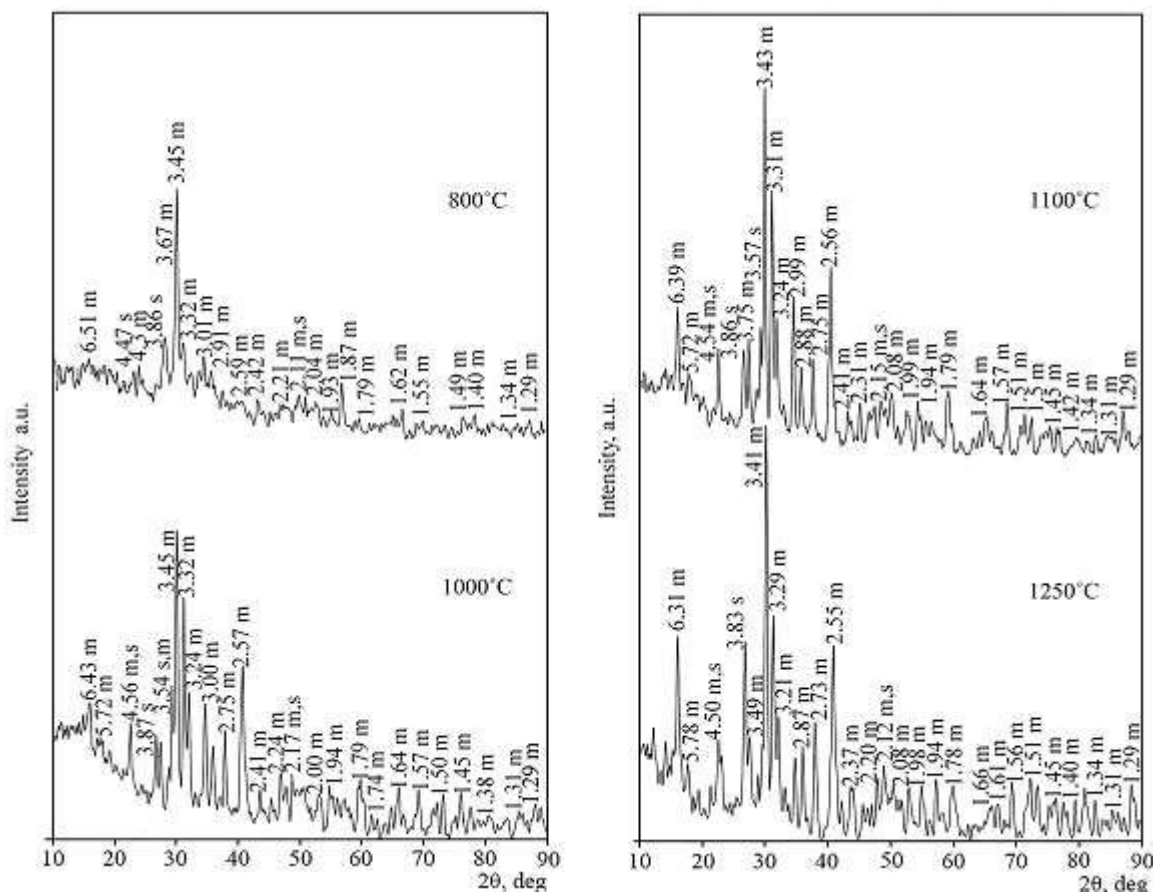


Fig. 4. X-ray patterns of celsian ceramics C-3 fired in the temperature range of 800–1250°C:

m – monoclinic celsian, s –  $\beta$ -spodumene

of the monoclinic form of celsian already at the temperature of 800°C (Fig. 4). The intensity of the diffraction maxima of the monoclinic celsian phase depends on the firing temperature and gradually increases with its rise to 1250°C. Along with monoclinic celsian,  $\beta$ -spodumene, which is a product of crystallization of LABS glass, is also found in the phase composition of ceramics. The presence of  $\beta$ -spodumene, obviously, determines the stability of LCTE values of celsian ceramics C-3  $(26.0\text{--}26.4)\cdot 10^{-7} \text{ deg}^{-1}$  in the wide firing temperature range of 800–1250°C.

It should be noted that the introduction of LABS glass provides the maximum degree of sintering of celsian ceramics and allows us to achieve zero water absorption at the temperature of 1250°C. Introduction of a part of components of celsian ceramics using BAS glass acts effectively on its sintering as well. In this case, the water absorption of the resulting material is 2.5%, which is significantly lower than the water absorption of a blank sample of celsian ceramics (11.0%).

### Conclusions

Thus, we studied the features of the formation of the celsian ceramics phase composition using BAS glass of eutectic composition and LABS glass of spodumene composition as modifying components. It was found that the introduction of BAS glass contributes to the complete transition of hexagonal celsian to monoclinic celsian at a reduced temperature of 1100°C. The maximum effect was achieved by the introduction of LABS glass. As a result, the temperature of formation of the monoclinic form of celsian was decreased as much as possible and was equal to 800°C. This temperature was 450°C lower than for a blank sample of the celsian ceramics.

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## ОСОБЛИВОСТІ ФОРМУВАННЯ ЦЕЛЬЗІАНОВОЇ ФАЗИ ПРИ ВИПАЛЕННІ ТЕРМОСТІЙКОЇ КЕРАМІКИ В СИСТЕМІ $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

*О.В. Зайчук, Ю.Р. Калишенко, О.А. Амеліна, Ю.С. Гордєєв, С.А. Галушка, О.С. Савченко, М.В. Нагорний*

При синтезі термостійкої кераміки у системі  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  актуальним є пошук шляхів інтенсифікації процесу переходу гексагонального цельзіану в моноклінний цельзіан без підвищення температури спікання. Моноклінна форма цельзіану характеризується більш високими термічними, електроізоляційними та механічними показниками властивостей. Ця стаття присвячена вивченню особливостей формування фазового складу цельзіанової кераміки при використанні в ролі модифікуючих компонентів BAS скла евтектичного складу і LABS скла сподуменового складу. Показано, що кінцевою кристалічною фазою, яка утворюється в кераміці, синтезованій на основі вуглекислого барію і каоліну, є моноклінний цельзіан. Формування моноклінного цельзіану відбувається ступінчато. Першим утворюється гексагональний цельзіан. Повний перехід гексагональний цельзіан→моноклінний цельзіан відбувається лише у процесі високотемпературного випалу при 1250°C. При цьому ступінь спікання кераміки залишається низьким (водопоглинання становить 11,0%). Введення BAS скла сприяє повному переходу гексагонального цельзіану в моноклінний цельзіан при зниженій температурі 1100°C. Максимальний ефект у формуванні моноклінного цельзіану досягається при введенні LABS скла. Як наслідок, температура, при якій формується дана модифікація, максимально знижується і становить 800°C. В цьому випадку при температурі 1250°C досягається повне спікання цельзіанової кераміки.

**Ключові слова:** термостійка кераміка, моноклінний цельзіан, гексагональний цельзіан, кристалізація, твердофазне спікання, температурний коефіцієнт лінійного розширення.

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## FEATURES OF FORMATION OF THE CELSIAN PHASE DURING FIRING OF HEAT-RESISTANT CERAMICS IN THE SYSTEM $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$

A.V. Zaichuk <sup>a</sup>, Y.R. Kalishenko <sup>a, \*</sup>, A.A. Amelina <sup>a</sup>,  
Y.S. Hordieiev <sup>a</sup>, S.A. Halushka <sup>b</sup>, O.S. Savchenko <sup>b</sup>,  
M.V. Nahorny <sup>b</sup>

<sup>a</sup> Ukrainian State University of Chemical Technology, Dnipro, Ukraine

<sup>b</sup> Sumy National Agrarian University, Sumy, Ukraine

\* e-mail: kalishenkoyuliya@gmail.com

In the synthesis of heat-resistant ceramics in the system  $\text{BaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  (BAS), it is important to find ways of intensifying the process of transition of hexagonal celsian to monoclinic celsian without an increase in sintering temperature. Monoclinic form of celsian is characterized by higher thermal, electro-insulating, and mechanical properties. This paper deals with the features of formation of the phase composition of celsian ceramics when using BAS glass of eutectic composition and glass in the system  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{B}_2\text{O}_3-\text{SiO}_2$  (LABS) of spodumene composition as modifying components. It is shown that monoclinic celsian is the final crystalline phase formed in ceramics synthesized on the basis of barium carbonate and kaolin. Monoclinic celsian is formed stepwise; and the hexagonal celsian appears first. The complete transition hexagonal celsian→monoclinic celsian occurs only in the process of high temperature firing at 1250°C. Notably, the degree of ceramic sintering remains low (water absorption is 11.0%). Introduction of BAS glass contributes to the complete transition of hexagonal celsian to monoclinic celsian at a reduced temperature of 1100°C. Maximum effect in the formation of monoclinic celsian is achieved by the introduction of LABS glass. As a result, the temperature of formation of this modification maximally decreases to 800°C. In this case, complete sintering of celsian ceramics is achieved at the temperature of 1250°C.

**Keywords:** heat-resistant ceramics; monoclinic celsian; hexagonal celsian; crystallization; solid-phase sintering; coefficient of linear thermal expansion.

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