

To create heat-resistant structural materials capable of operating at high temperatures (up to 1,400 °C), glass crystalline materials based on the SrO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> system are promising.

This paper reports the results of studying strontium-anorthite ceramics modified with boron-containing glass of the spodumene composition. It was established that in order to achieve a set of high physical and technical indicators of ceramics at reduced firing temperatures (1,200–1,300 °C), it is necessary to introduce glass in the amount of 20–30 % by weight. In this case, densely baked materials with low TCLE values were obtained  $(32.0–33.4) \cdot 10^{-7}$  degrees<sup>-7</sup>, which predetermine their high thermal resistance (not lower than 850 °C). The principal crystalline phase of the examined ceramics is a monoclinic modification of strontium anorthite that mainly forms its microstructure. The strontium anorthite crystals measuring from 1–2 μm to 3–4 μm are tightly connected via thin layers of the residual glass phase. In the glass phase, the β-spodumene crystals the size of 0.1–0.3 μm are evenly distributed. The observed microstructure features of ceramics determine zero values of water absorption and open porosity, as well as high density values (2.40–2.50 g/cm<sup>3</sup>) and mechanical compression strength values (237–246 MPa). The dense microstructure also makes it possible to achieve high dielectric indicators ( $\epsilon=4.4–4.8$ ;  $\text{tg}\delta=0.005–0.007$ ) in an ultra-high-frequency electromagnetic field. Therefore, the designed materials are promising as radio-translucent materials, including structural ones. In addition, the enrichment of the residual glass phase with the refractory components of the SAS system in the process of firing the examined ceramics predetermines its increased resistance to high-temperature heating during operation

**Keywords:** heat-resistant ceramics, Sr-anorthite, β-spodumene, glassy phase, baking, crystallization, microstructure of ceramics

# PATTERNS IN THE SYNTHESIS PROCESSES, THE MICROSTRUCTURE AND PROPERTIES OF STRONTIUM-ANORTHITE CERAMICS MODIFIED BY GLASS OF SPODUMENE COMPOSITION

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## 1. Introduction

Among the current priority tasks in materials sciences the creation of materials for modern areas of science

and technology with a wide range of operational properties that meet the requirements of specific consumers. Improved indicators of thermal stability and mechanical strength are the most important criteria when choosing

dielectric materials exposed to high thermomechanical loads [1].

The range of heat-resistant glass-ceramic and glass-crystalline materials is quite wide. Among them, an important role is given to quartz ceramics, as well as glass-crystalline materials, which are obtained in various aluminosilicate systems.

Quartz ceramics are characterized by high heat resistance and stability of dielectric properties over a wide temperature interval [2]. At the same time, despite all the efforts aimed at strengthening quartz ceramics, it demonstrates relatively low indicators of mechanical strength [3, 4].

More promising are glass-crystalline and glass-ceramic materials obtained on the basis of various aluminosilicate systems. These materials are characterized by a set of high physical and technical indicators [5]. However, the temperature of the effective use of existing lithium aluminosilicate materials is limited to 900 °C due to the insufficient temperature stability of dielectric, thermal, and mechanical properties [6, 7]. Glass-crystalline materials with a high level of functional characteristics, designed on the basis of cordierite ( $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$ ), have an operating temperature not exceeding 1,100 °C [8].

Therefore, to achieve the high-temperature stability of glass-crystalline materials, it is advisable to use alkaline-free or weakly alkaline aluminosilicate systems, specifically  $\text{SrO}\text{-Al}_2\text{O}_3\text{-SiO}_2$  (SAS).

Materials of this system can simultaneously demonstrate low values of dielectric characteristics, high indicators of mechanical strength, chemical resistance, heat resistance, as well as resistance to high temperatures (up to 1,400 °C). It is possible to achieve the set of necessary physical-chemical indicators due to the properties of the main crystalline phase – strontium anorthite (slavsonite –  $\text{SrO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ). The melting point of the mineral slavsonite is 1,654 °C. This significantly exceeds the melting point of eucryptite and spodumene (1,380 °C), as well as cordierite (1,465 °C), on the basis of which technical sitals and glass ceramics for industrial use are synthesized [9]. Therefore, strontium-anorthite-based glass-crystalline materials can be successfully used for the manufacture of high-temperature radio-transparent products, structural materials for engineering, energy and oil and gas industry.

Given the above, it is a relevant area of research to establish the features of microstructure formation and the phase composition of strontium-anorthite ceramics related to the physical and technical indicators during low-temperature firing.

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## 2. Literature review and problem statement

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Densely baked glass-crystalline materials in the  $\text{SrO}\text{-Al}_2\text{O}_3\text{-SiO}_2$  system are mainly obtained by using two technologies: conventional glass and ceramic. Glass technology implies the directed crystallization of glass by its thermal treatment. This technique, in addition to the typical high melting temperature of starting glasses (for glasses within a SAS system, it is mainly 1,600–1,700 °C), has a series of limitations and disadvantages. Namely, strict requirements for glass in terms of its melting and molding properties, the need for strict adherence to the heat treatment regime (at product firing and crystallization) [10]. In addition, conventional glass technology imposes restrictions on the possi-

bility of varying the phase composition of materials and the complexity of the shapes of products themselves [9].

At the same time, the ceramic technology of glass-crystalline materials (powder method) is more actively developing. Powder technology of glass-crystalline materials makes it possible to significantly expand the range of compositions of starting glasses. It is possible to use glass that is not suitable for classical glass technology, in particular, highly-viscous, “short”, as well as glass with different crystallization capabilities. The ceramic technique also makes it possible to significantly expand the range of products that are manufactured, increasing the complexity of their shapes, improving the stability and reproducibility of physical-chemical properties [11].

The main issues related to the application of ceramic technology of glass-ceramic production are the high melting temperatures of starting SAS glass (1,600–1,700 °C), the difficulty of making slips with the required rheological properties, insufficient strength of cast workpieces [12]. In addition, the difference in the grain composition of the dispersed phase of ceramic slips can be the reason for obtaining products (especially large-sized) with areas of different densities. This greatly complicates ensuring the stability and reproducibility of their physicochemical characteristics. It is also important to optimize the stages of heat treatment in order to directly regulate the processes of baking and crystallization, which are mutually competing, when obtaining material with a predefined structure.

The density of glass-ceramics is a critical factor in its use. The effect of different heat treatment modes on the Sr-anorthite glass-ceramics baking intensity is described in work [13]. Using a method of isostatic pressing followed by high-temperature baking, the authors obtained a material of the stoichiometric Sr-anorthite composition with a density of 94–97 % of the theoretical composition.

At the same time, it is difficult to receive the densely baked Sr-anorthite ceramics from the mixture of  $\text{SrCO}_3$ ,  $\text{SiO}_2$ , and  $\text{Al}_2\text{O}_3$  when using conventional technology. The crystalline phase of Sr-anorthite is actively formed starting from a temperature of 1,150 °C [14]. Despite this, even a high-temperature firing at 1,350 °C over 5 hours does not make it possible to achieve high rates of the physical and technical properties of such ceramics due to its unsatisfactory baking (a water absorption rate not less than 2 %) [15].

The long-lasting mechanic activation of the raw material mixture with a stoichiometric composition consisting of  $\text{SrCO}_3$ ,  $\text{SiO}_2$ , and  $\text{Al}_2\text{O}_3$  over 12 hours enabled an almost complete binding of the starting components into a Sr-anorthite phase at a firing temperature of 1,100 °C. However, the degree of compaction and the mechanical properties of synthesized materials were also not sufficient [16].

To intensify the processes of strontium anorthite formation in low-temperature synthesis, the authors of [17] investigated the effect of mineralizers ( $\text{Li}_2\text{O}$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{SnO}_2$ ) on the characteristics of SAS ceramics baking and its phase composition. The most effective, in terms of compaction of the material with a stoichiometric Sr-anorthite composition, is  $\text{Li}_2\text{O}$ , both separately and in the composition with  $\text{SnO}_2$ . The maximum level of baking (zero water absorption) is achieved when the content of additives is 2–3 % by weight at a temperature of 1,250–1,350 °C. At the same time, the authors failed to achieve high indicators of the mechanical strength of the synthesized materials.

The densely baked ceramics with a Sr-anorthite composition was obtained through a two-stage baking process at a

temperature of 900 °C with the addition of SrO·3B<sub>2</sub>O<sub>3</sub> as a flux component. This compound stimulates the process of formation of a densely baked microstructure and the re-crystallization of grains with the help of liquid-phase baking. However, obtaining SrO·Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub> during the first stage requires a long-term high-temperature firing (1,400 °C, 4 hours) of the powder mixture of SrCO<sub>3</sub> and kaolin [18].

Activation of the process of Sr-anorthite ceramics baking is also achieved when the borosilicate glass is added [19]. It is shown that the completion of the formation of the anorthite phase and complete compaction is achieved at the content of glass of 10 % by weight at a temperature of 1,350 °C. This composition has sufficiently high values of dielectric permeability, 8.42. With the increase in the content of glass, depending on the cooling conditions, a small amount of quartz or cristobalite is formed, which adversely affects the heat resistance of ceramics.

Another difficulty in the synthesis of SAS glass-crystalline and ceramic materials is that the Sr-anorthite phase has several polymorphic modifications, which are characterized by structural features and differ in properties.

Paper [20] investigated the processes of phase-formation in glass within the SrO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> system with a stoichiometric Sr-anorthite composition. The authors' focus is that one must not allow the formation of a hexagonal modification of Sr-anorthite because it is accompanied by a phase transition into the orthorhombic modification. This form is characterized by a 3 % change in volume due to the difference in the temperature coefficient of linear expansion (TCLE). At the same time, the results of X-ray phase examinations showed that the primary crystalline phases formed were the simultaneous hexagonal and monoclinic form of Sr-anorthite. When the temperature rises (up to 1,100–1,150 °C), the hexagonal Sr-anorthite completely transforms into monoclinic, which has better physical and mechanical indicators.

Work [9] reports the results of studying the effect of the chemical composition of SAS glass on temperature intervals and the sequence of formation of crystal phases during heat treatment. It was found that depending on the molar ratio Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub> and the concentration of titanium dioxide there are changes in the nature of the crystalline phases that are initially released. Thus, in glass with a molar ratio of oxides Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub>=1:1 and the TiO<sub>2</sub> content of 15 % by weight, in the first stages of heat processing, TiO<sub>2</sub> is released in the form of rutile, and there is the simplest hexagonal form of Sr-anorthite. In the process of raising the temperature to 1,030–1,070 °C, the polymorphic transformation of hexagonal anorthite into monoclinic modification is observed, as well as the crystallization of monoclinic anorthite from the glass phase. For glass whose molar ratio of oxides is Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub>>1, there is a formation, as the primary crystalline phase, of tialite (Al<sub>2</sub>O<sub>3</sub>·TiO<sub>2</sub>). Subsequently, monoclinic anorthite is crystallized, thereby omitting the stage of forming a hexagonal shape.

At higher temperatures, there is a formation of the monoclinic form of Sr-anorthite when using a sol-gel method [21]. The release of the monoclinic phase in the dominant amount occurs in the region of temperatures 1,250–1,300 °C. TCLE of such ceramics is 45·10<sup>-7</sup> degrees<sup>-1</sup>, and the strength limit at bending reaches 80 MPa after a heat treatment at a temperature of 1,350 °C. Even higher temperatures and the duration of baking are necessary at the conventional solid-phase synthesis.

Summarizing the above, when creating high-temperature-resistant glass-crystalline materials, it is advisable to use

the SrO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> system. Existing methods of achieving a high density of materials and dominance of the stable phase of monoclinic strontium anorthite are mainly based on high-temperature heat treatment, or do not make it possible to obtain a set of high physical and technical indicators. Therefore, it is necessary to devise a more perfect technological procedure for designing densely baked glass-crystalline materials with a Sr-anorthite composition. The technique implies the targeted regulation of the microstructure and phase composition of ceramics by introducing lithium aluminoborosilicate glass with aspodumene composition into the basic matrix whose role is to be performed by strontium anorthite. This approach was fully justified when obtaining densely baked cordierite ceramics. It is shown in [22] that the introduction of fusible lithium aluminoborosilicate glass significantly accelerates the progress of solid-phase reactions towards forming the crystalline phase of α-cordierite and predetermines the formation of a densely baked structure of ceramics. The fusibility of the experimental glass is ensured by the introduction of boron oxide into the system (10 mass fractions over more than 100 % by weight). Boron oxide contributes to the formation of a silicate glass melt at a relatively low temperature and to a decrease in its viscosity, as well as to improving the wetting ability of glass relative to the crystalline phase [23]. In addition, the composition of LABS glass is near eutectics with a temperature of 1,260 °C [24]. These two factors predetermine a decrease in the melting temperature of the experimental LABS glass to 1,350 °C compared to the LAS glass with a stoichiometric aspodumene composition (1,500–1,550 °C). Introducing such glass should ensure the production of densely baked strontium-anorthite ceramics with a set of high physical and technical indicators.

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### 3. The aim and objectives of the study

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The aim of this study is to develop technological aspects of obtaining densely baked strontium-anorthite ceramics with the addition of glass of aspodumene composition, as well as to investigate the physicochemical patterns in the formation of its microstructure and phase composition. This could greatly simplify the technology of glass-crystalline materials at the stage of preparing ceramic slips and production of semi-finished products, reduce energy consumption during heat treatment, control their microstructure and phase composition. In addition, a set of high physical and technical indicators of glass-crystalline materials would be ensured that could provide for a long period of effective operation.

To accomplish the aim, the following tasks have been set:

- to determine the physical and technical indicators of strontium-anorthite ceramics when adding the glass of aspodumene composition;
- to establish patterns in the formation of the phase composition and microstructure of strontium-anorthite ceramics related to the physical and technical indicators that determine its properties.

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### 4. Materials and methods to study strontium-anorthite ceramics

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The raw materials used to obtain strontium-anorthite ceramics included enriched kaolin, brand zref-1, and carbon dioxide strontium.

Fire clay, which was obtained by pre-baking kaolin of brand zref<sup>1</sup> at 1,150 °C, was used as leaner in the formulation of the examined compositions.

The composition of the base glass was selected in the oxide 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) and corresponded to the stoichiometric spodumene. It was melted in an electric furnace at a temperature of 1,350 °C for 1 hour. LABS glass is characterized by TCL Eequal to  $60.7 \cdot 10^{-7}$  degrees<sup>-1</sup> [25].

Starting components were used to prepare ceramic slips in a ball mill by the method of joint wet grinding until full passage through sieve No. 0063. After the aging process, the slips with a humidity of 28–30 % were used to pour samples into gypsum molds shaped as cylinders, square and cylindrical billets. After pulling the billets from the molds, they were dried to a residual humidity of 1 %. Dried samples were baked in an electric furnace in the air environment according to the specified temperature and time regime. Aging at a maximum temperature of 1,200–1,300 °C lasted for 1 hour.

Our experimental study was performed according to the standard procedures for determining the properties of ceramic materials.

Water absorption (*W*), apparent porosity (*P*), and imaginary density (*ρ*) of the samples were measured by saturation and subsequent weighing in the air and water.

The compression strength limit ( $\sigma_{st}$ ) was determined on cylindrical samples ( $d=h=10$  mm) using a hydraulic press.

Ceramic samples the size of 5×5×50 mm were used to measure the relative lengthening of ceramic samples ( $\Delta l$ ). The data obtained were used to calculate the average TCLE value in the range of 20–400 °C at a heating rate of 10 °C/min.

The mineralogical composition of the experimental ceramics was determined by X-ray phase analysis (XPA) at the Philips APD-15 diffractometer in Co-K $\alpha$  radiation.

Electron-microscopic studies of ceramic samples at rupture were conducted at the scanning electron microscope SEO-SEM Inspect S50-B. The elemental analysis of the glass phase in the local areas of these samples was performed by X-ray energy dispersion spectroscopy based on the magnitudes of energies in the characteristic X-ray peaks of each chemical element. The device SEO-SEM Inspect S50-B with a beryllium window can effectively determine elements with the atomic number  $Z > 5$ .

Heat resistance was determined on the basis of the maximum temperature difference, *K*, which the samples can withstand before showing the signs of damage.

The measurement of dielectric permeability ( $\epsilon$ ) and the tangent of the angle of dielectric losses ( $\text{tg}\delta$ ) was carried out at a measuring unit consisting of the generator G4-83, the spectrum analyzer C4-11, and a biconical resonator. The resonator was connected according to the scheme on the pass. The measurements were carried out at a frequency of  $10^{10}$  Hz at a temperature of 20 °C [26].

## 5. Results of studying strontium-anorthite ceramics

### 5.1. Results of studying the physical and technical indicators of strontium-anorthite ceramics with the addition of glass of the spodumene composition

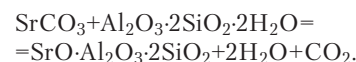
To obtain densely baked strontium-anorthite ceramics and achieve a set of high physical and technical indicators,

we additionally introduced LABS glass with aspodumene composition to its charge formulation.

The role of glass is to intensify the processes of solid-phase baking in order to form a dense microstructure, as well as the predefined mineralogical and phase composition of experimental ceramics. It should also ensure the finely dispersed crystallization of the  $\beta$ -spodumene phase, which is characterized by low thermal expansion ( $9 \cdot 10^{-7}$  degrees<sup>-1</sup>). The spodumene phase could help reduce TCLE for materials based on strontium-anorthite ceramics. During firing, the residual glass phase will be enriched with refractory components of the base SAS system, causing an increase in the resistance of the designed ceramics to high-temperature heating.

Given the need to achieve high dielectric indicators of ceramics, it is important to note the fact [27] that heavy cations, in particular Ba<sup>2+</sup> and Sr<sup>2+</sup>, are able to block the mobility of alkaline cations. Therefore, the presence of strontium oxide in the system could increase the resistance of glass and improve the dielectric properties of experimental ceramics. Boron oxide introduced to the glass formulation also increases their electrical strength [27]. Thus, the addition of LABS glass to SAS of the ceramic matrix should not significantly affect the dielectric properties of Sr-anorthite ceramics. The content of LABS glass in the formulation of the examined compositions S-1, S-2, and S-3 was 10, 20, and 30 % by weight, respectively.

In order to obtain a SAS matrix of the ceramic material, we used barium carbon dioxide and enriched kaolin in a stoichiometric ratio. The resulting process of its formation can be represented by the following equation:



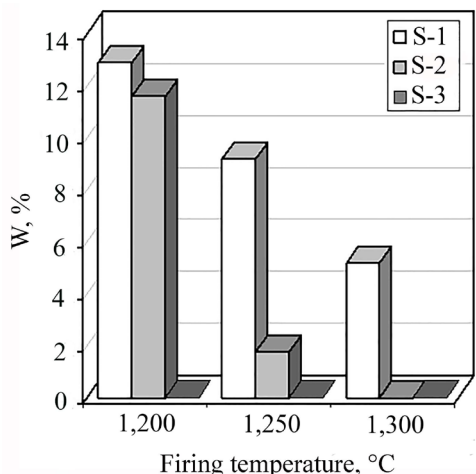
The presence of kaolin makes it quite easy to manage the rheological-technological properties of water slips and receive (after mold disassembly) billets with a strength enough for further technological stages.

To reduce the susceptibility to deformation and crack formation, kaolin was partially replaced with fire clay. Fire clay was introduced to the formulation of the examined charge in the amount of up to 25 % by weight; it replaced the appropriate amount of kaolin in terms of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>.

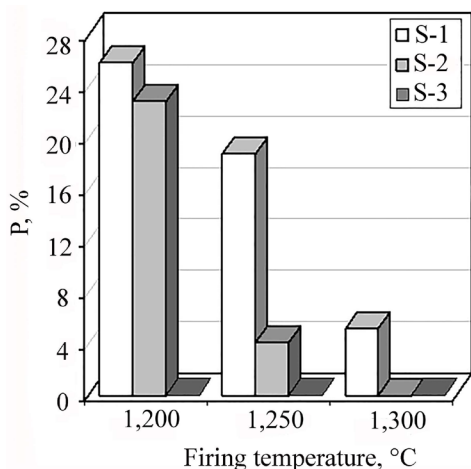
A very important technological stage of obtaining glass-crystalline materials is the process of their heat treatment. Its purpose is to ensure the predefined microstructure of the material through the targeted crystallization and subsequent baking. Moreover, the processes of crystallization and compaction are competing and can progress both sequentially and simultaneously. Therefore, it is necessary to establish the most rational parameters of heat processing, which would ensure the formation of the predefined mineralogical composition and microstructure of SAS ceramics.

Taking data on the differential and thermal studies of the original LABS glass into consideration [25], the stepped firing of experimental ceramic materials was carried out. In the first stage, at a nucleation temperature of 600 °C and a crystallization temperature of 760 °C of the original glass with isothermal aging for 2 hours. Subsequently, the maximum firing temperature reached 1,200–1,300 °C with aging over 1 hour.

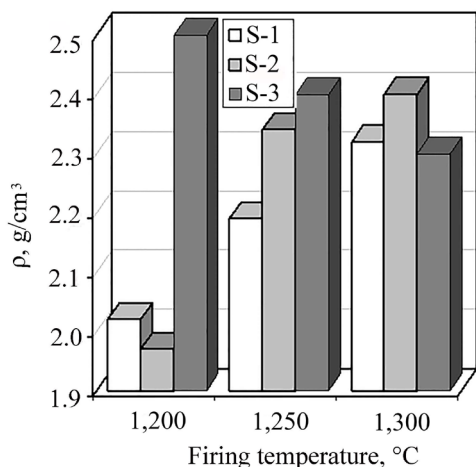
The results from measuring the physical and technical properties of experimental ceramics in the form of graphical dependences are shown in Fig. 1.



a



b

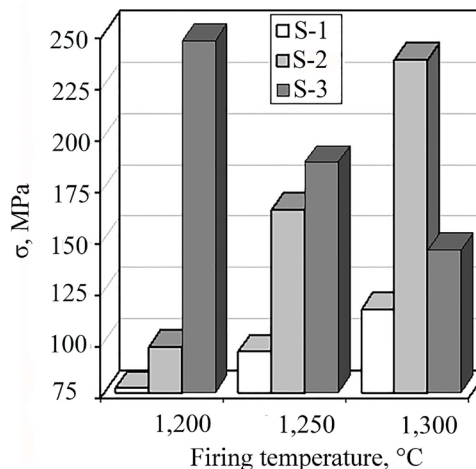


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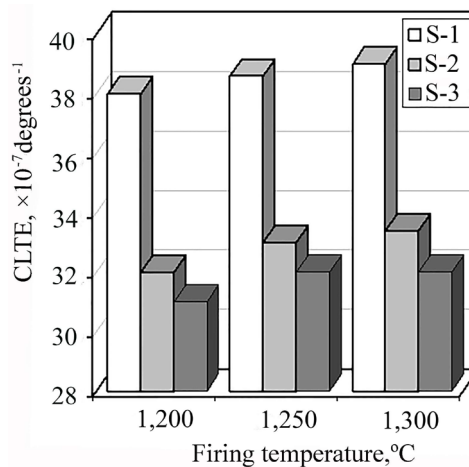
Fig. 1. Dependence of the physical-technical indicators of strontium-anorthite ceramics on the content of LABS glass and firing temperature: a – W, %; b – P, %; c –  $\rho$ , g/cm<sup>3</sup>

We have experimentally established that the LABS glass with aspodumene composition exerts a mineralization effect on the components of strontium-anorthite ceramics. The action effectiveness is enhanced with an increase in the content of glass from 10 % by weight up to 30 % by weight and in the firing temperature from 1,200 °C to 1,300 °C.

Considering the results of studying the physical and technical properties of experimental ceramics with the composition S-1, it is necessary to note that the introduction of glass in the amount of 10 % by weight is insufficient because it does not provide for the required degree of baking even at a maximum temperature of 1,300 °C. This is evidenced by the high water-absorption values (5.2 %) and apparent porosity (12 %) of the material. The imaginary density does not exceed 2.30 g/cm<sup>3</sup> while compression strength is 115 MPa. TCLE is characterized by the maximum value in a series of the considered compositions (S-1–S-3) and is (38–39)·10<sup>-7</sup> degrees<sup>-1</sup> (Fig. 2). However, even such values are much lower than the TCLE of pure strontium-anorthite ceramics, which is 45·10<sup>-7</sup> degrees<sup>-1</sup> [21].



a



b

Fig. 2. Effect of the LABS glass content and firing temperature on: a – limit of compression strength; b – TCLE of strontium-anorthite ceramics

Glass in the amount of 20 % by weight, added to the Sr-anorthite matrix (composition S-2), makes it possible to achieve a set of the highest physical and technical indicators at a baking temperature of 1,300 °C. This ceramic is characterized by zero values of water absorption and apparent porosity, as well as high values of  $\rho$  (2.40 g/cm<sup>3</sup>) and  $\sigma_{st}$  (237 MPa). TCLE in the range of 20–400 °C is  $33.4 \cdot 10^{-7}$  degrees<sup>-1</sup>.

The introduction of experimental glass in the amount of 30 % by weight (composition S-3) makes it possible to obtain densely baked ceramics at a temperature of 1,200 °C. Increasing the firing temperature of such a composition to 1,250–1,300 °C causes some reduction in material density, from 2.50 g/cm<sup>3</sup> to 2.40–2.30 g/cm<sup>3</sup>. In addition, there is a significant decrease in the indicator of mechanical compression strength, from a maximum value of 246 MPa to 144–187 MPa.

Taking this fact into consideration, increasing the firing temperature of strontium-anorthite ceramics of S-3 composition over 1,200 °C is inappropriate.

The comparison of TCLE of ceramic compositions S-2 and S-3 indicates that the increase in the content of the spodumene LABS glass from 20 % by weight to 30 % by weight has almost no effect on the value of this indicator. Thus, the low values of thermal expansion coefficient ( $32.0$ – $33.4$ ) $\cdot 10^{-7}$  degrees<sup>-7</sup> are achieved already when adding starting glass in the amount of 20 % by weight. This is 26–30 % lower than the TCLE value of conventional strontium-anorthite glass-crystalline materials.

For ceramic samples, characterized by the highest physical and technical indicators, we studied dielectric properties, as well as determined thermal stability. It was established that the ceramic compositions S-2 and S-3, baked at a temperature of 1,300 °C and 1,200 °C, respectively, has a low dielectric permeability  $\epsilon=4.4$ – $4.8$ . In addition, the experimental ceramics have low  $\tan \delta$  values, which are at 0.005–0.007, and its heat resistance is 850 °C.

## 5. 2. Results of studying the phase composition and microstructure of strontium-anorthite ceramics with the addition of LABS glass

The results of X-ray phase analysis showed that the mineralogical composition of the synthesized materials is mainly represented by strontium anorthite, which crystallizes in the monoclinic system (Fig. 3). With an increase in the firing temperature of the experimental compositions from 1,200 °C to 1300 °C, the intensity of the main diffraction peaks of this compound ( $d \cdot 10^{10}=6.39$ ; 4.51; 3.72; 3.23; 2.95; 2.73; 2.53; 1.78 m) increases. Strengthening the diffraction pattern for experimental compositions with a rising synthesis temperature is due to the formation of the Sr-anorthite phase with a more perfect structure and the growth in the size of crystals. X-ray phase analysis data clearly correlate with the results from electron microscopic studies (Fig. 4).

Thus, compositions of S-1 and S-2 formulations, fired at a temperature of 1,200 °C, are characterized by a fine crystalline structure (Fig. 4, a, c). The main crystalline phase (strontium anorthite) is represented by irregularly shaped grains that are no larger than 2  $\mu\text{m}$ . Strontium anorthite crystals are connected to each other through a vitreous phase. Also clearly distinguished are the evenly distributed small crystals of  $\beta$ -spodumene with a spherical shape. The  $\beta$ -spodumene phase is a product of crystallization of the original LABS glass. The size of

this crystalline phase does not exceed 0.5  $\mu\text{m}$ . It should be noted that the microstructure of such materials is characterized by a sufficiently large number of pores measuring mainly 3–6  $\mu\text{m}$ . This determines the low density ( $v=1.97$ – $2.02$  g/cm<sup>3</sup>) and mechanical strength ( $\sigma_{st}=77$ – $97$  MPa) indicators.

Increasing the firing temperature of experimental ceramics to 1,300 °C, and the content of LABS glass in its composition to 20 % by weight, causes the formation of dense microstructure. It consists of explicitly formed flat prismatic strontium anorthite crystals (Fig. 4, b, d), mainly of hexagonal shape. Those crystals dominate whose longitudinal size is within 3–5  $\mu\text{m}$  while their thickness does not exceed 1  $\mu\text{m}$ .

Increasing the LABS content in glass also significantly increases the intensity of the main lines corresponding to  $\beta$ -spodumene ( $d \cdot 10^{10}=5.82$ ; 3.90; 3.47; 1.33; 1.27 m), for ceramics fired at a temperature of 1,200 °C. The increase in the amount of  $\beta$ -spodumene explains the displacement of its main line ( $d \cdot 10^{10}=3.42$  m) in the radiograph of composition S-1 towards larger inter-plane distances ( $d \cdot 10^{10}=3.47$  m) for the composition of formulation S-3. The crystalline phase of  $\beta$ -spodumene is characterized by the main diffraction maximum at the level of  $d \cdot 10^{10}=3.49$  m.

The increase in the content of  $\beta$ -spodumene is manifested in the growth of the number of small spherical crystals in electronic images when moving from composition S-1 (Fig. 4, a) to composition S-3 (Fig. 4, e). These crystals are 0.1–0.3  $\mu\text{m}$  in size and are evenly distributed throughout volume.

Increasing the content of LABS in glass to 30 % by weight makes it possible to obtain a dense small-crystalline structure of ceramics already at a temperature of 1,200 °C. At the same time, the strontium anorthite crystals that are formed are flat and have a rounded shape. Their size is mainly 1–2  $\mu\text{m}$ .

The increase in the firing temperature of composition S-3 to 1,300 °C causes the growth of the size of strontium anorthite crystals to 3–4  $\mu\text{m}$  and their clearer design (Fig. 4, e). The crystals have a mostly hexagonal shape. At the same time, there is a partial melting of strontium anorthite crystals, expressed in the rounded shape of their corners. This process is activated by increasing the activity of residual glass melt when the firing temperature rises to 1,300 °C.

Simultaneously with the increase in the size of crystals, there is a decrease in the density of their arrangement in the structure of the material. In addition, the content of  $\beta$ -spodumene is significantly reduced, obviously due to its dissolution. This change in the microstructure and phase composition of ceramics leads to a significant deterioration in the indicator of mechanical compression strength (reduced from 246 MPa to 144 MPa).

For ceramic samples S-3, we carried out an elementary analysis of the residual glass phase. The results showed (Fig. 5) that the glass phase after firing the ceramics is enriched with strontium oxide. The content of this oxide increases from 12.35 % by weight to 18.05 % by weight when the firing temperature rises from 1,200 °C to 1,300 °C. This fact is a confirmation that in the process of baking experimental ceramics there is a change in the composition of the glass phase due to the partial dissolution of the crystalline phase in it, in particular strontium anorthite. Enriching the residual glass phase,

first of all, with strontium oxide increases its viscosity and, as a consequence, improves the resistance of ceramics to high-temperature heating during operation. Boron and

lithium, which are obviously present as part of the residual glass phase, are not registered, due to the small charge of the nucleus of their atoms.

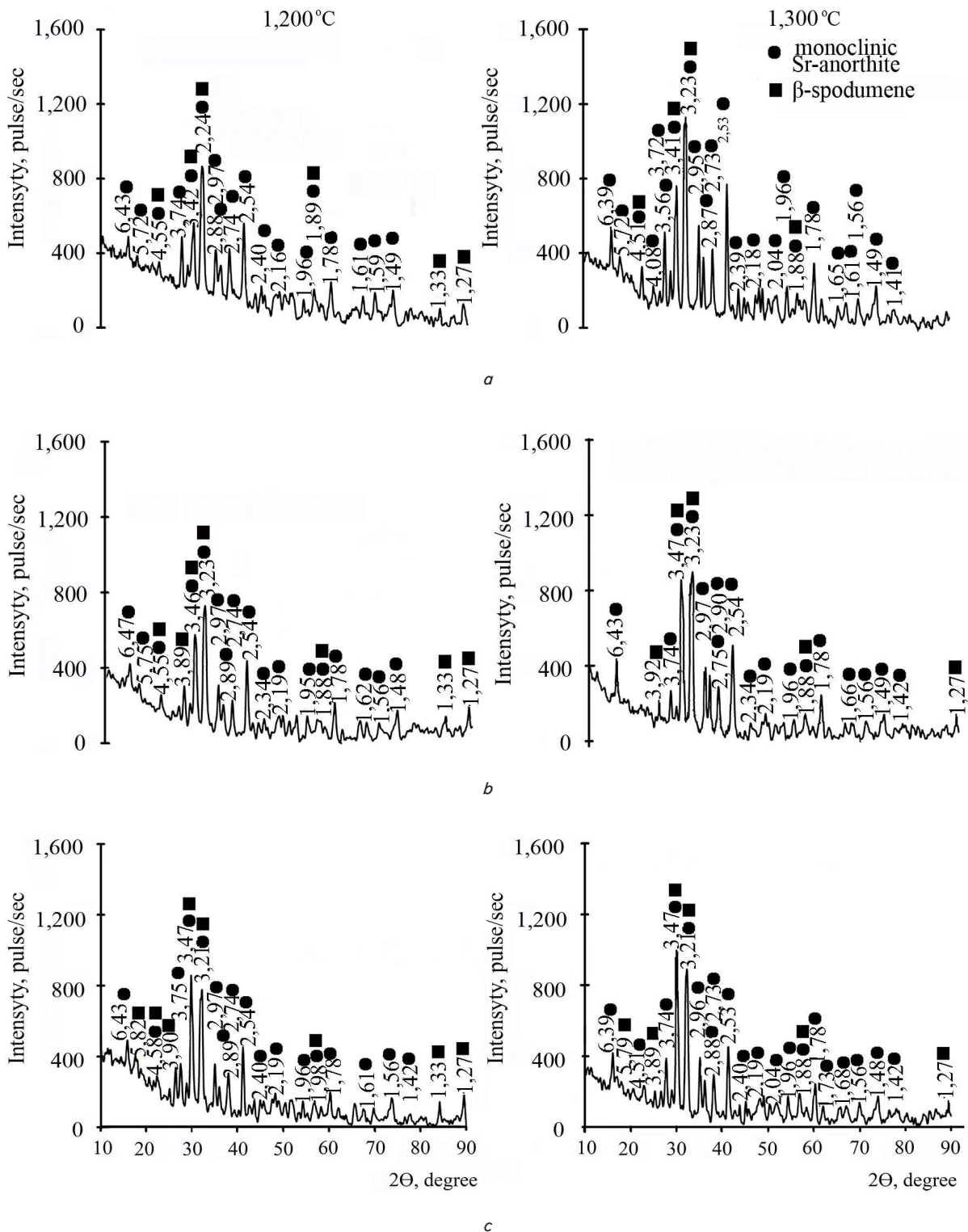


Fig. 3. Radiographs of experimental strontium-anorthite ceramics samples (a – S-1; b – S-2; c – S-3), fired at different temperatures

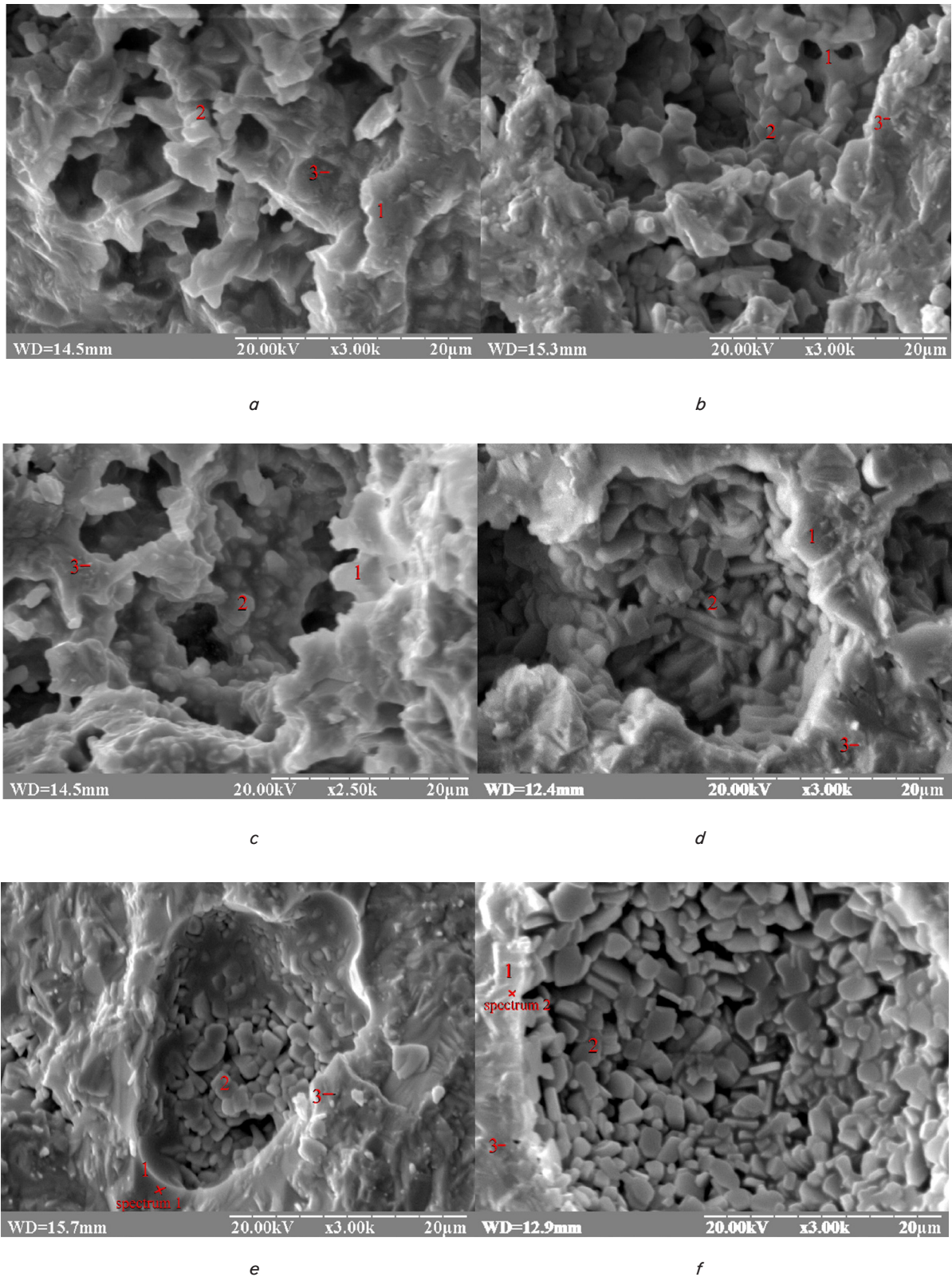
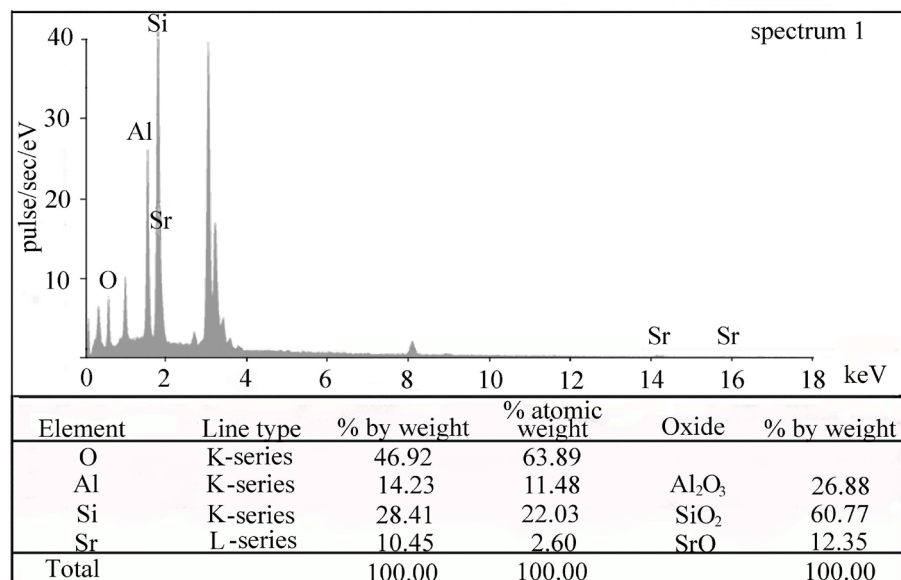


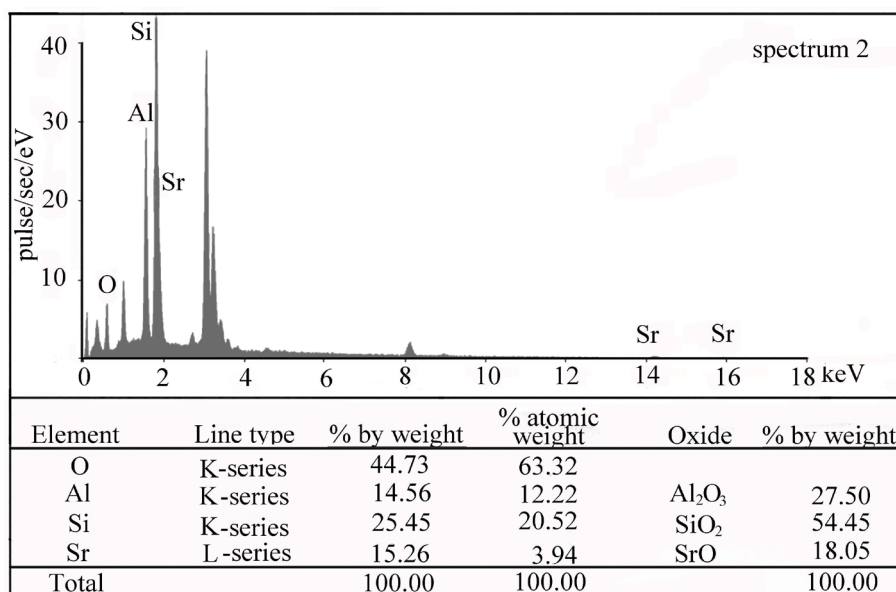
Fig. 4. Scanning electron microphotographs of experimental strontium-anorthite ceramics samples fired at different temperatures: *a* – S-1(1,200 °C); *b* – S-1(1,300 °C); *c* – S-2(1,200 °C); *d* – S-2(1,300 °C); *e* – S-3(1,200 °C); *f* – S-3(1,300 °C); 1 – glass phase; 2 – Sr-anorthite crystals; 3 – crystals of  $\beta$ - spodumene

Note: \*in Fig. 4, e, f, a cross indicates the local area of the glass phase whose element composition was studied by X-ray energy dispersion spectroscopy; the results are shown in Fig. 5





a



b

Fig. 5. Spectra of X-ray energy dispersion analysis of the residual glass phase of strontium-anorthite ceramics of S-3 composition fired at temperatures: *a* – 1,200 °C; *b* – 1,300 °C

## 6. Discussion of results of studying the microstructure and properties of strontium-anorthite ceramics modified by the glass of the spodumene composition

In this work, in order to obtain densely baked Sr-anorthite ceramics at a relatively low firing temperature, we examined the spodumene LABS glass as a mineralizing additive. Our study found that the physical-technical indicators of experimental ceramics, its quantitative mineralogical composition, as well as microstructure, are determined by the content of LABS glass and the temperature of firing. Increasing the concentration of the starting glass from 10 to 30 % by weight, as well as the firing temperature from 1,200 °C to 1,300 °C, significantly enhances the process-

es of baking ceramics. Moreover, in all cases, a monoclinic modification of strontium anorthite is formed, which is more beneficial in terms of achieving the set of the necessary physical and technical indicators (Fig. 3). This crystalline phase does form the principal matrix of experimental ceramics.

It is important to note that the thermophysical properties (TCLE) of ceramic materials are directly affected only by the content of LABS glass, the increase of which causes the quantitative growth of the crystalline phase of  $\beta$ -spodumene. This crystalline phase is a product of glass crystallization and is characterized by low thermal expansion. Effective action of  $\beta$ -spodumene on a decrease in the TCLE of strontium-anorthite ceramics to  $(32-33.4) \cdot 10^{-7}$  degrees<sup>-7</sup> is achieved already with the introduction of glass in the amount of 20 % by weight (Fig. 2, *b*). This is due to the finely-dispersed crystallization of the  $\beta$ -spodumene phase (the size of crystals is 0.1–0.3  $\mu$ m) and its uniform distribution in the structure of the material.

The physical and mechanical properties of the experimental ceramics are influenced by both the content of LABS glass and the temperature of firing. When introducing glass in the amount of 10 % by weight, there is a small-crystalline but porous microstructure of the material due to the insufficient glass phase content in the system. Increasing the concentration of starting glass to 20 % by weight, as well as the firing temperature to 1,300 °C, contributes to the formation of dense microstructure. This microstructure is formed by a close combination of the phase of strontium anorthite in the form of clearly formed hexagonal crystals and the glass phase (Fig. 4, *d*). Consequently, ceramics are characterized by zero indicators of water absorption and apparent porosity (Fig. 1, *a, b*), as well as high values of mechanical compression strength, 237 MPa (Fig. 2, *a*). Increasing the content of LABS glass to 30 % by weight can achieve an intensive baking strontium-anorthite ceramics by reducing the firing temperature to 1,200 °C. In this case, small structurally oriented crystals of strontium anorthite are formed, measuring 1–2  $\mu$ m, densely connected to each other by the thin layers of the glass phase (Fig. 4, *d*). It is this microstructure that causes the maximum value of the mechanical strength of experimental ceramics ( $\sigma_{st}=246$  MPa) – Fig. 2, *a*. The increased hardness and strength with a decrease in grain size to some critical

value is typical of almost all crystals [28]. The smaller the size of the crystals, the more often in the path of sliding dislocations there are obstacles at the grain boundary and, accordingly, the higher the stresses required for deformation of the material in the initial stages. In addition, the dense location of crystals of the main phase of strontium anorthite in the structure of the material brings the density of ceramics closer to theoretical, also increasing its strength indicators.

The dense structure of the S-3 composition formulation makes it possible to achieve high dielectric indicators ( $\epsilon=4.8$ ,  $\text{tg}\delta=0.007$ ) in an ultra-high-frequency electromagnetic field. Therefore, the materials that are being designed are promising as radio-translucent materials, including structural ones.

Increasing the firing temperature of composition S-3 to 1,300 °C causes the growth of the size of strontium anorthite crystals to 3–4  $\mu\text{m}$  and their clearer design. At the same time, with rising temperatures and, consequently, a decrease in the viscosity of the glass melt, the processes associated with the dissolution of the crystalline phase are activated. In particular, the content of  $\beta$ -spodumene is significantly reduced while strontium anorthite crystals are characterized by melting, as evidenced by their shape (Fig. 4, e). There is also a decrease in the density of the arrangement of strontium anorthite crystals in the structure of ceramics, which is the reason for reducing the strength of the material to 144.3 MPa (Fig. 2, a).

It should also be stated that during the firing of experimental ceramics, the composition of the glass phase undergoes significant changes. In particular, it is enriched with the refractory components of the base SAS system (Fig. 5), which leads to an increase in the resistance of ceramics to high-temperature heating. This is confirmed by the absence of signs of deformation and structure defects on the samples of experimental ceramics S-2 and S-3, fired repeatedly at 1,400 °C.

Thus, our technological technique implying the introduction of LABS glass to the SAS ceramics contributes to the significant intensification of the process of forming a monoclinic form of Sr-anorthite, as well as the baking of the resulting materials. This makes it possible to significantly reduce the energy costs of the process of making ceramic materials and effectively manage their microstructure and phase composition. As a result, at reduced temperatures of 1,200–1,300 °C, we synthesized Sr-anorthite ceramics, which is characterized by a set of high physical and technical indicators (zero water absorption, high mechanical compression strength, 237–246 MPa). The introduction of glass also causes a decrease in TCLE to  $(31.0\text{--}33.4)\cdot 10^{-7}$  degrees<sup>-7</sup>, causing the high thermal stability of ceramics (not lower than 850 °C). The dense microstructure and specified phase composition of experimental ceramics make it possible to achieve high dielectric indicators ( $\epsilon=4.8$ ,  $\text{tg}\delta=0.007$ ) and use it as high-temperature radio-translucent materials, including structural ones.

The devised compositions of strontium-anorthite ceramics make it possible to obtain articles of different shape complexity using all the basic molding methods, which are conventionally employed in ceramic technology. A wet technique for preparing ceramic mass provides for its high degree of homogenization and, consequently, good reproducibility of the properties of fired ceramics. At the same time, in order to obtain strontium-anorthite ceramics with a set of high physical and technical indicators, it is necessary, first of all, to strictly adhere to the temperature and time mode of firing and the optimal content of LABS glass.

In addition, further study should investigate the conditions for the formation of a small-crystalline structure of ceramics and ways to reduce the size of the phase of Sr-anorthite, which forms the main matrix of the material. Obtaining a fine-grained structure with a size of Sr-anorthite crystals less than 1  $\mu\text{m}$  is a significant reserve for improving the mechanical strength of the experimental ceramics.

This approach, which is well established in the synthesis of densely baked aluminosilicate ceramics, can in the future be implemented in the technology of other types of heat-resistant ceramics, provided that their specific features are taken into consideration.

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## 7. Conclusions

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1. Our study has defined patterns of change in the physical and technical indicators of strontium-anorthite ceramics depending on the content of glass with aspodumene composition and the temperature of firing. It was established that low TCLE values  $(32.0\text{--}33.4)\cdot 10^{-7}$  degrees<sup>-7</sup> are already achieved when adding original glass in the amount of 20 % by weight. This is 26–30 % lower than the TCLE value for conventional strontium-anorthite glass crystalline materials and provides high thermal stability of experimental ceramics (not lower than 850 °C). The densely baked strontium-anorthite ceramics were obtained by introducing glass in the amount of 20–30 % by weight and at firing in a temperature interval of 1,200–1,300 °C. Such ceramics are characterized by zero values of water absorption and apparent porosity, as well as high values of  $\rho$  (2.40–2.50 g/cm<sup>3</sup>) and  $\sigma_{st}$  (237–246 MPa). The dense structure of the experimental ceramics and its composition makes it possible to achieve high dielectric indicators ( $\epsilon=4.4\text{--}4.8$ ;  $\text{tg}\delta=0.005\text{--}0.007$ ) in an ultra-high-frequency electromagnetic field. Therefore, the materials that are being designed are promising as high-temperature radio-translucent materials, including structural ones.

2. The influence of the technological parameters of making strontium-anorthite ceramics on its microstructure and phase composition has been investigated in relation to their physical and technical indicators. It is determined that the main matrix of experimental ceramics is formed by a monoclinic modification of strontium anorthite. Based on data from microscopic studies, it is shown that increasing the synthesis temperature to 1,300 °C causes the formation of the Sr-anorthite phase with a more advanced structure and the growth in the size of crystals to 3–5  $\mu\text{m}$ . The increase in the content of LABS glass significantly increases the content of the crystalline phase of  $\beta$ -spodumene, which is the product of crystallization of such glass. These spherical crystals are 0.1–0.3  $\mu\text{m}$  in size and are evenly distributed throughout the volume, making it possible to significantly reduce the TCLE of strontium-anorthite ceramics. The densest and most durable microstructure of ceramics is demonstrated when the content of LABS glass is 30 % by weight and the firing temperature is 1,200 °C. This microstructure is formed by the crystals of strontium anorthite the size of 1–2  $\mu\text{m}$ , which are densely connected to each other through the thin layers of the residual glass phase. The vitreous phase hosts the small crystals of  $\beta$ -spodumene, evenly distributed. In addition, enriching the residual glass phase with the refractory components of SAS system predetermines an increase in the resistance of ceramics to high-temperature heating.

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*This paper proposes a technique to prevent the corrosion of steel reinforcement in concrete based on slag cement (SC) activated by Na(K) salts of strong acids (SSA) in the composition of by-pass cement kiln dust (BP). The technique implies using additional modifiers in the form of the Portland cement CEM I 42,5 R and the calcium-aluminate admixture (CAA)  $C_3A \cdot 6H_2O$ .*

*It is shown that adding the Portland cement contributes to enhancing the intensifying influence of BP on the SC hydration, accompanied by an increase in the strength of artificial stone. This effect is predetermined by the formation of hydrosilicates in hydration products with an increased crystallization degree in the form of CSH(I) and  $C_2SH(A)$ .*

*Modifying SC with CAA ensures the intensive formation of low-soluble AFm phases in the composition of hydration products, aimed at reliable binding the SSA anions ( $Cl^-$ ,  $SO_4^{2-}$ ) that are aggressive to steel reinforcement.*

*The study result has established the possibility to produce SC, activated by SSA, when using BP, the Portland cement, and CAA. Mathematical methods to plan the experiment were applied to produce an SC composition of "granulated blast furnace slag – BP – Portland cement – CAA", characterized by a strength class of 42.5 and a molar ratio of  $Cl^-/OH^-$  in a porous solution not exceeding 0.6. The resulting properties predetermine the feasibility of using SC in steel-reinforced concrete.*

*The relevance of this work is due to the modern trends in the development of the construction industry. The introduction of cement that contains mineral additives, in particular granulated blast furnace slag, contributes to improving the environment by reducing  $CO_2$  emission. The use of such cement as a base of steel-reinforced concrete ensures the increase in their functionality and durability*

**Keywords:** slag cement, steel reinforcement, cement kiln dust, AFm phase, structure formation

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# DESIGN OF SLAG CEMENT, ACTIVATED BY Na (K) SALTS OF STRONG ACIDS, FOR CONCRETE REINFORCED WITH STEEL FITTINGS

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## 1. Introduction

Current trends in the development of the construction industry predetermine the relevance of introducing the so-called "green" materials whose production involves resource- and energy-saving technologies [1] implying a responsible attitude towards the environment [2]. The types of cement containing mineral additives of artificial and natural origin fully match the trends of sustainable development of mankind [3]. As regards the environmental aspect, replacing part of the clinker in cement composition with mineral additives contributes to reduc-

ing the emission of  $CO_2$  [4]. At the same time, materials based on such types of cement are characterized by high quality, functionality, and durability. Thus, paper [5] shows the effectiveness of the use of multicomponent types of cement based on slag, zeolite, and fly ash in mortars by a high early strength [5]. The positive effect on the strength of concrete under various humid conditions of operation is exerted by admixtures of polydisperse zeolite tuff and perlite [6]. Applying admixtures in the form of zeolite or highly dispersed chalk in cement composition leads to an increase in strength indicators [7], crack resistance [8], and frost resistance of concrete [9].