Досліджено вплив комплексних інтенсифікаторів спікання з числа фторидів лужних металів, а також поєднання оксидів стануму та літію на спікання стронцієвої кераміки. Показано позитивний вплив добавки SnO_2–Li_2CO_3 на низькотемпературну активацію процесу синтезу стронцієвого анортиту і отримання щільноспеченого керамічного матеріалу. Встановлено, що за своїми діелектричними властивостями отриманий керамічний матеріал може бути віднесений до радіопрозорих матеріалів

Ключові слова: стронцієвий анортит, інтенсифікатор спікання, радіопрозора кераміка, евтектика, діелектрична проникність

Исследовано влияние комплексных интенсификаторов спекания из числа фторидов щелочных металлов, а также сочетание оксидов олова и лития на спекание стронциевой керамики. Показано положительное влияние добавки SnO_2–Li_2CO_3 на низкотемпературную активацию процесса синтеза стронциевого анортита и получение плотноспеченого керамического материала. Установлено, что по своим диэлектрическим свойствам полученный керамический материал может быть отнесен к радиопрозрачным материалам

Ключевые слова: стронциевый анортит, интенсификатор спекания, радиопрозрачная керамика, эвтектика, диэлектрическая проницаемость

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INFLUENCE OF COMPLEX ACTIVATORS OF SINTERING ON CREATING RADIOTRANSPARENT CERAMICS IN $SrO–Al_2O_3–SiO_2$

G. Lisachuk
Doctor of Technical Sciences, Professor*
E-mail: lisachuk@kpi.kharkov.ua
R. Kryvobok
PhD, Senior Research*
E-mail: krivobok_ruslan@ukr.net
A. Zakharov
Junior Researcher*
E-mail: zakharovartem106@gmail.com
V. Tsovma
PhD, Chief Technologist of Construction Materials*
Pology Chemical Plant Coagulant ALC
Lesi Ukrainki str., 243, Pology,
Zaporizhia region, Ukraine, 70605
E-mail: vitalii.cvv@gmail.com
O. Lapuzina
PhD, Professor*
E-mail: elapuzina@gmail.com
*Department of engineering ceramics, refractories, glass and enamel
National Technical University
«Kharkiv Polytechnic Institute»
Kyrpychova str., 2, Kharkiv, Ukraine, 61002

1. Introduction

Ceramics in terms of performance characteristics can be described a versatile material, and the aerospace industry is no exception. For receiving and transmitting antennas placed onboard, radiotransparent ceramic materials are essential components due to a number of properties that can be reproduced by neither metal-based nor organic substances-based materials. The main requirements for radiotransparent materials are: resistance to heat shock, high heat capacity and thermal conductivity, high strength over a wide temperature range, significant impact strength, low density (a weight reduction factor) and radiotransparency maximization by minimizing losses in reflection and absorption when interacting with electromagnetic waves of a high frequency range [1].

For radiotransparent ceramic materials, it is typical to have low dielectric losses ($tg\delta<0.001$) and high stability of properties under temperature changes. For example, the dielectric permittivity of glass ceramics does not change by more than ±1 %, and the dissipation factor does not change by more than ±20 % at a temperature change from – 60 °C to +1,200 °C [2].

One of the crystalline phases, characterized by relevant electrical and mechanical properties, is strontium anorthite. This phase, in addition to a low value of dielectric permittivity (ε~6.2÷6.8), has the lowest dielectric loss ($t\gamma\delta$~0.0001÷0.0002) among crystalline phases that are used in creating radioceramics [3]. However, the technology of obtaining strontium ceramics requires high temperatures of sintering – 1,500 °C with an additional pre-heating treatment at 1,300 °C [4].

The importance of the direction and the study itselt consists in ensuring a low-temperature synthesis of the strontium anorthite crystalline phase and obtaining densely sintered radiotransparent ceramic materials.

2. Literature review and problem statement

The problem of synthesizing a densely sintered material based on strontium anorthite has been considered by scientists in many countries, through using various methods.

The authors of [5] obtained stoichiometric strontium-anorthite glass ceramics, using cold isostatic pressing and

sintering. In this case, the density of the material reached 94 % and 97 % of the theoretical level.

The influence of various heat treatment modes on the sintering density of strontium-anorthite glass ceramics is described in [6]. This is important because the density of the resulting material is a critical factor in using it. For this study, the involved material is a strontium aluminosilicate composition, which produces monoclinic anorthite as the primary crystalline phase at a temperature of 1,200 °C.

In [7], it is undertaken to synthesize strontium anorthite by a sol-gel method. The peaks of the crystalline phase begin to appear clearly on the X-ray film of the synthesized material at a temperature of 1,100 °C, and at 1,250 °C the strontium anorthite of the monoclinic system amounts to 100 % of the crystalline phase.

It is important to note that in the described studies strontium anorthite is synthesized in the presence of a liquid phase. A liquid phase sintering is the most common process in ceramics technology, especially with the participation of aluminosilicates. The melted substance formed as a result of firing, plays a major role, for it dissolves raw materials and subsequently is a source of a new crystalline phase. Therefore, by changing the characteristics of the melt (viscosity, surface tension, and activity), it is possible to manage the process of synthesizing.

In [8], it is studied what effect is produced by the additives B_2O_3 and P_2O_5, which are glass-forming oxides, but in this case they are also used as mineralizers lowering the temperature of the initial crystallization of strontium anorthite down to 1,020 °C.

In [9], it is described how to obtain high-strength ceramic materials with low dielectric properties. It is also said that the additive B_2O_3, LiF, Cr_2O_3, and $ZrSiO_3$ facilitate the transition of strontium anorthite from a hexagonal form to a stable monoclinic form. This is due to the cracking of the ceramic item, which may happen at a stage of cooling during polymorphic transformations of the hexagonal form, with an increase in the volume by 3 %.

In [10], the temperature for obtaining ceramic materials based on strontium anorthite is reduced by using single-component additives, both as mineralizers and modifiers. However, after firing the samples at a temperature below 1,450 °C, their structure could not be characterized as densely sintered shards even if the material contained only the phase of strontium anorthite.

Synthesis of strontium anorthite is studied in [11]; it has been found that synthesis is not a direct reaction, and the first intermediate compound formed already at 800 °C is strontium silicate. These data suggest that the processes of synthesizing strontium anorthite can be activated by intensifying particular stages.

Therefore, it is advisable to conduct a study on the use of activators for the sintering process that would be capable of producing a melt in a temperature range of 600–800 °C and an impact on the synthesized material.

3. The purpose and objectives of the study

The aim of the research is to use the system $SrO–Al_2O_3–SiO_2$ to obtain densely sintered radiotransparent ceramics at a low temperature of firing them, by introducing activators of sintering with a fluxing effect.

To achieve this goal, it is necessary to do the following tasks:

– to make an informed choice of activators for sintering ceramics with a low temperature of firing;

– to study the effect of the sintering activators and their amounts on the processes of strontium anorthite phase formation under a low-temperature synthesis.

4. Justification of the choice of sintering activators

The issue of choosing activators for sintering is based on taking into account several factors:

– these should be additives to accomplish the task of reducing the sintering temperature for ceramic materials based on strontium anorthite;

– the amount and properties of the newly formed glass phase shall be such as not to impair the basic characteristics of the materials – the dielectric constant and the dissipation factor;

– subject to the first two conditions, the strontium anorthite phase of the monoclinic form should be synthesized completely.

It is at once assumed that the most acceptable additives for achieving the purpose of the study can be combinations of classic glass modifiers – compounds of alkali metals. It is also suitable for this purpose to use a combination of stannic oxide and lithium oxide due to the low-temperature eutectics.

One of the criteria for choosing alkali metal fluorides as activators is the characteristic properties of the melt on their basis. It is known [12] that such melts are highly active and with low viscosity, so adding this kind of activators even in small amounts is effective.

It is known that introduction of alkali cations to a glass composition increases their conductivity and dielectric loss [13]. This is due to the increased mobility of small-size metal cations in a glass grid. To reduce their mobility, as stated in [14], it is advisable to use complex input of (two or more) alkali metals whose cations in the glass grid will prevent the movement of each other. The resulting opposite effect achieved is called alkaline. This reduces the conductivity and dielectric loss of the melt and, therefore, the glass phases.

Consideration of physical and chemical processes in the diagrams of multicomponent systems LiF–NaF, LiF–KF, KF–NaF, LiF–NaF–KF, and $Li_2O–SnO_2$ reveals the presence of eutectics in a temperature range from 450 °C to 700 °C. According to the studies that have been collected into the database of the phase equilibria patterns, the systems LiF–NaF [15], KF–LiF [16], KF–NaF [17], KF–LiF–NaF [18], and $SnO_2–Li_2O$ [19] with eutectic compositions of the components, mol. % (X_{NaF}=0.39 and X_{LiF}=0.61; X_{KF}=0.5 and X_{LiF}=0.5; X_{KF}=0.6 and X_{NaF}=0.4; X_{KF}=0.42, X_{LiF}=0.46, and X_{NaF}=0.12; X_{SnO2}=0.4; X_{Li2O}=0.6) form melts in the temperature ranges of 646–652 °C, 487–493 °C, 699–721 °C, about 454 °C, and 600–700 °C, respectively.

5. The materials and methods for researching the impact of complex additives on the synthesis of strontium anorthite and the sintering process of the ceramic mass

At the preliminary stage [20], the study revealed the optimal compositions of masses for obtaining radiotransparent

strontium anorthite ceramics. This was based on investigating the subsolidus structure of the system $SrO–Al_2O_3–SiO_2$, with choosing two points of the composition as a result (Fig. 1). The choice of the composition "0", corresponding to the point of strontium anorthite stoichiometry, was based on the possibility to study the effect of additives on the process of synthesizing this phase. The composition "2" was based on the elementary triangle of $SrAl_2Si_2O_8–Al_6Si_2O_{13}–SiO_2$, where the temperature of the initial melting is 1,383 °C. Its choice was based on the possibility to study the effect of the additives in a bigger amount of the melt and their influence on crystallization of side phases (mullite and tridymite). The chemical compounds that correspond to the points in the diagram in weight percentage are: "0" (SiO_2 – 36.89; Al_2O_3 – 31.30; SrO – 31.81) and "2" (SiO_2 – 50.0; Al_2O_3 – 25.0; SrO – 25.0).

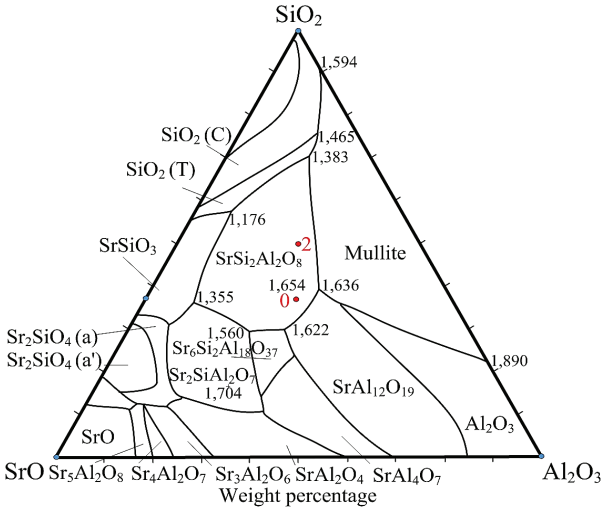


Fig. 1. A diagram of the three-component system $SrO–Al_2O_3–SiO_2$ according to Shukla [21]

The sample masses were supplemented with additives as complex activators of sintering in the amounts of 1, 2 and 3 wt % by 100 wt %. The phase diagrams of the multicomponent systems LiF–NaF, KF–KiF, KF–NaF, KF–LiF–NaF, and $SnO_2–Li_2O$, which characterize the thermal physical and chemical processes of the selected complex sintering activators, were used to calculate the raw material mixtures that produce the eutectic ratio (Tables 1, 2).

Table 1

The chemical composition of the raw materials

The raw materials	The component contents in wt%						
	SiO_2	Al_2O_3	Fe_2O_3	CaO	Na_2O	SrO	H.d.p.
Vyshnevetsky Quartz	99.35	0.421	0.06	0.039	–	–	0.13
Alumina G-00	0.03	98.7	0.02	–	0.25	–	1.0
Strontium carbonate	–	–	–	–	–	70.0	30.0

Given that the raw components of the model masses have no plastic properties, the formation of the samples was conducted by semidry pressing. The samples were produced according to the following flowchart (Fig. 2). In laboratory conditions, the raw materials were crushed in a porcelain mill and sieved through a sieve No. 0.15. Complex supplements were added in the quantities of 1, 2 and 3 wt % above the 100 wt % of the charge in terms of the active ingredient. A uniform distribution of the mineralizer was ensured by sieving it three times through the sieve No. 0.15. The samples were formed from the charge, using a temporary astringent (a dextrin solution). Cylinder-shaped samples with a diameter of 25 mm and a height of 5 mm were formed by semidry pressing on a laboratory press under the pressure of 25 MPa. The moulded semi-finished products were dried in a drying cabinet to the residual moisture of less than 1 %.

Table 2

The complex activators of sintering

The system of additives	The amounts of the raw components, wt %				
	$KF·2H_2O$	LiF	NaF	Li_2CO_3	SnO_2
LiF–NaF	–	49.14	50.86	–	–
LiF–KF	79.06	20.94	–	–	–
NaF–KF	66.22	–	32.78	–	–
KF–LiF–NaF	74.17	20.36	5.47	–	–
$Li_2O–SnO_2$	–	–	–	27.74	72.26

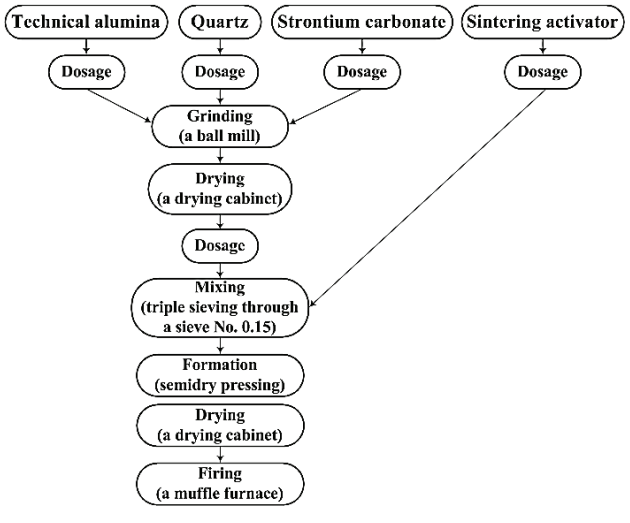


Fig. 2. A technological flowchart of producing strontium anorthite ceramics

The samples were fired in the laboratory muffle furnace Nabertherm HTCT 01/16 at a temperature of 1,250 °C with an exposure during 1 hour and cooled along with the furnace.

The phase composition of the test samples was determined by using the method of X-ray diffraction analysis (XRD analysis). The X-ray diffractions were filmed on the DRON-3M diffractometer with CuKα radiation and a nickel filter under conditions of its constant work. The phases were identified, using the American ASTM database.

The dielectric properties were determined by a device for measuring immittance – E 7-8. The measured values of the electrical capacitance of the samples were used to determine the dielectric constant. The calculation was carried out based on the formula of determining the electrical capacity:

$$\varepsilon = \frac{C \cdot d}{\varepsilon_0 \cdot \pi \cdot r^2}, \quad (1)$$

where C is the measured value of the electrical capacitance of a sample, d is the thickness of the sample, ε_0 is the electrical constant, and r is the radius of the electrodes.

The study of the morphological features of fracture surfaces of the samples were carried out by scanning electron microscopy (SEM) in the scanning electron microscope JSM-6390LV (JEOL, Japan). The tests were conducted in the mode of secondary electrons in the accelerating voltage of 10–20 kV and at low beam currents.

The main feature that can help make an indirect assessment of the impact of activating additives on the sintering degree is water absorption. Another important property is the apparent density, which allows making an indirect assessment of closed porosity. These characteristics, as well as open porosity, were investigated by "rapid vacuumization" under ISO 5017:2014.

6. The results of testing the properties of the obtained samples of ceramics

The results of determining water absorption, apparent density and open porosity of the synthesized ceramic samples depending on complex activators and their amounts are shown in Fig. 3.

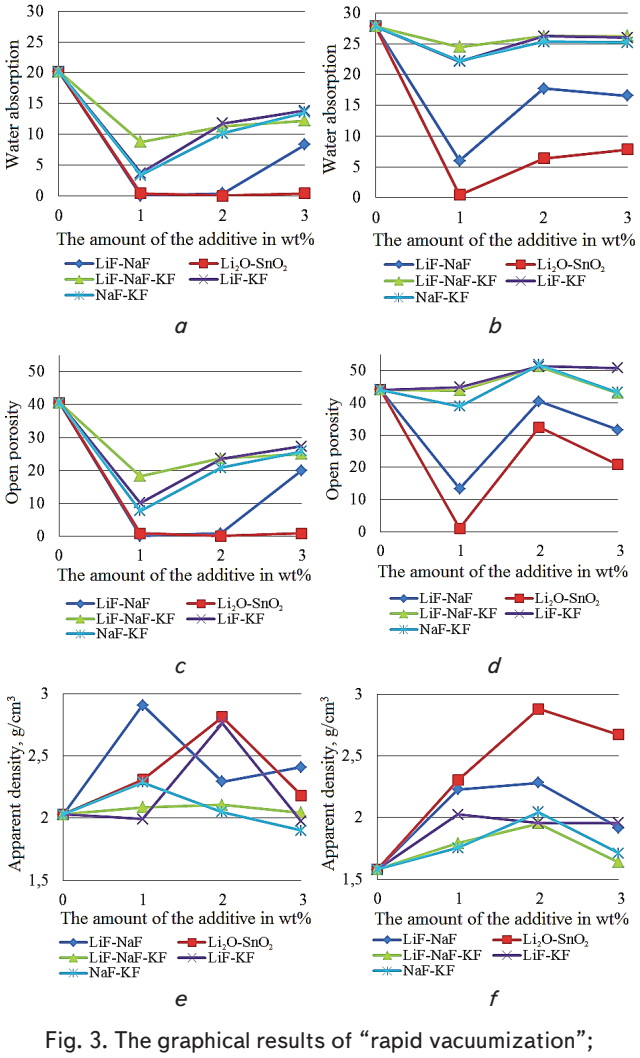


Fig. 3. The graphical results of "rapid vacuumization"; The left side displays the composition "2", whereas the right side displays the composition "0"; *a*, *b* show water absorption, *c*, *d* show open porosity, whereas *e*, *f* stand for the apparent density of the compositions

The properties of the fired samples indicate versatile actions of the additives. The results indicate that the most complex activator of sintering for the selected compositions is Li_2CO_3+SnO_2. Samples of the composition "2" have the water absorption rate of less than 0.4 %, and the biggest apparent density is achieved through adding Li_2CO_3+SnO_2 in an amount of 2 wt %. For the composition "0", water absorption with this activator has the smallest value of 0.47 % at 1 wt %, and it increases to 7.82 % at 3 wt %; the best apparent density is achieved if the content of the additive is in an amount of 2 wt %, which is 2.88 g/cm^3 and almost twice more than without this supplementation (1.58 g/cm^3). The properties are also significantly improved by the complex LiF+NaF; water absorption is less than 0.4 % at 1 wt % and 2 wt %, whereas at 3 wt % it slightly increases to 8.3 % of "2". For the composition "0", the smallest value of water absorption is recorded at 1 wt % (0.47 %). The complex LiF+NaF better affects the apparent density of the composition "2", and it has a maximum value of 2.91 g/cm^3 if its content of 1 wt %. Other mixtures at 1,250 °C have no significant effect on the studied properties.

Thus, at a firing temperature of 1,250 °C, all samples of "2" with the complex activators of sintering have signs of a high-temperature strain, which in turn makes it impossible to study the dielectric properties and to create radiotransparent materials for use above this temperature. When assessing the impact of sintering activators on the composition of "0" for a set of indicators, a noteworthy additive is Li_2CO_3+SnO_2 in an amount of 2 wt %. As for the other additions, they have no influence on the process of consolidating ceramics.

By using X-ray diffraction analysis, a sample of "0" with the addition of 2 wt % of Li_2CO_3+SnO_2 (Fig. 4) was investigated for the completeness of the reactions of forming the strontium anorthite phase and for the phase composition of heat treatment products. The bar radiograph shows only peaks of strontium anorthite, indicating a complete transformation of the raw materials. The bar radiograph also reflects an amorphous phase, confirming the fluxing mechanism of the mineralizer's action.

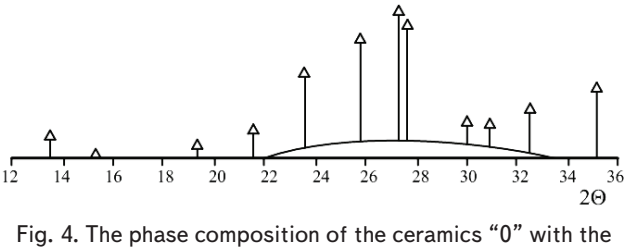
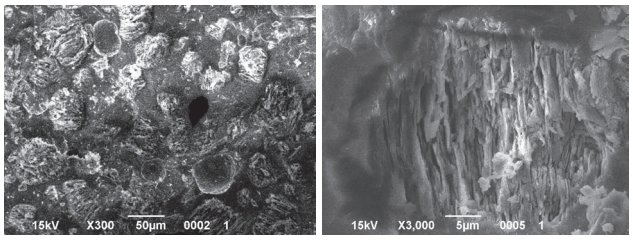


Fig. 4. The phase composition of the ceramics "0" with the addition of 2 % of Li_2CO_3+SnO_2: Δ – strontium anorthite

We also studied the microstructure of the fracture of the best sample (Fig. 5). The results show that the material in the glass phase contains randomly placed strontium anorthite crystals that are 35–40 microns in size, with closed pores of 50 microns. In general, there is a uniform dispersion of strontium anorthite crystals in the ceramics, and the presence of pores can be explained by a short interval of isothermal holding at the maximum temperature and a probably rapid mode of firing.

For a sample of the composition "0" with 2 wt % of Li_2CO_3+SnO_2, the received dielectric constant is 9.7 and the dielectric loss tangent is 0.06. According to the dielectric

values, the obtained material can be referred to the class of radiotransparent materials.



a *b*

Fig. 5. The microstructure of the ceramic fracture of "0" + Li_2CO_3–SnO_2 (2 wt %) fired at a temperature of 1,250 °C: *a* – zoomed in 300 times; *b* – zoomed in 3,000 times

7. The discussion of the research results about the impact of complex activators of sintering on the synthesis of strontium anorthite ceramics

In this study, attention was mainly directed to using activators of sintering in order to obtain a melt in a temperature range of 600–800 °C. The aim of this decision was the process of activating an interaction between SiO_2 and $SrCO_3$, which takes place in this temperature range, and the formation of a sufficient amount of a melt for a densely sintered shard already at 1,250 °C.

It is the selection and introduction of these complex activators that helped solve two problems. The first problem was to achieve low-temperature eutectics compared with an example of using one-component activators. The second problem was to neutralize the effect of alkali cations in order to improve the dielectric properties of the created ceramic material, which was achieved by the "polialkaline" effect.

The study made it fully possible to investigate the effect of the selected additives only in terms of the degree of sintering. For both compositions, the effects of the same additives turned out to be similar; as expected, there was a retained tendency of a stronger effect on the more fusible composition "2". For this composition under the pre-set mode of heat treatment at a temperature of 1,250 °C, the effects of the sintering activators were excessive. This was manifested in terms of a high-temperature deformation of the samples, which in turn caused excessive amounts of the melt.

The overall impact of fluoride complexes of alkaline metals in the composition "0" was low, probably because of their high volatility. It may have been caused by a high-speed mode of heat treatment at which the melt "had boiled out", failing to enter into reactions with the raw components.

All the subsequent tests were conducted with only one sample (the composition "0" with 2 wt % of Li_2CO_3–SnO_2), in which the set of properties was determined as the best. The roentgen-phase analysis showed a complete transformation of the raw materials and a synthesis of only strontium anorthite. Accordingly, it can be argued that the specific composition has confirmed the validity of the idea to activate an interaction between SiO_2 and $SrCO_3$ to the complete dissociation of the latter.

The microstructure of the sample clearly reveals a laminar "feldspar" structure of strontium anorthite and correctly rounded isolated closed pores. The microstructure results show that the goal has been achieved – the densely sintered ceramic material was obtained on the basis of strontium anorthite.

The dielectric properties within the normal range ($\varepsilon<10$) have an enlarged value (ε~9.7) compared with strontium anorthite (ε~6.2÷6.8). This is due to the increased amount of the glass phase; the values of the dielectric constant of oxides in the glass are the following: SrO – 18.0; Al_2O_3 – 9.2; SiO_2 – 3.8 [13].

The next step will be to research a temperature-time mode of firing for adjusting the amount of the glass phase, which is likely to reduce the dielectric properties and the number of closed pores.

7. Conclusions

The completed study has helped solve the problem of firing densely sintered ceramics at reduced temperatures due to adding the system SrO–Al_2O_3–SiO_2 with low dielectric properties:

1. It has been experimentally determined that it is advisable to use the complex sintering activators LiF–NaF and SnO_2–Li_2O due to their low-temperature eutectics and high activity of the melts, which can beneficially affect the structure and properties of strontium ceramics.

2. The study has revealed the effect of the complex activators of sintering on the basis of a fluxing action and their number on the low-temperature synthesis of strontium anorthite. The tests have confirmed the possibility of activating the reaction that produces the intermediate phase of silicate strontium, which reduces the temperature of firing strontium anorthite ceramics. It has been found that the best properties are produced by the composition "0", with the addition of 2 wt % of Li_2CO_3+SnO_2, a water absorption rate of 5.52 %, and an apparent density of 2.88 g/cm^3, with the dielectric constant being 9.7 and the dielectric loss tangent having the value of 0.06.

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