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REGULAR ARTICLE

Development of Radio-Absorbing Ceramics Based on SrO–BaO–TiO_2–SiO_2 System Compositions

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The paper investigates the features of the formation of radio-absorbing ceramics based on barium and strontium titanates for use in masking systems for special objects. The concept of creating heterophase materials with a controlled dielectric constant, which is provided by the controlled synthesis of target phases, is implemented. The physicochemical analysis of the SrO–BaO–TiO_2–SiO_2 system is carried out, a model state diagram of its subsolidus part is constructed. The phase relationships in the $BaTiO_3$–$SrTiO_3$–SiO_2 section are investigated, and the phase triangle $SrTiSiO_5$–$BaTiO_3$–$SrTiO_3$ is determined as the most technological region of compositions for the synthesis of target phases. Using two-stage solid-state sintering (1250 – 1350 °C – first firing, 1380 °C – second firing), materials were obtained, for which the phase composition, dielectric permittivity and electromagnetic properties were studied. The materials are characterized by a wide range of variation in dielectric permittivity (21.0-71.5), which is due to their phase composition. The formation of $SrTiO_3$, solid solutions of $Ba_{0.5}Sr_{0.5}TiO_3$ i $Ba_{0.9}Sr_{0.1}TiO_3$ and in various combinations was experimentally con-firmed, as well as the possibility of regulating their ratios in the final product by changing the composition of the initial compositions and the temperature of the firing. The attenuation and reflection coefficients of the electromagnetic wave, as well as the wave resistance of the materials at a frequency of 0.1 GHz, were deter-mined and it was established that they belong to ceramics with a medium and high level of radio wave ab-sorption. The feasibility of using the developed materials as functional and matching layers in gradient radio-absorbing compositions intended to protect special objects from microwave radiation is shown.

Keywords: Radio-absorbing ceramics, Strontium and barium titanates, Solid-state synthesis, Dielectric constant, Reflection coefficient, Absorption coefficient, Wave impedance.

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1. INTRODUCTION

Ceramic dielectrics are widely used as components in wireless communication systems, radar systems, unmanned aerial vehicle control systems, and electronic warfare systems. This technology operates in the radio frequency range of electromagnetic radiation, particularly in the ultra-high frequency range, which is particularly relevant for military applications. The requirements for the frequency and dielectric characteristics of microwave ceramics for military equipment and passive camouflage systems vary significantly depending on the intended use of the materials. For example, the permittivity of ceramic elements varies widely, from low values for antenna modules in unmanned aerial vehicles to high values for dielectric resonators and filters. This necessitates the search for effective materials science methods for controlling the dielectric properties of materials in a given direction, in accordance with the requirements of their specific application.

Technologies for removing microfiber ceramics are important to use on a traditional solid-phase sponge, which is characterized by high temperatures and will require a high level of purity to ensure due to the durability of the phase storage of materials. Based on research from the development of functional ceramics for aerospace technology [1, 2] based on the SrO–Al_2O_3–SiO_2 and BaO–Al_2O_3–SiO_2 systems, by which low values were achieved dielectric penetration of materials while ensuring a high level of their sintering and maximum level of radio transparency. The approach that has been implemented in these studies is the direct synthesis of the target monophase between the main oxide systems – slavsonite and celsian – with the minimization of by-products phase development processes.

In addition to this approach, in order to better regulate the dielectric characteristics over a wide range of values, a promising concept is the creation of heterophasic ceramic systems in which the conformity is controlled. The purpose of the phases is to ensure the dielectric penetration of the material. In this context, there is significant scientific and practical interest in the SrO–BaO–TiO_2–SiO_2 system, in which it is possible to instantly synthesize the titanate phases of strontium and barium with completely different dielectric values penetration. Variation of their high-quality properties opens up the possibilities for direct ceramic treatment

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due to the phase structure and functional properties of materials, which significantly expands the range of their practical hardening.

Further development of technical ceramics technology, based on the SrO–BaO–TiO_2–SiO_2 system, will require more detailed material analysis, which is transferred to the subsolid phase of the system, minds of the development of phases and their synthesis. Although the functional subsystems are already sufficiently developed, information about the phase changes between each component system of the infection is not consistent.

The aim of this investigation was to conduct physical-chemical analysis of the SrO–BaO–TiO_2–SiO_2 system, identification of promising compositions for the direct synthesis of titanates with strontium and barium and identification of technological parameters based on them radio-absorbing ceramics with controlled properties.

2. THEORETICAL PART

Considering that the basis of future materials will be the phases $SrTiO_3$ and $BaTiO_3$, an analysis of the literature data on the structure of ternary systems in which these compounds can be formed was carried out.

The subsolidus state of the SrO–Al_2O_3–TiO_2 system is described by the following phase relations: $SrTiO_3$ coexists with aluminates $Sr_3Al_2O_6$, $SrAl_2O_4$, $SrAl_{12}O_{19}$, titanate $Sr_3Ti_2O_7$ and two ternary compounds. The first of them (the so-called phase Y) coexists with $SrTiO_3$ and aluminates $SrAl_2O_4$, $SrAl_4O_7$, $SrAl_{12}O_{19}$, the second (phase X) – with $SrTiO_3$, $SrAl_{12}O_{19}$ and tialite $TiAl_2O_5$.

According to scientists from the Kharkiv School of Physical Chemistry of Silicates, the SrO–TiO_2–SiO_2 system in its subsolidus is divided into 10 elementary triangles [3]. Strontium titanate coexists with such new formations as $Sr_3Ti_2O_7$, $SrSiO_3$ and $SrTiSiO_5$. It is this elementary triangle that the authors recognized as promising for the technology of technical ceramics.

Data on phase equilibria in the BaO–TiO_2–SiO_2 system are limited to studies of the $BaTiO_3$–SiO_2 section. They indicate the presence of barium titanosilicates with different degrees of SiO_2 saturation in the system: $BaTiSiO_5$ (barium sphene), $BaTiSi_2O_7$, $BaTiSi_3O_9$ (benitoite, which exists up to a temperature of 1050 °C) [4], as well as the compound $Ba_2TiSi_2O_8$ (fresnoite) [5]. It is also known that $BaTiSiO_5$ was not obtained synthetically, but fresnoite with a similar X-ray pattern was formed. Barium titanate forms a solid solution with silica at a low concentration of SiO_2 (8 and 4 mol. %) for cubic and hexagonal $BaTiO_3$, respectively), which coexists with sphene up to a temperature of 1260 °C. This system is of practical interest for the synthesis of barium titanosilicates, which, due to their optical and photoluminescent properties, can be used to create functional materials and light-emitting devices [6]. This applies to $BaTiSi_2O_7$ to a greater extent. In contrast to Robbins' studies [5], according to [6], $BaTiSi_4O_{11}$ can be additionally synthesized in the BaO–TiO_2–SiO_2 system, and $BaTiO_3$ cannot coexist with any of the ternary compounds.

Scientists of the NTU "KhPI" have studied the complete subsolidus structure of the SrO–BaO–TiO_2 system (temperature 1380 °C), which includes 11 elementary triangles with six barium titanates and four strontium titanates. This system was studied to obtain ceramic ferroelectrics based on it and, according to the authors [7-9], the most promising phase triangle for their synthesis is $SrTiO_3$–Sr_2TiO_4–$BaTiO_3$, which has the highest probability of existence. It is important that between $BaTiO_3$ and $SrTiO_3$ solid solutions of variable composition $Ba_xSr_{1-x}TiO_3$ (x = 0.3-0.4) are formed, based on which ceramic energy storage devices with high dielectric constant are obtained [8, 9].

Based on the given data on phase relations in the considered ternary systems, a theoretical phase diagram of the SrO–BaO–TiO_2–SiO_2 system was constructed in this work (Fig. 1). In the figure, the formulas of the compounds are given in abbreviated form: the designation BTS_3 corresponds to the compound $BaTiSi_3O_9$, SrTS–$SrTiSiO_5$, etc. For the synthesis of ceramics with target phases of strontium and barium titanates, the cross-section of the $BaTiO_3$–$SrTiO_3$–SiO_2 system is of greatest interest, which has not been studied from the point of view of possible phase interactions and the probability of the existence of compounds in its subsolidus part.

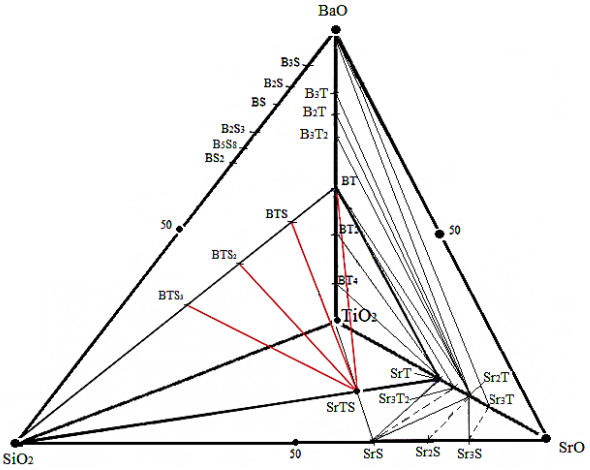


Fig. 1 – System state diagram SrO–BaO–TiO_2–SiO_2

As can be seen from Fig. 1, geometric analysis of the $BaTiO_3$–$SrTiO_3$–SiO_2 cross-section allows us to establish the existence of the SrTS–BTS_3 connode, which does not intersect with any other. At the same time, the existence of other connodes requires additional clarification, since they can compete with each other (Fig. 2).

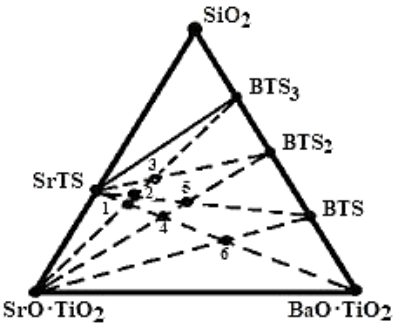


Fig. 2 – Cross-section of the $BaTiO_3$–$SrTiO_3$–SiO_2 phase diagram of the SrO–BaO–TiO_2–SiO_2 system

This clarification was carried out using thermodynamic analysis of six exchange reactions (Table 1) using both standard thermodynamic constants of substances and, in

their absence, calculated by the method of Taranenkova V.V. (enthalpy) and Wood-Fraser (entropy) [10]. According to Table 1, it can be concluded that all reactions are possible, the probability of their occurrence increases with increasing temperature and, therefore, the reaction products can coexist and be in equilibrium with each other. In the final version, the phase relations in the $BaTiO_3$–$SrTiO_3$–SiO_2 cross section are shown in Fig. 3.

Table 1 – Change in Gibbs free energy for reactions

Reactions	ΔGt, kJ/mol	
	1000 K	1500 K
$3SrTiO_3 + BaTiSi_3O_9 = 3SrTiSiO_5 + BaTiO_3$	– 146.7	– 219.2
$2SrTiO_3 + BaTiSi_3O_9 = 2SrTiSiO_5 + BaTiSiO_5$	– 34.1	– 58.4
$SrTiO_3 + BaTiSi_3O_9 = SrTiSiO_5 + BaTiSi_2O_7$	– 16.9	– 28.9
$2SrTiO_3 + BaTiSi_2O_7 = 2SrTiSiO_5 + BaTiO_3$	– 129.9	– 190.3
$SrTiO_3 + BaTiSi_2O_7 = SrTiSiO_5 + BaTiSiO_5$	– 17.2	– 29.5
$SrTiO_3 + BaTiSiO_5 = SrTiSiO_5 + BaTiO_3$	– 112.7	– 160.8

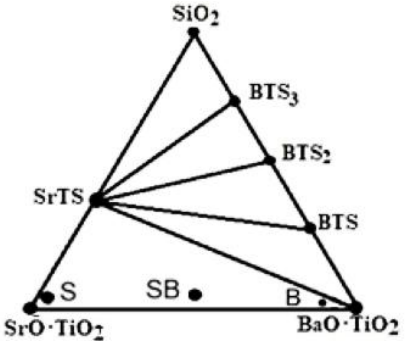


Fig. 3 – Phase relations in the $BaTiO_3$–$SrTiO_3$–SiO_2 section and figurative points of the compositions of experimental masses

Further, based on the above breakdown of the experimental section according to the method described by the authors [10], the lengths of connodes L_i between the connections of the section, the relative areas of all five elementary triangles and the degrees of their asymmetry were determined. Based on these data, the total areas of triangles in which specific phases may be present and the probability of their existence were determined (Table 2).

Table 2 – Characteristics of the cut phases $BaTiO_3$–$SrTiO_3$–SiO_2

Phase	Total relative area in which a phase can exist, ‰	Probability of existence, fractions of unity
$BaTiO_3$	444	0.148
$SrTiO_3$	300	0.100
SiO_2	400	0.133
$SrTiSiO_5$	1000	0.333
$BaTiSi_3O_9$	460	0.153
$BaTiSi_2O_7$	156	0.052
$BaTiSiO_5$	240.0	0.080

According to the information in Table 2, the phases $BaTiSi_3O_9$, $SrTiSiO_5$, $BaTiO_3$, SiO_2 and $SrTiO_3$ have the highest probability of existence within the experimental section in order of decreasing probability. The elementary triangle, which simultaneously contains the phases $BaTiO_3$ and $SrTiO_3$ (SrTS–BT–ST), has an asymmetry level of $\omega = 3.33$, which is less than the same value for another triangle, in which the titanium phase is present barium, – SrTS–BTS–BT with asymmetry step $\omega = 4.33$. This indicates the greater technological efficiency of warehouses with a composition based on the elementary triangle SrTS–BT–ST.

The generalization of the data of the geometric and topological analysis of the $BaTiO_3$–$SrTiO_3$–SiO_2 cross-section of the SrO–BaO–TiO_2–SiO_2 system showed that for the formation of heterophase ceramics with specified dielectric properties, it is advisable to choose the compositions of the initial compositions within the SrTS–BT–ST triangle near the BT–ST connode. This ensures the controlled synthesis of $BaTiO_3$ and $SrTiO_3$ as target phases and creates the prerequisites for the directed regulation of the dielectric properties of materials by varying their compositions.

3. EXPERIMENTAL PART

To obtain ceramic materials with the target phases $SrTiO_3$ and $BaTiO_3$, the compositions of the initial compositions were investigated, the figurative points of which are shown in Fig. 3. The compositions were designed in such a way as to investigate materials with the dominance of one of the target phases and their equal content, as well as to carry out an experimental verification of the phase relationships determined by the geometric and topological analysis of the cross section. The theoretical phase composition of the materials is given in Table 3.

Table 3 – Composition of the initial experimental masses

Composition code	Phase content in the composition, %		
	$SrTiO_3$	$BaTiO_3$	$SrTiSiO_5$
S	90.0	5.0	5.0
SB	45.0	45.0	10.0
B	5.0	90.0	5.0

Ceramic samples were obtained by dosing raw components (quartz sand, strontium and barium carbonates, titanium dioxide), their repeated mixing for homogenization with subsequent semi-dry pressing with a CMC solution as a binder. After drying the samples, the first firing was carried out (1250-1350 °C) with subsequent grinding of the intermediate product, sieving of the powder (sieve № 0063), moistening and repeated sieving (sieve № 05). Then the powder was subjected to semi-dry pressing, the samples were subjected to final firing (1380 °C) with subsequent mechanical processing. Two-stage firing provided a reduction in shrinkage, improved sintering and intensification of phase formation.

For the obtained ceramic samples, the following were investigated: apparent density ρ (DSTU B V.2.7-283:2011), dielectric permittivity ε and dielectric loss tangent tan δ (1 kHz) using a digital RLC immittance meter E7-8. The electromagnetic properties of the materials were calculated using the formulas given in [11].

In this case, the experimentally established values of ε of the materials at a frequency of 1 kHz were used.

Given that the dielectric loss tangent of ceramics is insignificant (0.005-0.01), the attenuation coefficient was determined using the simplified formula (1.1):

$$\alpha = \frac{\omega\mu\varepsilon}{2} tg\delta, \quad (1.1)$$

where α is the attenuation coefficient (Np/m), ω is the angular frequency (rad/s), and μ is the magnetic permeability (for non-magnetic materials $4\pi \cdot 10^{-7}$ H/m).

The reflection coefficient of an electromagnetic wave was calculated using formula (1.2):

$$RL = 20\, lg\left[\frac{\sqrt{\varepsilon_r}-1}{\sqrt{\varepsilon_r}+1}\right], \quad (1.2)$$

where RL is the wave reflection coefficient (dB), ε_r is the relative dielectric constant.

The wave resistance of materials was calculated by the formula (1.3):

$$Z = Z_0\sqrt{\frac{\mu}{\varepsilon}}, \quad (1.3)$$

where Z is the wave resistance of the material (Ohm), Z_0 is the wave resistance of air (Ohm), μ is the relative magnetic permeability ($\mu \approx 1$), ε is the dielectric permittivity.

The phase composition of the ceramics was determined by the method of X-ray analysis (DRON-3M diffractometer with CuKα-radiation).

4. RESULTS AND DISCUSSION

Ceramic samples demonstrated a high level of sintering, their density is close to the density of pure titanates. Thus, the ρ of the sample of composition S varies from 4.2 to 4.8 g/cm^3 at a density of 5.1 g/cm^3 for $SrTiO_3$, the density of the sample B – from 4.8 to 5.4 g/cm^3 (density of $BaTiO_3$ = 6 g/cm^3). This property weakly depends on the temperature of the first firing. At the same time, this temperature significantly affects the dielectric constant of the samples, which varies within quite wide limits (Fig. 4).

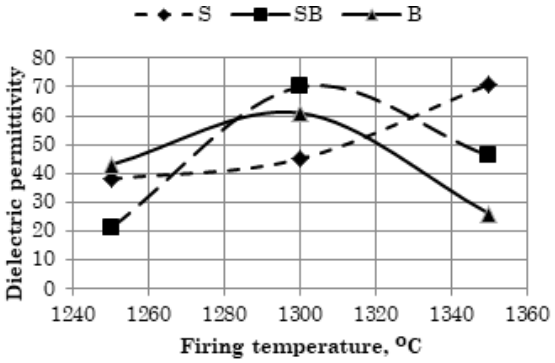


Fig. 4 – Effect of the temperature of the first firing on the dielectric constant of the samples (frequency 1 kHz)

Materials of compositions B and SB illustrate extreme dependences $\varepsilon = f(t)$ with maxima of ε values at a temperature of 1300 °C, in contrast to sample S, for which the dielectric constant increases gradually. It should be noted that for materials an atypical dependence between density and dielectric constant is observed. In general, a decrease in density leads to a decrease in ε due to the presence of air in the pores and a decrease in the volume of material capable of dissipating wave energy, but the nature of the pore distribution is important here. As the authors note [12], their uneven distribution can cause field localization and an increase in wave losses. This is probably the reason for the maximum value of ε = 71.5 for material S (1350 °C) at its lowest density of 4.2 g/cm^3.

To assess the correspondence of the phase composition of the materials to their theoretical composition established by the geometric-topological analysis of the $BaTiO_3$–$SrTiO_3$–SiO_2 cross-section, X-ray phase studies of samples obtained at the limiting temperatures of the first firing (1250 °C, 1350 °C) were carried out. It was established that in the S material at both temperatures, only the $SrTiO_3$ phase is formed. The phase composition of the *SB* sample is represented by a solid solution of $Ba^{0.5}Sr_{0.5}TiO_3$ and barium sphene $BaTiSiO_5$, the formation of which is quite probable according to the data of thermodynamic calculations of the reactions (Table 1). The basis of the material of composition B is a solid solution of $Ba_{0.9}Sr_{0.1}TiO_3$, barium sphene is present in a small amount, which disappears with an increase in the firing temperature. In general, the phase composition of the ceramic samples confirms the results of the theoretical analysis of the phase relations of the $BaTiO_3$–$SrTiO_3$–SiO_2 cross-section and indicates the possibility of controlled synthesis of target phases by purposeful regulation of the composition and heat treatment modes.

To achieve high values of dielectric permittivity of materials (60-70), any of the considered compositions can be used, but, taking into account technological and economic factors, compositions S and B should be considered optimal, which provide such values at a lower temperature (1300 °C). The defining feature of the obtained materials is the variability of dielectric permittivity indicators (ε = 21.0-71.5). This opens up the possibility of their application as functional layers of gradient radio-absorbing compositions. Such compositions were previously proposed by the authors [13] for their use as a building material for the protection of technical and biological objects located in the zones of influence of radio radiation sources. Their purpose is to minimize reflection and maximize absorption of EMR, which is ensured by a gradual change in dielectric permittivity and wave resistance between individual layers. The outer layer should be made of a material with a low dielectric constant to match the wave resistance of air and the material, the other layers should be made of dielectrics with a relatively high dielectric constant.

For the developed materials, their electromagnetic characteristics, such as the attenuation and reflection coefficients of the electromagnetic wave and the wave resistance, were determined by calculation, which are of key importance when used as layers of radio-absorbing compositions (Table 3).

Analysis of the table shows that according to the values of the attenuation coefficient, the materials are either medium-absorbing (10-100 Np/m) or those that will absorb the wave effectively (100-1000 Np/m). The reflection coefficient for effective absorbers should be less than 10 dB. This will mean that less than 10 % of

the wave energy will be reflected from the surface of the material [14]. According to this indicator, the samples are medium-effective materials. To minimize the reflection of the wave from the surface of the material, it is necessary that its wave resistance approaches the resistance of air (377 Ohm). The Z values of the materials are lower than those required for matching with air, however, when using materials as layers of a gradient composition, such wave resistance is acceptable if they are used in internal layers with wave resistances that are matched to each other.

Table 3 – Electromagnetic properties of materials

Composition code	First firing temperature, °C	Attenuation coefficient a (0.1 GHz), Np/m	Reflection coefficient RL, dB	Impedance Z, Ohm
S	1250	72.80	– 2.90	62.03
SB		42.00	– 3.82	81.70
B		88.60	– 2.60	56.20
S	1300	90.00	– 2.59	55.74
SB		139.10	– 2.10	45.00
B		123.00	– 2.20	47.70
S	1350	141.00	– 2.06	44.60
SB		90.30	– 2.60	55.70
B		52.00	– 3.40	73.33

From the above data, it can be concluded that in general all materials are effective for absorbing radio waves in the ultrahigh frequency range, but their level of effectiveness differs. High-dielectric ceramics (with ε = 60-70) due to intensive interaction with the electromagnetic field will be actively polarized and will provide effective dissipation of wave energy, which determines the feasibility of using it as an active radio-absorbing layer in gradient structures. Ceramics with an average dielectric permittivity (ε = 21-46), increased wave resistance and reduced reflection coefficients are suitable for use as matching layers in radio-absorbing compositions. Fig. 5 shows a diagram of such a composition as one of the possible options for combining the developed materials.

Taking into account the economic aspect of the technology of research materials (two-stage firing and high synthesis temperatures), the following are recommen-ded for practical use: high-dielectric ceramics of compositions *SB* and *B* with a first firing temperature of 1300 °C and ceramics with an average dielectric constant obtained at a temperature of 1250 °C based on any of the considered compositions.

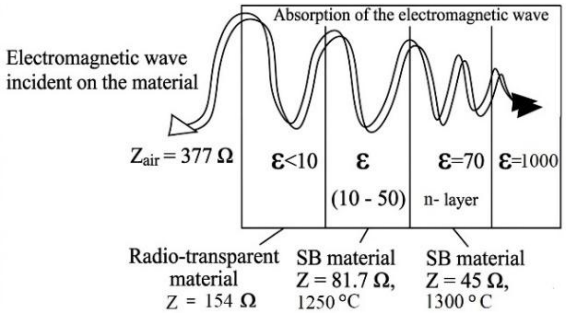


Fig. 5 – Scheme of a radio-absorbing composition using developed materials

5. CONCLUSIONS

As a result of the physicochemical and experimental study of the SrO–BaO– TiO_2-SiO_2 system, the prospects of its use for the creation of heterophase radio-absorbing ceramics with controllable dielectric characteristics were established. Based on the geometric-topological and thermodynamic analysis of the $BaTiO_3$–$SrTiO_3$–SiO_2 cross-section, the phase relations were determined and the feasibility of choosing compositions within the SrTS–BT–ST triangle for the simultaneous synthesis of $BaTiO_3$ and $SrTiO_3$ was substantiated. The possibility of forming $SrTiO_3$ phases, solid solutions of $Ba_{0.5}Sr_{0.5}TiO_3$, $Ba_{0.9}Sr_{0.1}TiO_3$ in different ratios by changing the composition of the compositions and heat treatment modes was experimentally confirmed. The obtained ceramic materials are characterized by a high degree of sintering and a wide range of dielectric permittivity values (ε = 21.0-71.5), which creates prospects for their use in functional layers of gradient radio-absorbing structures with coordinated dielectric and electromagnetic properties. It has been established that the materials provide an average and high level of absorption of electromagnetic waves in the radio frequency range. The most promising for practical application are the compositions *SB* and *B* obtained at a first firing temperature of 1300 °C. The developed materials can be used as elements of wireless communication systems, radio-location and radio-masking of objects.

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Розробка радіопоглинаючої кераміки на основі композицій системи $SrO–BaO–TiO_2–SiO_2$

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У роботі досліджені особливості формування радіопоглинаючої кераміки на основі титанатів барію і стронцію для використання в системах маскування спеціальних об'єктів. Реалізована концепція створення гетерофазних матеріалів з керованою діелектричною сталою, що забезпечується контрольованим синтезом цільових фаз. Проведений фізико-хімічний аналіз системи $SrO–BaO–TiO_2–SiO_2$, побудована модельна діаграма стану її субсолідусної частини. Досліджені фазові співвідношення в перерізі $BaTiO_3–SrTiO_3–SiO_2$, визначений фазовий трикутник $SrTiSiO_5–BaTiO_3–SrTiO_3$ як найбільш технологічна область складів для синтезу цільових фаз. Із застосуванням двоетапного твердофазного спікання (1250-1350 °C – перший випал, 1380 °C – другий) отримані матеріали, для яких досліджені фазовий склад, діелектрична проникність та електромагнітні властивості. Матеріали характеризуються широким діапазоном варіювання діелектричної проникності (21,0-71,5), яка обумовлена їх фазовим складом. Експериментально підтверджено формування $SrTiO_3$, твердих розчинів $Ba_{0.5}Sr_{0.5}TiO_3$ і $Ba_{0.9}Sr_{0.1}TiO_3$ в різних комбінаціях, а також можливість регулювання їх співвідношень у кінцевому продукті шляхом зміни складу вихідних композицій і температури випалу. Визначені коефіцієнти затухання і відбиття електромагнітної хвилі, а також хвильовий опір матеріалів на частоті 0,1 ГГц і встановлено, що вони відносяться до кераміки середнього та високого рівня поглинання радіохвилі. Показана доцільність застосування розроблених матеріалів як функціональних та узгоджувальних шарів у градієнтних радіопоглинаючих композиціях, призначених для захисту спеціальних об'єктів від надвисокочастотного випромінювання.

Ключові слова: Радіопоглинаюча кераміка, Титанати стронцію і барію, Твердофазовий синтез, Діелектрична проникність, Коефіцієнт відбиття, Коефіцієнт поглинання, Хвильовий опір.