

*G. V. LISACHUK, V. Yu. BAHlai***TECHNOLOGY FOR THE MANUFACTURE OF COMPOSITE RADIO-ABSORBING CERAMICS**

In the modern world, electromagnetic radiation constantly accompanies a person, affecting both his health and the operation of technical devices. One of the promising solutions to this problem is the use of special ceramic materials that can effectively absorb radiation and can significantly increase the level of environmental safety. The purpose of the work is to manufacture facing tiles based on composite radio-absorbing ceramics with the addition of silicon carbide and strontium titanate. Experimental composite ceramic tiles consist of three layers. Spectral characteristics were determined - transmittance, absorption and reflection coefficients in the frequency range of 20 - 40 GHz. The results of experimental studies of the developed ceramic tiles based on facing ceramics with electrically conductive additives of silicon carbide in the amount of 30 wt. % are presented and strontium titanate in the amount of 10, 20 and 30 wt. %. To determine the parameters of the interaction of electromagnetic radiation with samples, an upgraded meter of the standing wave coefficient and attenuation was used - a generator unit P2-65 with an indicator Я2Р-67. The developed composite ceramic materials meet the basic performance requirements for this class of materials. They can be effective for reducing the intensity of the electrical component of the electromagnetic field in the middle of rooms located in the areas of exposure to radio emission sources, as well as used to improve the environment to reduce the level of the electromagnetic field outside the premises where work related to electromagnetic radiation is located.

Keywords: radio absorption; composite ceramics; three-layer tiles; silicon carbide; strontium titanate; electrodynamic characteristics; transmission coefficient; reflection; absorption coefficient.

*Г. В. ЛИСАЧУК, В. Ю. БАГЛАЙ***ТЕХНОЛОГІЯ ВИГОТОВЛЕННЯ КОМПОЗИЦІЙНОЇ РАДІОПОГЛИНАЮЧОЇ КЕРАМІКИ**

У сучасному світі електромагнітне випромінювання постійно супроводжує людину, впливаючи як на її здоров'я, так і на роботу технічних пристроїв. Одним із перспективних вирішень цієї задачі є використання спеціальних керамічних матеріалів, здатних ефективно поглинати випромінювання та можуть значно підвищити рівень безпеки навколишнього середовища. Метою роботи є виготовлення облицювальної плитки на основі композиційної радіопоглинаючої кераміки з додаванням карбіду кремнію та титанату стронцію. Дослідна композиційна керамічна плитка складається з трьох шарів. Визначали спектральні характеристики – коефіцієнти передачі, поглинання та відбиття у діапазоні частот 20 – 40 ГГц. Представлені результати експериментальних досліджень розробленої керамічної плитки на основі облицювальної кераміки з електропровідними добавками карбіду кремнію в кількості 30 мас. % та титанату стронцію в кількості 10, 20 та 30 мас. %. Для визначення параметрів взаємодії електромагнітного випромінювання зі зразками використовували модернізований вимірювач коефіцієнту стоячої хвилі та ослаблення – генераторний блок Р2-65 з індикатором Я2Р-67. Розроблені композиційні керамічні матеріали задовольняють основні експлуатаційні вимоги, що висуваються до даного класу матеріалів. Вони можуть бути ефективними для зниження інтенсивності електричної складової електромагнітного поля в середині приміщень, розташованих у зонах впливу джерел радіовипромінювання, а також використовувати з метою покращення екології для зниження рівня електромагнітного поля за межами приміщень, де розміщені роботи, пов'язані з електромагнітним випромінюванням.

Ключові слова: радіопоглинання; композиційна кераміка; тришарова плитка; карбід кремнію; титанат стронцію; електродинамічні характеристики; коефіцієнт передачі; коефіцієнт відбиття; коефіцієнт поглинання.

Entry. It is well known that the absorption efficiency of microwaves of a material is provided by its electromagnetic properties, in particular dielectric and magnetic constant, which mainly depend on the composition and microstructure of absorbents. The morphological distribution of absorbents in carriers (coatings or composites) plays an important role in improving the microwave absorption capacity of integral absorbent materials that contain absorbents and a matrix. The orderly distribution of absorbents in the matrix contributes to the formation of structured conductive connections with multi-level transitions even with low filler content. This lengthens the path of propagation of electromagnetic waves due to repeated reflection and scattering, which, in turn, increases the material's ability to lose electromagnetic energy [1,2].

The main requirement for the electrical properties of ceramic materials for use in devices is a combination of dielectric constant in the range from 300 to 600 when retuning the electric field not worse than 10-20 % (electric field strength $\sim(2-5)$ V/ μ m) and low dielectric losses in the microwave range (dielectric loss angle

tangent $\delta \leq 0.005$). To achieve the required level of dielectric constant in a system of solid solutions (Ba, Sr) TiO_3 with the structure of perovskite can increase the content of strontium titanate, which leads to a shift in the Curie temperature of ferroelectrics towards lower temperatures and reduces the tangent of the dielectric loss angle to $(1-3) \cdot 10^{-3}$ in a wide range of frequencies, including microwave. However, for such materials, the rearrangement of the electric field of the ϵ almost disappears. and alkaline earth oxides and compounds. Such compositions should provide a combination of high tunability of ϵ and low dielectric losses [3,4]. Machining and sintering determine the basic characteristics of materials, such as microstructure and the presence of defects, which makes knowledge of the evolution of microstructure during the machining process very important. Numerous methods can be used to obtain materials based on SrTiO_3 , such as sol-gel, hydrothermal, incineration method and polymer precursor method [9-13]. Most of these methods require high-purity chemicals, making them too expensive for industrial production. Therefore, commercial SrTiO_3 is still predominantly produced by solid-state reaction. Modern applications

require homogeneous, low-impurity micrograins of ceramic materials, which makes alternative low-cost efficient methods for large-scale production of phase-pure and nano-sized SrTiO₃ particles an important topic.

The use of mechanical activation in high-energy mills can affect the evolution of the microstructure and the sintering properties of the material [14]. Mechanical grinding leads to a decrease in particle size, resulting in a change in the dielectric properties of the final ceramic material [15]. It has been found that reducing the grain size to ~1 μm in some perovskites leads to an increase in the dielectric constant value (ε'r), while a further decrease in the grain size leads to decrease in the value of ε'r [16,17]. Also, the value of the relative permittivity is additionally influenced by the density values of different activated sintered samples, i.e. different porosity. A sample that has a high degree of porosity, i.e. lower density, will have lower relative permittivity values [18]. In general, microstructure has a great influence on the dielectric properties of SrTiO₃-based ceramics, and sintering is an important process that affects microstructure evolution, grain growth, and compaction of the final product [19,20].

Strontium titanate, as a material with high dielectric constant, is an important raw material for the creation of electrical ceramics. Products based on it have advantages such as low dielectric loss and good thermal stability, so it is widely used in the electronics industry. In the ceramic industry, it is used for the production of capacitors, piezoelectric materials, ceramic sensing components and microwave components, and is also applied to the automatic adjustment of heating elements and the manufacture of demagnetized parts. It can be used in optical glass, carbon powder, enamel, heat-resistant materials, insulation materials and other industries.

In connection with the above, the purpose of this work is to manufacture facing tiles based on composite radio-absorbing ceramics with the addition of silicon carbide and strontium titanate with specified electrodynamic characteristics.

Research methods.

The study of the electrodynamic characteristics of the samples - transmission, absorption and reflection coefficients, was carried out in the frequency range of 20 - 40 GHz. Standard rectangular waveguides with a cross section of 7.2 x 3.4 mm² were used. The samples that were examined completely overlapped the cross-section of the waveguide and had a thickness of 2.4 mm.

When a wave passes through a composite material, the interaction of radiation with it is determined by the magnitude of S_{21} transmission, S_{11} reflection and S_A absorption [21]

$$S_{21} = S_{11} + S_A + S_{MR},$$

where S_{MR} is the value of multiple reflection.

The amount of reflection S_{11} in dB, equal to

$$S_{11} = 20 \lg \left| \frac{VSWR - 1}{VSWR + 1} \right|,$$

where $VSWR$ is the voltage coefficient of the standing wave.

The amount of *absorption* of S_A is calculated in dB and is equal to:

$$S_A = 20 \lg \cdot e^{d/\delta}, \delta = \left(\sqrt{\pi f \mu \sigma} \right)^{-1},$$

where d is the thickness of the sample,
 f is the frequency of radiation,
 μ is the magnetic permeability of the sample,
 σ is the conductivity of the sample.

S_{MR} Multiple Reflection Value

$$S_{MR} = 20 \lg \left| 1 - e^{-2d/\delta} \right|.$$

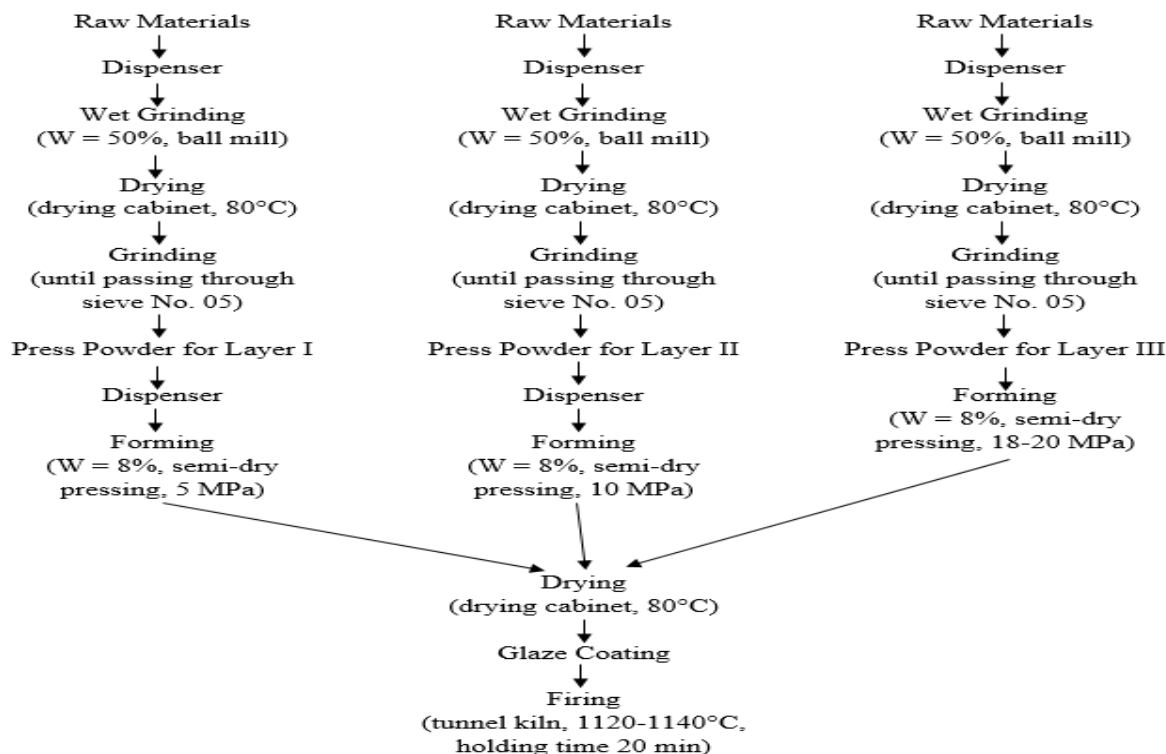
Typical values of multiple reflection in composites with applied concentrations of a filler with high conductivity are negligible and can be ignored.

Experimental part

A promising vector of scientific research is the formation of highly efficient composite materials based on a dielectric matrix and electrically conductive additives (carbon black, graphite, carbonyl iron, silicon carbide, titanates, iron (III) oxide, copper (II) oxide, etc.) [22-24]. In previous studies, it was found that for the creation of electrically conductive composite ceramics based on charge for facing tiles, the optimal amount of additive is 30% silicon carbide. concentrations of the additive samples after firing retained a sufficient amount of silicon carbide in its original form, which made it possible to effectively protect against the effects of electromagnetic radiation [25,26]. Taking into account the requirements for conductive additives [27-29], which are mainly used to obtain electrically conductive composite ceramic products with protective properties against the effects of electromagnetic radiation, the following additives were selected for research in the work: silicon carbide (TCLR – $5 \cdot 7 \cdot 10^{-6} \text{ deg}^{-1}$, $T_{pl} = 2700 \text{ }^\circ\text{C}$) and strontium titanate (TCLR – $9 \cdot 11 \cdot 10^{-6} \text{ deg}^{-1}$, $T_{pl} = 2080 \text{ }^\circ\text{C}$). The ceramic mass was chosen based on the minimum temperature and firing time, taking into account the possibility of oxidation, burnout, or interaction of the additive with the dielectric matrix. The technological scheme of the production process of composite ceramic tiles is presented in Fig. 1. The preparatory stage of the components of the ceramic mass must ensure that each of them meets the specified chemical and mineralogical composition, the required level of purity, as well as optimal physical parameters and humidity, which are critical for further processing. It includes the main technological processes of preparation, including: optimal physical parameters and humidity, which are critical for further processing. It includes the main technological processes of preparation, including:

- Enrichment or ordering of mineral raw materials, in particular washing with water;
- Initial grinding of raw materials;
- Classification of material by sorting, magnetic or sieve separation, chemical cleaning and other methods of removing unwanted impurities;

- Removal of excess moisture by drying to a level that allows efficient subsequent grinding;
- Pre-heat treatment (firing), which contributes to the necessary phase transformations, compaction of the material and elimination of volatile compounds.



Experimental composite ceramic tiles consist of three layers, the arrangement of which is schematically shown in Fig. 2. The encryption of the samples contained the following information: samples of ceramic tiles with the addition of silicon carbide and strontium titanate (SCTS), the first digit of the numerical component of the cipher indicates the amount of silicon carbide (3 – 30 %), and the second – the amount of strontium titanate (1 – 10 %, 2 – 20 %, 3 – 30 %). Charge compositions for I, II and III layers of ceramic tiles are shown in Table 1.

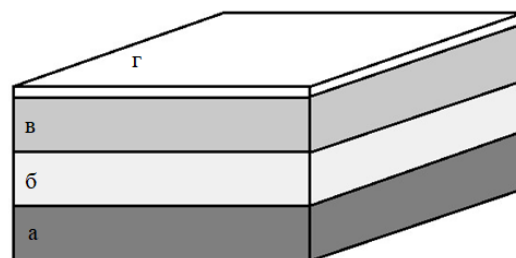


Fig. 2 – Schematic arrangement of layers in tiles:
a) I layer; b) Layer II; c) Layer III; d) Watering

Table 1 – Charge components for composite ceramic tiles

Name of raw materials	Mass content of materials, mass %				
	Layer I	Layer II			Layer III
		1	2	3	
Andriyivska Clay	29.93	37.31	33.62	29.93	41.00
Granite screenings	8.76	10.92	9.84	8.76	12.00
Crushed chalk	4.02	5.00	4.52	4.02	5.50
Quartz sand	24.31	30.30	27.30	24.31	33.30
Tile Battle	5.98	7.47	6.72	5.98	8.20
Silicon carbide	27.00	-	-	-	-
Strontium titanate	-	9.00	18.00	27.00	-

The press powder for each of the three layers (I, II and III) was kneaded separately using the required

amount of feedstock. The components were weighed, moistened, crushed in a ball mill, after which the resulting

slipper was dried in a drying oven, crushed and sieved through sieve No. 05.

At the first stage, the press powder for the first layer was brought to a moisture level of 8 %, the required mass was measured and poured into the mold for pressing, applying a pressure of 5 MPa. Further, they worked with layer II in a similar way: the press powder prepared to 8 % humidity was measured, added to the mold and for pressing, using a pressure of 10 MPa. At the final stage, the press powder for layer III (also with a moisture content of 8 %) was poured into the mold and pressed with a pressure of 18–20 MPa.

The resulting blanks were dried, after which they were covered with watering and placed in a drying cabinet. The final stage involved firing in a silite furnace at temperatures of 1120–1140 °C with isothermal exposure for 20 minutes.

Research results and discussion

To determine the parameters of the interaction of electromagnetic radiation with samples, upgraded standard equipment was used - a meter of the standing wave coefficient and attenuation as part of the generator unit P2-65 with an indicator Я2Р-67. The determination of the transmission coefficient was carried out according to the scheme shown in Fig. 3.

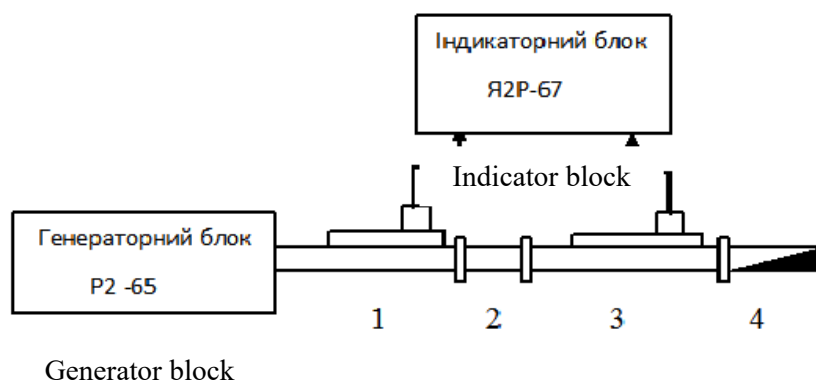


Fig. 3. Block diagram of the experimental installation for determining the transmission coefficient: 1,3 - directional branches; 2 - waveguide with a sample; 4 – Coordinated load

The results of studies of the electrodynamic characteristics of the developed composite ceramic tiles are presented in Table. 2.

Table 2 - Electrodynamic characteristics of developed ceramic materials

Composition code	Frequency, GHz	Nper, dB	Quidb, dB	Size, dB
SCTS-30	20	-12.4	-3.8	-18.8
	40	-14.1	-5.2	-21.1
SCTS-31	20	-12.1	-3.9	-20.1
	40	-13.7	-5.4	-23.6
SCTS-32	20	-11.8	-4.8	-20.5
	40	-13.3	-6.2	-25.8
SCTS-33	20	-11.6	-4.9	-20.7
	40	-13.4	-6.5	-25.9

The analysis of the data obtained indicates an improvement in the absorption properties of prototypes when an additional layer with strontium titanate is introduced into the composite ceramic tiles, however, with an increase in the concentration of SrTiO₃ from 20 to 30 wt.%, there are no significant changes in electrodynamic characteristics, the absorption coefficient reaches Kpogl – -25.9 dB at a frequency of 40 GHz.

The results of studies of electrodynamic indicators of materials confirm the legitimacy of using the developed composite ceramic tiles as materials for EMV attenuation in the millimeter wave range.

Conclusions and prospects for further development of this direction

The electrodynamic characteristics of electrically conductive composite ceramics based on facing ceramics with the addition of SiC and SrTiO₃ in the microwave range from 20 to 40 GHz have been investigated. Based on the studies carried out, the following conclusions can be drawn:

- experimentally shown that the reflection coefficient of the test samples in the range of 20-40 GHz increases with an increase in the concentration of the conductive additive from 10 to 30 wt.%. At the same time, the absorption coefficient of electromagnetic radiation

increases and ceramics passes to the class of radio-absorbing materials;

- developed and manufactured composite ceramic materials with the appropriate concentration of electrically conductive additives meet the basic requirements for the use of radio-absorbing materials and can be used to weaken the electrical component in the electromagnetic field of premises located in places with radio emission and in order to protect the environment from the outside of the premises where work is carried out using EMF.

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