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INFLUENCE OF THE FIRING TEMPERATURE ON THE DIELECTRIC PROPERTIES OF CERAMICS BASED ON BARIUM TITANATE

The object of the research is the firing temperature of ceramic materials based on barium titanate. In laboratory conditions, barium titanate was synthesized from raw materials of barium carbonate and titanium dioxide using ceramic technology, taking into account the stoichiometric composition of the compound. In order to study the effect of the firing temperature on the properties of the ceramic material, three temperatures were selected: 1270, 1300, and 1350 °C. The physical properties of the samples (imaginary density, water absorption, open porosity) were determined by the method of hydrostatic weighing in water. The samples were saturated with water after their preliminary evacuation. The dielectric characteristics of the obtained materials were measured on an E7-8CLR automated device (Ukraine) at a frequency of 1 kHz. The structural and morphological features of ceramics based on synthesized barium titanate were investigated by direct scanning electron microscopy and X-ray phase analysis. On the basis of the complex of studies carried out, the technological parameters of the production of ceramics were selected. Thus, the duration of grinding at the first and second stages is 10 and 30 minutes; moisture content of the press powder – 8 %; pressing pressure – 20 MPa; temperature of the first firing – 1000 °C; temperature of the second firing – 1350 °C. The regularity of the change in the dielectric constant on the firing temperature of ceramics based on barium titanate was established. The investigated samples, obtained according to the given technological regime, are characterized by the following indicators: dielectric constant – 259.9; open porosity – 0.02 %; water absorption – 0.01 %; imaginary density – 5.45 g/cm^3. The resulting material can be used to create composite ceramic materials that protect biological and technical objects from the effects of electromagnetic radiation, and can also be used to create new functional materials for space, aerospace, electronic engineering and medicine.

Keywords: *ceramic technology, semi-dry pressing, thermal sintering, dielectric constant, imaginary density, open porosity.*

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1. Introduction

Everyday human life takes place in conditions of high concentration of electromagnetic radiation:

– computer, office and home appliances;
– analog and cellular communication;
– medical equipment;
– terrestrial radio broadcasting;
– navigation systems and many other sources [1].

Exceeding the permissible levels of electromagnetic radiation for humans can lead to such disorders as fatigue, central nervous system disorders, clouding of the lens of the eye and others [2–4]. For electronic devices, these radiations adversely affect their technical condition, sometimes up to their failure, which in the spread of automated process control systems is extremely dangerous [5, 6].

The creation of new materials with new electrophysical properties largely determines the scientific and technological progress in space, aerospace, electronics and radio electronics. Currently, special ceramic materials, designed to absorb and transmit electromagnetic radiation in the radio frequency range, are increasingly used for these areas [7]. Materials with high values of dielectric constant are used to create effective data protection systems, design and installation of moonless cameras, ensure optimal settings of electronic equipment, security in the event of an electromagnetic field, masking equipment from radars and more.

The greatest interest is the creation of inorganic composite ceramics based on barium titanate ($BaTiO_3$) due to the relatively high and almost constant values of dielectric properties. According to various data, the dielectric constant in the wide frequency range of radio waves is $\varepsilon = 155–255$ at a frequency from 1 kHz to 10 GHz, in comparison with other known crystalline phases [8]. Barium titanate and its products are the most studied universal electroceramic materials due to their intensive use in multifunctional applications in the electronics and electrical industry in many countries [9–11]. Since the synthesis product is strongly influenced by the homogeneity of the composition, it is necessary to maintain the same particle size distribution of raw materials. To maintain the stable characteristics of the finished products, it is necessary to select the optimal technological parameters for the production of composite ceramics [12].

Based on this, *the object of the research* is the firing temperature of ceramic materials based on barium titanate. *The purpose of this work* is to develop technological parameters for the creation of ceramic materials with a given level of electrophysical characteristics that will ensure a long period of effective operation, including the absorption of electromagnetic radiation.

2. Methods of research

The calculation of the charge compositions of the masses for the synthesis of barium titanate was carried out taking into account the stoichiometric composition of the compound. The following raw materials were used for the research: barium carbonate GOST 2149-75 and titanium dioxide GOST 9808-84. The percentage of barium carbonate in the composition of barium titanate is, mass %: $BaCO_3$ – 65.75; TiO_2 – 34.25.

Under laboratory conditions, the raw materials in a given amount were weighed and wet ground in a planetary mill for 10 minutes at a speed of 400 rpm. The resulting slurry was dried in an oven to 1 % humidity, crushed and passed through a sieve No. 0063. Then formed briquettes and calcined them at a temperature of 1000 °C for 2 hours.

The obtained briquettes were manually ground to a fraction of 10–20 mm, after which wet grinding was carried out in a planetary mill for 30 minutes at a speed of 200 rpm. The resulting suspension was dried to a residual moisture content of not more than 1 % and the obtained press powder was passed through a sieve No. 0063.

Given that the components of the model masses do not have plastic properties, the formation of samples with a diameter of 50 mm and a height of 3 mm was carried out by semi-dry pressing on a hydraulic press at a pressure of 20 MPa with a total moisture content of 8 %. The dried samples were fired in a silite furnace in an oxidizing medium at temperatures of 1270, 1300, 1350 °C, at a maximum temperature of 2 hours. The calcined samples were subjected to machining for further study of electrophysical and physical properties.

The dielectric characteristics of the obtained materials were measured on an automated device «E7-8CLR» (Ukraine) in accordance with GOST 22372-77 [13]. The examination of the samples was performed at a frequency of 1 kHz. To ensure a tight fit of the electrodes, the samples for the research were ground.

The phase composition of the experimental samples was determined using the method of X-ray phase analysis using a diffractometer DRON-3M (Russia) with a Nickel filter and CuK_α-radiation under standard operating conditions. The study of the microstructure of the experimental samples was carried out by direct scanning electron microscopy using a scanning electron microscope PhenomPro (USA).

3. Research results and discussion

Under laboratory conditions, the physical properties of the obtained ceramics were studied by the method of hydrostatic weighing in water with preliminary evacuation of the samples for 15 minutes. The results of the studies on the measurement of the physical and dielectric properties for the samples, fired at different temperatures, are given in Table 1. The result was the arithmetic mean of three parallel tests with rounding to 0.01.

Table 1

Properties of barium titanate samples fired at different temperatures

T, °C	Dielectric constant	ρ_i, g/cm^3	*W*, %	P_o, %
1270	154.8	5.00	0.02	0.10
1300	159.4	5.12	0.02	0.08
1350	259.9	5.45	0.01	0.02

From the obtained data we see that with increasing firing temperature from 1270 to 1350 °C the indicators of water absorption and open porosity decrease to 0.01 % and 0.02 %, respectively, and the imaginary density of the samples increases to 5.45 g/cm^3. Also with increasing firing temperature there is an increase in dielectric constant. The improvement in the characteristics of the samples in this case can be explained by the fact that at a temperature of 1350 °C the intensive growth of barium titanate crystals begins.

The structural and morphological features of ceramics were studied for the samples, obtained at a firing temperature of 1350 °C. The results of the study of the phase composition of the ceramic mass are presented on the radiograph of Fig. 1.

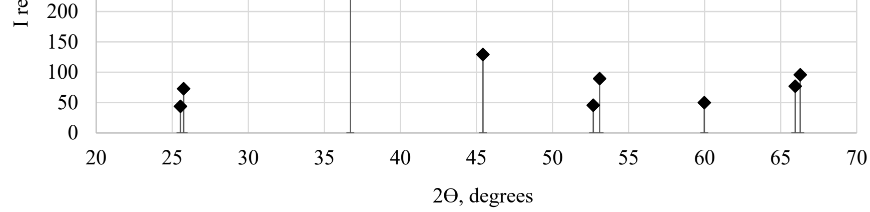
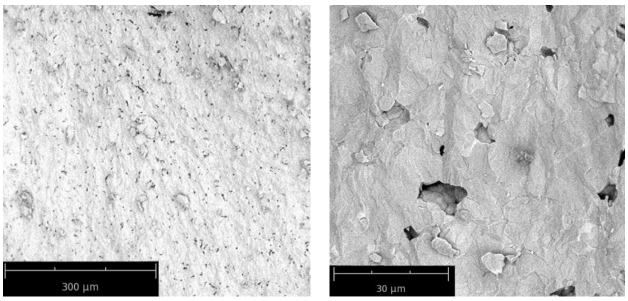


Fig. 1. Radiograph of the ceramic mass of barium titanate fired at temperature 1350 °C: ◆ – $BaTiO_3$

As can be seen from Fig. 1, the samples of the ceramic material contain only the $BaTiO_3$ phase, which indicates the complete course of the phase synthesis reaction. The microstructure of the obtained ceramics was investigated by scanning electron microscopy. Photomicrographs of fractures of the prototypes are shown in Fig. 2.



a *b*

Fig. 2. Microstructure of a sample of composite ceramics fired at a temperature of 1350 °C: *a* – magnification of 250 times; *b* – magnification of 2000 times

In the obtained images (Fig. 2) we see that the structure of the ceramics is homogeneous, without cracks and defects. The pore size is from 2 to 10 microns.

X-ray phase analysis and scanning electron microscopy revealed that the increase in the dielectric constant of the test samples, obtained at a firing temperature of 1350 °C, is due to the formation of the crystal structure of the barium titanate phase.

According to the research results, the composition and technology of obtaining ceramic materials based on barium titanate in laboratory conditions using the two-stage ceramic technology are proposed. In the first stage ($BaTiO_3$ synthesis), technical raw materials (barium carbonate and rutile) were used to create the charge in a stoichiometric ratio with a grain fraction of 50–60 µm. The briquettes were pressed under a pressure of 20 MPa, the calcination temperature of the briquettes was 1000 °C for 2 hours. The second stage involved firing the samples in a silite furnace in an oxidizing medium at a temperature of 1350 °C and holding for 2 hours, followed by cooling together with the furnace.

Further research is aimed at studying the dielectric properties of ceramic materials based on barium titanate in the frequency range 26–37 GHz. The research results can be used as a ceramic tile to attenuate the electrical component of the electromagnetic field inside the premises, located in the area of radio sources. It is also possible to use for environmental purposes, to reduce the intensity of the electromagnetic field outside the premises where the work with electromagnetic radiation.

4. Conclusions

The performed work allowed to solve the problem of obtaining composite ceramics based on barium titanate. Prototypes were made by the two-stage ceramic technology. The first stage of the synthesis took place at a temperature of 1300 °C with exposure to the maximum temperature for 2 hours. The second stage of firing took place at temperatures of 1270, 1300 and 1350 °C with exposure for 2 hours.

Based on the research, the optimal technological parameters of ceramic material production have been developed. Thus, the duration of grinding in the first and second stages – 10 and 30 minutes; humidity of press powder – 8 %; pressing pressure – 20 MPa; temperature of the first firing – 1000 °C; temperature of the second firing – 1350 °C. According to the results of the X-ray phase analysis, only the barium titanate phase is present in the samples, the structure is homogeneous and without defects, and the obtained material has the following characteristics: dielectric constant – 259.9; open porosity – 0.02 %; water absorption – 0.01 %; imaginary density – 5.45 g/cm^3.

The generalization of the obtained data allows us to conclude that the developed composition of the composite ceramic material based on barium titanate is promising for the creation of new functional materials with radio-absorbing properties.

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