The influence of titanium compounds on the electrophysical characteristics of the epoxy composite

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Abstract. This article discusses influence of titanium compounds on the electrophysical characteristics of materials. Sequence of operations for receive the materials possessing improved radio-technical characteristics is shown. Material characteristics such as permittivity, shrinkage and hardness are described. Higher permittivity \(\varepsilon\) of composites is presented and subsequence and modes fabrication body parts of products. Facts are given that testify that the insertion is 30 \% TiO\(_2\) raises permittivity by 1.4 times; interposition BaTiO\(_3\) in the same ratio - 1.7.

1. Introduction

Currently, invention of composites possessing a given set of functional characteristics is an urgent trend in the evolution of present materials science. Fundamental nature of the conclusions obtained in the study of these composites and, on the other hand, to their constantly growing practical application. For the evolution of microwave electronics, in technologies of which dielectrics are used, it is required to development materials with a large permittivity. Such materials must also have mechanical strength, moisture resistance, shock resistance, provide a wide range of operating temperatures [1].

In production, a material is used to manufacture the bodies of spiral antennas [2]. The antenna body made of this material has strength, small shrinkage, provides a temperature range from -60°C + 150°C [3]. However, to expand the frequency range of the antenna while maintaining a minimum size, it is necessary to use a material possessing a large permittivity. This parameter can be the best by introducing a filler with high \(\varepsilon\) values into this material [4]. Titanium compounds such as titanium dioxide (TiO\(_2\)) and barium titanate (BaTiO\(_3\)) can be considered as such additives [5, 6]. From the above it follows that introduced of titanium compounds is the material will allow to develop new composites possessing enhanced electrophysical characteristics.

Determination of permittivity mathematically of heterogeneous systems is conducted out according to the Lichtenecker formula [7]. This formula connects permittivity of the matrix and filler and their content and also predict the permittivity.

\[
\ln \varepsilon = v_1 \ln \varepsilon_1 + v_2 \ln \varepsilon_2
\]

(1)

where \(\varepsilon_1, \varepsilon_2\) are dielectric constant of the 1st and 2nd components;

\(v_1, v_2\) are volume fractions of the 1st and 2nd components.

A composite material filled with TiO\(_2\) was studied in [9]. However, a comparative study of electrophysical and mechanical properties of composite materials filled with titanium compounds has not been carried out.
In this regard, the main task of this research was to create composite materials filled TiO$_2$ and BaTiO$_3$, with improved radio engineering and mechanical characteristics, and a comparative analysis of their properties. And also checking the ability of fabrication structural parts from these composites.

2. Research part

Main objects of this research were: diane resin, diaminodiphenylmethane. Additives - titanium dioxide grade RK [5], barium titanate grade TBK-1 [6].

Fillers were introduced into the epoxy base in different proportions - TiO$_2$ and BaTiO$_3$. Then, within 5 minutes, the components were mixed in special equipment.

Composite materials filled with titanium compounds were obtained using this technology. These materials contained different amounts of fillers - 7, 15, 30%.

Next stage of work was production of details from composites with different contents of titanium compounds. Parts were made by high-temperature pressing. First of all, parts are made to check material characteristics. And then the product bodies were made by the same method. Technological parameters are given in table 1.

| Manufacturing parameters | Additive | TiO$_2$ | BaTiO$_3$ |
|--------------------------|----------|---------|-----------|
| Temperature range, °C    |          | 150 — 160 | 125 — 145 |
| Pressure range, MPa      |          | 15 — 25  | 20 — 35   |
| Preparation time, min    |          | 20       | 25        |
| - samples for measuring properties | | 16 | 20 |
| - antenna housing        |          |          |           |

After the manufacture of parts, thermal stabilization is mandatory. Stage is very important for the process as it relieves internal stresses of parts and allows the production of precision parts. Thermal stabilization is carried out at a temperature of 150 — 170 °C for 8 hours.

The definition of shrinkage of details was carried out following GOST 34206-2017 [10]. The water absorption of details was determined following GOST 4650-2014 (ISO 62: 2008) [11]. Also, with the help of certified equipment, the hardness of the parts was determined.

The permittivity was calculated using Lichtenecker formula (1). Since the antennas under study operate in microwave range, then permittivity was determined in it. To determine dielectric constant, we used samples for a waveguide (figure 1). Depending on the range of ε values, the height of the samples varied from 4 to 8 mm. It should be noted that the pressed flat parts were machined to size by mechanical means, while there were no chips or cracks, that is, the composite material is perfectly processed.
3. Results of the study
Table 2 summarizes generalized data on the study of the electrophysical and mechanical characteristics new substance. The first part of the table shows data on filling with titanium dioxide, and in the second - with barium titanate.

Table 2. Distinctive qualities of filled materials.

| Measures                  | Additives content, mass. % |                      |                      |                      |
|---------------------------|-----------------------------|----------------------|----------------------|----------------------|
|                           | 0                           | 7                    | 15                   | 30                   |
| TiO₂                      |                             |                      |                      |                      |
| Appearance and color      | Coarse powder of green color| Medium-dispersed powder of light green color | Fine white powder with a light green tint |
| Permittivity calculated   | 4.4                         | 4.9                  | 5.5                  | 6.5                  |
| Permittivity measured     | 4.4                         | 4.9                  | 5.4                  | 6.4                  |
| Shrinkage, %              | 0.5                         | 0.4                  | 0.3                  | 0.2                  |
| Shore hardness, HD        | 80                          | 81                   | 83                   | 85                   |
| Water absorption, %       | 0.09                        | 0.09                 | 0.08                 | 0.08                 |
| BaTiO₃                    |                             |                      |                      |                      |
| Appearance and color      | Coarse powder of green color| Medium-dispersed powder of light green color | Fine white powder with a light green tint |
| Permittivity calculated   | 4.4                         | 5.5                  | 6.8                  | 8.0                  |
| Permittivity measured     | 4.4                         | 5.1                  | 6.5                  | 7.6                  |
| Shrinkage, %              | 0.5                         | 0.2                  | 0.1                  | 0.1                  |
| Shore hardness, HD        | 80                          | 82                   | 84                   | 87                   |
| Water absorption, %       | 0.09                        | 0.09                 | 0.08                 | 0.07                 |

When finding permittivity the values of the matrix and filler are of great importance, therefore, formula (1) was used for calculations. The results of this study have demonstrated that introduction of even a small amount of titanium compounds significantly increases the permittivity of the composite. For compositions filled with titanium dioxide, permittivity rises with a large introduction to 6.5. For materials filled with barium titanate, the value of permittivity increases to 8.0. It should be noted that in the course of the study it was shown that the $\varepsilon$ was determined using the device and the $\varepsilon$ calculated are consistent. The dependences of the permittivity of the samples on the content of fillers are presented below.
Figure 2. Permittivity of composites:
I – measured data (filler TiO$_2$);
II – calculated by (1) formula (filler TiO$_2$);
III – measured data (filler BaTiO$_3$);
IV – calculated by (1) formula (filler BaTiO$_3$).

In previous studies, it was found that insertion of titanium dioxide, barium titanate increases permittivity of the press material [9, 12], however, this study found that when BaTiO$_3$ was used in a similar ratio as TiO$_2$, permeability additionally increases from 10 to 30 % as the filler content increases.

It was found that the introduction of titanium compounds insignificantly increases the hardness. A sample with a maximum TiO$_2$ content has 85 HD, and the hardness of a sample filled with BaTiO$_3$ in the same ratio is 87 HD.

It is shown that with an increase in the content of fillers, the water absorption index (water adsorption) decreases from 0.09 to 0.07 %. The diffusion of moisture into the filled composites decreases, respectively, these materials are more resistant to the external liquid medium, which expands the scope of their application.

Further, antenna elements were made of composite materials (see figure 3). Technological modes of pressing parts are presented in table 1. Composite samples are a homogeneous mass, and the resin evenly impregnates the fillers, thereby the composite material has excellent fluidity and evenly fills a structurally complex mold.
Figure 3. 3D model of the antenna body.

Shrinkage during molding was defined as difference between dimensions of the molded product and the cavity of the mold in which the molding was performed. Shrinkage value as it is filled with titanium compounds reaches: 0.2% for TiO$_2$, and 0.1% for BaTiO$_3$.

The made the product bodies of composites has no cracks, under-compressions, delaminations.

It was found the introduction of titanium compounds, such as TiO$_2$ and BaTiO$_3$ allows the manufacture of new materials possessing enhanced physico-mechanical and electrophysical characteristics.

The choice of filler between TiO$_2$ and BaTiO$_3$ for the creation of composite materials is determined only by the range of values of the properties of the final parts.

4. Conclusion
In the course of scientific investigation, potentiality of invention of new technological composites that can be used to obtain special parts is demonstrated.

Determined that insertion of 30 % by weight of titanium dioxide increases permittivity by 1.4 times; while filling with barium titanate in same ratio - by 1.7 times.

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