Dielectric Properties of Nanocomposites based on Epoxy Resins and Titanium Dioxide

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Abstract—In this work, polymer composite materials based on nanoparticles of titanium dioxide and epoxy polymer have been obtained. The dielectric properties of the obtained composite are investigated. Studies have shown that the introduction of TiO_2 nanoparticles into an epoxy polymer leads not only to a slowdown in relaxation processes, but also increases the value of the through conduction. It was shown that the greatest influence of the introduction of nanoparticles on relaxation processes is observed on the α-relaxation.

Keywords—Composites, resins, titanium dioxide, conductivity.

I. Introduction

HIGH-PERFORMANCE polymer composite materials are now finding more and more applications in our daily life [1]-[18]. A composite is usually a combination of a matrix and a filler; in the case of nanocomposites, the filler is nano-sized along one of the measurement axes [19]-[35].

Modern composites have not only a wide range of physical and mechanical properties, but are also capable of directionally changing them, for example, increasing fracture toughness, regulating rigidity, strength, and other properties. These possibilities are expanded when fibers of different nature and geometry are used in composites, i.e., when creating hybrid composites. In addition, these materials are characterized by the appearance of a synergistic effect (coordinated joint action of several factors in one direction).

The properties of the interface or interfacial zone, first of all, the adhesive interaction between the fiber and the matrix, determine the level of properties of composites and their retention during operation. Local stresses in the composite reach their maximum values just near or directly at the interface, where material destruction usually begins. The interface must have certain properties to ensure efficient transfer of the mechanical load from the matrix to the fiber. The adhesion bond at the interface should not be destroyed under the action of thermal and shrinkage stresses arising from the difference in the temperature coefficients of linear expansion of the matrix and fiber or as a result of chemical shrinkage of the binder during its curing.

When creating nanocomposites, the key tasks are the development of efficient, reliable, and affordable production technologies for mass production, which make it possible to obtain materials with stable characteristics. The hand lay technique, also called wet lay, is the simplest and most widely used process for producing flat reinforced composites. The process consists of laying layers of a polymer in successive layering using an epoxy matrix. Wet-laying is a molding process that combines layers of reinforced carbon fiber with epoxy to create a high-quality laminate. Before starting the installation process, you must prepare the appropriate form. This preparation consists of cleaning the table and applying a release agent to the surface. The manual laying process can be divided into four main steps: mold preparation, epoxy coating, laying and curing. Form preparation is one of the most important steps in the installation process. This process requires dry reinforcement layers and the application of a wet epoxy matrix. They are connected together - reinforcing material, impregnated with a matrix - epoxy resin.

Titanium dioxide is one of the promising materials as a nanofiller due to its optical, thermal, photocatalytic and electrophysical properties. The application potential of nanodispersed TiO_2 is very high: titanium dioxide and materials based on it can be used as an additive in plastics, an ultraviolet light blocker, an energy converter in solar batteries, an agent for photocatalytic degradation of bacteria and photochemical degradation of toxic chemicals, for wastewater treatment. Due to their chemical inertness, low toxicity, photocatalytic activity, high refractive index and other beneficial properties, titanium dioxide (TiO_2) nanoparticles have attracted the attention of many researchers and are used in the food, paint and varnish industry, etc. Previous studies have shown that the introduction of TiO_2 nanoparticles improves some properties of epoxy resin. But the process of interaction and the mechanism of hardening of epoxy resin are not fully understood [44]-[52]. There are few works on epoxy nanocomposites with TiO_2 nanoparticles; therefore, it is relevant to obtain new examples of such nanocomposites and study their physical and mechanical properties, since due to the presence of TiO_2, it is possible to use such nanocomposites in biomedicine, as bactericidal and photocatalytic surfaces. The aim of this work is to create an epoxy nanocomposite based on TiO_2 nanoparticles and to study its physical and mechanical properties depending on the concentration of nanoparticles.

The matrix can be a thermosetting polymer - epoxy resin, which has already found many applications: from structural composites to adhesives and surface coatings. Epoxy resins already have a number of unique qualities among polymers: no shrinkage during curing, high adhesion to various substrates, good dielectric and other valuable properties [36]-[44]. Nanocomposites using thermoplastic polymers are well known and studied to improve mechanical, electrical, thermal and insulating properties [45]-[78]. However, nanocomposites using thermosetting polymers have not been studied as widely, especially using TiO_2. The aim of this study was to investigate the dielectric properties of nanocomposites based on epoxy resin and titanium dioxide nanoparticles in view of their potential application as construction and functional materials.

II. Numerical Calculations of the Dielectric Properties of Polymer Nanocomposites

To identify the elastic properties of a monolayer from the known values of the elastic moduli of composite samples with different reinforcement schemes, we will use the classical model of layered composites.

An analytical calculation to determine the effective elastic properties of a monolayer will be carried out in the Digimat-MF module using the Mori-Tanaka averaging method. Separately, we will also evaluate the effect of the filler on the elastic modulus of the matrix, considering the material containing only nanoparticles as inclusions. Based on these calculations, we will determine the "effective" volumetric content of inclusions, taking into account the influence of interphase layers formed around the inclusions. The influence of these layers, as will be shown below, cannot be neglected, since in this case underestimated values of the elastic characteristics of the matrix will be obtained. Therefore, in fact, knowing the modulus of elasticity of the nanomodified matrix from experiments, the content of the filler will be selected such that the calculation and experiment will coincide. The found value of the effective volumetric content of inclusions is further used in analytical and numerical calculations of the properties of a monolayer.

Numerical calculations will be carried out using the Digimat-FE module. The size of a cubic representative fragment was set by the system automatically. The effective elastic properties were calculated by determining the ratio of the volume-averaged representative fragment of the stress level to a given value of homogeneous deformations. In this case, the boundary conditions and the geometry of the fragment itself are periodic. The calculations were carried out using the finite element method.

Nanoparticles, even with a very low volumetric content (less than 1%), are contained in such a fragment in a very large amount, and it is impossible to model their effect at this scale level. For example, a cubic fragment of a 1 μm matrix contains more than thousand nanoparticles for a given volumetric content. Therefore, in particular, the nano-modified binder is white, while the usual binder is yellow. To model such materials, it is necessary to resort to multiscale approaches and to carry out a consistent determination of effective properties at various scale levels. This task is greatly simplified if the properties of the nanomodified matrix are known from experiments. In particular, it is known that its Young's modulus is 2.5 GPa. The missing characteristic is Poisson's ratio, which can be approximately taken unchanged, or estimated on the basis of analytical calculations using the found value of the "effective" volumetric content of the filler, which was done. Further, it suffices to numerically solve the averaging problem on a representative fragment containing only nanoparticles.

To obtain an analytical assessment of the properties of the nanomodified sample, the “effective” volumetric content of nanoparticles in the matrix was preliminarily determined, at which the calculated value of the effective Young's modulus of the nanomodified matrix coincides with the known experimental value (2,5 GPa). It was found that if we do not take into account interfacial effects, then the calculation predicts the effective Young's modulus equal to 2,04 GPa, with an initial value of 2 GPa. That is, with such a low content (1%) of even very hard inclusions, they should not have a significant effect on the properties of the material. The experimentally found increase in the matrix modulus can be explained by the influence of hardened and rigid interphase zones formed around the inclusions. For an approximate assessment of their influence, the concept of "effective" volumetric content of inclusions is introduced. It is assumed that the properties of the interphase zones and inclusions are the same, and the calculation should use the value of the "effective" volumetric content of inclusions, which is the sum of their real volumetric content and the content of interfacial zones. This value was approximately 10% for the considered composite. In this case, the predicted Young's modulus of the modified matrix is 2,5 GPa, Poisson's ratio is 0,39, and the thermal expansion coefficient is $5{,}76 \cdot 10^{-5}$.

Based on the results of experimental studies, the effective characteristics of a monolayer made of a composite material based on both conventional and nanomodified matrices were determined, and a solution to the corresponding inverse problem was obtained. It was found that the addition of nanoparticles within the recommended standard range of 10% leads to a slight increase in the longitudinal elastic modulus and shear modulus of the monolayer. In this case, there is an almost twofold decrease in the elastic modulus in the transverse direction and a decrease to zero Poisson's ratio. The reliability of the developed numerical models is confirmed by a good correlation between the results of both numerical and analytical solutions and the obtained experimental data on the study of the thermomechanical characteristics of nanomodified materials.

The effect of TiO_2 nanoparticles with concentrations from 0 to 3% by weight on the electrical conductivity and dielectric characteristics of the epoxy polymer in a wide range of frequencies and temperatures was studied.

The image (Figure 1) shows the dependence of the imaginary ε'' part of the complex permittivity $\varepsilon^* = \varepsilon' - j\varepsilon''$ on the frequency and temperature in the original polymer film.

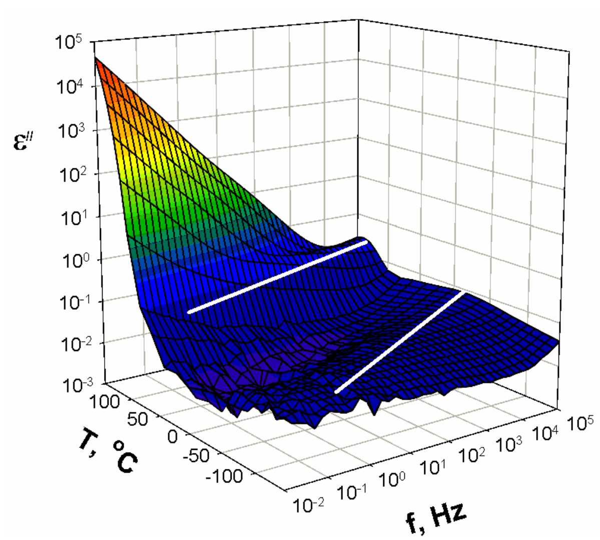

Fig. 1 Dependence of ε'' on frequency and temperature in epoxy resin.

It can be seen from the figure that a complex relaxation pattern is observed in the measured range of frequencies and temperatures, due to comparable contributions to ε* of electric dipole relaxation and electrical conductivity $\sigma^* = \sigma' + j\sigma''$ (σ' - is real, σ'' - the imaginary part of the specific complex conductivity) (observed at high temperatures). There are two main relaxation peaks. Both peaks shift to higher frequencies with increasing temperature. The image (Figure 2) shows the dependence of the low-temperature relaxation peak P1 for different contents of TiO_2 nanoparticles in the epoxy oligomer. It can be seen that, upon the addition of nanoparticles with a concentration of 3% by weight, a shift of the peak towards lower frequencies is observed.

III. Results of the Experimental Study of Dielectric Properties of Polymer Nanocomposites

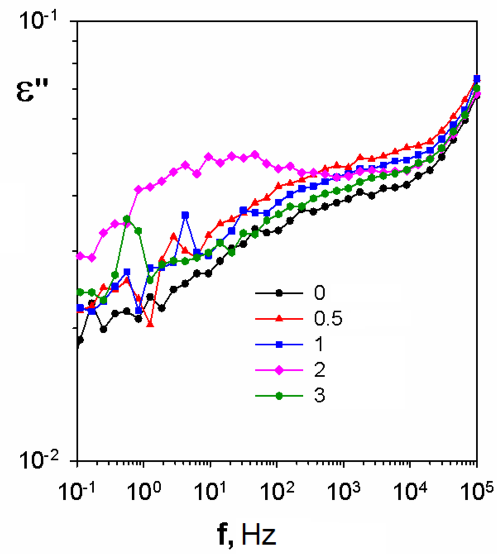

Fig. 2 Dependence of ε'' on frequency in an epoxy polymer containing different concentrations of TiO_2 nanoparticles (wt.%). Measurement temperature -60 °C.

The second relaxation peak P2 (Figure 3) is observed at higher temperatures. It can be seen that a shift of the relaxation peak towards lower frequencies is also observed for different contents of TiO_2 nanoparticles in the epoxy polymer.

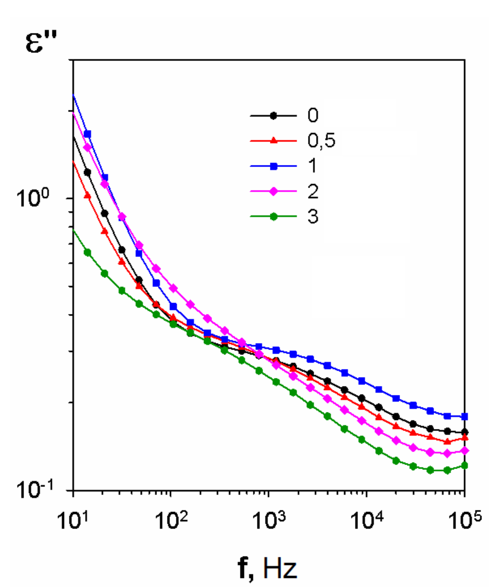

Fig. 3 Dependence of ε'' on frequency in an EP polymer containing different concentrations of TiO2 nanoparticles. Measurement temperature 120 °C.

At high temperatures, measurements of more than 100 °C at low frequencies on the ε(f), dependence, in addition to the through conduction, there is a step, which with an increase in the measurement temperature shifts towards higher frequencies (Figure 4).

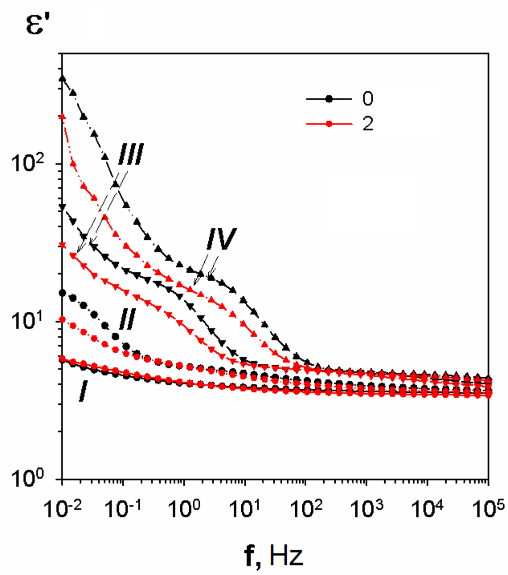

Fig. 4 Dependence of ε/ on frequency in epoxy polymer (black curves) and polymer containing 2% TiO2 nanoparticles (red curves) for different temperatures. I - 373; II - 383; III - 403; IV - 423 K.

From the Fig. 4 it can be seen that when nanoparticles are introduced, ε shifts towards lower frequencies. A weakly pronounced maximum of P3 was also observed in the ε'' (f) curves.

The image (Figure 5) shows the dependence of the real part σ' of the complex electrical conductivity σ* = σ'+ *j*σ''.

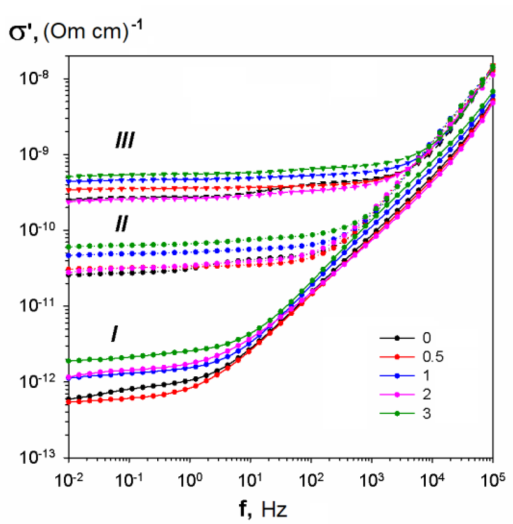

Fig. 5 Dependence of σ' on frequency in an epoxy polymer containing different concentrations of TiO2 nanoparticles for different temperatures: I - 110; II - 130; III – 150 °C.

It can be seen that a plateau corresponding to the through conduction is observed in the figure at low frequencies. With an increase in the concentration of introduced nanoparticles, the value of the through conduction increases.

Thus, our studies have shown that the introduction of TiO_2 nanoparticles into the ED-20 epoxy polymer leads not only to a slowdown in relaxation processes, but also increases the value of the through conduction. According to the position of peak P1 on the temperature axis, it should be attributed to β-relaxation, peaks P2 and P3 - α- relaxation.

As it can be seen from the data obtained, the greatest influence of the introduction of nanoparticles on relaxation processes is observed on the α- relaxation. And since α-relaxation occurs with the participation of the free volume of the substance, it is natural to assume that the introduction of TiO_2 nanoparticles leads to a decrease in the free volume. The presence of a wide peak of β-relaxation may indicate that a wide range of side groups are involved in relaxation.

IV. Conclusion

Studies of the dielectric properties have shown that the introduction of TiO_2 nanoparticles into the ED-20 epoxy polymer slows down the relaxation processes and also increases the through conduction regardless of temperature. The greatest influence on relaxation processes is observed during α-relaxation.

This opens new prospective for tailored fabrication of polymer nanocomposites with desired structure and properties. Further research can be aimed toward the development of nanocomposites with advanced mechanical properties.

To determine the structure of the obtained nanocomposite, the method of IR spectroscopy can be further used. IR spectroscopy allows first to get evidences of the interaction between nanoparticles and polymer matrix and, second, get the information about the structure of the resulting nanocomposite.

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Contribution of Individual Authors to the Creation of a Scientific Article (Ghostwriting Policy)

A. Getmanov synthesized the nanocomposites.
Tran Quyet Thang studied the properties of nanocomposites.