Improvement of Mechanical Properties of Polymer Materials by the Nanosized Ceramic Particles

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Abstract—As a result of the experiments, epoxy nanocomposites based on titanium dioxide nanoparticles in the form of opaque blue-gray films were obtained. The composition and structure of epoxy nanocomposites were studied by scanning electron microscopy and infrared spectroscopy. The properties of the obtained film nanocomposites were investigated to determine the glass transition temperature, and the mechanical properties of the films were tested in tension, where the tensile strength, elastic modulus, and relative deformation were determined.

Keywords—Composites, mechanical properties, polymers, nanoparticles.

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1. Introduction

EPOXY resins are commonly used as polymer matrices in high performance composites due to their good heat resistance, durability, electrical, chemical and mechanical properties. However, its range of applications is limited due to its fragility and poor toughness. One of the ways to overcome this problem is to change the properties of epoxy matrices by introducing a rigid filler into the polymer matrix [1],[2],[3],[4],[5],[6],[7],[8],[9],[10],[11],[12]. A new approach in nanotechnology proposes the use of fillers at the nanometer scale, since nanofillers have a large surface area, which makes them chemically very reactive and helps them bind better to the matrix. Nanoparticles embedded in a polymer matrix are attracting more and more interest due to the unique mechanical, optical, electrical and magnetic properties exhibited by nanocomposites and significantly improve them [13],[14],[15],[16]. These improvements depend on the volumetric content of the filler as well as the type and characteristics of the nanofillers [17],[18],[19],[20],[21],[22],[23],[24],[25],[26],[27],[28],[29],[30],[31],[32],[33]. Nanofillers of a metallic or inorganic type can be considered an excellent candidate for hardening polymer matrices [34],[35],[36],[37],[38],[39],[40],[41],[42],[43].

The development of modern technology requires the creation of new structural materials with high elastic-strength characteristics, and, on their basis, structures with more effective weight data. The creation of polymer composites based on nano-modified binders has been one of the priority areas of research in the field of composite materials manufacturing technologies for many years [1],[2],[3],[4],[5],[6],[7],[8],[9],[10],[11],[12]. Significant progress has been made in this area [13],[14],[15],[16],[17],[18],[19],[20],[21]. The development of composite materials that improve their operational limits is based on the reinforcement of two or more fibers into a single polymer matrix, which leads to an improved material system called hybrid composites with a wide variety of material properties [22],[23],[24],[25].

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 (TiO_2) 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 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. 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

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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 [44],[45],[46],[47],[48],[49],[50],[51],[52],[53],[54],[55],[56],[57],[58],[59],[60],[61],[62],[63],[64],[65],[66],[67],[68],[69],[70],[71],[72],[73],[74],[75],[76],[77]. Nanocomposites using thermoplastic polymers are well known and studied to improve mechanical, electrical, thermal and insulating properties. However, nanocomposites using thermosetting polymers have not been studied as widely, especially using TiO_2.

Nanoparticles are usually introduced into the polymer matrix using various methods. Dispersion processes are necessary in order to transfer nanoparticles from an agglomerated state to a uniformly dispersed state [26],[27],[28],[29],[30],[31],[32],[33],[34]. The most popular are live streaming with the use of chemical methods and the use of high shear forces in the process of mechanical dispersion of the powder. Chemical methods are capable of generating individual and non-agglomerated nanoparticles within a thermosetting or thermoplastic polymer. For mechanical dispersion, ultrasonic treatment is often used, which also improves the dispersion state of nanoparticles.

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.

2. Modelling the mechanical properties of nanocomposites

We use the model of spherical inclusions to model the properties of the filled matrix, assuming that the reinforcing particles of titanium dioxide are spheres. We assume that the particles are absolutely solid and do not collapse (the upper estimate). Bulk content 0,6%. We use the Digimat - MF module, the averaging method of Mori - Tanaka. Strength criterion - according to the maximum principal stresses acting in the matrix.

If the initial volumetric content of inclusions is set to 0.6%, the model predicts that the properties of the matrix will not change, since there are too few inclusions. The effect of the interfacial layer must be taken into account. For this, we will carry out a calculation with the setting of the effective volumetric content (volumetric content of the filler + volumetric content of the interfacial layer, under the assumption that their properties are equal). Let us select the effective volumetric content that allows us to describe the obtained experimental data in relation to the elastic modulus and ultimate strength.

If we select according to ultimate strength, then the effective volumetric content of inclusions should be 50%, and the modulus of the composite should be 6 GPa according to the calculation. If we select by modulus, then the effective volumetric content of inclusions should be 11%, and the ultimate strength of the composite according to the calculation should be 23 MPa.

To describe the experiment, we can assume that the effective volumetric content of inclusions is 11% (we obtain the coincidence of the calculation and experiment in modulus), and the strength of the matrix increases when a filler is added up to 30 MPa (we obtain a coincidence of the calculation and experiment in strength). The diagram of $\sigma - \varepsilon$ samples with different volumetric content of inclusions (in "DIGIMAT-MF"), (green-50%, blue-11%, red-0%) is presented in Fig. 1.

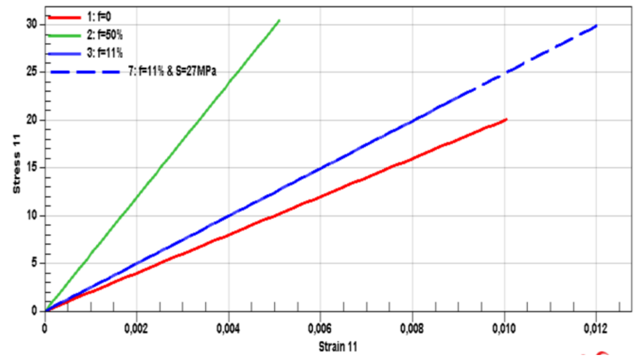

Fig. 1 Diagram of $\sigma - \varepsilon$ samples with different volumetric content of inclusions (in "DIGIMAT-MF"), (green-50%, blue-11%, red-0%).

For the found volumetric content of inclusions, by selection, we determine what the volumetric content of inclusions should be, so that the calculation and the experiment on measuring the CTE of the composite coincide. For 11%, we find that the CTE of the filler (and the surrounding interfacial layer) should be $85 \cdot 10^{-6}$ s^{-1}. The obtained high value of the CTE of the filler and the experimentally established phenomenon of an increase in the CTE of composites with a nanomodified matrix may be associated with a change in the structure of the polymer matrix or may be a consequence of the ongoing chemical reactions between the filler and the matrix.

The initial data for modeling the process of degradation of the mechanical properties of the test samples are the characteristics of the monolayer.

"DIGIMAT" program is designed for fast and highly accurate prediction of the nonlinear behavior of multicomponent materials, such as plastics, polymers, carbon and fiberglass, nanomaterials, etc., for accurate assessment of the local and global behavior of multicomponent structures using the finite element method, for preparing storage and confidential exchange of material models, for easy and highly efficient design of honeycomb sandwich panels. Also "DIGIMAT" presents to the user a number of interfaces for finite element software systems of computer engineering ("ANSYS", "LS DYNA", "SIMULIA / Abaqus", etc.), intended for computer modeling and research of problems of mechanics of a deformable solid body, mechanics of structures and software systems for finite element modeling of plastic molding processes ("MOLDEX3D", "MOLDFLOW", etc.).

Fig. 3 shows the σ–ε diagrams obtained as a result of finite element analysis in conjunction with "DIGIMAT". Similarly, the diagram of stress-strain for unidirectional specimen and specimen with packing in «DIGIMAT MF» is presented in Fig. 2.

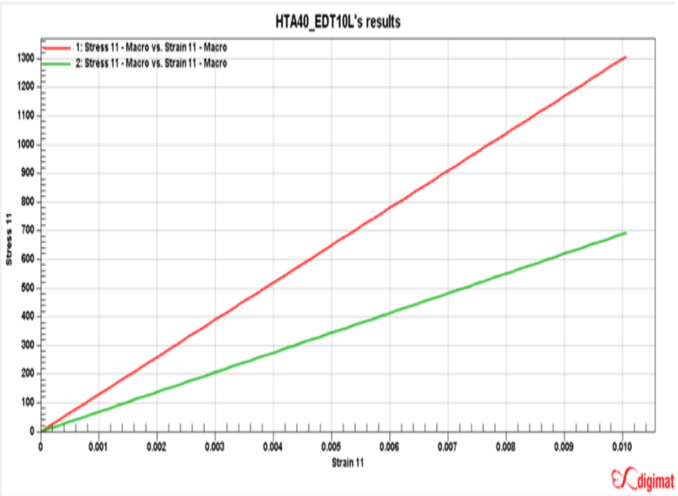

Fig. 2 Diagram of stress-strain for unidirectional specimen and specimen with packing in «DIGIMAT MF».

Two analyzes were given: a unidirectional sample and a sample with longitudinal transverse packing. These diagrams exactly matched the diagrams obtained when testing these two types of samples.

3. Investigation of the physical and mechanical properties of nanocomposites

Tests carried out by the method of differential scanning calorimetry with an increase in temperature from 20 to 200 ^{o}C and a test speed of 5 ^{o}C/min in a nitrogen atmosphere gave the result shown in Fig. 3. As it follows from Fig. 3, the glass transition temperature of the obtained epoxy nanocomposites weakly depends on the TiO_2 concentration, however, there is a tendency to a slight decrease: at concentrations less than 1 wt. % the glass transition temperature decreases to 167 °C, and at concentrations up to 5 wt. % rises and reaches 173 °C. Such a decrease in the glass transition temperature may indicate a change in the viscosity and elasticity of the system, an increase in the flexibility of molecules and their mobility, and a further increase in the concentration of nanoparticles leads to an increase in the rigidity and, consequently, to an increase in the glass transition temperature of the system.

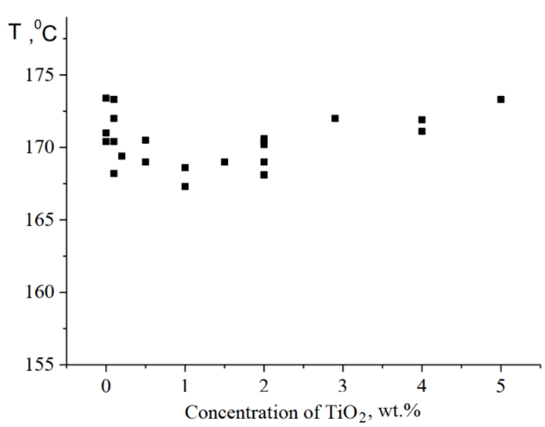

Fig. 3 Dependence of the glass transition temperature of TiO_2/EP nanocomposites on the concentration of TiO_2.

Based on the results obtained, it can be concluded that the presence of titanium dioxide nanoparticles in the system leads to a slight decrease in the glass transition temperature at a concentration of 1% TiO_2.

In physical and mechanical tensile tests, the efficiency of filling the composite with TiO_2 nanoparticles was monitored according to the following parameters: tensile strength (Rm, MPa), elastic modulus (E, GPa), relative deformation (ε, %). The test included control samples not modified with titanium dioxide and samples of nanocomposites with different contents of TiO_2 nanoparticles. The size of the samples was 10×90 mm, the thickness was 0,08 mm. The tensile test speed was 2 mm/min. The samples were securely fixed in rubberized clamps and a load was applied until complete destruction. Fracture occurred in the middle of the sample. After carrying out all the necessary tests and calculations, diagrams of the dependence of strength, modulus, and deformation on the concentration of TiO_2 nanoparticles were obtained. As shown in Fig. 4, tensile strength drops sharply at n-TiO_2 concentrations greater than 3 wt. %, which is possibly associated with secondary processes of agglomeration of n-TiO_2. Therefore, the working concentration should not exceed 3 mass. % n-TiO_2.

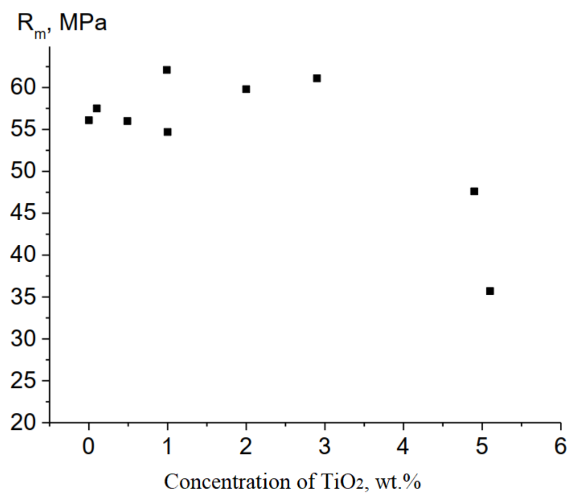

Fig. 4 Dependence of the tensile strength of n-TiO2/EP nanocomposite on the concentration of TiO2.

The dependence of the modulus of elasticity on the concentration of TiO_2 is given in Fig. 5. At concentrations above 1 wt. % increase in the modulus of elasticity is 20-25%, which indicates the effect of antiplasticization of the nanocomposite.

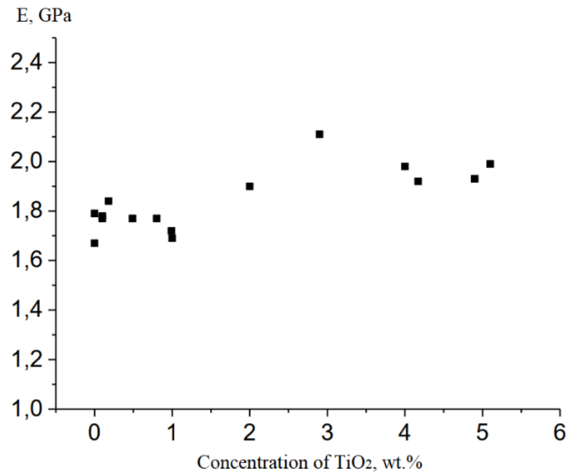

Fig. 5 Dependence of the modulus of elasticity of TiO2/EP nanocomposite on the concentration of TiO2.

The change in the relative deformation with varying nanofiller content is complex: the deformation increases at TiO_2 concentrations less than 1 wt. %, and decreases at TiO_2 concentrations above 1 wt. %. The dependence of the relative deformation of the TiO2/EP nanocomposite on the TiO2 concentration nanocomposite on the TiO2 concentration is presented in Fig. 6.

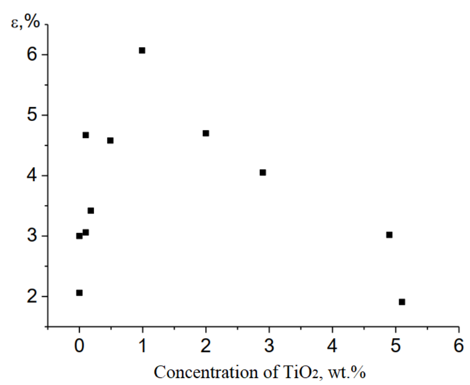

Fig. 6 Dependence of the relative deformation of the TiO2/EP nanocomposite on the TiO2 concentration.

Thus, the mechanical properties depend on the concentration of TiO_2 nanoparticles. The values of the modulus of elasticity and tensile strength are maximum at a nanoparticle concentration of 3 wt. %. The relative deformation increases with increasing concentration up to 1 wt. %.

4. Discussion of the results obtained

The obtained value of the average Young's modulus of the packet differs from the test. It is known that when using test data for a unidirectional material in the calculation of the properties of a layered package, errors can occur, therefore, it is usually necessary to use data on the stiffness of several versions of packages with different layering of layers. If using the values of the modules, then it is not possible to describe the test data. In this work, for the properties of a monolayer, we will use an overestimated value of the transverse modulus equal to 28 GPa, which is higher than the experimental data obtained for unidirectional samples (6,5 GPa). In this case, it is possible to reliably describe the obtained experimental data on Young's modulus of composite samples with symmetric packing.

The conducted studies allowed to investigate residual deformations in panels with an asymmetric reinforcement scheme based on the obtained analytical solution, as well as numerical modeling. Comparison of the results of analytical and numerical solutions with the obtained experimental data confirms the reliability and validity of the developed mathematical models and research methods for effective thermomechanical characteristics and residual stress-strain state of panels made of layered nanomodified materials.

5. Conclusion

Nanocomposites based on ED-20 epoxy resin and titanium dioxide nanoparticles have been obtained by introducing finished nanoparticles into epoxy resin at the stage of its curing, and the effect of nanoparticle concentration on physical and mechanical properties has been investigated. The physical and mechanical properties of epoxy nanocomposites have been investigated. It is shown that the introduction of 1 mass. % TiO_2 nanoparticles leads to an increase in the relative deformation by 35-40%. The maximum tensile strength values are achieved at a concentration of 3 wt. % TiO_2. The increase in the elastic modulus is 20-25% at TiO_2 concentrations above 1%. Improved mechanical properties indicate the formation of strong interfacial interactions.

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. For the details of the IR characterization of the polymer nanocomposites, it is referred to the literature [59],[60],[61].

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