Synthesis and Structure of Nanocomposites based on Linear Polymers and Nanoparticles of Titanium Dioxide

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Abstract—Nanocomposites based on titanium dioxide and epoxy polymer nanoparticles have been obtained and investigated by the in situ method at the stage of curing with preliminary ultrasonic dispersion and evacuation. The composition and structure of the obtained TiO₂ nanocomposites have been studied by IR spectroscopy and scanning electron microscopy. It is shown that with an increase in the content of nanoparticles, their average size increases to 88 nm at a TiO₂ concentration of 1% as a result of secondary aggregation processes.

Keywords—Nanocomposites, polymers, nanoparticles, titanium dioxide.

I. INTRODUCTION

To improve the existing and introduce new properties of the matrix, it is often reinforced with various fillers. The addition of micro-sized particles often has a negative effect on some properties, for example, impact strength. A new approach to solving this problem is associated with nanotechnology and the use of fillers on the nanometer scale [1]-[9]. Nanoparticles embedded in a polymer matrix are attracting more and more interest due to the unique mechanical, optical, electrical, magnetic, and other properties exhibited by nanocomposites [10]-[17]. Due to the nanometer size of these particles, their physicochemical characteristics differ significantly from those of molecular materials. Nanoparticle-reinforced polymers combine the properties of both the main polymer matrix and discrete nanoparticles. By

controlling parameters such as the rate of rejection and the amount of nanoparticles, the final properties of the nanocomposite can be influenced [18]-[25].

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).

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.

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 (TiO₂) is one of the promising materials as a nanofiller due to its optical, thermal, photocatalytic and electrophysical properties. The application potential of nanodispersed TiO₂ 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 TiO₂ 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 TiO₂ 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 TiO₂, 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 TiO₂ 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]-[64]. 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 TiO₂.

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]-[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.

II. METHODS FOR PREPARING TIO₂-BASED NANOCOMPOSITES

Methods for the synthesis of nanocomposite materials (or nanocomposites) are divided into two main large approaches: in situ and ex situ. The creation of nanocomposites by the in situ method makes it possible to simultaneously (in one stage) obtain both a matrix and nanoparticles, obtaining a nanocomposite at the output. The advantage of the method is that it prevents particle agglomeration while maintaining a good spatial distribution in the polymer matrix. The main disadvantage of the method is that all the products of the synthesis of nanoparticles remain in the nanocomposite, which can deteriorate the quality and purity of the obtained material.

When using the ex situ method, each stage of nanocomposite creation is brought into a separate process: from the synthesis of nanoparticles to the preparation of a nanocomposite. This method is more energy and labor intensive compared to in situ, and requires much more time. Also, special attention should be paid to the dispersion of the nanocomponent in the matrix, since during long-term storage nanoparticles are collected into larger ones and they must be dispersed by various methods, for example, by ultrasonic treatment [1]-[8]. The ex situ synthesis method is more suitable for large scale industrial applications than the in situ method.

The ex situ method, despite all its drawbacks, is used more often than the more technologically advanced in situ method. First of all, this is due to the simplicity of the approach based on the fact that in such reactions the synthesis of nanoparticles is not complicated by additional reactions with the polymer matrix, the resulting nanocomposite will not contain byproducts associated with the formation of nanoparticles in the polymer matrix, etc.

III. STRUCTURE AND PROPERTIES OF TIO₂-based NANOCOMPOSITES

The structure of nanocomposites, as well as properties, directly depend on many factors, such as: the type of particlematrix bond, mass composition, modification of the particle surface, size and dispersion of particles in the matrix, etc. As a result of composite synthesis, the properties of the matrix can improve, deteriorate, or acquire new properties.

Polymer nanocomposites reinforced with inorganic nanoparticles attract considerable interest due to their light weight, low cost, and controlled physical and mechanical properties. They find their application from the paint and varnish industry to the aviation and aerospace industries. For their practical application, it is necessary to create new materials based on them with the necessary physical and mechanical characteristics. In this regard, studies aimed at improving the mechanical and functional properties of nanocomposite materials based on epoxy polymers and revealing the role of nanofillers in providing the required characteristics are important.

Nanocomposites based on $TiO_2/epoxy$ polymer nanoparticles were obtained by the in-situ method at the stage of curing an epoxy resin with pre-dispersed titanium (IV) oxide nanoparticles in an epoxy resin matrix. To improve the dispersion of the introduced nanoparticles in a viscous epoxy resin, we used the method of ultrasonic treatment at a frequency of 40 kHz for 10 min. The curing process was carried out in a two-stage temperature conditions at 90 °C - 3 h, 160 °C - 3 h.

The elemental composition of the obtained nanocomposites was investigated using energy dispersive X-ray spectroscopy. The X-ray diffraction pattern of the synthesized nanocomposites (Figure 1) showed the presence of crystalline structures. Diffraction peaks are narrow with high intensity.



Fig. 1 edrs spectrum of TiO₂/EP nanocomposite.

As the data shows, no titanium oxide was found in the pure epoxy film (Figure 2), which confirms its purity (results presented are expressed as percent by weight). The opposite is shown by the results of analyzes of nanocomposite samples containing titanium dioxide (Figure 3, 4). The data obtained make it possible to assert the presence of titanium dioxide in the samples.

Spectrum 2	Spectrum	C	0	Ti	Total
	Spectrum 1	82.91	17.09	0.00	100.00
Spectrum 3 Spectrum 1	Spectrum 2	83.18	16.82	0.00	100.00
- 2µm Electron Image 1	Spectrum 3	79.64	20.36	0.00	100.00

Fig. 2 edrs results for a pure epoxy sample.

	Spectrum		0		Total
Excton Imge 1	Spectrum 1	74.95	20.74	4.31	100.00
	Spectrum 2	79.04	20.96	0.00	100.00
	Spectrum 3	90.48	9.52	0.00	100.00

Fig. 3 edrs results for TiO₂/EP composite with titanium mass content of 0.5%.

	Spectrum		0		Total
0	Spectrum 1	76.48	19.51	4.01	100.00
Spectrum 3	Spectrum 2	83.89	16.11	0.00	100.00
Spectrum 2	Spectrum 3	86.15	13.85	0.00	100.00

Fig. 4 edrs results for TiO₂/EP composite with 1% titanium mass content.

Further research was carried out on a Fourier transform spectrophotometer, which turned out to be more indicative in our study. Figure 5 shows the absorption spectra for pure titanium dioxide, pure epoxy resin and nanocomposites with a mass content of 1% and 4% nanofiller.



Fig. 5 Absorption spectra of IR spectroscopy with Fourier transform for samples containing 4%, 1% and 0% TiO₂ and pure TiO₂.

In the region of 3600-3200 cm⁻¹, one broad weakly pronounced absorption band of stretching vibrations of -OH groups of the matrix is observed, and hydroxyl groups on the surface of TiO₂ particles can also appear here. The -OH groups also include the peak at 1629 cm⁻¹, some literature indicates that the peak at 1629 cm⁻¹ also indicates the presence of hydroxyl groups on the surface of titanium dioxide. Adsorbed -OH ions play a central role in photocatalysis as they capture charge carriers to produce reactive -OH radicals, which are the driving force behind the photocatalytic process. In addition, they act as adsorbents for pollutant molecules. The epoxy groups of the matrix manifest themselves at weakly pronounced peaks at 840, 907 cm⁻¹ (valence asymmetric) and 1268 cm⁻¹ (valence symmetric). This indicates that after curing, a small amount of unopened rings remains during the polymerization reaction of epoxy groups. Vibrations of the -NH- group are not manifested, since nitrogen becomes tertiary as a result of the reaction and manifests itself in stretching vibrations of C - N bonds at a peak of 1125 cm⁻¹, which indicates a past polycondensation reaction between the hardener and epoxy groups. A number of weak peaks in the range of 2950-2870 cm⁻¹ are attributed to symmetric and asymmetric vibrations of the -CH₂- and -CH₃ groups. In addition, some signals in the 1295-1180 cm⁻¹ region indicate the presence of aliphatic chains. Stretching vibrations -C = C - of aromatic rings of diaminodiphenylmethane manifest themselves as a peak at 1510 cm⁻¹. The Ti – O bond manifests itself at peaks at 892 cm⁻¹, and partially coincides with the vibrations of epoxy groups, and 2360 cm⁻¹, and this region can be enhanced by the Ti - O - C bond, which manifests itself at 1180 cm⁻¹. The Ti - O - C bond also appears as a peak at 1040 cm⁻¹. According to some sources, the broad peak at 1230 cm⁻¹ refers to pure titanium dioxide. In addition, according to the literature, in the region less than 750 cm^{-1} inaccessible on the graph, there are peaks of the O – Ti – O bond at 692 cm⁻¹ and the Ti – O bond at 485 and 435 cm⁻¹.

The structure of the obtained nanocomposites was studied using scanning electron microscopy, and histograms of the particle size distribution were plotted. In addition to the study of the obtained samples, a control study of pure TiO_2 was done (Figure 6). The micrograph shows a large number of spherical nanoparticles. The data confirm that the nanoparticles have an average diameter of 46 nm.



Fig. 6 sem image (left) and histogram of particle distribution (right) in the original TiO₂.

In the nanocomposite containing 0.2% TiO₂ nanoparticles, aggregates of particles are observed, and therefore the average diameter increases from 46 nm to 64 nm (Figure 7). The micrograph shows single rounded particles, the distribution of particles is uniform.



Fig. 7 sem image (left) and histogram of particle distribution (right) in a nanocomposite containing 0.2% TiO₂.

In the sample containing 0.5% TiO₂ based on the total mass (Figure 8), an increase in the average particle diameter up to 79 nm is also observed. The image shows clear individual rounded particles as well as small aggregates. No irregular accumulations of particles are observed.



Fig. 8 sem image (left) and histogram of particle distribution (right) in a nanocomposite containing 0.5% TiO2.

In the TiO₂/EP nanocomposite with a nanoparticle content of 1% of the total mass (Figure 9), the growth trend of nanoparticles continues: the average diameter is 88 nm. The micrograph shows a good distribution of particles, as well as the formed aggregates.



Fig. 9 sem image (left) and histogram of particle distribution (right) in a nanocomposite containing 1% TiO2.

As the data show, with an increase in the concentration of the nanocomponent, the particle size also grows (from 46 to 88 nm at 1% of the nanocomponent). Obviously, with an increase in the concentration of the nanocomponent in the matrix, the distance between the particles decreases, which increases the probability of the formation of aggregates – the closer the particles are located to each other, the easier to them to overcome the disjoining forces caused by polymer molecules. Perhaps this problem can be solved by choosing the parameters of ultrasonic dispersion. 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 [48]-[50].

IV. CONCLUSION

TiO₂/EP nanocomposites were prepared and investigated by the in situ method at the stage of curing in the presence of 4,4diaminodiphenylmethane with preliminary ultrasonic dispersion and evacuation. The composition and structure of TiO₂/EP nanocomposites have been studied. It was shown that with an increase in the content of nanoparticles, their average size increases to 88 nm at a TiO₂ concentration of 1% as a result of secondary aggregation processes. 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.

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