DOI: 10.46300/9106.2021.15.155 Volume 15, 2021

Study of Thermophysical Properties of Polymer Materials Enhanced by Nanosized Particles

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Received: March 10, 2021. Revised: August 15, 2021. Accepted: September 12, 2021. Published: September 14, 2021.

Abstract— In this work, the object of study is an epoxy nanocomposite based on TiO_2 nanoparticles and epoxy resin, and the subject is the preparation and physical and mechanical properties of TiO_2/epoxy nanocomposites. The characteristics of the properties and methods of synthesis of the initial components for the synthesis of epoxy nanocomposites - epoxy resins and nanoparticles of titanium dioxide are given, and data on epoxy nanocomposites based on nanoparticles of titanium dioxide are presented. It was found that the addition of TiO_2 to the epoxy matrix reduces the coefficient of friction and significantly increases the wear resistance of such nanocomposites.

Keywords—Composites, polymers, titanium dioxide, adsorption.

I. Introduction

In order for the polymer matrix to withstand high mechanical loads, it is reinforced with fillers. Nanoparticles are more effective fillers than coarse particles. Due to the small size and high surface energy of nanoparticles, the improvement of the properties of nanocomposites is achieved at a much lower filler concentration [1]-[9].

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]-[74]. However, nanocomposites using thermosetting polymers have not been studied as widely, especially using TiO_2.

TiO_2 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 [10]-[17]. There are two ways to obtain nanocomposite materials. The first in situ method is a one-step method, which consists in the fact that the synthesis of nanoparticles and a polymer matrix occurs simultaneously, resulting in the formation of a nanocomposite. The disadvantage of this method is that the products of the synthesis of nanoparticles remain in the nanocomposite. Another way to obtain nanocomposites is a multistage ex situ method. In the multistage method, each of the stages of nanocomposite formation is separated into a separate process. It is necessary to synthesize stable nanoparticles, isolate them, then disperse them in a polymer matrix or in an oligomer, followed by polymerization [18]-[27]. The difficulty in creating nanoclusters by the ex situ method lies in the dispersion of nanoparticles in the bulk, which requires additional equipment (high-performance mills, ultrasound, etc.), the use of surfactants, etc. [28]-[35]. In a number of works, titanium-containing coatings were obtained by photopolymerization of nanocomposites based on epoxy resin [36]-[40]. The initial nanocomposite was obtained in two ways: by dispersing preformed TiO_2 nanoparticles and by in situ using a sol-gel process. According to the results of the analysis of transmission electron microscopy (TEM), it was shown that using the in situ method it is possible to achieve a more uniform distribution of nanoparticles in the matrix, without the formation of agglomerations.

The one-stage method of forming nanocomposites is more technological, does not require additional equipment and additional energy and labor costs for synthesis, isolation of nanoparticles, their stabilization and combination with a polymer matrix, as required in a multistage method.

However, nanocomposites using thermosetting polymers have not been studied so widely, especially with the use of TiO_2, and there is a need for more experimental and theoretical studies of nanocomposites filled with TiO_2 nanoparticles.

II. Study of the Properties of Nanocomposites

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.

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.

DOI: 10.46300/9106.2021.15.155 Volume 15, 2021

The properties of the obtained nanocomposites depend on many factors: size, shape, concentration of nanoparticles, etc. The unique properties of the nanocomposite can also be achieved by good dispersion of nanoparticles in the surrounding epoxy matrix. In the article [3], it was found that TiO_2 nanoparticles can be homogeneously dispersed in epoxy resins using ultrasound, which improved mechanical properties and scratch resistance compared to traditional epoxy resins with microparticles. It was shown in [4] that for the best distribution of nanoparticles in the matrix, the ultrasonic dispersion time should be 2 minutes. Increasing the dispersion time further increases the temperature and viscosity of the resin. For even distribution, the loading percentage of nanoparticles should be 1%. It is believed that a larger particle size improves dispersion, since smaller particles have a greater Van der Waals force between themselves. This is proved by the TEM results in Figure 1.

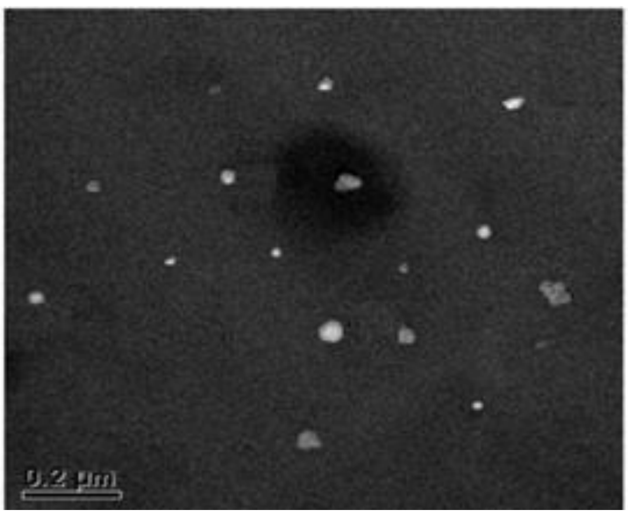

Fig. 1 Tem of TiO_2-epoxy nanocomposite (5 nm, 1% TiO_2).

TiO_2 nanofillers have a large surface area, which makes them chemically very reactive and helps them to better bond with the matrix, therefore nanosized particles have advantages over others. This is confirmed in Figure 2, where it can be seen that composites with nanosized TiO_2 (~ 50 nm) are superior in mechanical properties to microcomposites (~ 50 µm).

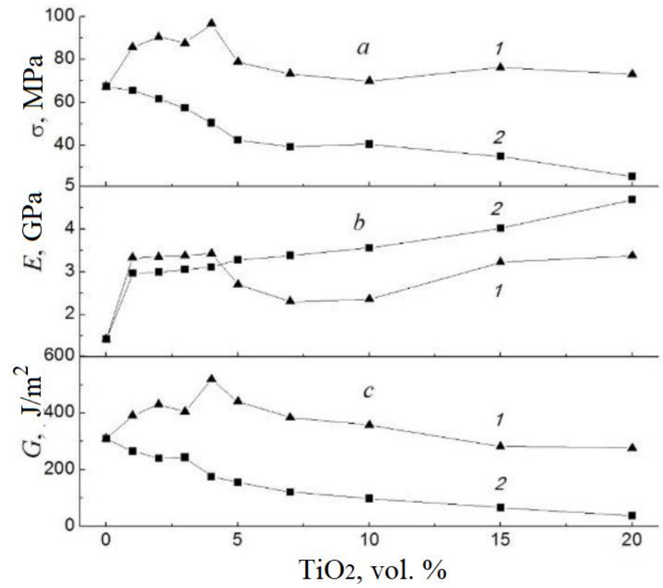

Fig. 2 Dependence of the mechanical properties of the composite on the concentration of nano- (1) and microparticles (2) TiO_2: a - strength, b - modulus, c - fracture energy.

As can be seen from Figure 2, all mechanical characteristics increase until the concentration of titanium dioxide nanoparticles in the composite reaches 4%. The subsequent fall is probably due to the enlargement of particles due to their agglomeration. Figure 3 shows that the tensile strength is maximum at 10% loading of nanoparticles, and the fracture strain and impact strength at 5%, which is 26%, 18% and 54%, respectively, more compared to pure epoxy resin. There is also evidence that improved mechanical properties are observed even at a very low content of TiO_2 nanoparticles ~ 1%.

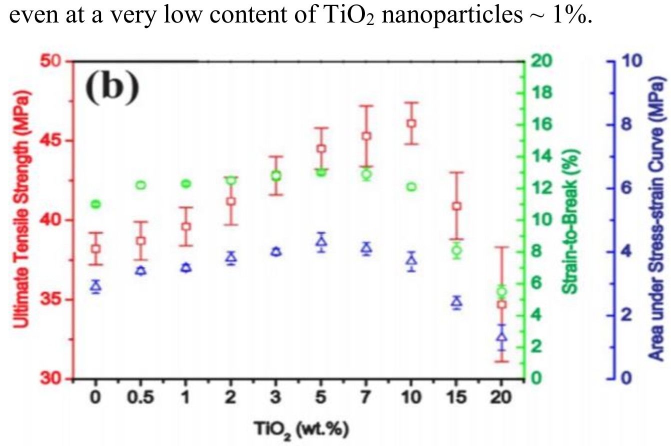

Fig. 3 Change in tensile strength (red graph), fracture strain% (green graph) and impact strength (blue graph) depending on the content of TiO_2 nanoparticles.

To understand the bonding mechanism, Fourier transform infrared spectroscopy was performed. IR spectra show a slight shift of the peak towards a lower wavelength (3418 cm^{-1}), which is associated with the stretching of the OH hydroxyl group on the surface of nanoparticles and epoxy resins, which tells us about their hydrogen bonding. Another article also talks about the hydrogen bond between TiO_2 nanoparticles and epoxy resin. The strong absorption band at 610-630 cm^{-1} corresponds to the Ti-O-Ti bonds, which prove that TiO_2 has been successfully prepared and incorporated into the epoxy matrix.

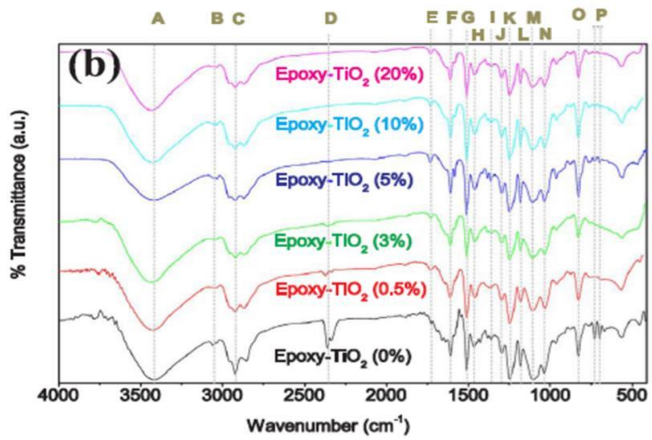

Fig. 4 ir spectra of epoxy nanoclusters with TiO_2 nanoparticles.

It was found that the addition of TiO_2 to the epoxy matrix reduces the coefficient of friction and significantly increases the wear resistance of such nanocomposites. The decomposition characteristics of a sample of a polymer paste reinforced with epoxy resin and TiO_2 were investigated. Sample exposed into phosphoric acid for 90 days (pH = 3,0-4,0). The results showed that the sample reinforced with epoxy resin and TiO_2 nanoparticles had low water absorption rates and was more resistant to penetration of water, acid, and other solutions. The degradation depth is 8-10% less compared to the control sample, which can be seen in Figure 5.

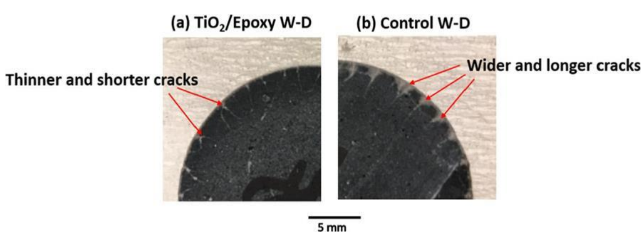

Fig. 5 Enlarged portions of the samples after 90 days of exposure to the phosphoric acid cycle and the environment, (a) a TiO_2/epoxy sample and (B) a control sample.

Epoxy nanocomposites can be used as refractive index tunable films. It has been investigated and shown that the refractive index of hybrid films can increase linearly with a percentage of TiO_2 and reach 1,731 at a titanium dioxide content of 20% (Figure 6).

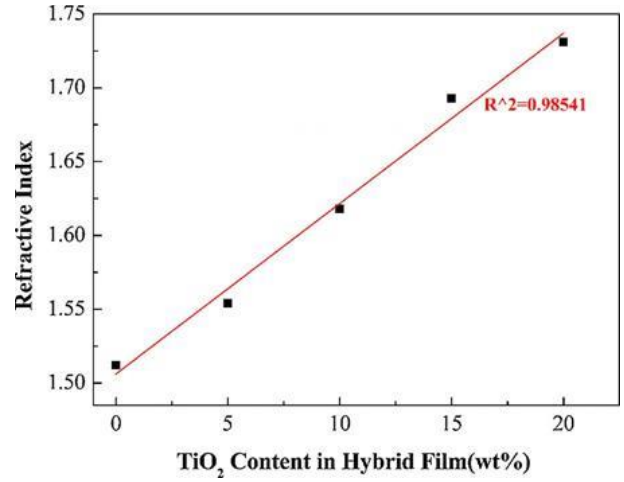

Fig. 6 Influence of TiO_2 content on the refractive index of epoxy/TiO_2 hybrid films.

III. Conclusion

Thus, the resulting epoxy nanocomposites with the advantages of good thermal, physical, mechanical, optical and other properties have great prospects for application in modern optical, medical, and structural purposes. It was found that the addition of TiO_2 to the epoxy matrix reduces the coefficient of friction and significantly increases the wear resistance of such nanocomposites.

It was shown, that all mechanical characteristics increase until the concentration of titanium dioxide nanoparticles in the composite reaches 4%. The subsequent fall is probably due to the enlargement of particles due to their agglomeration.

IR spectra show a slight shift of the peak towards a lower wavelength (3418 cm^{-1}), which is associated with the stretching of the OH hydroxyl group on the surface of nanoparticles and epoxy resins, which tells us about their hydrogen bonding.

Summarizing, the application of ceramics nanoparticles as fillers of polymer matrix allows for obtaining of composites with improved mechanical characteristics. Such composites can find their application as functional and construction materials.

This study was funded by RFBR, project number 20-08-00880.

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Sources of Funding for Research Presented in a Scientific Article or Scientific Article Itself

This study was funded by RFBR, project number 20-08-00880.