

Mechanoactivated silicatecontaining modifiers for thermoplastic polymers

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Abstract: Compositions of mechanoactivated silicatecontaining particles used as modifiers of polymer matrices have been developed. The morphology, particle sizes of the modifier, the nature of their distribution in the polymer matrix, as well as the topography of the friction surface of samples from modified polymers were studied using scanning electron and optical microscopy. Introduction of mechanically activated particles in concentrations from 0.01 to 2 wt. % into the polymer matrix of a reactoplastic polymer based on epoxy resin leads to multilevel modification, expressed in a change in the morphology of the test sample at the supramolecular, intermolecular and molecular levels. The effect of additives of nanophase modifiers on the wear rate of the developed nanocomposite materials is investigated. The introduction of composite mechanically activated nanodispersed particles into a reactive polymer based on epoxy resin leads to a decrease in the coefficient of friction in the studied range of modifier concentrations, loads and test speeds. Studies of the morphology of the surface of the analyzed samples have shown the presence of a separation layer on the surface of rubbing bodies, contributing to a significant decrease in the coefficient of friction. In the structure of this coating, the presence of nanophases is observed, which have a positive effect on the wear resistance of the developed composite material.

KEYWORDS: MECHANICAL ACTIVATION, NANOCOMPOSITES, NANOPARTICLES, THERMOPLASTICS.

1. Introduction

The concept of technology for producing mechanoactivated composites is quite extensive, as it includes various areas of production activity, meaning obtaining a finished product by high-energy combination of several types of raw materials with subsequent processing [1]. One of the many definitions of the concept of technology of mechanically activated composite materials can be the following formulation: all theoretical, practical skills and abilities used in mixing, molding and combining materials of various nature and origin in order to obtain a technological product characterized by predetermined technical parameters according to technical or commercial documentation [2].

Among the components of composite materials, it is necessary to distinguish active ingredients that perform the main specified function, and base materials that are subject to modification with the introduction of active fillers [3]. Mechanically activated composites usually consist of microdisperse or nanodisperse phases that are located in the polymer matrix and look homogeneous on a macroscopic scale and heterogeneous on a microscopic scale. The main requirement for composite materials, including mechanically activated ones, is the stability of the composition, structure and physico-mechanical characteristics of the materials obtained [4]. The resulting composite materials are usually anisotropic and heterogeneous, which is due to the production technology, structure and physico-mechanical characteristics of the polymer matrix and the mechanically activated modifier [5]. Thus, the nature of the high-molecular compound, the charge of the mechanically activated modifier, the amount of modifier in the polymer, and the manufacturing process used are parameters that have a decisive effect on the properties of the composite material [6]. Mechanoactivated particles can be introduced into metal, ceramic or polymer matrices, which makes it possible to obtain a huge combination of mechanoactivated composite materials [7].

Usually composite materials generally consist of one or more dispersed phases randomly distributed as micro- or nanoscale particles in the volume of the modified material. If two or more types of fibers are introduced into the polymer matrix, the size of which exceeds a micron diameter, we can talk about hybrid composite polymer materials. Currently, a new class of composite materials has appeared, including mechanoactivated ones – nanohybrid composite materials [8]. The production of composites from polymer materials with complementary physical and mechanical properties (strength, Young's modulus, heat resistance, frost resistance, wear resistance) makes it possible to manufacture parts of complex shapes, including those with a lower density compared to a metal or ceramic matrix.

Thus, the development of compositions, manufacturing technology, as well as ways to modify the created mechanoactivated composites is an urgent task of modern polymer materials science.

The purpose of the research is to study the structure and physico-mechanical characteristics of composite polymer materials modified with mechanoactivated silicatecontaining particles.

2. Materials and methods of research

As initial matrices for the creation of polymer composite materials, traditional multi-tonnage polymers produced by the domestic industry were used: epoxy resins of the ED-20 type.

Two types of particles were used in the course of the research:

- 1) mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (PVB) (20 wt. %), activation time 4 min, acceleration 60 g, irradiation with a dose of 200 kGy;
- 2) mechanically activated kaolinite particles (80 wt. %) with polyvinyl alcohol (PVA) (20 wt. %), activation time 4 min, acceleration 60 g, irradiation with a dose of 200 kGy.

Mechanical activation of composite particles was carried out in an AGO-2 type mill.

Additional irradiation treatment made it possible to obtain radiation crosslinking inside the high-molecular compound and directly increase the number of charge centers on the surface of the inorganic core and the organic shell to increase the interaction between them (Figure 1).

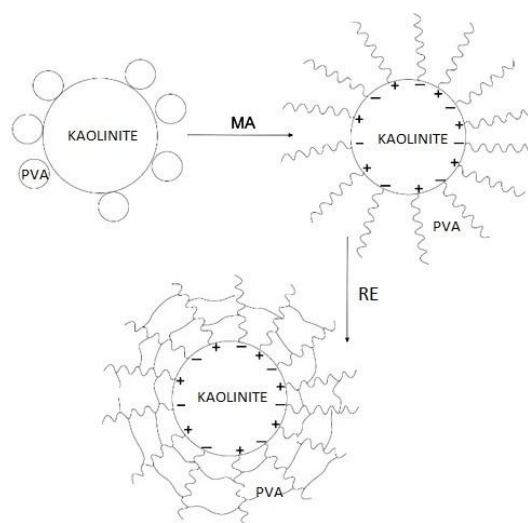


Figure 1 Diagram of the formation of a mechanoactivated nanoscale particle based on kaolinite and polyvinyl alcohol (PVA)
MA – mechanical activation; RE – radiation exposure

These mechanically activated particles were introduced into a polymer matrix. The content of composite particles of the modifier in the polymer volume ranged from 0.01 to 2 wt. %. Samples for testing, depending on the composition of the matrix, were obtained by free casting into a mold on specialized equipment under the conditions recommended for processing the base polymer material. The study of the structure of polymers, composite materials and modifiers was carried out using scanning electron and optical microscopy.

Optical studies of the morphology of nanomodifiers and the surface of the chips of samples, the size of the modifier particles, the nature of their distribution in the polymer matrix, as well as the topography of the friction surface of samples from modified polymers were carried out in reflected electrons on an electronic scanning microscope Mira/Tescan, in reflected light on an optical microscope MMR-1600T.

The samples for scanning electron microscopy were mechanically activated particles and chips of samples of initial and modified polymers obtained at liquid nitrogen temperature, or initial samples after injection molding or pressing. Scanning electron microscopy images were obtained in secondary electrons.

Block samples of compositions exposed to abrasive particles of various fractional composition or a metal counterbody were used to study the topography of friction surfaces on an optical microscope.

The effect of additives of nanophase modifiers on the wear rate of the developed nanocomposite materials was carried out on the FT-2 friction machine "Microtestmachines" according to the "sphere – plane" scheme in the sliding friction mode.

The temperature in the friction zone was measured with a chromel-nickel thermocouple installed in the sample at a distance of 0.5 mm from the friction surface. The experiments were carried out at ambient temperature (20 ± 5) °C. The samples were run-in at a sliding speed from 0.1 m/s to 0.5 m/s and a load of 30 N, the tests were carried out at a sliding speed of 0.1 m/s and a load of 30 N.

3. Results and discussion

The effectiveness of the modifying action of mechanoactivated polymers is related to the size of the filler and the interaction at the filler–matrix interface. Two aspects can be distinguished in the effect of the action of the mechanically activated filler on the polymer matrix:

- 1) the probability of defects causing premature failure of a polymer product during operation decreases with a decrease in the size of mechanically activated solid particles;
- 2) the dependence of the nature of the origin of the mechanically activated particle on the nature of the modified polymer. A number of studies have shown that nanometer parts of the same nature with a modifiable matrix behave like ideal crystals, and their physico-mechanical characteristics approach theoretically calculated parameters [9].

The morphology of composite materials modified by mechanoactivated nanophase particles has been studied by optical and scanning electron microscopy, according to the results of which it is possible to assume that an increase in the dispersion values of mechanoactivated particles leads to an increase in the modifying effect in the polymer, polymer-oligomeric and oligomeric matrix.

Figures 2, 3 show images of the morphology of composite particles obtained by mechanical displacement of kaolinite (80 wt. %) with polyvinyl alcohol (20 wt. %).

It is shown that during mechanical activation of inorganic and organic dispersed systems, nanoscale particles are formed, the properties of which will differ from those of the bulk substances forming these particles. An increase in the values of the specific surface of these particles is observed during mechanical activation. It is possible to expect a high modifying activity of these substances when introducing particles into a polymer matrix of various natures. The effect is due to the high energy states of highly dispersed mechanically activated particles. In the structure of the modified polymer matrix (thermoplastic, reactoplastic), in general, a distribution of cluster structures of various structures is observed.

Regardless of the structure of mechanically activated particles and their activity, agglomeration of these low-dimensional objects is observed in some cases with the formation of micron-sized structures. These structures in the polymer volume are lamellar, visceral, and globular. It should be noted that the use of mechanically activated particles encapsulated in a polymer shell makes it possible to reduce the sharp interface between the modified polymer matrix and the mechanically activated modifier.

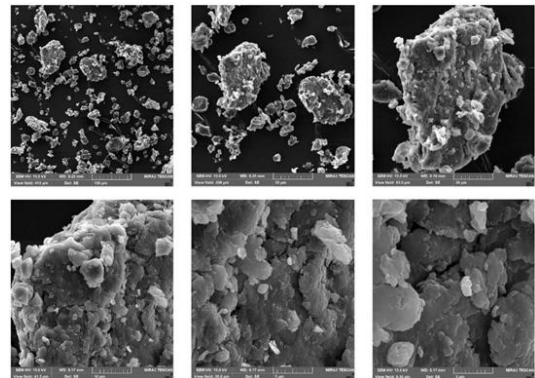


Figure 2 Morphology of nanodisperse mechanoactivated kaolinite particles (80 wt. %) with polyvinyl alcohol (20 wt. %)

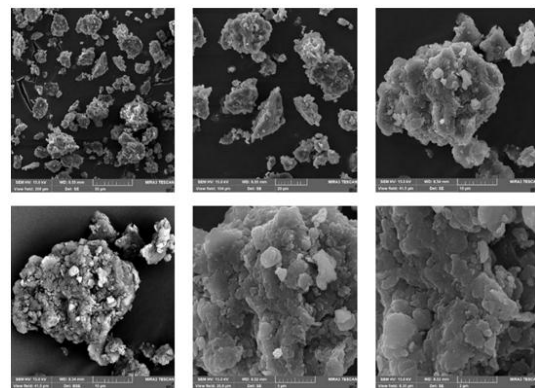


Figure 3 Morphology of nanodisperse mechanoactivated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %)

In [10], a physical model of the formation of mechanoactivated particles based on the formation of charge active centers during the joint dispersion of inorganic and organic components is proposed. It is shown that with an increase in the number of active centers on the core of a mechanoactivated particle, the activity of the formed mechanoactivated particle increases. An increase in the activity values of the mechanically activated particle leads to a greater modifying effect when these objects are introduced into the polymer matrix.

The results of studies on the morphology of chips of reactoplasts containing mechanoactivated particles based on kaolinite and polyvinyl alcohol, as well as kaolinite and polyvinyl butyral, are shown in Figures 4, 5.

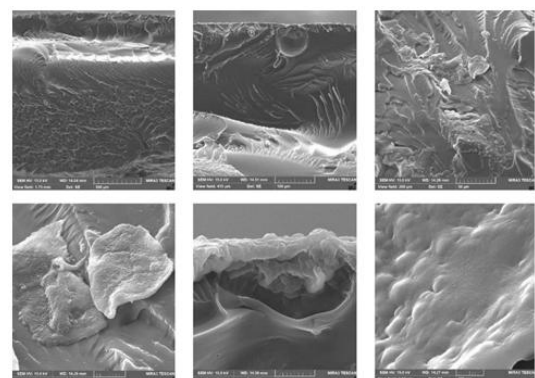


Figure 4 Morphology of chips of composite material based on epoxy resin ED-20, modified for 2 wt. % nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl alcohol (20 wt. %)

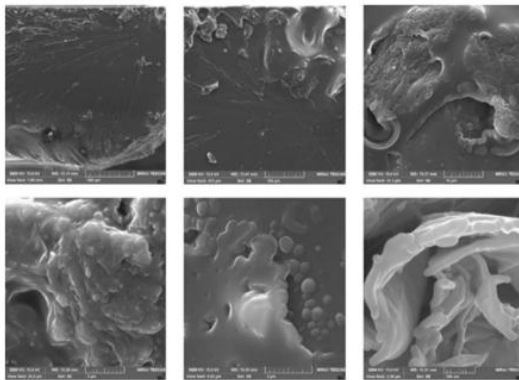


Figure 5 Morphology of chips of composite material based on epoxy resin ED-20, modified for 2 wt. % nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %)

So, for a sample of epoxy resin containing 2 wt. % of mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %), at an magnification of $\times 32$, the formation of a mesh structure on the chip surface was established. The magnification of $\times 140$ makes it possible to establish the presence of globular and whisker objects in the structure of the epoxy composite, the geometric dimensions of which range from several micrometers to $100\ \mu\text{m}$ or more.

A further increase in the resolution of the microscope $\times 900$ shows the presence in the composite structure of objects of lamellar, whisker habit of $20\ \mu\text{m} \times 30\ \mu\text{m}$, as well as globular particles in the geometric range of sizes from $1\ \mu\text{m}$ to $5\ \mu\text{m}$. When magnified to $\times 2000$, it is shown that these globular particles can concentrate in the surface layers of the polymer to form larger agglomerates. Further magnification of the image of the studied polymer morphology in the nanoscale of the geometric dimensions of the sample showed the presence of globular formations with a size from $\sim 20\ \mu\text{m}$ to $\sim 500\ \mu\text{m}$. A similar morphological pattern is observed for samples of epoxy resin modified by 2 wt. % nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %).

Thus, the introduction of mechanoactivated particles into the polymer matrix of a reactoplastic polymer based on ED-20 epoxy resin leads to multilevel modification, expressed in a change in the morphology of the test sample at the supramolecular, intermolecular and molecular levels.

The operating conditions of rubbing machine components are becoming increasingly difficult due to the rapid development of modern transport and industrial production [11]. Frequent operation in the "start – stop" mode, high specific loads lead to the fact that rubbing products work in harsh conditions of mixed and even boundary lubrication, which leads to critical wear of the operated parts and, in some cases, to destruction [12]. In a sufficiently large number of cases when friction units operate in heavily loaded conditions, the service life of metal-to-metal friction pairs does not meet operational requirements [13]. Thus, it is necessary to create highly efficient tribomaterials to replace traditional metal-metal friction pairs, which will significantly increase the wear-resistant characteristics of parts and mechanisms used in transport and mechanical engineering.

Polymer-based composites are increasingly used as self-lubricating materials due to their high tribotechnical characteristics, mechanical properties and corrosion resistance [14].

Great successes over the past two decades have been achieved both in tribology and in the field of triboengineering related to the development and application of self-lubricating polymer materials. Currently, a large number of self-lubricating composite materials

have been developed for various tribotechnical applications [15]. The frictional characteristics of polymer materials under friction without supply of lubricant have been studied [16]. It has been shown that the formation and presence of a stable highly efficient separation film between rubbing bodies can significantly reduce the wear of composite materials of various origin [17].

The presence of a lubricant, unlike the so-called "dry" friction, makes it difficult to form a separation layer. In addition, in the case of friction with the supply of external lubrication, the chemical and tribochemical processes occurring in the friction zone undergo significant changes, since the lubricating medium absorbs a significant amount of heat released during the friction process [18]. As a result of the formation of a separating tribofilm, its growth can be greatly slowed down due to the presence of a lubricant in the friction zone. Thus, the formation of a separating film is a key factor in the creation of new highly efficient polymer composite materials for tribotechnical purposes, including those operated under conditions of lubricant application.

Available studies have shown that the introduction of nanoparticles into the polymer matrix significantly improves its tribotechnical characteristics, expressed in a decrease in the coefficient of friction and an increase in the wear resistance of nanocomposites [19], [20]. The positive role of nanoparticles in friction is explained by their capture by the separating tribofilm, as a result of which its bearing capacity increases. However, conventional nanofillers are difficult to distribute in the polymer volume due to their strong tendency to agglomeration and poor compatibility with the polymer matrix [18] – [20]. Therefore, the task of uniform dispersion of nanofillers in a polymer matrix remains at the moment one of the main tasks in the creation of nanocomposites.

Various types of technological approaches make it possible to create hybrid nanocomposites consisting of a solid inorganic core and an organic polymer-oligomeric shell. Moreover, the mechanochemical approach makes it possible to ensure the formation of strong covalent or ionic bonds between the inorganic core and the organic shell.

The existence of a shell having a specific structure different from the initial polymer or oligomer effectively reduces the high interaction between nanoparticles and the formation of agglomerates [18] – [20]. Mechanically activated particles have good lubricating properties when used as an independent lubricant or when introduced as an additive into base lubricants. It has been shown that this type of particle is particularly promising to use under extreme operating conditions of a lubricant [18] – [20].

It has been proven that the mechanical properties of composites, such as flexural modulus, strength, toughness, and hardness, are significantly improved by the addition of mechanically activated particles [18] – [20]. However, the tribological behavior of polymers modified by mechanoactivated particles has not been sufficiently studied.

Figures 6–10 show the results of tribotechnical studies of composite materials based on ED-20 resin modified with mechanically activated particles with concentrations from 0.01 wt. % up to 2 wt. %.

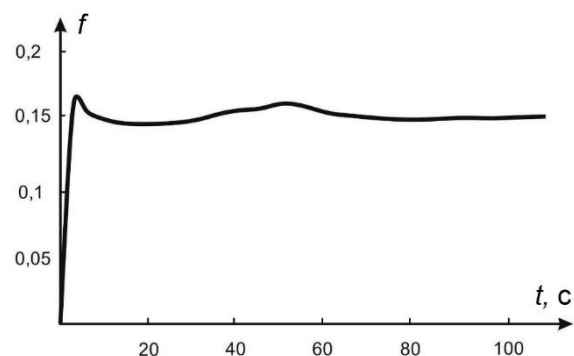


Figure 6 Dependence of the coefficient of friction f of an ED-20 epoxy resin polymer on the test time t

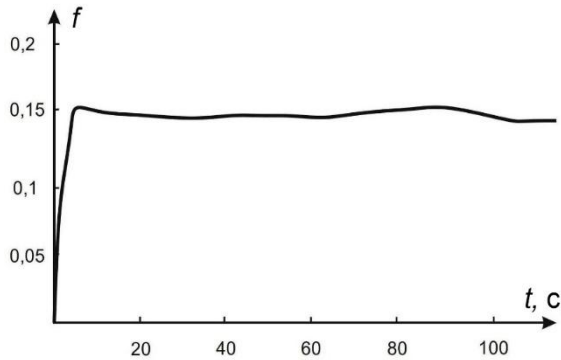


Figure 7 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %) with a concentration of 0.01 wt. % of the test time

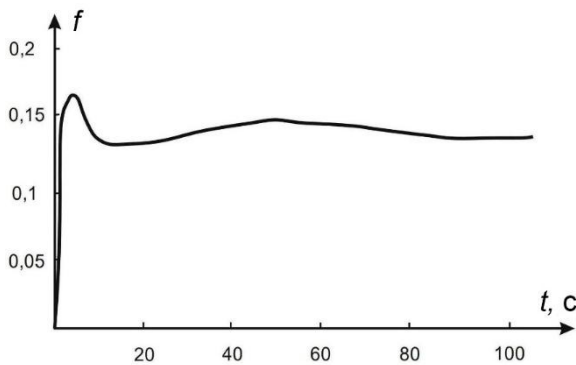


Figure 8 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %) concentration of 0.1 wt. %, from the time of testing

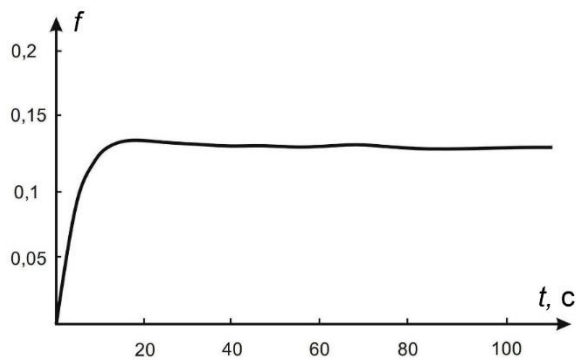


Figure 9 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %) with a concentration of 0.5 wt. % of the test time

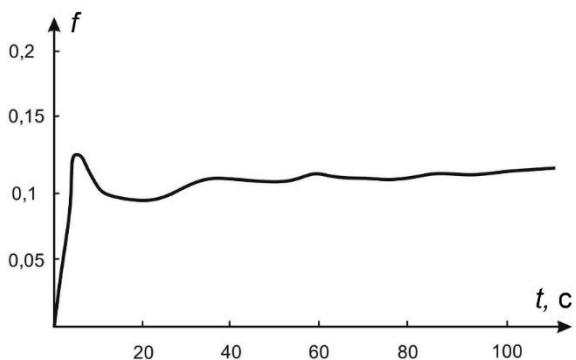


Figure 10 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %) with a concentration of 2 wt. %, from the time of testing

According to the data obtained, the introduction of composite mechanically activated nanodispersed particles into a reactoplastic polymer based on epoxy resin leads to a decrease in the coefficient of friction in the studied range of modifier concentrations, loads and test speeds (Figure 11). This effect is most likely associated with the formation of a separation film between the contacting bodies, which has high bearing capacities, as well as the possibility of repeated reshaping without destruction.

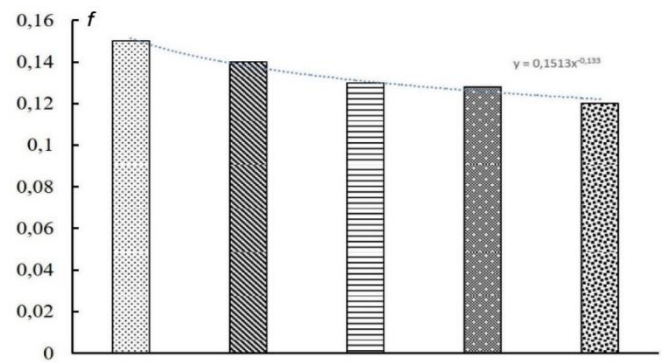


Figure 11 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %). Modifier concentration: 1 – initial polymer; 2 – 0,01 wt. %; 3 – 0,1 wt. %; 4 – 0,5 wt. %; 5 – 2 wt. %

The conducted studies of the morphology of the surface of the studied samples showed the presence of this separation layer on the surface of rubbing bodies (Figure 12). According to the data obtained, presented in Figure 12, a separation layer is formed, which contributes to a significant decrease in the values of the friction coefficient of the pair "composite based on ED-20 – 100Cr6 steel". In the structure of this coating, the presence of nanophases is observed, which have a positive effect on the wear resistance of the developed composite material.

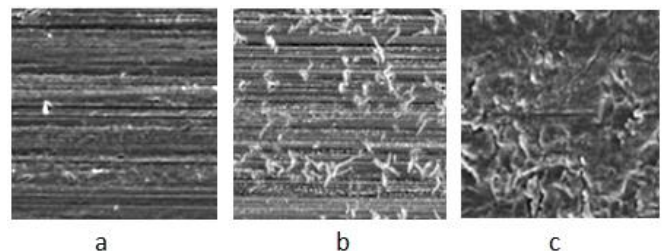


Figure 12 Morphology of the friction surface of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVA (20 wt. %), $\times 800$. The concentration of the modifier: a – the initial polymer; b – 0,1 wt. %; b – 2 wt. %

Studies of tribological characteristics of composite materials based on ED-20 resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %), show that the introduction of this modifier leads to similar results as in the case of the use of mechanoactivated modifiers based on kaolinite and polyvinyl alcohol (Figures 13–16).

Figure 17 shows the results of studies on determination of coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %). As for the case of using mechanoactivated particles based on kaolinite (80 wt. %) with polyvinyl alcohol (20 wt. %), there is a steady decrease in the values of the coefficient of friction.

Figure 18 shows the morphology of the friction surface of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with polyvinyl butyral (20 wt. %) with different concentrations.

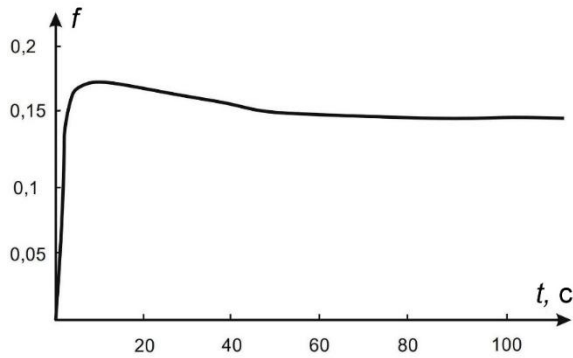


Figure 13 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVB (20 wt. %) with a concentration of 0.01 wt. % of the test time

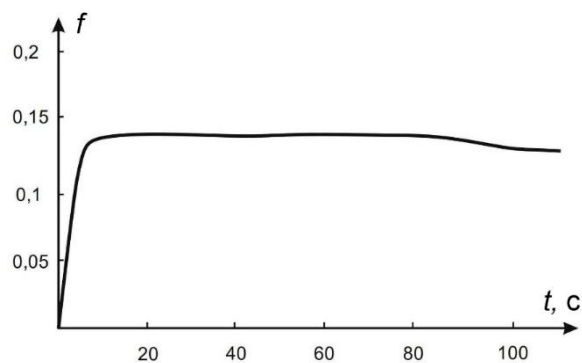


Figure 14 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVB (20 wt. %) concentration of 0.1 wt. %, from the time of testing

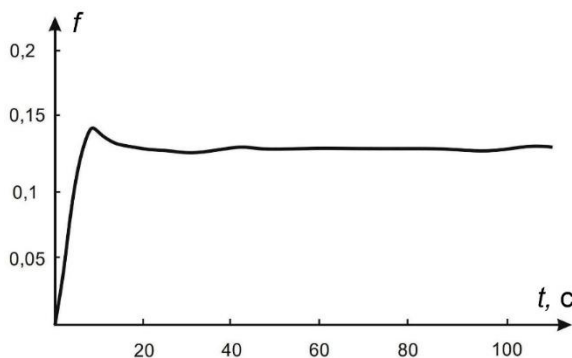


Figure 15 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVB (20 wt. %) with a concentration of 0.5 wt. % of the test time

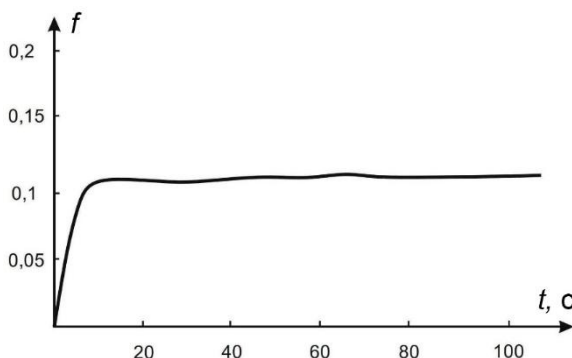


Figure 16 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVB (20 wt. %) with a concentration of 2 wt. %, from the time of testing

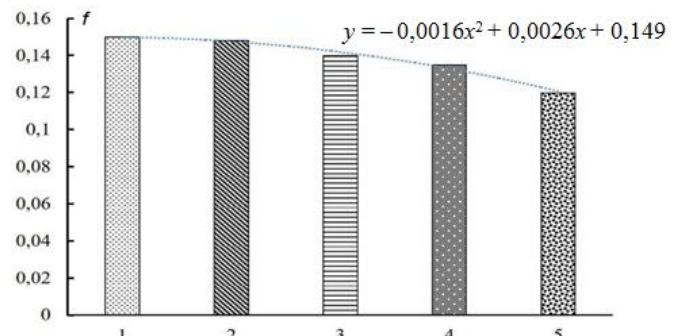


Figure 17 Dependence of the coefficient of friction of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles (80 wt. %) with PVB (20 wt. %). Modifier concentration: 1 – initial polymer; 2 – 0,01 wt. %; 3 – 0,1 wt. %; 4 – 0,5 wt. %; 5 – 2 wt. %

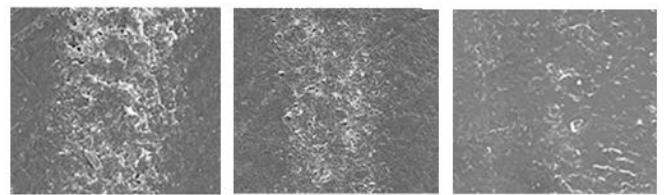


Figure 18 Morphology of the friction surface of a composite material based on ED-20 epoxy resin modified with nanodisperse mechanically activated kaolinite particles

The specific structure of polymer-oligomeric silicate mechanically activated particles makes it possible to form transferred layers on solid substrates. Under the action of tangential stresses, the polymer-oligomeric fraction of the mechanically activated particle forms an adhesive layer fixed in the micro-dimensions and defects of the substrate of any composition, both non-metallic and metallic. The formation of such a layer is also facilitated by the active charge state of macromolecules that are part of the polymer-oligomeric layer surrounding an ultrafine or nanoscale kaolinite particle [18] – [20].

Polymer-oligomeric nanoscale fragments are oriented in the direction of relative displacement under the action of shear, forming areas with increased strength. The thermodynamic compatibility of oligomeric and polymer fragments of cluster particles with the polymer matrix of the base polymer determines the high stability of the formed composite, capable of reshaping at tangential stresses, and the active charge state and polydispersity of the oligomeric components contribute to the implementation of alternating transfer (Figure 19).

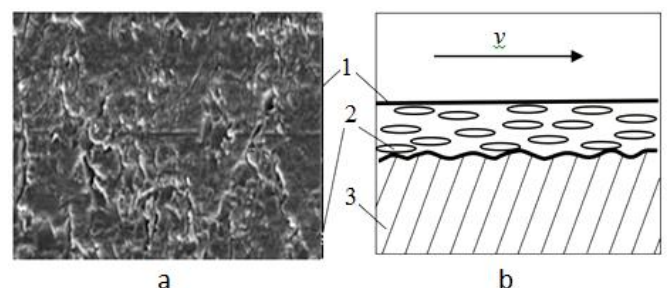


Figure 19 A characteristic view of the transferred layer on the surface of 100Cr6 steel (a) and a diagram of its structure (b). 1 – substrate; 2 – mechanically activated particles; 3 – polymer matrix of composite material

Thus, the cluster structure of mechanically activated particles "kaolinite – PVA", "kaolinite – PVB", representing a combination of nanoscale elements of the polymer fraction and oligomeric products of various masses located on the surface of an ultra-small or nanoscale particle, allows the formation of transferred

(separation) layers in the friction contact zone, synergistically combining the most favorable tribological characteristics of metal polymer friction pair.

4. Conclusion

Modern designs and parts of machines and mechanisms for various purposes are made from a wide range of composite materials. This makes it possible to significantly change the operational characteristics of machines and devices used in various types of production. Solving the problem of creating basic materials used as matrices for modification is one of the urgent problems of modern materials science that needs to be solved. However, there is another promising direction in the field of creating composite materials – the production of new modifiers that can significantly change the materials being modified to give them the necessary physical, chemical, technical and other characteristics.

To create new types of modifiers, various types of technologies based on physical, chemical, physico-chemical principles of generation are used. In recent decades, to obtain modern modifiers used in the chemical industry, medicine, and mechanical engineering, the technology of mechanochemical synthesis has been used, which consists in the simultaneous course of both mechanical action on the modified material and targeted chemical synthesis. As a result of the synergistic effect, materials are formed that differ qualitatively in properties from the starting materials involved in the synthesis of the modifier.

In the course of this work, the morphology of mechanically activated particles is considered. According to the data presented, composite materials based on epoxy resin modified with nanodisperse mechanically activated particles of polyvinyl alcohol and polyvinyl butyral are characterized by a developed morphology. Tribotechnical characteristics of composite materials based on an epoxy matrix modified with mechanically activated particles "kaolinite - polyvinyl alcohol", "kaolinite – polyvinyl butyral" have been studied. It is shown that the cluster structure of mechanically activated particles "kaolinite – polyvinyl alcohol", "kaolinite – polyvinyl butyral", representing a combination of nanoscale elements of the polymer fraction and oligomeric products of various masses located on the surface of an ultra-small or nanoscale kaolinite particle, allows the formation of transferred layers in the friction contact zone, synergistically combining the most favorable tribological characteristics for metal polymer friction pairs.

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