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This paper reports the analytical and experimental studies aimed at designing and modeling fluoropolymer anti-friction materials. The optimal ratios of the total surface of polymer particles to the total surface of filler particles for different brands of fluoropolymer-4 have been determined, as well as the critical concentrations of the modifiers of fluoropolymer anti-friction materials. The calculations of antifriction carboplastics' elasticity modules indicate the existence of adhesion between carbon fiber and polytetrafluoroethylene. When constructing composites that combine high durable and tribotechnical characteristics, it is advisable to combine modifiers with different dispersion and polymer-oligomeric matrices, which enables the implementation of the principle of multilevel modifying. It has been established that the adhesion between carbon fiber and polytetrafluoroethylene can be improved by applying a fluoropolymer coating onto the surface of carbon fibers or by modifying with zirconium oxide nanopowders. The binary fluoropolymer matrix applied to the surface of carbon fiber can be used as an effective base for composite materials. This study has demonstrated that filling polytetrafluoroethylene (PTFE) with coke, carbon fibers (18–19.5 % by weight), and zirconium oxide nanopowders in the amount of up to 2 % by weight produces materials with high mechanical characteristics and durability. It has been shown that the existence of an oligomer component improves the thermodynamic compatibility at the interface and promotes the plasticization of the PTFE boundary layers. Molecular structure with a certain orientation of coke and carbon fiber in the interphase areas is inherent in the materials with improved physical and mechanical properties

Keywords: anti-friction materials, composite, durability, fluoropolymer coatings, nanopowder, zirconium oxide, carboplastics

UDC 678.6-1

DOI:10.15587/1729-4061.2020.214533

DESIGN AND STUDY OF NANOMODIFIED COMPOSITE FLUOROPOLYMER MATERIALS FOR TRIBOTECHNICAL PURPOSES

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Received date 02.09.2020

Accepted date 05.10.2020

Published date 30.10.2020

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1. Introduction

The development of technology for the development and creation of polymeric composite materials based on polytetrafluoroethylene (PTFE) has for a long time been

based mainly on empirical research, due to the complexity of interphase interactions in the multicomponent polymeric composite systems. The combination of theoretical research into the structural and phase transformations occurring during the introduction of fillers to the composite's fluoro-

polymer matrix with experimental studies makes it possible to formulate a scientifically-based approach to forecasting and purposeful regulation of the properties of fluoropolymer materials.

An important task that determines the technical and economic efficiency of the use of new composite materials is the forecasting of operational properties and the associated modeling of the composition, structure, and properties of polymeric composite materials. The modern industry requires the production of new composite materials with a high level of operational characteristics. This task can be solved by defining the estimation-analytical and experimental-technological principles to design and model fluoropolymer anti-friction materials based on the targeted adjustment of their components. Therefore, it is a relevant task to design fluoropolymer anti-friction materials.

2. Literature review and problem statement

The process of designing fluoropolymer anti-friction materials can be divided into two stages: estimation-analytical and experimental-technological [1]. The first stage implies analyzing the predefined loading conditions and determining a technique to design a composite with the specified properties. However, the difference in interaction mechanisms hinders the construction of universal estimation dependences based on the studies that model the behavior of anti-friction composite materials exposed to stresses by employing the variational principles of solid mechanics [2, 3]. A characteristic feature of the technology of functional fluoropolymer anti-friction materials is the desire to ensure the optimal level of interaction between the matrix polymer and filler's particles, which determines the structure and characteristics of the boundary layers. This interaction is complicated due to the specific structure of the molecular chain of polytetrafluoroethylene (PTFE), which predetermines a pronounced chemical inertness towards a large number of solid-phase components [4, 5]. In other words, there are aspects that determine the probability of the course of the physical-chemical processes that form the boundary layers with the optimal parameters of adhesive and durable characteristics. These aspects should be taken into consideration when choosing the composition of a modifier, the particles' dispersion, and the combination of components for composites of different formulations.

For a long time, there was a perception that the introduction of various solid fillers in PTFE, without changing the friction coefficient, significantly improves its wear resistance and mechanical characteristics [6–10]. However, the enhanced wear resistance and mechanical characteristics when applying fillers are almost always accompanied by an increase in the friction coefficient and a decrease in the tensile strength. This is due to the fact that modifiers do not have a complex nature of the action in the process of intermolecular interaction and the formation of the molecular structure.

Consequently, the research is needed aiming at compensating for the negative effects of fillers on PTFE. An example of such research is the use of a mixture of functional components with the participation of nanomodifiers [11–14], as well as the development of composites with an increased adhesion between the polymer and filler [15].

Also efficient is the structural modification of physical and mechanical properties without changing the chemical composition of the polymer and its molecular mass as a result of mechanical activation, radiation exposure, the use of magnetic and electromagnetic fields, shockwave processing, etc. [16–20]. However, these modifying technologies, except for mechanical activation, are quite expensive.

The chemical modification in the interaction between a polymer and the chemical or physical agents, as well as the use of substances interacting with the main monomer, are considered in work [21]. The results of synthesizing polymeric matrices of different compositions and structure are reported in [22, 23]. At the same time, these technologies are time-consuming and are difficult to implement industrially.

Natural and synthetic components of different compositions and dispersion are widely used to target the modifying of fillers for fluoropolymer matrices. Among the wide range of fillers and modifiers for PTFE special role belongs to carbon-containing components – natural graphite and its modifications: thermally split graphite, technical carbon, coke, carbon fibers. For composites with increased durable and tribotechnical characteristics, promising modifiers are carbon fibers (CF).

It was found that the application to carbon fibers or fabrics of the fluoropolymer coating significantly increases their wear resistance [24, 25]. At the same time, that improves their strength, elasticity module, heat resistance, and chemical stability.

To enhance the tribotechnical characteristics of composite materials, the dispersed filler must have an orderly effect on the boundary layers of the matrix. As a result, a molecular structure with the increased resistance against re-deformation and destruction is formed.

In this regard, nanometric dispersion fillers are quite effective, which have an uncompensated charge and form orderly “quasi-crystalline” areas in the volume of the composite [26, 27].

Thus, the construction of the estimation-analytical and experimental principles to design and model fluoropolymer anti-friction materials is necessary for the development of new materials for tribotechnical purposes.

3. The aim and objectives of the study

The aim of this study is to form the basic technological principles for designing and modeling the nanomodified fluoropolymer anti-friction materials for tribotechnical purposes. This would make it possible to predict the properties and scope of the effective application of the developed anti-friction materials.

To accomplish the aim, the following tasks have been set:

- to determine, based on the calculation-analytical studies, the optimal characteristics for the polymer's and filler's components for different brands of fluoropolymer and concentration of modifiers of fluoropolymer anti-friction materials;

- to experimentally investigate the tribotechnical and strength properties of antifriction carboplastics based on the Tekarm fabric with an applied fluoropolymer coating;

- to experimentally study the tribotechnical and strength properties of the nanomodified fluoropolymer composite materials and analyze their resulting structures.

4. The study materials and procedure

In the study, we used the composite materials of tribo-technical purpose based on polytetrafluoroethylene: F-4 PN (GOST 10007-80), an analog to Teflon 7 (TCI, USA) (ASTM D4895, ISO 12086), and fluoropolymer-4MB brand P (Formoplast, Russia) (GOST 30333-2007), an analog to Teflon FEP (TCI, USA), modified with carbon fibers and other components.

Carbon fibers made of the fabrics Tekarm (Russia) (TU 48–20–17–77) and Ural T15 (Russia) (GOST 28005-88) were used as a filler; they were obtained on the basis of hydrate-cellulose tissue. For grinding the fibers and mixing the formulation, we used the crusher-grinder MRP-1 (Granat, Russia) with double knives and a frequency rotation of 7,000 rpm. The average length of fibers after grinding was 50...200 μm .

Finely-dispersed foundry coal coke of KL-1 brand and the dry colloidal-graphite preparation C-1 were also used to obtain the formulation.

ZrO₂ zirconium oxide nanopowder was used as a functional additive. The powders were analyzed by X-ray diffraction (XRD) methods at the Dron-3 X-ray diffractometer (IC Bouvestnik, Russia) under Cu-K α radiation to determine the size of the crystallites and the quantitative phase analysis. Powder particle sizes were estimated using transmission electron microscopy (TEM) (JEM 200, Jeol, Japan). According to XRD and TEM data, the particle sizes were 18 \pm 2 nm.

The structure of the PTFE composite modified with zirconium oxide nanopowder was studied at the electron microscope SEM, JSM 6490 LV, Jeol (Japan); the local chemical analysis – using the installation EDS – Inca Oxford (Germany).

The colloidal-graphite preparation C-1 (TU 113-08–48–63–90) (TOV Ukrspetsmasla), the chromium oxide pigment OHP-1 (Infrahim, Russia) (GOST 2912-79), diphenylsilanediol (TU 6-02-623-76), and a laminar graphite compound that is intercalated with FeCl₃ were used to fill the composition for a fluoropolymer coating.

The procedure to investigate composite materials implied determining the tensile strength and wear intensity rate of the composite materials. Due to the chemical inertness of PTFE macromolecules, chemical bonds are not formed at the interface with a filler. The low surface energy and high viscosity prevent the high-quality wetting of the filler surface with a melt. As a result, the interphase layer is not capable of load transfer; when composites are examined for tensile strength, the reinforcing filler does not contribute to increasing the resistance of the sample to rupture. Therefore, the value of the limit of strength during stretching is an indicator of the quality of the filled PTFE. Unlike other polymers, filling it with any component in the use of conventional technologies leads to a reduction in the strength limit when stretching a composite.

When testing the tensile strength, a sample was put between the platforms of the tensile testing machine MP-05-1 (TOV ASMA, Ukraine) and stretched at a speed of 15 mm/min. For mechanical tests on uniaxial stretching, ring samples were used. We studied the tensile strength of antifriction carboplastics according to ISO R527 (ASTM D638). The limit of tensile strength σ_t was determined from the following formula:

$$\sigma_s = \frac{P}{2h(R-r)}, \quad (1)$$

where P is the breaking force, N; h is the sample height, mm; R is the outer radius, mm; r is the inner radius, mm.

The tensile strength limit σ_t , determined from formula (1), is not a true characteristic of the composite material since the tensile and bending deformations occur near the places of semi-discs disconnection as a result of changes in the ring curvature. Therefore, tests with ring samples were performed only for comparison.

To increase the adhesive interaction at the interface between the filler and matrix, we applied a wear-resistant fluoropolymer coating onto the surface of the carbon fiber by electrostatic spraying. The method of applying thin-layer polymeric coatings in an electrostatic field is based on depositing the charged polymer's particles on the surface of the part with an opposite charge. The scheme of charging and precipitation of polymer's particles on the product in the field of a crown discharge is shown in Fig. 1. The application of coatings exploited the phenomenon of crown discharge, arising under the influence of a high voltage current. In this case, ions 1, having the same charge with crown electrode 4, are pushed away from it, and move in the opposite direction to the charged electrode. In the space between the electrodes, an electric field of high voltage is generated. If the interelectrode space includes the particles of polymer 6, the ions charge them. The charged particle is sent to the electrode (part) with an opposite charge and settles at its surface 7. A special spray gun was used to implement the spraying process.

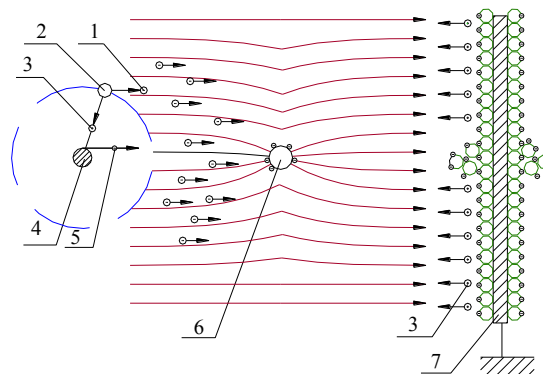


Fig. 1. Schematic charging and depositing the polymer's particles on a product in the field of crown discharge: 1 – ion (–); 2 – air molecule; 3 – ion (+); 4 – crowning electrode; 5 – electron; 6 – polymer's particles; 7 – product

The carbon fabric was washed and dried before coating. The fluoropolymer 4MB, boron nitride, the graphite C-1, a laminar compound of graphite, chromium oxide, and diphenylsilanediol were stirred in the mill MRP-1. Next, we performed fractionation of the powder in order to obtain a composition whose particle sizes are 40...80 μm , and then thermally treated it for one hour at 150 °C. Fluoropolymer coatings were applied using the installation "Optima-01C" (Zaporizhzhia, Ukraine, "Elem").

To apply a layer of greater thickness, we repeated the electrical deposition of the material on the preceding melted layer. The effectiveness of such deposition depended on the electrophysical properties of the sublayer. We applied the

coating on the surface of the carbon fabric by the electrostatic spraying of the first-layer powder at an electric field intensity of 50 kV; for subsequent layers – 60...70 kV. After that, the coated carbon fabric was aged at 360...370 °C for two hours and cooled to room temperature at a rate of 30...40 °C per hour.

To obtain the compositions of an antifriction material, polytetrafluoroethylene powder was mixed with the carbon fiber, crushed to 5...500 μm (% by weight: carbon, 49.5...90.0; hydrogen, 0.1...3.5; oxygen 0.1...9.5; boron, 0.08...4.8; phosphorus 0.1...4.7; ash, 4.5...25.5).

The samples of the materials for our study were obtained using the technology of cold pressing of compositions (pressing pressure, P_{pr} =60 MPa), with subsequent free heat treatment of tablet blanks in the air at a temperature of 365 ± 5 °C at a heating-cooling speed of 30 °C/h.

The wear of the composites was studied at a modified friction testing machine by rubbing the samples studied on metallic counter-bodies. The tests were carried out according to the “sphere-plane” contact scheme.

Based on the results from testing, the volumetric wear intensity was calculated for the path of friction $\Delta S_1=0...3$ km and $\Delta S_2=3...23$ km:

$$I_1 = \frac{\Delta V_{1i}}{N_i \cdot \Delta S_1}, \quad (2)$$

$$I_2 = \frac{\Delta V_{2i}}{N_i \cdot \Delta S_2}, \quad (3)$$

where ΔV_{1i} is the change on the volume of the i -th sample at the interval of the friction path from 0 to 3 km (the nonlinear dependence of wear on the path of friction); ΔV_{2i} is the change in the volume of the i -th sample at the interval of the friction path from 3 to 23 km (the linear dependence of wear on the path of friction).

The wear of the materials was determined under a normal load of 100 N and a sliding speed of 0.3 m/s. The temperature was measured at a distance of 0.5...1 mm from the surface of the counter-body. Tests were performed without lubrication on steel 30Kh13 (HB, 4.2 GPa) after working (2 km) on the path of friction of 2...18 km. The output roughness of the samples is $Ra=0.22 \pm 0.02$ μm. The cylindrical samples of the examined materials had a diameter of 10 mm with a resulting sphere of 12.7 mm in diameter.

5. Results of the estimation-analytical study into the properties of anti-friction composite materials

The properties of a filled polymeric material can be described with the help of a model shown in Fig. 2, whose elements are the dispersed environment, a polymeric binder or a matrix, the dispersed phase, a filler, and the interphase area.

An analysis of this model and the experiments on the destruction of filled compositions show that the weakest link in this system is the interface between a polymer and a filler. In this regard, one of the main conditions for improving the mechanical properties of composite materials is to ensure high adhesion strength at the interphase at minimal internal stresses. This can be achieved when the binder properly wets the surface of the filler and at a certain ratio between the modules of elasticity of the filler and binder.

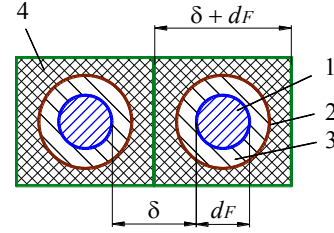


Fig. 2. A model of the filled polymeric composition: 1 – filler, 2 – interphase boundary, 3 – boundary layer, 4 – polymeric matrix, δ – the thickness of a polymeric inter-layer, d_F – filler’s size

In this model, the filler in the form of spherical particles with diameter d_F is located in the center of elementary cubic cells the size of $d_F + \delta$. For a single-fractional ball-shaped filler, the volume it occupies in the system will be equal to:

$$V_F = n \frac{\pi}{6} d_F^3, \quad (4)$$

where n is the number of filler particles.

Then the volume of the polymer can be defined as the difference between the volume of the cubic cell and the volume of the filler:

$$V_M = n \left[(d_F + \delta)^3 - \frac{\pi}{6} d_F^3 \right]. \quad (5)$$

Determine a correlation between the volume of the polymer and filler from the following formula:

$$K_V = \frac{V_M}{V_F} = \frac{(d_F + \delta)^3 - \frac{\pi}{6} d_F^3}{\frac{\pi}{6} d_F^3}. \quad (6)$$

By solving this equation relative to the thickness of the polymer interlayer δ , we obtain:

$$\delta = d_F \left[\sqrt[3]{\frac{\pi}{6} (K_V + 1)} - 1 \right] \quad (7)$$

or

$$\delta = d_F \left[0.806 \sqrt[3]{K_V + 1} - 1 \right]. \quad (8)$$

In actual composite materials, the shape of the surface of the filler particles differs from spherical, so the previous formula is supplemented with a shape factor that characterizes the ratio of areas of the actual and spherical particles. For the composite material F4K20, the shape factor is 1.8...2.1. For the composite F4VV15, the shape factor is 10.5.

It was found that for the composite F4K20 with a particle size from 20 to 36 μm, the optimum thickness of the polymeric interlayer δ is from 11 to 19 μm. And for the composite F4VV15 that contains carbon fiber made of “Tekarn” fabric in the amount of 20 % by weight, and whose particle size is from 80 to 100 μm, the optimum thickness of the polymeric interlayer (δ) is from 32 to 40 μm.

In the theoretical model of a filled material (Fig. 2), the polymer is represented in the form of a continuous phase, the particles of which have lost their independent significance. Such a structure is established after the completion of all

stages in the processing of fluoropolymer anti-friction material. The initial structure of the composite material is formed at the stage of mixing powder and fiber components. This determines the uniformity of the filler distribution inside the polymeric matrix and the nature of the interaction between the filler and the polymer.

If the filler particles are much larger than the polymer's particles, and its volumetric content is small, the effect of strengthening the composite material from the introduction of a filler is negligible. In this regard, when developing a composite material, it is necessary to take into consideration, in addition to the filler dispersion, the dimensions of the polymer's particles as well.

To determine the correlation between the surface of the polymer and filler, a dimensionless coefficient K_S was introduced, which is the ratio of the total surface of polymer's particles to the total surface of the filler's particles:

$$K_S = \frac{n_{M1}S_{M1} + n_{M2}S_{M2} + \dots + n_{Mi}S_{Mi}}{n_{F1}S_{F1} + n_{F2}S_{F2} + \dots + n_{Fi}S_{Fi}}, \quad (9)$$

where n_{Mi} , n_{Fi} is the number of particles of the i -th fractions of the polymer and filler, respectively; S_{Mi} , S_{Fi} is the surface area of the elementary particle of the i -th fraction in the polymer and filler, respectively.

For the single-fraction filler and polymer, this formula takes the following form:

$$K_S = \frac{n_M S_M}{n_F S_F}. \quad (10)$$

Assume that the filler and polymer consist of ball-shaped particles. Determine the number of particles using the ratio of volumetric parts of the filler and polymer φ_M and φ_S , and, taking into consideration the expressions for the surface areas, we obtain:

$$K_S = K_V \frac{d_F}{d_M}. \quad (11)$$

For the case of the multi-fraction filler and polymer, the formula takes the following form:

$$K_S = K_V \frac{d_{F,eq}}{d_{M,eq}}, \quad (12)$$

where $d_{F,eq}$, $d_{M,eq}$ are the equivalent diameters of the filler and polymer, respectively.

Thus, K_S is directly proportional to the ratio d_F/d_M , that is, the larger the filler particles and the smaller the polymer particles, the larger K_S , and vice versa.

It was established that the maximum strength, relative elongation, density, minimum wear intensity at $K_V=4$ (20 % by volume) corresponds to composites in the interval of the magnitude values: $4 \leq K_S \leq 8$.

K_S has been determined for the F4VV15 material based on fluoropolymers by different brands with different particle sizes: for a material based on the fluoropolymer 4PN, $K_S=3.4...4.2$; based on the fluoropolymer 4T, $K_S=7.5...8.0$; based on the fluoropolymer 4PN₂₀, $K_S=17...21$. Thus, the optimum values of K_S correspond to the composites F4VV15 based on the fluoropolymer 4PN and fluoropolymer 4T.

It is promising to use highly-filled polymeric materials. A highly-filled polymeric material is a two-phase system in

which particles of one phase (filler) are connected via a thin layer of another phase (interphase layer). The thickness of the binder layer in such a system should be minimal, so that the entire polymer, if possible, can transfer into a boundary structured state. The proportion of a filler, at which the entire binder enters the interphase layer, is termed a critical φ_{cr} and can be calculated from the following formula:

$$\varphi_{cr} = \frac{V_{max}}{\left(\frac{\delta_F}{r_{eq}} + 1\right)^3}, \quad (13)$$

where V_{max} is the volumetric fraction of the filler at the densest location; δ_F is the half distance between filler particles; r_{eq} is the equivalent radius of particles.

For a single-fraction ball-shaped filler, $V_{max}=74\%$; for a polydisperse one, $V_{max}=81\%$.

Consequently, in the fluoropolymers filled with micro-dispersed fillers, a coagulation structural mesh from the filler particles occurs already at its relatively small concentrations. When the concentration of filler increases, the distance between its adjacent particles become commensurate with the size of the elements of the molecular structure of the fluoropolymer 4. Further increase in concentration may lead to that that the structure becomes more unordered, there could occur micro defects and empty spaces, leading to a decrease in the strength and mechanical characteristics of the material.

The critical concentration of a filler for materials based on fluoropolymer 4 and coke can be determined from the dependence of material density on the concentration of the filler. A linear dependence is maintained to the concentrations of 25 % by volume for the materials based on fluoropolymer 4 and coke; after that, the density of the composites becomes less than the additive values.

An analysis of the tensile strength dependences, the Poisson coefficient, and the elasticity module of the composites based on fluoropolymer 4 and coke shows that these characteristics change their value at the filler content of 25 % by volume.

For the anti-friction carboplastic based on the fluoropolymer 4 and the carbon fiber made of "Tekarm" fabric, the critical concentration of the filler, derived from the calculation results, is 31 % by volume, which is confirmed by the experimental studies of mechanical characteristics [6].

Based on the amount of a polymeric component located in the interphase layer, at a critical degree of filling, one can determine the average thickness of the interphase layer. For the fibrous filler with the same fiber diameter, the average thickness of the interphase layer can be calculated from the following formula:

$$\delta \geq 0.07d + 0.52a, \quad (14)$$

where d is the fiber diameter, a is the minimum distance between fibers, equal to:

$$a = \frac{d(1 - \sqrt{1 - 1\varphi_F})}{1,1\varphi_F}. \quad (15)$$

For the anti-friction material based on the fluoropolymer 4 and the carbon fiber made of "Tekarm" fabric (31 % by volume): $a=12.2 \mu\text{m}$ and $\delta \geq 7 \mu\text{m}$.

The composite elasticity module has a maximum value when the entire binder enters the interphase layer. As a result of pore formation due to the insufficient amount of the binder between particles in the composite, the elasticity module decreases.

The elasticity module of the composite material with a filler between the upper (E_{max}) and lower (E_{min}) boundaries is equal to:

$$E_{max} = (1 - \varphi_F)E_M + \varphi_F E_F \tag{16}$$

$$E_{min} = \frac{E_M \cdot E_F}{[(1 - \varphi_F)E_F + \varphi_F E_M]} \tag{17}$$

where E_M is the module of a polymer elasticity.

It is established that the upper limit of the elasticity module corresponds to the same deformation of both components in the presence of adhesion between them, the lower one corresponds to the same stresses in the components in the absence of adhesion.

To calculate the elasticity module of a composite material with a large difference in elasticity modules, the following ratio can be used:

$$E = E_M \left[1 + \frac{\varphi_F}{3} \left(\delta_1 \frac{E_F}{E_M} - 1 \right) \right] \tag{18}$$

where δ_1 is an empirical coefficient that takes into consideration the nature of the polymeric and filler adhesion and a change in the deformation field, $0 \leq \delta_1 \leq 1$.

Determining the elasticity module of the anti-friction carboplastic F4VV15 has shown that the best match between the experimental data ($E_e=550...700$ MPa) and the estimation data is observed in determining the elasticity module according to formulae (16) and (18), respectively: $E_{max}=567.4$ MPa, $E=653.1...656$ MPa at $\delta_1=0...1$. When calculating using formula (17): $E_{min}=152.7$ MPa. Because E_{max} is closer to experimental data, it can be argued that there is adhesion between the carbon fiber and polymer across the entire contact surface. Given this, a hypothesis is stated: at the surface of carbon fibers, a chemical bond with the macro radicals of PTFE is realized. This takes place at the mechanical destruction of the polymer macromolecules below its glassing temperature, as well as a result of the mechanical-thermal processes during the heat treatment of composites.

Thus, composite materials are the systems with a clearly expressed heterogeneity of the structure. Their elastic, rheological, and durable characteristics are defined not only by the mechanical properties and the sizes of components but also by the conditions of interaction at the interface and interaction between the elements of the non-regularities.

6. Results of an experimental study into the properties, structure, and chemical analysis of fluoropolymer materials

The compositions' formulations and comparative data on the wear resistance of the examined fluoropolymer anti-friction materials are given in Table 1.

Our analysis has shown that the anti-friction carboplastics based on the "Tekarm" fabric with a fluoropolymer coating applied significantly outperform, in terms of wear

resistance, similar carboplastics without a fluoropolymer coating. In addition, such carboplastics demonstrate durability higher than that in the carboplastics coated with the fluoropolymer 4MB, grade P.

Table 1

The wear intensity of antifriction materials based on PTFE containing the carbon fiber made from the "Tekarm" fabric, $10^{-7} \text{ mm}^3/(\text{Nm})$

Coating type	Concentration of carbon fiber from the «Tekarm» fabric, % by weight					
	2	25	30	40	45	50
No fluoropolymer coating	10.2	6.4	7.2	8.5	10.4	39.5
Applied coating from the fluoropolymer 4 MB, grade P	8.1	4.3	5.3	6.3	7.0	18.0
Applied fluoropolymer coating	6.5	3.25	3.15	3.3	3.5	9.4

The results of our studying the tensile strength are given in Table 2.

Table 2

The tensile strength of antifriction materials based on PTFE containing the carbon fiber from the "Tekarm" fabric, MPa

Coating type	Concentration of carbon fiber from the «Tekarm» fabric, % by weight				
	5	15	20	25	45
No fluoropolymer coating	15	16.3	17.0	16.1	14.1
Applied coating from the fluoropolymer 4 MB, grade P	16	17.2	18.0	17.2	16.3
Applied fluoropolymer coating	17	18.4	19.1	18.3	17.5

It was obtained that the proposed carboplastics coated with a fluoropolymer coating outperform, in terms of tensile strength, the carboplastics without a coating, and those coated with the fluoropolymer 4MB, grade P.

It was established in [4, 6] that the carboplastics of the flubon type are promising anti-friction materials (F4VV15, F4VV20, materials based on polytetrafluoroethylene and carbon fiber, obtained by special technology, and modified with special additives). To improve the physical-mechanical and anti-friction characteristics of these materials, the principle of multilevel modification of the polymeric matrix was applied [19]. To implement this principle, fillers with different dispersion and composition such as coke, carbon fibers, and zirconium oxide nanopowders were introduced to the PTFE formulation. It was established in [15] that one of the best modifiers for fluoropolymer carboplastics is the nanopowder $\text{ZrO}_2+3\% \text{Y}_2\text{O}_3$, 700 °C. This nanopowder was used to create the fluoropolymer antifriction materials. We tested the developed anti-friction materials for wear resistance at a modified friction machine. The results of our study are shown in Fig. 3.

The method of electron microscopy was used to examine the microstructure of the friction surface after the end of testing the PTFE-composite material F4K10VV10, modified with zirconium oxide nanopowder $\text{ZrO}_2+3\% \text{Y}_2\text{O}_3$, 700 °C (Fig. 4).

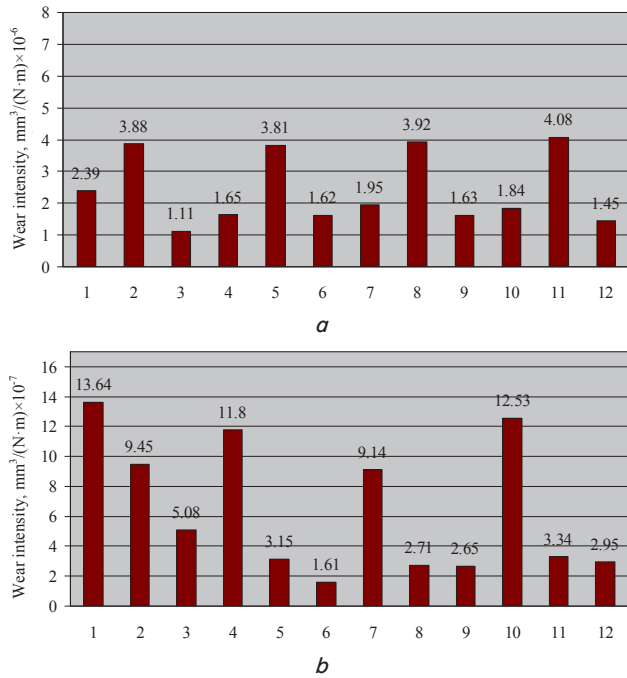


Fig. 3. The wear intensity histogram of antifriction materials F4K20, F4VV20, and F4K 10VV10, modified with zirconium oxide nanopowders: *a* – the first stage of research; *b* – the second stage of research. 1 – F4K20; 2 – F4VV20; 3 – F4K 10VV10; 4 – F4K20+1 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 5 – F4VV20+1 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 6 – F4K 10VV10+1 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 7 – F4K20+2 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 8 – F4VV20+2 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 9 – F4K 10VV10+2 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 10 – F4K20+3.0 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 11 – F4VV20+3 % by weight (ZrO₂+3 % Y₂O₃, 700 °C); 12 – F4K10VV10+3 % by weight (ZrO₂+3 % Y₂O₃, 700 °C)

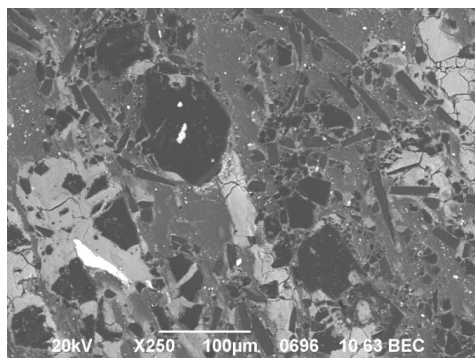


Fig. 4. Microstructure of the PTFE-composite material F4K10VV10+2 % by weight ZrO₂, 700 °C (×50)

The method of energy dispersion analysis was applied to determine the chemical composition of the friction surface of a sample from the material F4K10VV10+2 % by weight of ZrO₂, 700 °C, after testing along the friction path of 23 km under the following friction conditions: the normal load per sample, *N*_i=100 N; the sliding speed, *V*=0.3 m/s; the temperature measured at a distance of 0.5–1 mm from the surface of the counter-body, *T*=(323±2) K (Fig. 5).

Data from the chemical analysis of the sample's friction surface are given in Table 3.

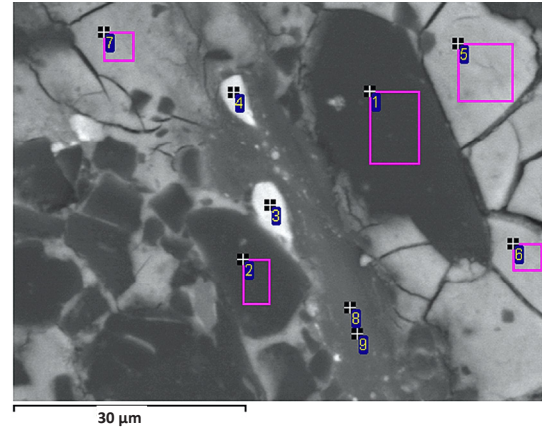


Fig. 5. The friction surface microstructure (a friction path of 23 km) of a sample made from the material F4K10VV10+2 % by weight of ZrO₂, 700 °C, with the designated zones for determining the chemical composition using an energy dispersion analysis: 1, 2 – coke and carbon fiber; 3, 4 – zirconium oxide; 5, 6, 7 – the spectra containing carbon, fluoride, iron, and oxygen; 8, 9 – fluoropolymer

Table 3

The results of the energy dispersion analysis of the friction surface of a sample made from the material F4K10VV10+2 % by weight of ZrO₂, 700 °C. Spectrum number corresponds to the analysis zones in Fig. 5

Spectrum number	C	O	F	Fe	Zr
1	96.41	3.28	0.01	0.19	0.11
2	94.76	4.51	0.29	0.37	0.07
3	40.1	9.11	10.67	0.48	39.64
4	39.47	13.8	14.07	1.34	31.32
5	23.53	26.97	8.41	37.49	3.6
6	24.84	24.91	8.15	38.34	3.76
7	28.22	22.4	18.85	27.33	3.2
8	47.37	2.23	49.87	0.3	0.23
9	50.45	2.38	43.53	0.41	3.23

Our energy dispersion analysis of the friction surface of a sample made from the material F4K10VV10+2 % by weight of ZrO₂, 700 °C, has revealed the presence of modifying impurities, carbon, and zirconium oxide, in the matrix of the fluoropolymer. The shape of carbon inclusions is rectangular and arbitrary, corresponding to coke particles and carbon fibers, respectively (Fig. 4). Their carbon content is 94–96 % (spectra 1, 2 in Fig. 5). Zirconium oxide particles (spectra 3, 4 in Fig. 5) are arranged in clusters from 0.2 to 10 µm, due to the process of their introduction into the material. The presence of oxidized iron (spectra 5, 6, 7) in the areas of the fluoropolymer (spectra 8, 9) is due to the transfer, in the process of friction, to the surface of the fluoropolymer of iron from the counter-body and its consistent oxidation (Fig. 5).

The results of testing the tensile strength of the antifriction materials F4K20, F4VV20, and F4K10VV10, modified with zirconium oxide nanopowders ZrO₂+3 % Y₂O₃, 700 °C, are shown in Fig. 6

Our analysis of the tensile strength limit of the antifriction fluoropolymer materials modified with zirconium oxide nanopowders has shown that the introduction to the composites F4K20 and F4VV20 of ZrO₂+3 % Y₂O₃ (700 °C) in the amount of 2 % by weight increases the

tensile strength by 11 % and 39 %, respectively; and, for the composite FK10VV10, the introduction of 1 % by weight of $ZrO_2+3\% Y_2O_3$ (700 °C) changes the tensile strength only slightly (Fig. 6).

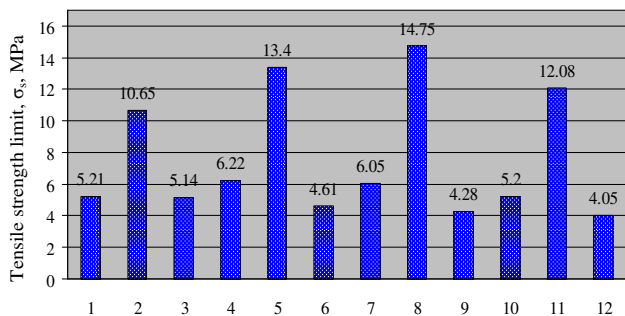


Fig. 6. The tensile strength limit histogram of the antifriction materials F4K20, F4VV20, F4K10VV10, modified with zirconium oxide nanopowders: 1 – F4K20; 2 – F4VV20; 3 – F4K10VV10; 4 – F4K20+1 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 5 – F4VV20+1 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 6 – F4K10VV10+1 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 7 – F4K20+2 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 8 – F4VV20+2 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 9 – F4K10VV10+2 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 10 – F4K20+3.0 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 11 – F4VV20+3 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C); 12 – F4K10VV10+ 3 % by weight ($ZrO_2+3\% Y_2O_3$, 700 °C)

In order to find other effective nano-modifiers from a zirconium oxide group, we have studied the composite material F4K10VV10, modified with the nanopowder of zirconium oxide $ZrO_2+3\% Y_2O_3$, at 900 °C. Investigating the wear resistance of the composite material F4K10VV10, modified with the nanopowder of zirconium oxide $ZrO_2+3\% Y_2O_3$, 900 °C, has not shown any positive result from the introduction of the nanomodifier. A tensile strength histogram of the antifriction materials F4K10VV10, modified with zirconium oxide nanopowder $ZrO_2+3\% Y_2O_3$, 900 °C, is shown in Fig. 7.

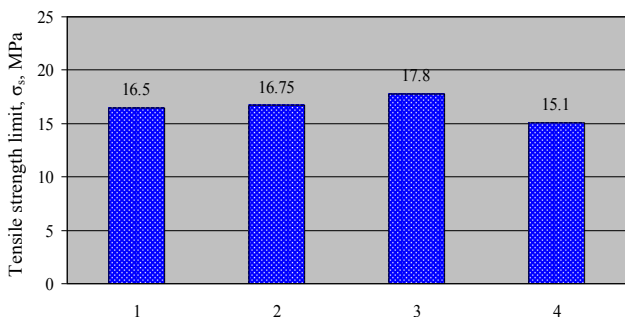


Fig. 7. A tensile strength limit histogram for the following antifriction materials: 1 – F4K10VV10; 2 – F4K10VV10+1 % by weight ($ZrO_2+3\% Y_2O_3$, 900 °C); 3 – F4K10VV10+2 % by weight ($ZrO_2+3\% Y_2O_3$, 900 °C); 4 – F4K10VV10+3 % by weight ($ZrO_2+3\% Y_2O_3$, 900 °C)

The analysis of our study results has established that the most effective is the introduction to the materials F4K20 and F4VV20 of 2 % by weight of $ZrO_2+3\% Y_2O_3$, 700 °C, to the material F4K10VV10 – 1 % by weight of $ZrO_2+3\% Y_2O_3$, 700 °C.

7. Discussion of results of the estimation-analytical and experimental studies into the properties and structure of fluoropolymer materials

An analysis of the analytical studies of fluoropolymer composites has shown that in the development of a composite antifriction material it is necessary to take into consideration, in addition to the filler dispersion, the size of the polymer's particles. Our calculations of the anti-friction carboplastics' elasticity modules indicate the presence of adhesion between the carbon fiber and polytetrafluoroethylene, which can be improved by applying onto the carbon fibers' surface a fluoropolymer coating.

The analysis of 2 has demonstrated that enhancing the adhesion properties of a carbon fiber coating is achieved through the introduction of C-1 graphite and the laminar graphite compound, intercalated by $FeCl_3$, to its composition. The higher the melting point, the better the adhesive characteristics and quality of the coating. A significant advantage of this coating is that the unmodified powder fluoropolymer 4MB, grade P, begins to decompose at 360 °C. The proposed composition can be heated up to 370 °C within one hour; no destruction occurs in this case.

The heat resistance of the coating increases due to the introduction of optimal amounts of chromium oxide and diphenylsilanediol to its composition. The tensile strength of the examined composites (Table 2) also increases.

The thickness of the polymeric layer depends on the duration of electrical deposition, the resistance of polymeric particles, and the intensity of an electrostatic field. For each material, at a certain electric field intensity, there is a limiting thickness of the layer of particles that settle. This is due to the accumulation, by a polymeric layer, of the charge that compensates for the outer field. The higher the electrostatic field intensity, the greater the charge acquired by particles, and the faster the layer is formed, which prevents further settling. As the field intensity increases, the density of the layer and adhesion grow. Increasing the density of the layer is contributed to by a decrease in the specific resistance of the material and a reduction in the size of the polymer's particles.

The disadvantages of the electrostatic application of fluoropolymer coatings include the fact that with a significant increase in the field intensity there is a decrease in the thickness of the polymeric layer. This is caused by the occurrence of the phenomenon of reverse coronation or discharge processes for materials with high electrical conductivity. In addition, the humidity of the ambient air has a significant impact. Thus, an increase in the relative air humidity by 15 % leads to a decrease in the amount of material, which settles, by larger than 2 times.

It was established that the increase in the specific surface of the filler particles leads to an increase in the share of the boundary layer in the system "matrix – filler" and the interface length in the composite materials. When using fibrous modifiers based on thermoplastic matrices, stresses may occur around the fibers under the action of mechanical load that significantly exceed the corresponding indicators in composites with dispersed fillers. Since the fibers are much stronger than the thermoplastic matrix and dispersed fillers, the strength of the created fibrous composites is limited to the possibility of constructing a strong adhesion bond at the interphase boundary.

By many indicators, the PTFE-based composites, reinforced with carbon fibers, outperform other polymeric

composites, bronze, and color alloys. At the same time, the low adsorption activity of PTFE in interaction at the interphase boundary and the lack of a viscous-liquid state in the range of composite processing temperatures prevent the formation of a strong adhesion bond at the interphase boundary. As a result, the possibility of obtaining high strength and elasticity module of fibrous fillers in PTFE-based composites decreases. Therefore, it is advisable to introduce to the anti-friction materials based on PTFE and carbon fibers the third component with a predefined functional purpose.

It is believed that dispersed and fibrous modifiers exert a different effect on the process of the structural modification of the matrix. Because the length of carbon fiber fragments (50...500 μm) is large compared to the size of the spherulites of the crystalline phase, they are distributed mainly in the amorphous phase of the matrix. As a result of grinding and mechanic activation of the carbon fiber, during the subsequent pressing and heat processing, the active segments of macromolecules are adsorbed on the active centers of the filler. This leads to a certain orientation of macromolecules in the amorphous phase and changes the ratio between the crystalline and amorphous components of the matrix. Thus, the fibrous filler is evenly distributed in PTFE and most of the fibers are involved in the interaction with the macromolecules of the matrix, which ensures an increase in the degree of its crystallinity.

In the composite, the fibrous modifier will perform the function of a strengthening agent, and the ultra-dispersed one – a wear inhibitor as a result of the participation of the modifier's particles in the process of forming a stable layer on a metal counter-body. Due to this, the wear resistance of the anti-friction composites modified with zirconium oxide nanopowders increases (Fig. 3).

The devised technological process of obtaining zirconium oxide nanopowder makes it possible to receive non-agglomerated powders with nanoscale particles. Its main advantages over others are a low production cost and the possibility to obtain powders with the predefined composition industrially. Due to minimal interaction with metals, zirconium oxide is well suited for sliding friction pairs. Consequently, zirconium oxide-based nanomaterials are promising for modifying fluoropolymer materials for tribotechnical purposes.

The greatest effect of increasing the wear resistance of a metal-polymeric friction node can be achieved when generating, in the friction zone, those components that have the properties of wear inhibitors. This phenomenon can be achieved through the directional use of tribochemical processes in the friction zone. The introduction of functional modifiers contributes to the implementation of thermal and tribo-cracking processes in a polymeric material. As a result, a lubricant is formed at the surface of friction in the form of low molecular products of the tribodestruction of the polymer with a high adhesion capacity.

The introduction of nanomodifiers contributes to the intensive structuring of the matrix, as nanoparticles can create ensembles by cluster type. As a result, a reinforced polymeric system is created, characterized by increased strength and tribotechnical characteristics.

The effect of increasing the adhesion interaction between the PTFE matrix and the filler particles is realized as a result of the effect of nanoparticles on the macromolecules of the boundary layer and the formation of microspherulite molecular formations within the volume of the composite.

The nanomodifier particles increase the adhesion interaction between the polymer and filler and enhance the mobility of PTFE structural elements. This facilitates the course of the deformation processes and leads to an increase in the strength characteristics of the composite. These effects can be explained by the fact that zirconium oxide particles are the additional centers of PTFE crystallization, resulting in reduced dimensions of the microstructure elements (Fig. 4).

Spectral analysis of the friction surface of a sample made from the material F4K10VV10+2 % by weight of $\text{ZrO}_2+3\% \text{Y}_2\text{O}_3$, 700 °C, has shown that due to the fact that the scattering area of reverse scattered electrons accepts a fluoropolymer matrix, the carbon content is 40 %, and fluoride is 10–15 %. Iron, which is part of the counter-body, is oxidized and transferred after friction to the surface of the fluoropolymer (Fig. 5).

It was established that the effect of the oxide modifier on the wear resistance of fluoropolymer carboplastic is most significant at the optimum amount (18...19.5 % by weight) of carbon fiber in the composite. When the concentration of carbon fiber is reduced in the modified fluoropolymer carboplastic, its durability decreases as a result of increasing the degree of deformation of the surface layer and narrowing of areas and the elastic-plastic deformation in interaction with the counter-body. With the increase in the composite of the concentration of carbon fiber, part of the interphase layers in the composite material grows while defects in the polymer structure decrease. The mechanism of the modifying action of fillers on the structure of PTFE is the increase in the density of structural elements as a result of the formation of interphase layers with a certain arrangement of molecules at the interface (Fig. 4).

It was found that the excess carbon fiber content in PTFE leads to a decrease in the cohesion strength of the composition due to the increase in the specific surface of fillers and a decrease in the thickness of the interphase layer in the system.

Our study into the tensile strength of the anti-friction fluoropolymer materials F4K20, F4VV20, and F4K10VV10, modified with zirconium oxide nanopowders, has shown quite a large efficiency of the introduction to their composition of 1–2 % by weight of $\text{ZrO}_2+3\% \text{Y}_2\text{O}_3$, 700 °C. As regards the composition material F4K10VV10, modified with 2 % by weight of $\text{ZrO}_2+3\% \text{Y}_2\text{O}_3$, 900 °C, its tensile strength limit increases by only 7 %; such a modifier is not effective enough (Fig. 6, 7).

At the same time, it should be noted that the effectiveness of the proposed technological measures is limited to the high cost of zirconium oxide nanopowders. Therefore, the area of further research is the search for cheaper and more effective nanomodifiers for anti-friction fluoropolymer materials.

The developed nanomodified fluoropolymer materials for tribotechnical purposes are recommended to increase the wear resistance of friction units in modern instrumentation and mechanical engineering.

8. Conclusions

1. It was established that when designing a composite anti-friction fluoropolymer material, it is necessary to take into consideration, in addition to the filler dispersion, the size of the polymer's particles. The optimum values of the

dimensionless coefficient K_S , which is the ratio of the surface of polymer's particles to the surface of filler particles, correspond to the composites F4VV15, based on the fluoropolymer 4PN and fluoropolymer 4T. The critical degree of a filler for the anti-friction carboplastic based on the fluoropolymer 4PN is 31 % by volume of the carbon fiber from fabric "Tekarm"; and, for the anti-friction fluoropolymer material modified with coke, is 25 % by volume of coke. Determining the elasticity module of the anti-friction carboplastic F4VV15 showed a good agreement between the experimental and estimation data. This indicates the presence of adhesion between the carbon fiber and polytetrafluoroethylene, which can be improved by applying, onto the carbon fibers' surface, a fluoropolymer coating.

2. Based on the results of experimental studies, it was established that the developed anti-friction carboplastics outperform, in terms of durability, by 1.97 times, and, in terms of tensile strength, by 13.7 %, carboplastics without a fluoropolymer coating and carboplastics coated with a coating made from the fluoropolymer 4MB, grade P.

3. We have determined the optimal concentration of a nanomodifier for the materials F4K20 and F4VV20 – 2 % by weight of $ZrO_2+3\% Y_2O_3$ (700 °C), for the material

F4K10VV10 – 1 % by weight of $ZrO_2+3\% Y_2O_3$ (700 °C) with a maximum structured effect. When introducing, to the materials F4K20 and F4VV20, $ZrO_2+3\% Y_2O_3$ (700 °C) in the amount of 2 % by weight, the tensile strength of the composites increases by 11 % and 39 %, respectively, while wear resistance increases by 1.49 and 3.49 times. For the material F4K10VV10, the introduction of 1 % by weight of $ZrO_2+3\% Y_2O_3$ (700 °C) slightly changes the tensile strength of the composite, while wear resistance increases by 3.16 times. The mechanism of the modifying effect of nanomodifiers on the structure of PTFE is the increase in the density of structural elements as a result of the formation of interphase layers with a certain location at the interface polymer-filler.

Acknowledgments

This study was funded from the State Budget of Ukraine within the framework of scientific project No. 0120U102070 "Forecasting the wear resistance and reliability of bearing units and the optimization of their parameters" commissioned by the Ministry of Education and Science of Ukraine.

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