Composite Material Based on Polytetrafluoroethylene and Al–Cu–Fe Quasicrystal Filler with Ultralow Wear: Morphology, Trybologic, and Mechanical Properties

Samples of composites with polytetrafluoroethylene as matrix and powder of 0, 1, 2, 4, 8, 16, and 32 vol.% Al–Cu–Fe quasicrystal as filler were prepared. Electron microscopy studies of the sample structure were carried out, the influence of the filler on sample crystallinity degree and melting and destruction temperatures was investigated, mechanical tensile tests and tribological ones were performed. Samples of composites with the filler contents 4, 8, 16, and 32 vol.% showed ultralow wear with coefficient $K < 5$ Ч 10–7 mm3/N·m. The highest wear resistance exceeding that of unfilled polytetrafluoroethylene in 2200–3100 times was registered in composites with 16 vol.% filler. Increasing in the wear resistance is associated with forming on the friction surface of a thin crust including quasicrystal particles of 0.2–0.3 μm in size, revealed by scanning electron microscopy in combination with energy dispersive analysis.

INTRODUCTION

One promising direction will be materials for friction units is an example possessing high antifriction properties of polymers such as polytetrafluoro-ethylene (PTFE), polyethylene and others. Advantage of polymeric antifriction materials consists in their high chemical resistance, low specific mass, good strength characteristics, low cost.

In a series of polymers, perspective for tribological applications, a special place is occupied by PTFE due to the most low coefficient of friction, the values of which according to different authors, are between 0.04 to 0.15 [1–7].

The disadvantage of PTFE is relatively low wear resistance, which can but enhanced by the introduction of fillers. Introduction fillers can pursue other goals, for example, increased mechanical strength, hardness and cost reduction.

A large number of studies are devoted to the impact on tribological properties of composites based on PTFE and other polymers of ceramic such as nitrides, oxides and oxynitrides transition metals and aluminum, thanks to their hardness, strength, wear and heat resistance [8].

A polymer fillers similar in properties to ceramics quasicrystalline alloys (Al-Cu-Fe and others), which are characterized by low surface energy, high hardness, low wettability and friction coefficient [17] are less studied [9–16].

The prospect of composites preparation with dispersed quasicrystalline fillers was noted in [18, 19]. Powder injection quasi-crystalline Al-Cu-Fe alloy into various polymers gave a positive effect in terms of wear resistance [9–11, 16].

Characteristics of copolymer-based composites ethylene – tetrafluoroethylene (ETFE) growth [16] was explained under the assumption of good particle adhesion filler to the fluoropolymer matrix, in the ultrahigh molecular weight polyethylene (UHMWPE) / quasicrystalline composites Al-Cu-Fe [15], where improvement was not observed due to the alleged chipping of filler particles from the polymer matrix.

In continuation of researching work of the influence of Al-Cu-Fe quasicrystalline filler on tribological and other properties of polymer composites [14–16] in this work composites in which PTFE was used as a matrix are studied.

Wear resistance, friction coefficient were measured, gravimetric and differential-thermal analyzes of composites with fillers of different concentrations were carried out.

Since mechanical properties such as modulus of elasticity, the yield strength and strength are the most important characteristics of the material, determining the possibility of its practical use [20–22]; to determine these parameters, tests on uniaxial tension were also carried out.

EXPERIMENTAL PART

PTFE powder of the fluoroplast-4 PN brand was used as a matrix of the composites. Composite samples containing 0, 1, 2, 4, 8, 16, and 32 vol.% of the quasicrystal were made. Preparation of quasi-crystalline powders, used as a filler, and their diagnostics are described in [15].

Powder is a single-phase quasicrystal of Al-Cu-Fe. The particle size distribution curve was characterized by a maximum at 6 μm and a significant fraction of particles of submicron size.

The process of polymer composites obtaining consists of three operations. In the first stage, PTFE powder was sieved through a sieve with a size cells of 1 mm and mixed in a certain proportion with the powder of quasicrystal Al-Cu-Fe in two-screw mixer at room temperature and screw speeds of 50 rpm for 10 min.

In the second stage the disks with a diameter of 50 mm and a thickness of 2 mm. are made of the mixture of powders at Hydraulic hand press at room temperature and specific pressure 29.4 ± 2.4 MPa (300 ± 25 kg / cm2), keeping under pressure for 10 minutes.

Then the mold with the workpiece was placed in an electric furnace. The temperature was raised to 350 ° C at a rate of 6 degree / min, then to 375 ° C at a rate of 1 degree / min. At a temperature of 375 ± 5 ° C, the sample was held for 13 h, then cooled to 200 ° C at a rate of 6 degrees / min. Then the furnace was turned off, and after cooling to a temperature of 50 ° C, the sample was removed from the oven.

Mechanical tests were performed using with an Instron 5965 tensile machine according to standard ASTM D 638 in constant speed mode movement of clamps (1 mm / min) with continuous fixing deformation and load on the specimen up to the gap.

To determine temperature and enthalpy of melting, as well as the degree of crystallinity samples differential PerkinElmer DSC 8500 bumper calorimeter is used.

The measurements were carried out in a stream of nitrogen (speed flow 20 ml / min) in the following mode: incubation for 1 min at 50 ° C, heating to 350 ° C at a rate of 20 degrees / min.

To determine the heat resistance, the PerkinElmer Pyris1TGA thermogravimetric analyzer was used. The measurements were carried out in a dynamic mode with a heating rate of 10 degrees / min in a stream of nitrogen 100 ml / min.
Morphology and elemental analysis of samples were examined in Helios 600 and Versa 3D raster electron-ion microscopes (FEI, USA) with accelerating voltage of 2–30 kV, equipped with a system of energy dispersive X-ray microanalysis (EDAX, USA).

The Electron detectors and detectors of back scattered electrons were used secondary. In the last, in case of heavier filler atoms appear lighter and stand out well on background of the polymer matrix.

At the same time, work in the mode of secondary electrons more information about the details of the relief and the best spatial resolution are available.

To obtain information on the transverse profile of the surface layer, a method of etching with a focused ion beam was used: a vertical wall hole was etched and an electron microscopic image of this wall was obtained.

To eliminate artifacts before ion etching onto the surface of the sample in the microscope chamber using a gas injection systems sprayed a film of platinum, and the finish etching of the wall was carried out at a small value ion current.
Material for electron microscopic studies cut from specimens subjected to rupture during mechanical testing (from the neck of the rupture and the intact part). Before the experiment, the samples were cut and chopped in liquid nitrogen.

In most cases, amorphous carbon was sprayed onto the cleaved surface to reduce the effect of charging the surface under an electron beam.

To obtain information about the change in the morphology and composition of the surface during friction, the samples were examined after tribological experiments. In this case on the sample surface was also sprayed amorphous carbon.

The friction coefficient was measured using the device T-01M (Institute for sustainable technologies, Poland) according to the “pin-on-disk” scheme, when a disk-shaped sample with a diameter of 4 mm and a thickness of 2 mm was pressed against a steel rotating disk with a diameter of 70 mm.

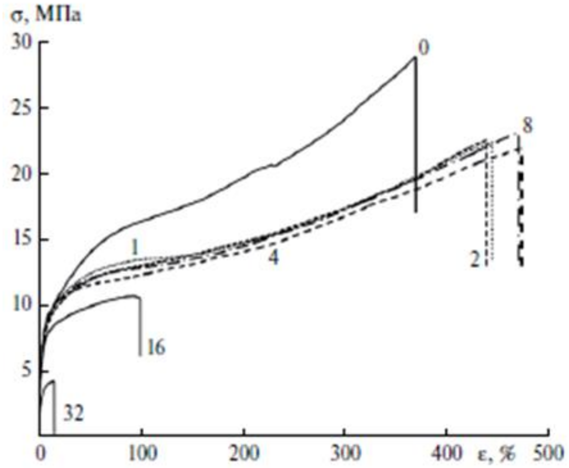
Samples for measuring the friction coefficient were cut out of the same plate as for mechanical testing. The diameter of the friction track was 50 mm, the load 20 N, rotation speed 300 rpm.

Before and after measuring of the friction coefficient the sample was weighed, and the mass loss served as a measure of wear.

RESULTS AND ITS DISCUSSION

Mechanical properties. In Pic. 1 stress – strain curves constructed based on tensile test unfilled PTFE and samples of composite materials PTFE / quasicrystalline Al-Cu-Fe with different content of filler are presented. On the deformation curves of all samples was observed linear section, the slope of which is determined by Young's modulus, then deviation from linearity and a plot close to linear, but with less steep tilt.

With further stretching of the samples with concentrations of filler from 0 to 4% by volume there was a steeper rise, the voltage reached a maximum value (in this case equal to the tensile strength) σmax, after which the gap occurred.



Pic. 1. Deformation curves of PTFE samples And Composites PTFE / Quasicrystalline Al-Cu-Fe. The numbers beside the curves indicate the content filler in vol.%.

Curves for samples with a higher filler content were different in point, when the area with a steeper rise was not observed, and σmax and the maximum relative elongation εmax with increasing concentration fell rapidly.

In all tested samples, the gap occurred by defects, which increased the scatter of σmax and εmax. Evolution of the nature of deformation curves with an increase with can be interpreted as a consequence reduce the mobility of macromolecules due to their interaction with filler particles: first, the section with flow deformation disappears, then the area of forced elastic deformation is reduced.

In fig. 2 shows the dependences of E, σmax and εmax on c.
With an increase in the amount of filler, the mechanical properties of the composite change significantly: E has a maximum at 8 vol.%, And σmax values and εmax are reduced, dependencies are observed plateau in the amount of 1–4 vol.%.The degree of crystallinity of the PTFE composites / quasicrystalline Al – Cu – Fe. Many factors affect the deformation-strength and tribological characteristics of polymers, one of which is the degree of crystallinity.
The degree of crystallinity, temperature and heat melting of investigated samples of composites are given in table. 1 (in the calculations for the degree crystallinity 100% accepted heat of fusion 68.5 J / g [23]).

The introduction of 1% by volume of filler leads to an increase in the volume of the crystalline phase (by almost 10%).
Further, with an increase in the filler content, there is a general tendency to a slight increase in the degree of crystallinity. This result is not surprising since dispersed the fillers can play the role of crystallization centers as a result of their interaction with the polymer matrix on the basis of physical adsorption or chemical bonding [24], and in many polymers the introduction of such fillers leads to an increase in degree of crystallinity.

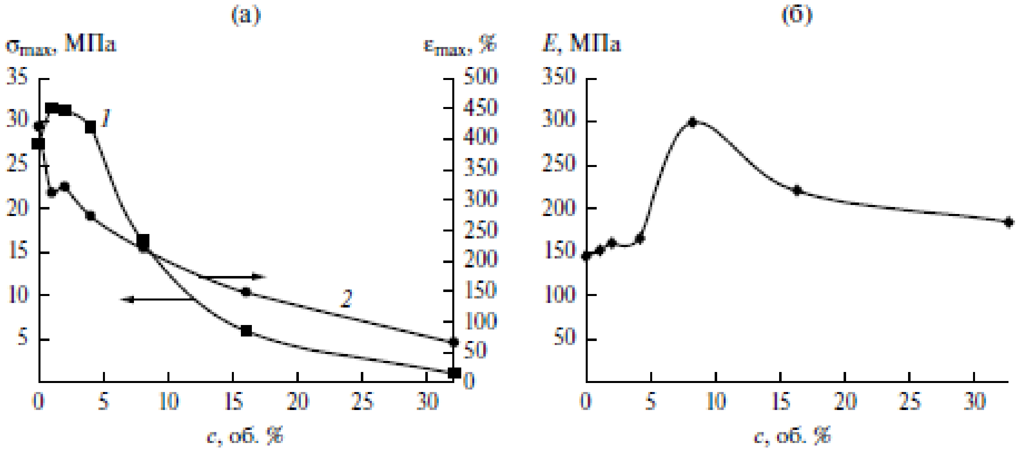
Moreover, the influence of the surface as a center for the formation of embryos is realized with moderate interaction: a strong interaction of the polymer with the surface slows down the crystallization, and the weak does not affect it [24].

Thus, you can make an assumption about moderate adhesion of quasicrystalline Al-Cu-Fe and PTFE.

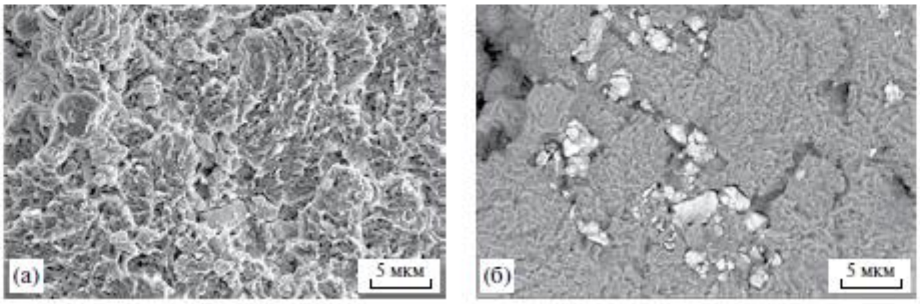
The reason for moderate adhesion can be the fact that under certain conditions the fluoropolymer can become chemically active with respect to aluminum at temperatures well below the pyrolysis temperature [25].

The increase in the degree of crystallinity in partially crystalline polymers, as a rule, leads to an increase in strength and elastic modulus, this primarily due to the higher density of the crystalline phase. However in the case of PTFE with increasing crystallinity tensile strength decreases [26].

Introduction to PTFE of a quasicrystalline also did not lead to an increase in strength. An increase in the filler content is accompanied by a decrease in σmax (Pic. 2). This can be explained by the fact that in filled polymers a large difference in the elastic moduli results in the process of deformations at the polymer – filler interface to local overvoltages that contribute to the premature formation of a trunk cracks.



Picture 2. Dependence of ultimate strength σmax (1), limiting relative elongation εmax (2) (a) and modulus of elasticity E (b) on the concentration of the filler c.



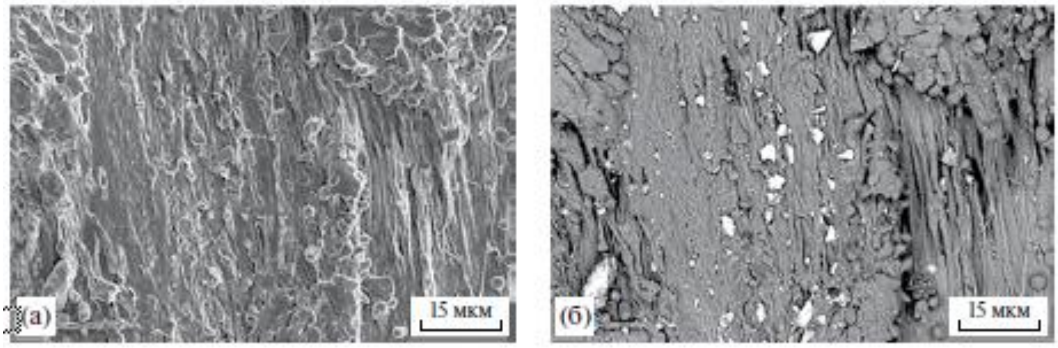
Picture 3. Electron microscopic image of the cleaved composite sample PTFE + 1% vol. Filler, obtained in the mode: a - secondary electrons; b - back scattered electrons.

This is typical for filled systems in which the size of the filler particles exceeds a certain critical value (as typically, several hundred nanometers) [27]. Also It is known that the concentration of the introduced filler is predominant in amorphous areas of the polymer, which can make it difficult for the macromolecules in these areas to stretch under tension and lead to a

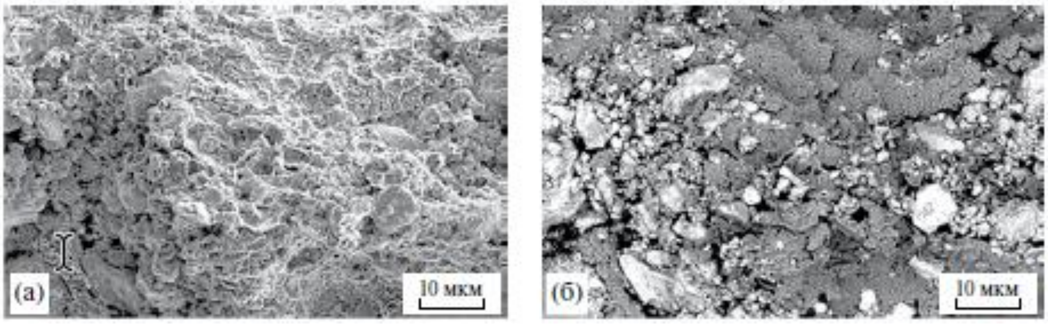
decrease in strength with increasing filler content [28]. How will It is shown below when considering the results obtained by the method of raster electron microscopy (SEM), in the studied composites the preferential concentration of the filler is observed in amorphous regions.
Study of composite samples of PTFE / quasicrystalline Al – Cu – Fe using SEM picture 3 shows the images of the cleaved nondeformed part of the sample containing 1 vol.% quasicrystal obtained in the collection mode secondary and back scattered electrons.
On picture 3b filler particles are clearly visible, while in picture 3a the same particles stand out weakly. On the other hand, the subtle features associated with the structure of the polymer matrix are more clearly shown in picture 3a. It should be noted characteristic spherical formations - “globules”, observed in the upper left corner of picture. 3b. On picture 3a, they are not clearly visible, but the lamellar structure of PTFE is more clearly visible.

Picture 4 shows images of the cleaved part of a sample containing 1% by volume of a quasicrystal, deformed (elongated) during mechanical testing. The main difference from the previous one pattern consists in the formation of fibrillar structure elongated in the direction of deformation.
“Globules” observed in picture 3b, under tension they unfold, forming fibrils with a characteristic diameter of ~ 300 nm.

In picture 5 and 6 are similar data for the sample containing 16 vol.% quasicrystal - images of chips made on the non-deformed part of the sample (picture 5) and near the fracture (picture 6). In this case in picture 6, the formation of a fibrillar structure is not observed, which is consistent with the deformation curve (picture 1). Supramolecular formations that are clearly visible in picture 5a, can be identified as spherulites, as was done, for example, in [29, 16].



Picture. 4. Electron microscopic image of the cleaved composite sample PTFE + 1% vol. Filler near the point of rupture, obtained in the mode: a - secondary electrons; b - back scattered electrons.



Picture. 5. Electron microscopic image of the cleaved composite sample PTFE + 16% vol. Filler, obtained in the mode: a - secondary electrons; b - back scattered electrons.

Picture 5b shows that the filler is distributed in dimensional matrix is uneven. In areas of more or less regular supramolecular structure (crystalline part of the matrix), the filler is almost absent. Large part of the filler is in the amorphous regions.

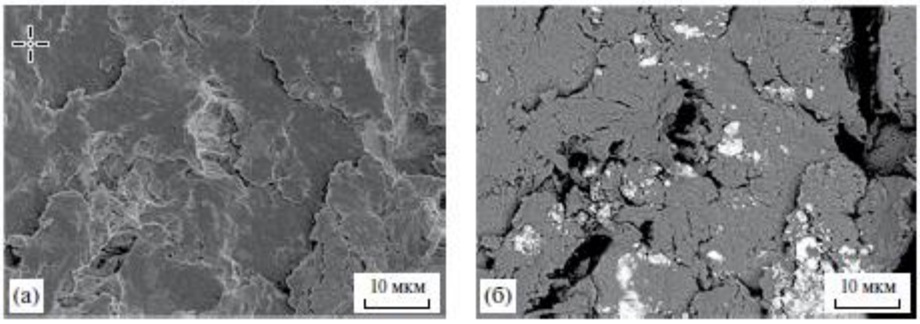
The study of the tribological properties of PTFE / Quasicrystalline Positive Samples Al – Cu – Fe.

The results of the tribological study properties are shown in picture. 7 and 8. The friction coefficient f (picture. 7) of unfilled PTFE is lower, than composites with different filler contents, but the wear resistance is so low that a0fter ~ 15 min of the experiment, the sample is almost completely abraded.

Table 1. The degree of crystallinity, temperature and heat of fusion of the investigated samples of composites

Surround Content of Quasicrystal %	Massive content PTFE,%	Melting Temperature, ° C	Heat melting (total mass) Joul / g	Heat melting (by weight PTFE) Joul / g	Crystallinity level,%
0	100	337	14.9	14.9	22 ± 3
1	98.1	335	21.2	21.6	32 ± 3
2	96.2	341	23.1	24.0	35 ± 3
4	92.6	334	20.2	21.8	32 ± 3
8	85.8	342	18.2	21.8	32 ± 3
16	73.2	336	16.6	22.7	33 ± 3
32	49.8	334	12.8	26.0	38 ± 3

Samples of composites are tested within 2 or 4 hours. The friction coefficient changes relatively strongly in the first 300–1300 seconds of experiment (picture 7), after which weaker changes may occur within ~ 10%, and in some cases there is a tendency to decrease *f.* By increasing of filler content friction coefficient in steady state (at the end of the test) *fs* experiencing a sharp rise at low seconds (up to 2–4 vol.% filler) and then slightly changes when a further increase from (picture. 8a). Adding 1 vol.% of quasicrystalline filler reduces wear of PTFE by ~ 70 times, and 16 vol.% - 2200–3100 times (picture 8b). This effect is weakened only with a higher filler content (composite with filler concentration 32 vol.% exceeds the wear resistance of unfilled PTFE by 940 times).



Picture 6. Electron-microscopic image of the cleaved composite sample PTFE + 16% vol. Filler near the point of rupture, obtained in the mode: a - secondary electrons; b - back scattered electrons.

It should be noted that the nature of temporary dependences of the friction coefficient (picture. 7) on dependencies observed in samples of quasicrystalline composites Al-Cu-Fe [14]. In the latter case, observed areas of a sharp rise in friction coefficient, moreover, with increasing filler concentration, they shifted to shorter times, which can be explained by chipping of quasicrystalline particles and their abrasive action.

The increase in wear with increasing filler concentration in UHMWPE [15] could also be explained by the chipping effect. In composites samples PTFE, as well as ETFE [16], with quasicrystalline Al-Cu-Fe as a filler, this no effect due, apparently, more

higher than in the case of UHMWPE, the adhesion of the filler to the polymer matrix.

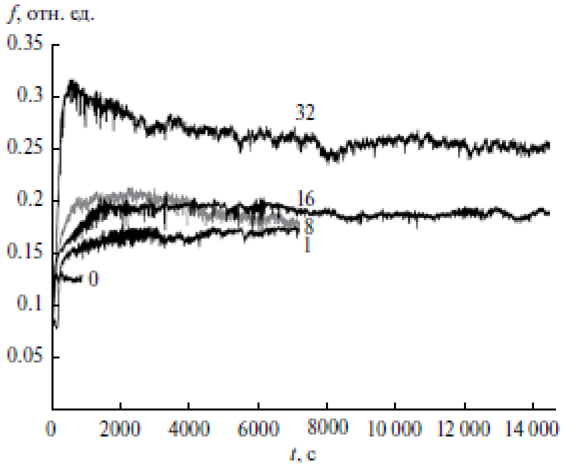
The positive effect of filling the polymer with quasicrystalline Al-Cu-Fe in increasing the wear resistance of PTFE manifests itself much stronger than in ETFE [15], and in UHMWPE [16] it generally was negative. In tab. 2 shows the data on wear rate determined by

formula:

$$K = V (m / m_0) / (Ps),$$

where V is the sample volume, m / m0 is the relative mass loss due to the passage of the friction path s under load P. In picture. 8b shows a graph of the relative wear resistance K (0) / K (c) from s.

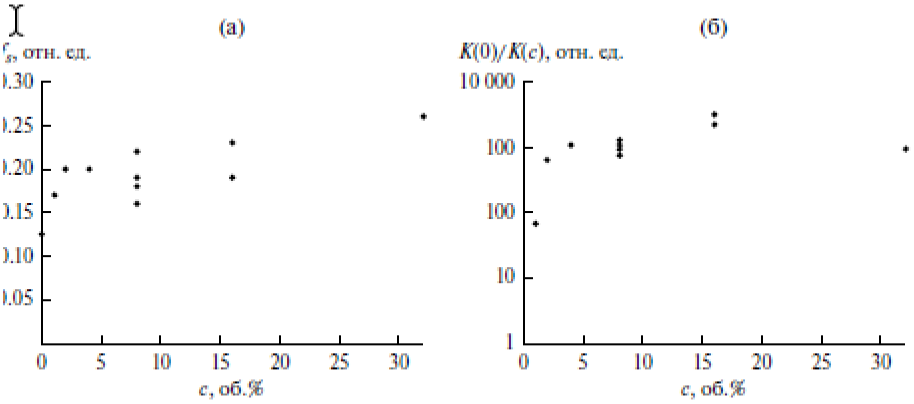
In some works, for example, in [30], the term “ultra-low wear” by the criterion K <5 × × 10–7 mm3 / N · m. These materials include PTFE-based composites, mainly with α-Al2O3 nanoscale fillers, but also graphene, carbon and so on. From the results of this work, it follows that PTFE, filled with quasicrystalline Al – Cu – Fe can also be attributed to these materials.



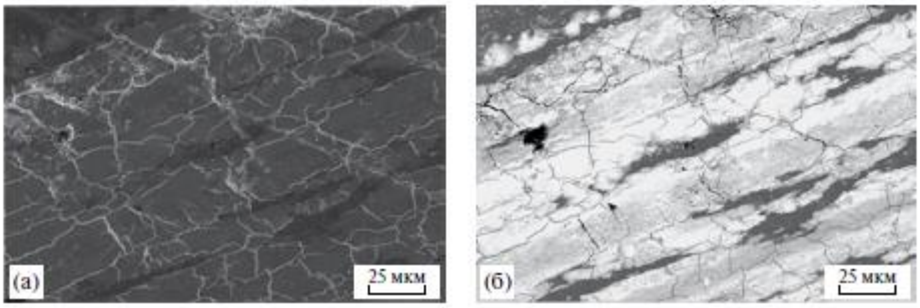
Picture. 7. Dependence of the friction coefficient f on the time of the tribological test t for samples composites and unfilled PTFE. Numbers near curves indicate the content of the filler in vol.%.

Investigation of surface modification in the process of friction using SEM.

In picture. 9 is an electron microscopic image friction surface of composite sample PTFE + 1% by volume.



Picture. 8. Dependences of friction coefficient fs in steady state (a) and relative wear resistance K (0) / K (c) (b) on the concentration of the filler c.



Picture. 9. Electron microscopic image of the friction surface of a composite sample of PTFE + 1 vol.% filler, obtained in the mode: a - secondary electrons; b - back scattered electrons.

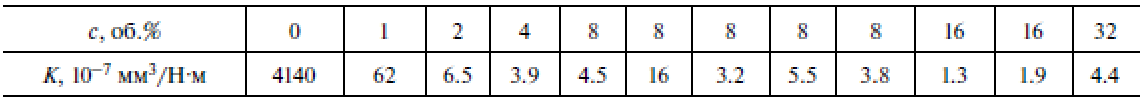
The surface is smooth and, as can be seen in pic. 9b, consists of bands containing a quasicrystal, elongated in the direction of friction. Between the bands have dark spots indicating the absence of a quasicrystalline coating in these places. Picture 10 shows a transverse cut of the surface layer made by ion etching. It can be seen that the layer, directly lying beneath the friction surface, is a crust 0.3–1 µm thick (Pic. 10). In the image, the particles forming ~ 0.2 µm in size. The composition of Al – Cu – Fe particles is confirmed by energy dispersive analysis. This crust, apparently, provides an increase in the wear resistance of composites by compared to unfilled PTFE. The part of the cut that lies below the quasicrystalline crust, practically consists of pure PTFE. It is seen in picture 11 and 12 the similar sample of PTFE + 16% by volume of filler. A very similar picture is observed: a thin, not solid crust of a quasicrystal is formed, but now in many places filler particles are visible through it.

Picture 12 shows two surface sections: under the surface large particle is visible; under the surface there are no large particles. It can be seen that the thickness of peel does not depend on the concentration of the filler in the bulk of the crystal.

The size of the filler particles forming the crust (~ 0.2 µm) is less than their average size in volume (6 µm). This can be explained by the fragmentation of particles as a result of friction.

Increased concentration of Al_2O_3 filler particles of smaller size, at the surface of friction of the composite, based on PTFE, was also registered in [31] by X-ray microtomography and electron transmission microscopy.

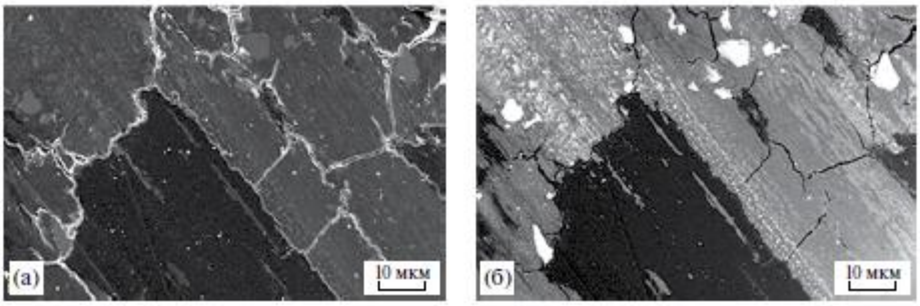
Table 2. Wear coefficient K for samples of composites with different content of the filler



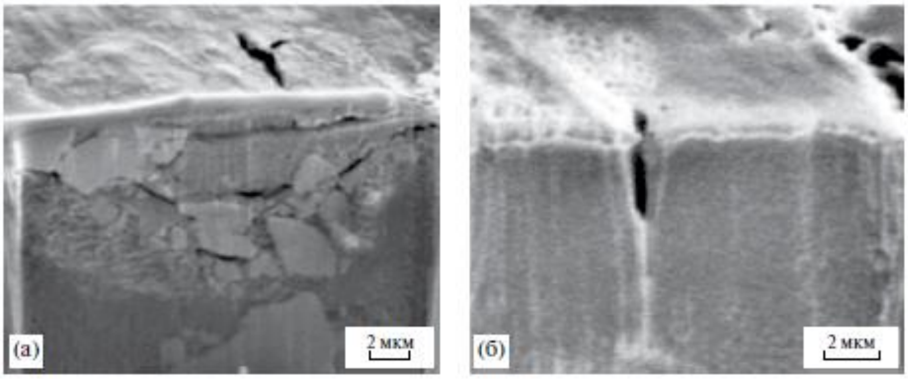
c, об.%	0	1	2	4	8	8	8	8	8	16	16	32
K, 10^{-7} мм^3/Н·м	4140	62	6.5	3.9	4.5	16	3.2	5.5	3.8	1.3	1.9	4.4

Enrichment of the of the polymer matrix friction surfaces with filler particles was observed in composites based on PTFE and other researchers [32–34]. In accordance with the concepts developed in [24, 30–38], it can be assumed that in the case under study, the crust, apparently, consists of filler particles between which there are thin layers of polymer. This crust is formed due to increasing adhesion of the filler to the polymer as a result of tribochemical processes, including tribodestruction and tribosynthesis. The local temperature flashes play the Important role in these processes. They arise from solid collisions of filler particles with protrusions of the counterbody. These processes lead to increased nucleation, possibly the formation of a cross-linked structure, and together with the formation of a transfer film on counterbody - to increase wear resistance. It is experimentally established [30], the surface layer under friction in ultra-wear-resistant PTFE / Al_2O_3 composites. It should be noted that for ultra low wear essential apparently has no original particle filler size, and their ability due to their brittleness shattered as a result of friction to nano-sizes.

As a result, a large specific surface area is possible to create. Results obtained in the present work, suggest that the state of the surface friction is equally important properties of the transfer film [30], actively studied by tribologists since their discovery in the seventies of the last century.



Picture. 11. Electron-microscopic image of the surface friction of a composite sample of PTFE + 16% by volume filler, obtained in the mode: a - secondary electrons; b - back scattered electrons.



Picture 12. Electron-microscopic image of the cross section of the friction surface of a composite sample of PTFE + 16% by volume of a filler obtained by ion etching in the mode of secondary electrons: a - there is a large particle under the surface; b - under the surface there are no large particles of filler.

CONCLUSION

Composites based on PTFE (matrix) and quasicrystalline Al-Cu-Fe (filler) with ultra low wear were prepared. The wear coefficient K <5 × 10–7 mm3 / N · m is registered in samples of composites with a content of filler 4, 8, 16 and 32%.

Ultra low deterioration of investigated composites, apparently, is equally connected with the quasicrystalline crust formation on the friction surface of the tested samples and a film transfer on the counterbody, whose existence follows from sets of published data.

In both cases, the key role belongs to the proximity of the polymer layers to the metal, thanks to which the crust and transfer film are acquired as a result of tribochemical processes properties that allow to reduce by three orders of magnitude degree of wear of the tribosystem.

The brittleness of the Al – Cu – Fe quasicrystalline filler plays an important role, due to which, under the tribomechanical effect, the filler particles are crushed to sizes of ~ 100 nm. This is established using scanning electron microscopy in combination with energy dispersive analysis.

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