

# Composite Material Based on Polytetrafluoroethylene and Al–Cu–Fe Quasi-Crystal Filler with Ultralow Wear: Morphology, Tribological, and Mechanical Properties

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**Abstract**—Samples of composites with polytetrafluoroethylene as the matrix and a powder of 0, 1, 2, 4, 8, 16, and 32 vol % Al–Cu–Fe quasi-crystal as the filler are prepared. Electron microscopy studies of the sample structure are carried out, the influence of the filler on the degree of crystallinity and the melting and destruction temperatures of the samples is investigated; mechanical tensile tests and tribological tests are performed. The composite samples with filler contents of 4, 8, 16, and 32 vol % show ultralow wear with the coefficient  $K < 5 \times 10^{-7} \text{ mm}^3/\text{N m}$ . The highest wear resistance exceeding that of unfilled polytetrafluoroethylene by 2200–3100 times is recorded in composites with 16 vol % filler. An increase in the wear resistance is associated with formation on the friction surface of a thin crust containing quasi-crystal particles 0.2–0.3  $\mu\text{m}$  in size, revealed by scanning electron microscopy in combination with energy dispersive analysis.

**Keywords:** wear resistance, quasi-crystal, composite, coefficient of friction, polytetrafluoroethylene, tribological properties, fluoropolymer

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## INTRODUCTION

One of the promising directions of fabricating materials for friction units is the use of the high-anti-friction properties of polymers such as polytetrafluoroethylene (PTFE), polyethylene and others. The advantage of polymer anti-friction materials is their high chemical resistance, low specific mass, good strength characteristics and low cost. Among the polymers promising for tribotechnical applications, PTFE has a special place due to the lowest coefficient of friction, the values of which according to the data of different authors are within 0.04 and 0.15 [1–7]. The disadvantage of PTFE is the relatively low wear resistance, which can be increased by the introduction of fillers. The introduction of fillers can also pursue other purposes, for example, increasing the mechanical strength, hardness, and also reducing the cost price. A large number of studies are devoted to the effect of PTFE-based composites and other polymers of ceramic fillers such as nitrides, oxides and oxynitrides of transition metals and aluminum on the tribotechnical properties due to their hardness, strength, wear and heat resistance [8]. Quasi-crystalline alloys (Al–Cu–Fe and others) close to ceramics in terms of their properties are less studied [9–16] as fillers of polymers, while they are characterized by a low sur-

face energy, high hardness, and a low wettability and coefficient of friction [17]. The viability of the preparation of composites with dispersed quasi-crystalline fillers was noted in [18, 19]. The introduction of quasi-crystalline Al–Cu–Fe alloy powder into different polymers gave a positive effect in terms of wear resistance [9–11, 16]. The increase in the characteristics of composites based on ethylene-tetrafluoroethylene (ETFE) copolymer [16] was explained within the framework of the assumption of good adhesion of the filler particles to the fluoropolymer matrix, unlike ultrahigh molecular weight polyethylene (UHMWPE)/quasi-crystalline Al–Cu–Fe composites [15], where no improvement was observed due to the alleged chipping of the filler particles from the polymer matrix. Continuing the study of the effect of quasi-crystalline Al–Cu–Fe filler on the tribological and other properties of polymer composites [14–16] in this work we study composites, in which PTFE is used as the matrix. The wear resistance and the coefficient of friction are measured and the gravimetric and differential-thermal analyses of the composites with fillers of different concentrations are carried out. Since the mechanical properties such as the modulus of elasticity, yield strength and strength, are the most important characteristics of the material, which determine the possibility of its practical use [20–22], uniaxial tensile

tests are carried out in order to determine these parameters.

## EXPERIMENTAL

Fluoroplast-4-PN PTFE powder was used as the matrix of the composites. Composite samples containing 0, 1, 2, 4, 8, 16, and 32 vol % of the quasi-crystalline powders used as a filler and their diagnostics are described in [15]. The powders were a single-phase Al–Cu–Fe quasi-crystal. The curve of the size distribution of particles was characterized by a maximum at 6  $\mu\text{m}$  and a considerable fraction of submicron-sized particles.

The process of manufacturing polymer composites consisted of three operations. At the first stage the PTFE powder was sieved through a sieve with a cell size of 1 mm and mixed in a certain proportion with the Al–Cu–Fe quasi-crystal powder in a twin-screw mixer at room temperature and a screw rotation speed of 50 rpm for 10 min. At the second stage disks with a diameter of 50 mm and a thickness of 2 mm were made from a mixture of powders using a hydraulic hand press at room temperature and specific pressure of  $29.4 \pm 2.4$  MPa ( $300 \pm 25$  kG/cm<sup>2</sup>) by keeping them under pressure for 10 min. Then the mold with a billet was placed in an electric oven. The temperature was increased to 350°C with a rate of 6 degrees/min, then to 375°C with a rate of 1 degree/min. The sample was kept for 13 h at a temperature of  $375 \pm 5^\circ\text{C}$ , after which it was cooled to 200°C with a rate of 6 degrees/min. Then the oven was switched off, and after cooling to a temperature of 50°C the sample was removed from the oven.

Mechanical tests were carried out using an Instron 5965 testing machine according to the ASTM D 638 standard in the mode of the constant clamping speed (1 mm/min) at continuous recording of the strain and load on the sample until breakage.

A PerkinElmer DSC 8500 differential scanning calorimeter was used for determination of the melting temperature and enthalpy, and also the degree of sample crystallinity. The measurements were carried out in a nitrogen flow (flow rate of 20 mL/min) in the following mode: holding for 1 min at 50°C, heating to 350°C with a rate of 20 degrees/min. A PerkinElmer Pyris1TGA thermogravimetric analyzer was used for determination of the heat resistance. The measurements were carried out in the dynamic mode with a heating rate of 10 degrees/min in a nitrogen flow of 100 mL/min.

The morphology and element analysis of the samples were examined using Helios 600 and Versa 3D (FEI, the United States) scanning electron microscopes (at an accelerating voltage of 2–30 kV) equipped with a system for energy-dispersive X-ray microanalysis (EDAX, the United States). Secondary

electron detectors and backscattered electron detectors were used. In the last case, the heavier filler atoms appear lighter and are distinguished well against the background of the polymer matrix. At the same time, when working in the mode of secondary electrons, more information is available on details of the relief and a better spatial resolution is achieved.

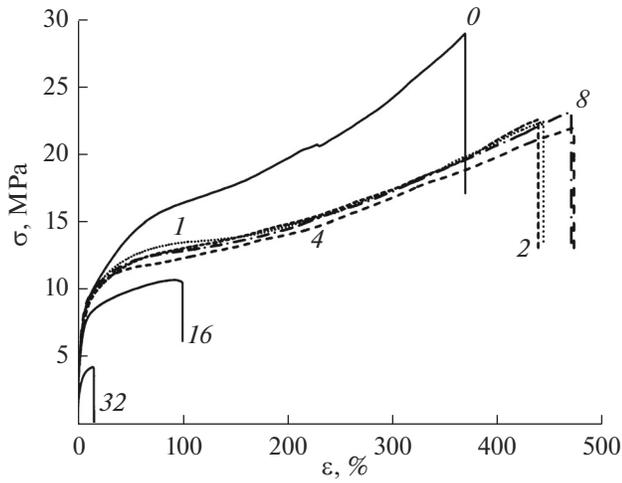
To obtain information on the transverse profile of the surface layer, an etching technique with a focused ion beam was used: a well with a vertical wall was etched and an electron microscopic image of this wall was obtained. To remove artifacts prior to ion etching, a platinum film was sputtered onto the surface of the sample in the microscope chamber using a gas-injection system, and final etching of the wall was carried out with a small ion-current value.

The material for electron microscopy studies was cut from samples that were broken off during mechanical testing (from the neck of the break and the undamaged part). Before the experiment, the samples were cut and cleaved in liquid nitrogen. On the cleavage surface, in most cases, amorphous carbon was deposited to reduce the effect of charging the surface under the electron beam. To obtain information on changes in the morphology and composition of the surface during the friction process, samples were studied after tribological experiments. In this case, amorphous carbon was also deposited onto the surface of the sample.

The coefficient of friction was measured using a T-01M device (Institute for sustainable technologies, Poland) according to the “pin-on-disk” scheme, when a sample in the shape of a disk with a diameter of 4 mm and thickness of 2 mm was pressed to a steel rotating disk with a diameter of 70 mm. Samples for measuring the coefficient of friction were cut from the same plate as for mechanical tests. The diameter of the friction track was 50 mm, the load was 20 N, and the rotation speed was 300 rpm. Before and after measuring the friction coefficient, the sample was weighed, and the weight loss served as a measure of wear.

## RESULTS AND DISCUSSION

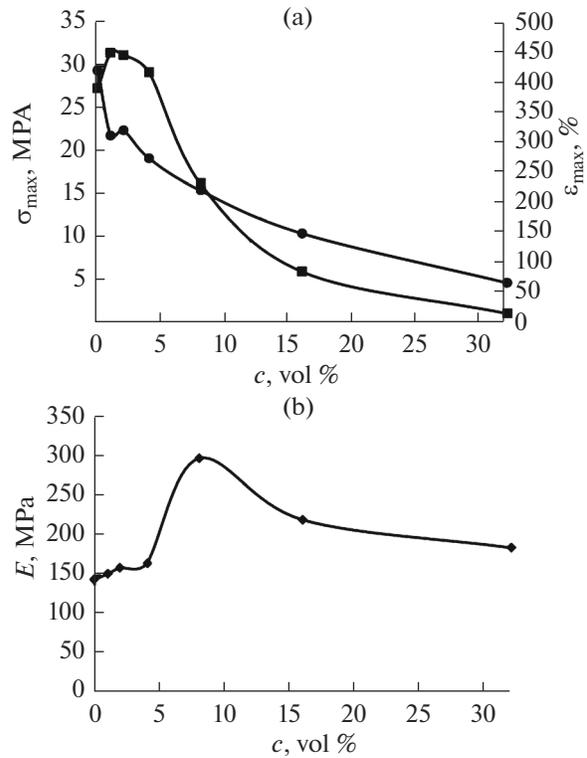
**Mechanical properties.** Figure 1 shows the stress–deformation curves plotted according to the results of tensile tests of the sample of unfilled PTFE and samples of the PTFE/quasi-crystalline Al–Cu–Fe composite materials with different filler contents. A linear section was observed on the deformation curves of all the samples, the Young’s modulus was determined from its slope, then the deviation from linearity and the section close to linear, but with a less steep slope. With further stretching of the samples with filler concentrations  $c$  from 0 to 4 vol % a steeper increase was observed, the stress reached the maximum value (in this case equal to the ultimate strength)  $\sigma_{\text{max}}$ , after which there was a break. The curves for samples with a



**Fig. 1.** Deformation curves of samples of PTFE and PTFE/quasi-crystalline Al–Cu–Fe composites. The numbers next to the curves indicate the filler content in vol %.

higher filler content differed in that the section with a steeper increase was not observed, and  $\sigma_{\max}$  and the maximum relative elongation  $\epsilon_{\max}$  with increasing concentration  $c$  fell fast. In all tested samples, the break occurred at defects, which increased the spread of  $\sigma_{\max}$  and  $\epsilon_{\max}$ . Evolution of the nature of the deformation curves with increasing  $c$  can be interpreted as a consequence of a decrease in the mobility of macromolecules due to their interaction with filler particles: first the region with deformation of the flow disappears, then the region of forced elastic deformation. At high  $c$  the character of the curves becomes typical for brittle fracturing. Figure 2 shows the dependences  $E$ ,  $\sigma_{\max}$  and  $\epsilon_{\max}$  on  $c$ . As the amount of filler increases, the mechanical properties of the composite significantly change:  $E$  has a maximum at 8 vol %, and the value  $\sigma_{\max}$  and  $\epsilon_{\max}$  decrease, the dependences show a plateau in the region 1–4 vol %.

**Degree of crystallinity of the PTFE/quasi-crystalline Al–Cu–Fe composites.** The deformation-strength and tribological characteristics of polymers are



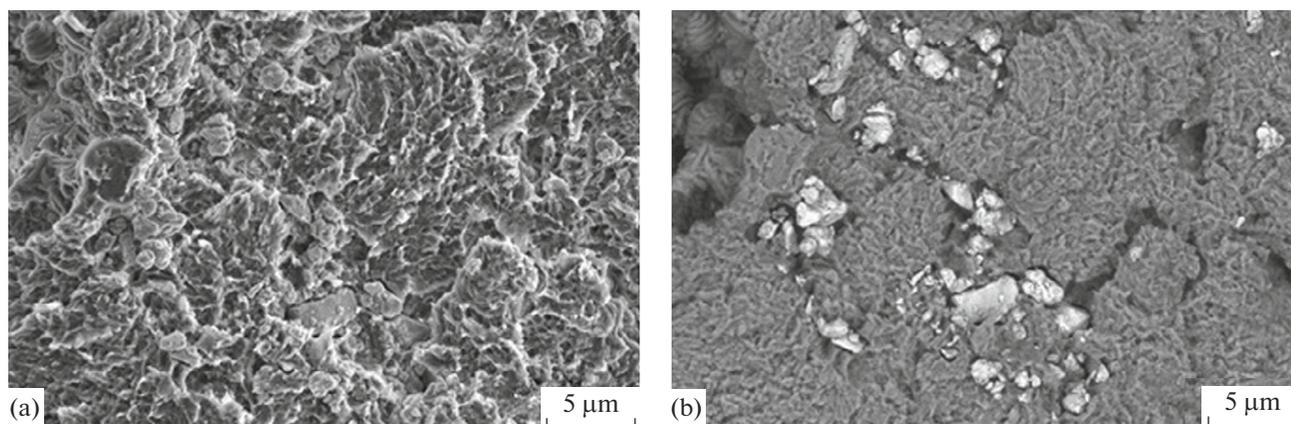
**Fig. 2.** Dependence of the maximum strength  $\sigma_{\max}$  (1), maximum relative elongation  $\epsilon_{\max}$  (2) (a) and modulus of elasticity  $E$  (b) on the filler concentration  $c$ .

affected by many factors, one of which is the degree of crystallinity. The degree of crystallinity, melting temperature and heat of the investigated samples of the composites are given in Table 1 (melting heat of 68.5 J/g is assumed for a degree of crystallinity of 100% in the calculations [23]).

The introduction of 1 vol % of the filler leads to an increase in the volume of the crystal phase (by almost 10%). Further, with increasing filler content, there is a general tendency toward a slight increase in the degree of crystallinity. This result is not surprising, since the dispersed fillers can play the role of centers of crystal-

**Table 1.** Degree of crystallinity, melting temperatures and heat of the studied samples of composites

Volume content of the quasi-crystal, vol %	PTFE mass content, %	Melting temperature, °C	Melting heat (per total mass), J/g	Melting heat (per PTFE mass), J/g	Degree of crystallinity, %
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



**Fig. 3.** Electron microscopy image of a cleavage of the PTFE + 1 vol % filler composite sample obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.

lization as a result of their interaction with a polymer matrix based on physical adsorption or chemical bonding [24], and in many polymers the introduction of such fillers leads to an increase in the degree of crystallinity. Moreover, the effect of the surface as a nucleation center is implemented at moderate interaction: strong interaction of the polymer with the surface slows down the crystallization, and weak interaction does not affect it [24]. Thus, one can assume the moderate adhesion of quasi-crystalline Al–Cu–Fe and PTFE. The origin of moderate adhesion may be the fact that under certain conditions fluoropolymer can become chemically active with respect to aluminum at temperatures well below the temperature of pyrolysis [25].

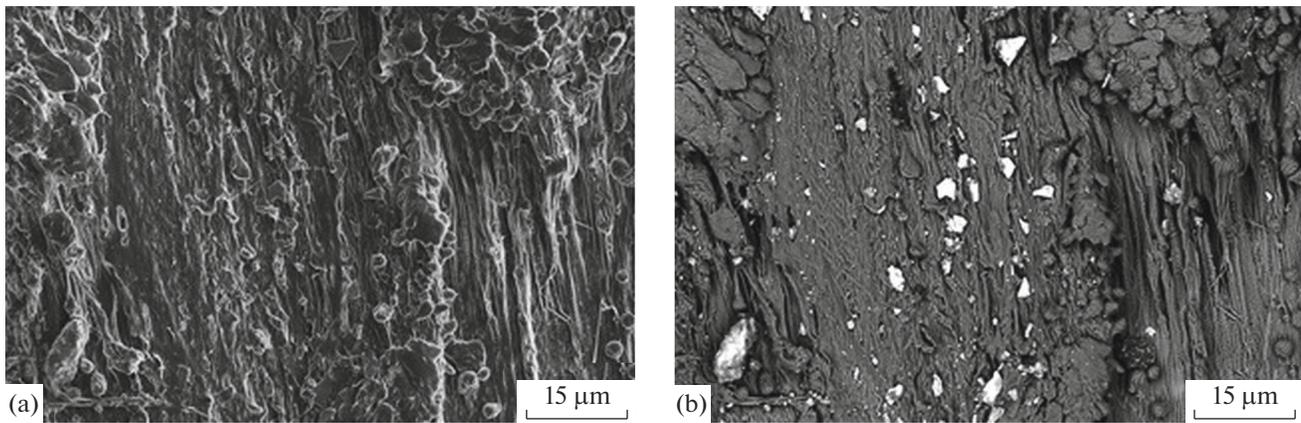
The increase in the degree of crystallinity in partially crystal polymers, as a rule, leads to an increase in the strength and modulus of elasticity, and this is due primarily to a higher density of the crystalline phase. However, in the case of PTFE, as the degree of crystallinity increases, the tensile strength decreases [26]. The introduction of the quasi-crystalline filler in PTFE also did not lead to an increase in the strength. The increase in the filler content is accompanied by a decrease in  $\sigma_{\max}$  (Fig. 2). This can be explained by the fact that in the filled polymers, a large difference in the elastic moduli results in deformation at the polymer-filler interface to local over stresses which promote the premature formation of main cracks. This is typical for filled systems in which the particle size of the filler exceeds a certain critical value (usually several hundred nanometers) [27]. The fact of the predominant concentration of the injected filler in amorphous regions of the polymer is also known, which can make it difficult to orient the macromolecules in these regions under tension and lead to a decrease in strength with increasing filler content [28]. As will be shown below when examining the results obtained by scanning electron microscopy (SEM), in the studied composites, the predominant filler concentration is observed in amorphous regions.

**Study of the composite PTFE/quasi-crystalline Al–Cu–Fe samples using SEM.** Figure 3 shows an image of the cleavage of the nondeformed part of the sample containing 1 vol % of the quasi-crystal obtained in the mode of the collection of secondary and backscattered electrons. Figure 3b shows filler particles very well, while in Fig. 3a the same particles are weakly distinguished. On the other hand, fine features associated with the structure of the polymer matrix are more distinct in Fig. 3a. One should note the characteristic spherical formations – “globules” observed in the upper left corner of Fig. 3. In Fig. 3a they are not so distinct, but the lamellar structure of PTFE is more clearly visible.

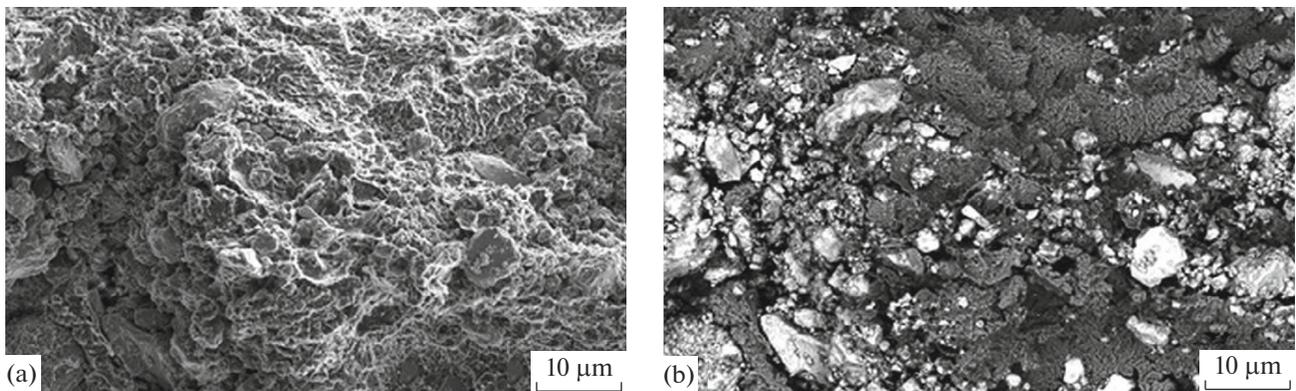
Figure 4 shows the image of the cleavage of the fraction of the sample containing 1 vol % of the quasi-crystal deformed (elongated) during mechanical tests. The main difference from the previous figure is the formation of a fibrillar structure elongated in the direction of deformation. The “globules” observed in Fig. 3b under tension unfold forming fibrils with a characteristic diameter of  $\sim 300$  nm.

Figures 5 and 6 show analogous data for the sample containing 16 vol % of the quasi-crystal – images of cleavages made on the undeformed part of the sample (Fig. 5) and near the break (Fig. 6). In this case, the formation of a fibrillar structure is not observed in Fig. 6, which is in agreement with the deformation curve (Fig. 1). Supramolecular formations, which are clearly visible in Fig. 5a, can be identified as spherulites as is done, e.g., in [29, 16]. Figure 5b shows that the filler is distributed unevenly in the polymer matrix. In the regions with a more or less regular supramolecular structure (the crystalline part of the matrix), the filler is practically lacking. Most of the filler is in amorphous regions.

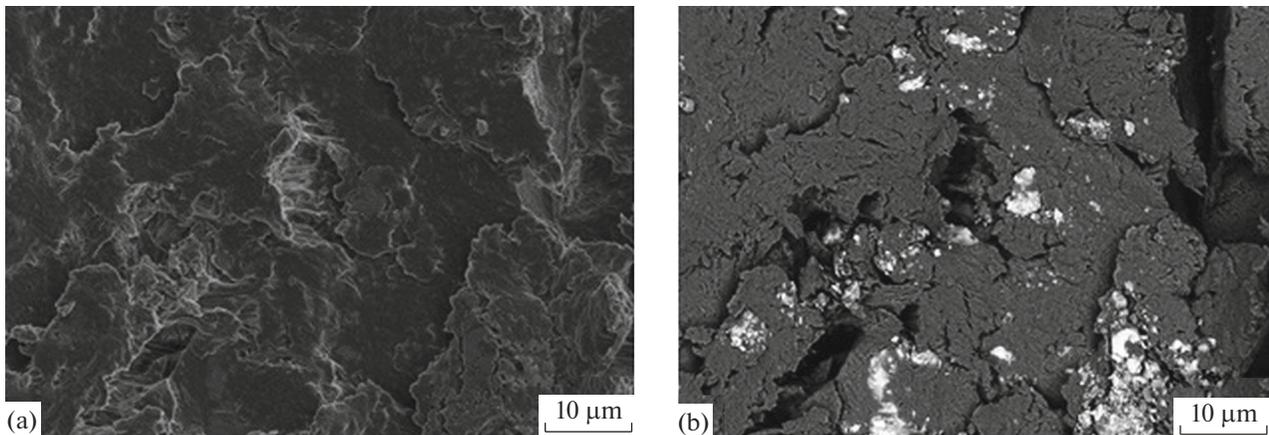
**Study of the tribological properties of PTFE/quasi-crystalline Al–Cu–Fe composite samples.** The results of studying tribological properties are presented in



**Fig. 4.** Electron microscopy image of a cleavage of the composite PTFE + 1 vol % filler sample near the place of a break obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.



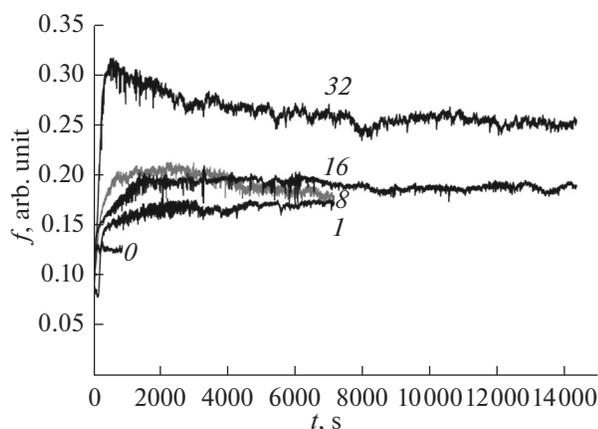
**Fig. 5.** Electron microscopy image of a cleavage of the PTFE + 16 vol % filler composite sample obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.



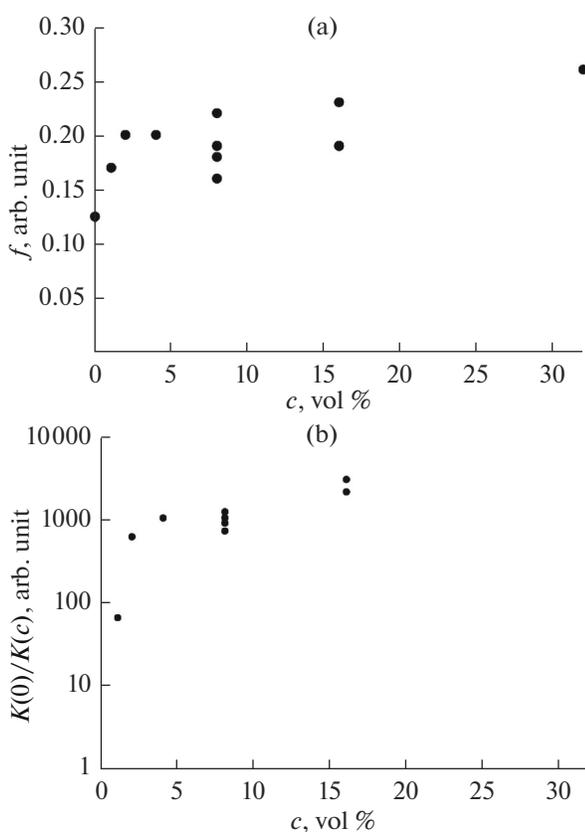
**Fig. 6.** Electron microscopy image of a cleavage of the PTFE + 16 vol % filler composite sample near the place of a break obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.

Figs. 7 and 8. The coefficient of friction  $f$  (Fig. 7) of the unfilled PTFE is lower than that of composites with different filler contents but the wear resistance is

so low that within  $\sim 15$  min of the experiment onset the sample is almost completely abraded. The composite samples were tested for 2 or 4 h. The coefficient of fric-



**Fig. 7.** Dependence of the coefficient of friction  $f_s$  on the time of the tribological test  $t$  for samples of composites and unfilled PTFE. The numbers near the curves indicate the filler content in vol %.



**Fig. 8.** Dependences of the coefficient of friction  $f_s$  in the steady-state mode (a) and the relative wear resistance  $K(0)/K(c)$  (b) on the filler concentration  $c$ .

tion changes relatively strongly in the first 300–1300 s of the experiment (breaking in) (Fig. 7), after which there may be slighter changes within  $\sim 10\%$ , moreover in some cases a tendency to a decrease in  $f_s$  is observed. When the content of the filler is increased, the coefficient of friction in the steady state (at the end of the test)  $f_s$  experiences a sharp rise at small  $c$  (to 2–4 vol % filler) and then slightly changes at a further increase in  $c$  (Fig. 8a). The addition of only 1 vol % of the quasi-crystalline filler reduces the wear of PTFE by  $\sim 70$  times, and that of 16 vol % by 2200–3100 times (Fig. 8b). This effect is attenuated only with a higher filler content (the composite with a filler concentration of 32 vol % exceeds the wear resistance of the unfilled PTFE only by 940 times).

It should be noted that the character of the time dependences of the coefficient of friction (Fig. 7) differs from the nature of the dependences observed in samples of the UHMWPE/quasi-crystalline Al–Cu–Fe composites [14]. In the latter case, areas of a sharp increase in the coefficient of friction were observed, and with an increase in the filler concentration they shifted toward smaller times, which can be explained by the chipping of quasi-crystalline particles and their abrasive action. The increase in wear with increasing filler concentration in UHMWPE [15] could also be explained by the effect of chipping. This effect is lacking in samples of PTFE composites, the same as of ETFE [16] with quasi-crystalline Al–Cu–Fe as a filler apparently due to the greater, than in the case of UHMWPE, adhesion of the filler to the polymer matrix.

The positive effect of filling the polymer with quasi-crystalline Al–Cu–Fe in increasing the wear resistance of PTFE is much stronger than in ETFE [15], and in UHMWPE [16] it was generally negative. Table 2 presents data on the coefficient of wear determined according to the formula:

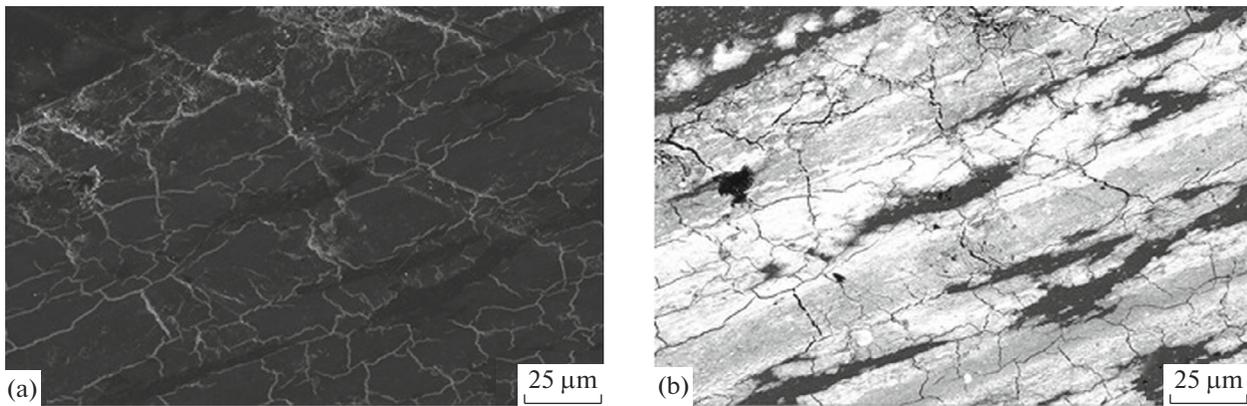
$$K = V(m/m_0)/(Ps), \quad (1)$$

where  $V$  is the sample volume,  $m/m_0$  is the relative mass loss as a result of passage of the friction path  $s$  at the load  $P$ . Figure 8b shows the plot of the dependence of the relative wear resistance  $K(0)/K(c)$  on  $c$ .

In some works, e.g., in [30], as applied to a number of materials, the term “ultra-low wear” is used according to the criterion  $K < 5 \times 10^{-7} \text{ mm}^3/\text{N m}$ . Such materials are PTFE-based composites mainly with nanoscale  $\alpha\text{-Al}_2\text{O}_3$  fillers but also graphene, carbon etc. It follows from the results of this work that PTFE

**Table 2.** Coefficient of wear  $K$  for samples of composites with different filler contents  $c$

$c$ , vol %	0	1	2	4	8	8	8	8	8	16	16	32
$K$ , $10^{-7} \text{ mm}^3/\text{N m}$	4140	62	6.5	3.9	4.5	16	3.2	5.5	3.8	1.3	1.9	4.4



**Fig. 9.** Electron microscopy image of the friction surface of the PTFE + 1 vol % filler composite sample obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.

filled with quasi-crystalline Al–Cu–Fe can also relate to these materials.

**Study of modification of the surface during the process of friction using SEM.** Figure 9 shows an electron-microscopy image of the friction surface of the PTFE + 1 vol % composite sample. The surface is smooth, and, as can be seen from Fig. 9b, consists of bands containing quasi-crystals elongated in the direction of friction. There are dark spots between the bands indicating the absence of a quasi-crystalline coating in these places. Figure 10 shows a cross-sectional view of the surface layer made by ion etching. It can be seen that the layer immediately below the friction surface is a crust with a thickness of 0.3–1 μm (Fig. 10). The image shows the particles forming the crust with a size of ~0.2 μm. The composition of Al–Cu–Fe particles is confirmed by energy-dispersive analysis data. Apparently, this crust provides an increase in the wear resistance of the composites in comparison with unfilled PTFE. A part of the section below the quasi-crystalline crust consists essentially of pure PTFE. Figures 11 and 12 show analogous images for the PTFE + 16 vol % filler sample. A very similar pattern is observed: a thin, non-continuous crust of the quasi-crystal is formed, but now in many places one can see filler particles through it. Figure 12 shows two cuts of the surface: a large particle is visible beneath the surface; under the surface there are no large particles. It can be seen that the thickness of the crust does not depend on the filler concentration in the volume of the crystal.

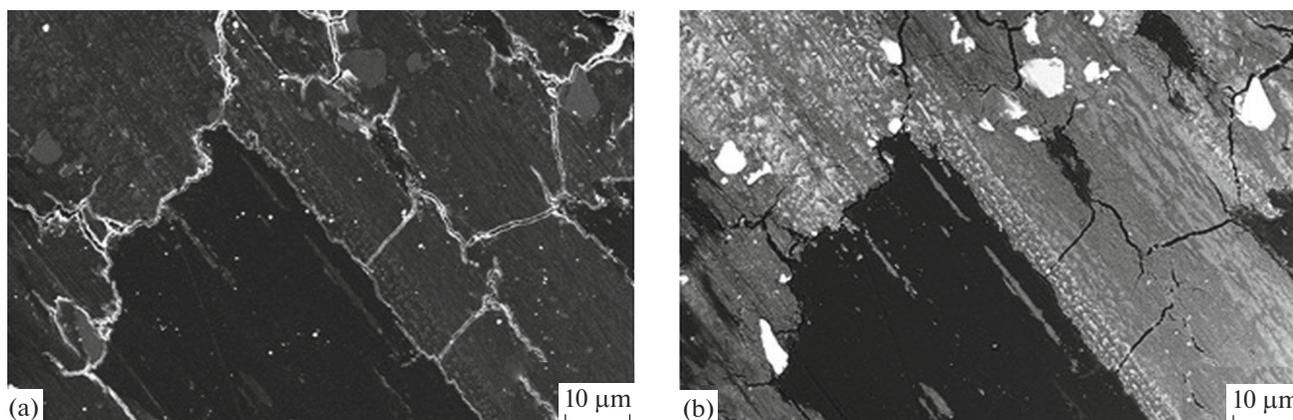
The size of filler particles forming the crust (~0.2 μm) is less than their average size in the volume (6 μm). This can be explained by the fragmentation of particles as a result of friction. The increased concentration of Al<sub>2</sub>O<sub>3</sub> filler particles of smaller size than in the volume near the friction surface of the PTFE-based composite was recorded also in [31] by X-ray microtomography and transmission electron microscopy. The enrichment of friction surfaces of the poly-

mer matrix with filler particles was observed in PTFE-based composites also by other researchers [32–34].

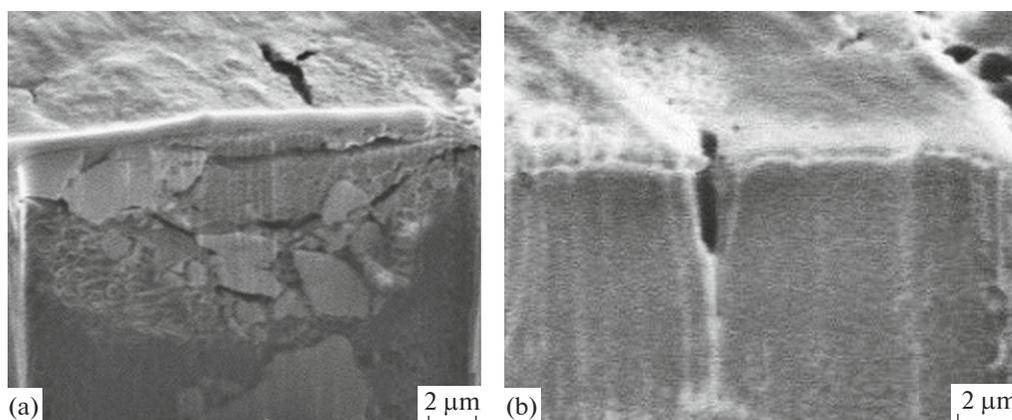
According to the ideas developed in [24, 30–38], it can be assumed that in the case under investigation, the crust appears to consist of filler particles, between which are thin polymer layers. It is formed due to an increase in the adhesion of the filler to the polymer as a result of tribochemical processes involving tribo-destruction and tribo-synthesis. An important role in these processes is played by local temperature flares that occur upon collisions of solid filler particles with protrusions of the counterbody. These processes lead to an increase in nucleation, possibly the formation of a cross-linked structure, and together with the formation of a transfer film on the counter-piece to an increase in wear resistance. Hardening of the surface layer upon friction in ultra-wear-resistant PTFE/Al<sub>2</sub>O<sub>3</sub> composites was experimentally established in [30]. It should be noted that for ultralow wear, the initial size of the filler particles does not seem to be of significant impor-



**Fig. 10.** Electron microscopy image of the cross section of the friction surface of the PTFE + 1 vol % filler composite sample fabricated by ion etching in the mode of secondary electrons.



**Fig. 11.** Electron microscopy image of the friction surface of the PTFE + 16 vol % filler composite sample obtained in the mode of: (a) secondary electrons; (b) backscattered electrons.



**Fig. 12.** Electron microscopy image of the transverse cross section of the friction surface of the PTFE + 16 vol % filler composite sample fabricated by ion etching in the mode of secondary electrons: (a) there is a large particle under the surface; (b) no large filler particles under the surface.

tance; of importance is their ability to split into nanosizes due to their brittleness as a result of friction. As a consequence, a large specific surface is achieved. The results obtained in this paper suggest that the state of the friction surface is no less important than the transfer properties [30] actively studied by tribologists from the time of their discoveries in the 1970s.

## CONCLUSIONS

Composites based on PTFE (matrix) and quasi-crystalline Al–Cu–Fe (filler) with ultra-low wear were fabricated. The coefficient of wear  $K < 5 \times 10^{-7} \text{ mm}^3/\text{N m}$  was recorded in samples of composites with a filler content of 4, 8, 16, and 32 vol %.

The ultra-low wear of the investigated composites seems to be related equally to the formation of both a quasi-crystalline crust on the friction surface of the test samples found in the present study and a transfer

film on a counter body, the existence of which follows from the set of published data.

In both cases the key role is apparently played by the proximity of the polymer layers to the metal, due to which the crust and the transfer film acquire properties as a result of tribochemical processes, which reduce the degree of wear of the tribosystem by three orders of magnitude.

An important role is played by the fragility of the Al–Cu–Fe quasi-crystalline filler, due to which under tribomechanical action the filler particles are crushed to a size of  $\sim 100 \text{ nm}$ , which is established by scanning electron microscopy in combination with energy-dispersive analysis.

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