

Development of Self-lubricating Polymeric Composites Based on PTFE

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ABSTRACT

Results of investigation of developed antifriction composites on the basis of polytetrafluoroethylene (PTFE) are presented. These composites were obtained with utilizing of mineral engine oil of M-8V brand. It has been developed several techniques for obtaining of oil-containing composites depending on the method of introducing liquid engine oil into the volume of the polymer composite. Activated natural zeolites are used as fillers, which in this case play the role not only of solid-state structurally active fillers, but as additives that intensify the process of oil absorption.

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1. INTRODUCTION

It is known that the main criterion for the reliability and durability of machines and mechanisms is the operability of parts as well as operability of tribological pairing, which include sliding bearing units, bearing shells, guiding elements, hinges, etc. The working surfaces of such parts undergo intense wear during operation, because they operate in a wide range of speeds and loading conditions - dynamic loads and vibrations; the effect of abrasive and chemically active environments; insufficient lubrication [1-6]. Such operating conditions quickly lead these parts to achieve maximum wear of their working

surfaces and, as a result, to the loss of performance of important components of the machine. The development of wear-resistant parts and using of bearings and other sliding elements able to lubricating-free functioning leads to an increase in the manufacturability of machine maintenance. In this regard, the use of self-lubricating materials in friction units is becoming increasingly important in mechanical engineering. Self-lubricating antifriction materials have an advantage over conventional materials requiring fluid and grease lubricants [7,8]. Thus, a promising direction for the creation of self-lubricating materials is the impregnation of porous materials with liquid lubricants.

Lubrication-free plane bearings can be made of antifriction materials by powder metallurgy [2,9,10]. Powder metallurgy is used to make porous friction parts sintered from powders (porous bearings, multilayer, metal-plastic and metal-glass antifriction materials), which are impregnated with liquid lubricant before use in friction units [11-13]. Parts made of such materials are used in friction pairs with insufficient lubrication or when other lubrication systems are inadmissible. Such oil-saturated porous parts have good strength and antifriction properties and are widely used in various friction units.

Recently, a promising direction is the development of oil-filled polymer composites [14-16]. Antifriction self-lubricating composite materials under the brand name "Maslyanit" are known [17-19]. Using polymers of various classes as the basis of the material, especially polyamides, introducing multifunctional fillers and additives into the polymer-plasticizer-lubricant system, it was possible to create more than 50 "Maslyanit" modifications with various physical, mechanical, thermophysical, tribological and special properties [20]. "Maslyanit" modifications are successfully operated in friction units in space and aeronautical engineering, in agricultural and automotive engineering, as sliding bearings - in hydraulic structures [21,22].

This paper presents the results of studies on the development of polymer composite materials based on polytetrafluoroethylene (PTFE), obtained using mineral engine oil M-8V brand. Several techniques for producing oil-filled PTFE-based composites are presented, depending on the method of introducing liquid engine oil into the volume of the polymer composite. Activated natural zeolites were used as fillers for PTFE. Due to the fact that zeolites are aluminosilicate microporous adsorbents, in this case zeolites are used not only as solid-phase structurally active PTFE fillers, but also as additives that intensify the process of oil absorption.

2. EXPERIMENTAL

2.1 Materials and methods

Polytetrafluoroethylene, grade PN (GOST 10007-80 [23]) and composites based on it were used in this research. A natural zeolite rock of the

Khonguruu deposit (Republic of Sakha (Yakutia) was used as a filler of polymer composite materials (PCM) [24,25]. Zeolites are sorbents with microporous structure, have high adsorption activity and molecular sieve properties. Before using zeolites has been mechanically activated in AGO-2 planetary mill (Russia) for 2 minutes at 3000 rpm.

As a liquid lubricant engine oil M-8V brand was used (GOST 10541-78 [26]). Engine oil M-8V brand by designation should be understood: M - engine (motor) oil; 8 - viscosity grade, means this summer oil with kinematic viscosity, mm² / s, at a temperature of 100°C - 7.0-9.3; V - universal oil for medium-boosted diesel and carburetor engines.

Tribological characteristics were determined on a friction machine "SMT-2" ("Tochpribor", Russia) according to R 50-54-107-88 [27]. "Shaft-bushing" scheme was used with a contact pressure of 0.5-2 MPa and a sliding speed of 0.5 m/s. The mass wear rate was estimated by the weight loss of the samples per time interval. The measurements were carried out on a ViBRA HT analytical balance (SHINKO DENSHI Co., Ltd, Japan).

To obtain information about the composition of engine oil, as well as, to register changes in the friction surface of composite before and after wear tests, IR-spectroscopic analysis was carried out. Varian 7000 FT-IR apparatus was used to collect spectra of the samples. All spectra consisted of 32 scans with a spectral resolution of 4 cm⁻¹. The spectra were collected by using PIKE MIRacle attenuated total reflectance (ATR) accessory with diamond crystal.

Structural studies were carried out using a JSM-6480LV scanning electron microscope (JEOL, Japan) with an X-ray spectral attachment and TM1000 tabletop scanning electron microscope (Hitachi, Japan).

Optical microscope BX51 (Olympus, Japan) was used to taking images of counterbody surface after wear tests.

Studies of the composition of the extract of the oil residue from PCM were carried out by gas-liquid chromatography on a GCMS-QP2010 Plus gas chromatography mass spectrometer.

2.2 Samples preparation

The first technique for producing oil-filled polymer composite materials (PCM) is to add oil into the porous polymer sample, followed by sintering the sample. Porosity of the samples was provided by reducing the pressure during samples molding from polymer powder.

Standard pressure applied to form solid sample from PTFE powder is 50 MPa and in this case this value was reduced by 2 or more times. The experimentally established minimum pressure to form suitable samples corresponds to 12.5 MPa. Density, porosity and oil content in the samples before and after sintering were calculated according to GOST 18898-89 [28] (table 1). The impregnation of porous materials with liquid lubricant is based on the phenomena of immersion wetting and the process of spontaneous impregnation, without the application of external forces. Spontaneous (free) impregnation of polymer porous preforms is carried out when they are completely immersed in the liquid phase, which ensures comprehensive penetration of oil into the volume of the composite [29].

Table 1. Dependence of the total porosity and content of oil in the sample before sintering on the molding pressure of samples.

Material	Porosity, %	P _{press} , MPa	T _{imp} , °C	C _{oil} , wt. %
PTFE	4.3	50	25	-
			150	0.24
	15.4	12.5	25	3.19
			150	6.04
	7.5	25.0	25	0.61
			150	2.84
PTFE+5 wt.% zeolite	17.4	12.5	25	-
	8.3	25.0		-
			25.0	150

Note: P_{press} - molding pressure; C_{oil} - content of oil in the sample; T_{imp} - impregnation temperature

As can be seen from table 1, a decrease in the molding pressure leads to a decrease in the density of the sample by 3-13%, while the porosity of the samples increases by 3.2-11%. The introduction of zeolites with their own porosity into PTFE leads to an increase in the total porosity of the composites.

3. RESULTS AND DISCUSSION

The results of tribological tests of PCM obtained by impregnation at two different temperatures and tested at two different contact pressure are given in table 2.

Table 2. Dependence of the tribological characteristics of PTFE composites + 5 wt.% activated zeolite on the temperature of impregnation and contact pressure.

Material	P _{press} , MPa	T _{imp} , °C	f		I, mg/h	
			1 MPa	2 MPa	1 MPa	2 MPa
PTFE	50	-	0.24	0.24	106.4	312.3
PTFE+5 wt.% zeolite+ M-8V oil	12.5	25	0.24	0.23	1.5	3.1
	12.5	150	0.21	0.16	0.2	0.5
	25.0	25	0.23	0.19	0.1	2.0
	25.0	150	0.19	0.17	0.1	0.3
F4K20 (PTFE+20 wt.% coke)	50	-	-	0.22	-	0.2

Note: f - coefficient of friction; I - mass wear rate.

The polymer composites obtained by this technique are up to 100-1000 times wear resistant depending on the contact pressure and have the same level of friction coefficient like initial PTFE. It can also be noted that the wear resistance of the developed materials is comparable to that of the commercially available material F4K20.

As can be seen from table 2, to obtain more wear-resistant oil-modified composites with zeolite, it should be made at a specific pressing pressure of 25 MPa. In this regard, mechanical tests were carried out on samples obtained at a specific pressing pressure of 25 MPa (table 3.)

Table 3. Compressive strength and compressive modulus of elasticity of samples.

Material	P _{press} , MPa	σ _{comp} at 25% deformation, MPa	E, MPa
PTFE	50	23 ± 0.9	328 ± 12
porous PTFE	25	22 ± 0.7	267 ± 10
PTFE+5 wt.% zeolite composite (modified by engine oil)	25	26 ± 1.1	390 ± 15

Note: σ_{comp} - compressive strength; E - compressive modulus of elasticity.

Table 3 shows the results of mechanical tests of samples under compression. It is clearly shown that the value of the compressive strength of the sample obtained at 25 MPa practically does not change in comparison with usually prepared PTFE samples, but the compressive modulus of elasticity decreases. When preparing a composite with a zeolite using a pressure of 25 MPa and modifying it with oil, an increase in both the compressive modulus of elasticity and the compressive strength is observed in comparison with the initial PTFE obtained using the standard pressing pressure. An increase in these parameters determines the stability of the material under load without plastic deformation, which is accompanied by an increase in the wear resistance of the composites.

To find out the more reason for the increase in wear resistance of the composites, IR spectroscopic studies were carried out.

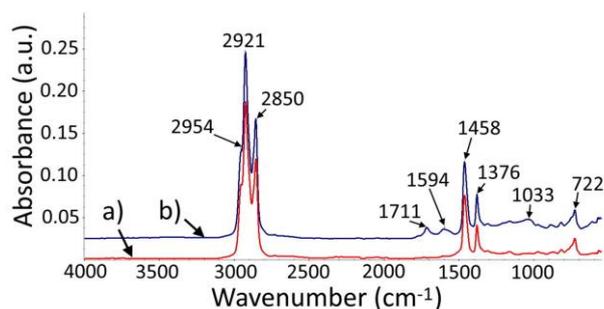


Fig. 1. IR-spectra of initial (a) M-8V oil and after heating (b).

In fig. 1 IR spectra of engine oil M-8V brand before and after exposure for 1.5 hours at a temperature of 375°C are shown. It was established that engine oil M-8V brand consists of a mixture of saturated hydrocarbons of a linear structure (Fig. 1, a). For alkanes, such components are CH₃ methyl groups (absorption bands 1376, 2954 cm⁻¹), also methylene CH₂ groups (absorption bands 1458, 2850, and 2921 cm⁻¹) and main-chain vibrations -CH₂-CH₂-CH₂- (absorption band 722 cm⁻¹) [30].

The absorption bands at 1458 cm⁻¹ are more intense than 1376 cm⁻¹, which indicates the absence of oxidized groups. There is weak absorption in the 1300-1100 cm⁻¹ region, which indicates wagging of the CH₂ groups, which also indicates the absence of oxidized groups. It was found that the absorption band corresponding to

a frequency of 1376 cm⁻¹ does not split into a doublet, which indicates that the hydrocarbons contained in the M-8V engine oil do not have branches, i.e. linear structure.

The spectra of engine oil aged at the temperature of processing of PTFE (Fig. 1, b) show additional peaks of average intensity at a frequency of ~ 1711 cm⁻¹ and a broad average intensity band of 1594 cm⁻¹. The first peak indicates the presence of a carbonyl group, and the second peak indicates the presence of double bonds conjugated to the carbonyl group. The presence of the oxidized group and double bond is explained by the fact that during the processing of the oil-filled composite based on PTFE, the process of oxidative dehydrogenation occurs, as a result of which oxygen-containing compounds with double bonds are formed [31]. Figure 2 shows the IR spectra of the surfaces of the composites before and after friction.

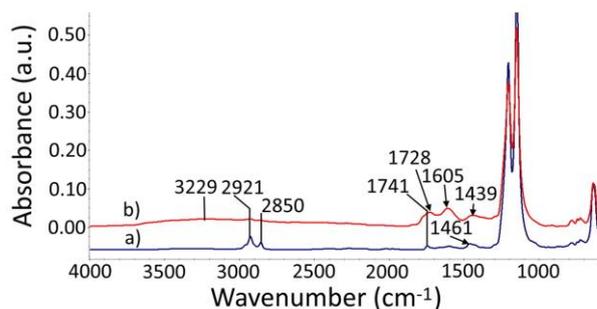


Fig. 2. IR spectra before and after wear test of PCM, modified with engine oil: a) before friction; b) after friction.

A peak at 1741 cm⁻¹, corresponding to stretching vibrations of the carbonyl group C = O, as well as peaks at 2921, 2850 and 1461 cm⁻¹, related to vibrations of methylene groups (Fig. 2), were detected in the IR spectra of the surface before friction. The peak intensities at 2921 and 2850 cm⁻¹ in the IR spectra of composites decrease after friction. This is due to the fact that in the process of friction, hydrocarbon groups undergo oxidation. This can be confirmed by the fact that the intensity of the peak at 1728 cm⁻¹, which indicates the presence of a carbonyl group, increases. On the friction surface of the samples, absorption peaks are observed at 1605 and 1439 cm⁻¹, which indicates the presence of carboxylic acid salts on the friction surface, since the vibrations at 1600 and 1400 cm⁻¹ are peaks of

asymmetric and symmetric stretching vibrations of C-O in the carboxylate anion, respectively [32]. The presence of a peak at 1728 cm^{-1} not participating in the formation of the carboxylate anion simultaneously with the presence of a wide peak at 3229 cm^{-1} , which refers to -OH, can serve as evidence of existing of carboxylic acids on the friction surface [33].

Identifying of the extract of the oil residue from PCM by gas-liquid chromatography showed that the engine oil after heat treatment at a processing temperature of PTFE ($375\text{-}380^\circ\text{C}$) contains higher (solid) aliphatic hydrocarbons, various oxygen-containing and polycyclic aromatic compounds.

In oil-modified PCMs the process of oxidation of engine oil intensifies with temperature increasing, which leads to an increase in the content of acidic components in the PCM volume. Acidic components contained in composite act as inhibitors of wear of the metal-polymer tribosystem [34]. The function of inhibitors is that the components of oils, especially those of an acidic nature, have the properties of surface-active substances (surfactants), and chemically react with the counterbody metal, form substances such as salts (soaps), which play the role of a boundary lubricating layer [35].

The wear process of the initial PTFE is accompanied by the formation of large, flocculent wear debris. The transfer of the polymer to counterbody surface occurs discretely in the form of "islands" of a predominantly round shape up to about 5 microns in size (Fig. 3, a), which are easily carried away from the friction zone. This fact leads to intensive formation of wear debris.

In the case of wear tests of oil-modified composites, the transfer of material to the counterbody surface also occurs discretely, but the shapes of these formations are more elongated along the friction direction and the amount of material transferred to the counterbody is noticeably greater (Fig. 3, b), while the formation of wear debris is minimal. This indicates an increase in the adhesive interaction of wear debris with a metal counterbody, as a result a strong transfer film is formed, which protects the composite from wear.

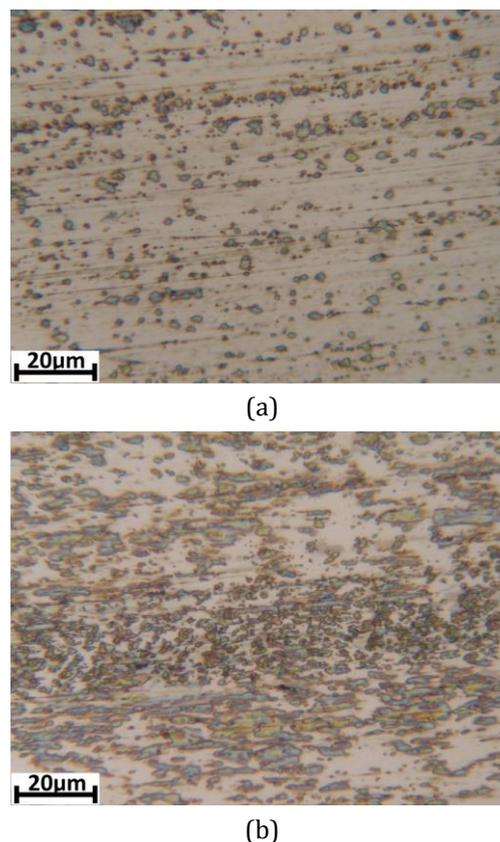


Fig. 3. Images of counterbody surface after wear tests of: a) initial PTFE; b) PTFE based oil-modified composite. Magnification 1750x.

Structural studies have shown that the components of a liquid lubricant are directly involved in the processes of polymer structure formation. It was revealed that during oil modification, the tape structure of PTFE is transformed into a spherulite type structure (Fig. 4). Along the boundaries of spherulitic formations, small round formations are registered (Fig. 5 a, b). Similar formations were registered by Okhlopkova et al. [36] when PTFE powder particles were heated. According to their observations, these formations are formed on that part of the surface of the powder particles that does not come into contact with other particles. Therefore, these formations in the volume of the composite could form only in those areas where the pores are located. For a more detailed clarification, low-temperature PCM chips were studied by scanning electron microscopy with EDS analysis (Fig. 5 c, d). It has been established that the dark regions correspond to the polymer, and the regions with small round formations are pores with the content of residual oil components. Presence of Cl, Ca, F, Na, Al, Si was registered in the spectrum at point located on round formation. This fact indicates the presence of engine oil components in pores of composites.

The formation of the spherulite structure is due to the fact that, firstly, the oil components act as nucleating agents, and secondly, the oil being in the interchain regions in the polymer increases the mobility of macromolecules and the reorientation of fibrils to spherulites occurs.

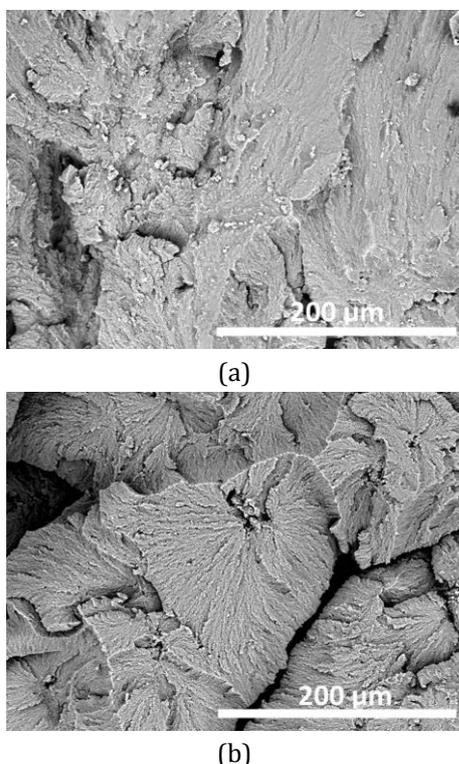


Fig. 4. The supramolecular structure of the initial PTFE (a) and PTFE modified with engine oil (b).

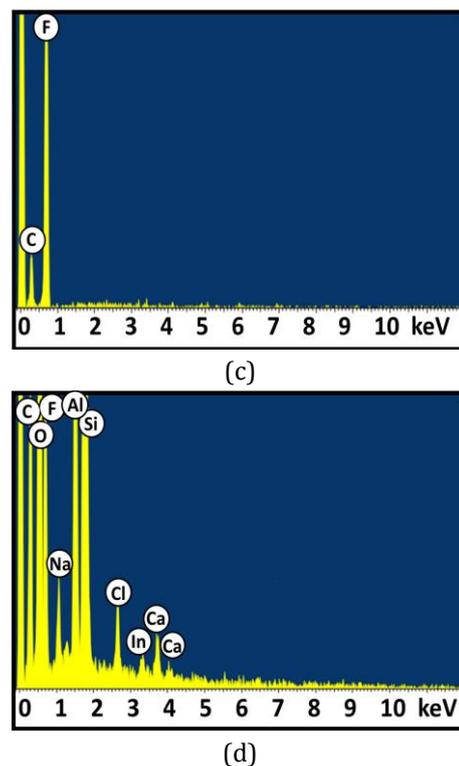
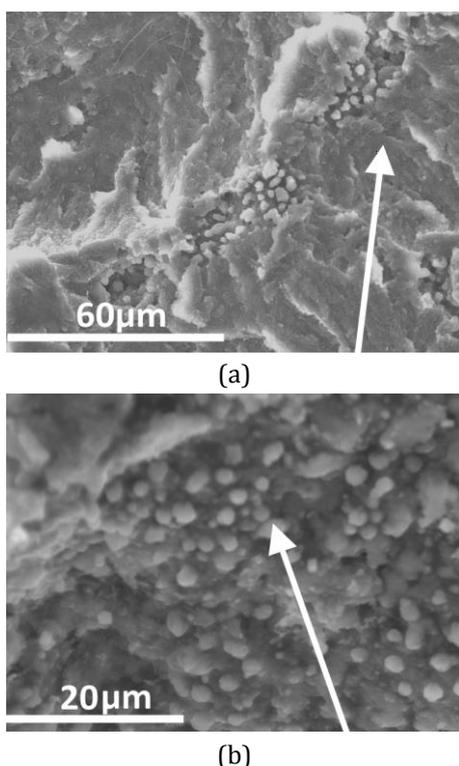


Fig. 5. EDS spectra (c, d) of oil-modified PCM and locations of points (a, b).

In work [22], by introducing plasticizers, fillers and additives, an anti-friction self-lubricating material based on P-610 polyamide was developed. The plasticization process consists of introducing into the polymer during its processing liquids that facilitate the processing of the polymer and change its properties. As a plasticizer, automotive lubricant No. 158 was chosen, made on the basis of MS-20 aviation oil and possessing a number of required properties. The introduction of lubricant into the polymer in large quantities (up to 30%) is technologically not possible. In order to increase the content of introduced lubricant method of preliminary adsorption of the lubricant by filler was used in study. As filler mixture of graphite and molybdenum disulfide was used, which has a layered structure and high antifriction properties. Utilizing of this complex filler improved the lubricating properties of the plasticizer by 40%.

In this regard, the next method of introducing liquid lubricant into the polymer composite was utilizing activated natural zeolites, previously impregnated with M-8V brand engine oil. Further, the oil-saturated zeolite was used as a filler for PTFE. Test samples were prepared using standard PTFE processing

technology. It was found that the oil content in composites filled with 2 and 5 wt.% zeolite was 0.6 and 1.4 wt.%, respectively. When PTFE filled with zeolite, previously impregnated with engine oil, a decrease in the mass wear rate up to 63-600 times was established depending on the contact pressure during friction. The friction coefficient decreased up to 2 times (Table 4).

Table 4. Tribological characteristics of PCM at contact pressure 1 and 2 MPa.

Material	1 MPa			2 MPa		
	I, mg/h	f	T, °C	I, mg/h	f	T, °C
PTFE	106.4	0.24	-	312.3	0.24	-
PTFE+2 wt.% zeolite oil adsorbed	2.3	0.23-0.30	40-46	29.5	0.16-0.24	55-60
PTFE+5 wt.% zeolite oil adsorbed	1.7	0.16-0.20	50-55	4.8	0.18-0.22	65-70

Using of these two methods leads to increased wear resistance and decreased coefficient of friction. However, oil in these composites undergoes profound changes during the heat treatment.

Table 5. Comparison of the tribotechnical characteristics of porous composites based on PTFE and natural zeolites.

Material	I, mg/h	f	T, °C
PTFE	106.4	0.21-0.24	65-70
PTFE+20 wt.% NH ₄ Cl+M-8V	206.2	0.07-0.17	60-65
PTFE+25 wt.% NH ₄ Cl+M-8V	489.5	0.07-0.12	60-65
PTFE+30 wt.% NH ₄ Cl+M-8V	663.4	0.10-0.13	60-65
PTFE+5 wt.% zeolite+20 wt.% NH ₄ Cl+M-8V	1.2	0.11-0.13	37-42
PTFE+5 wt.% zeolite+25 wt.% NH ₄ Cl+M-8V	4.6	0.11-0.17	38-40
PTFE+5 wt.% zeolite+30 wt.% NH ₄ Cl+M-8V	5.7	0.08-0.17	40-45
PTFE+5 wt.% zeolite oil adsorbed	1.7	0.09-0.11	50-55
PTFE+5 wt.% zeolite	1.4	0.27-0.32	65-73

In this regard, materials based on PTFE with an open-porous structure have been developed to give them the ability to absorb oils after the heat treatment stage. Activated natural zeolites are also used as fillers. Ammonium chloride was used as a blowing agent. Oil addition in these composites was carried out after the heat treatment stage. The oil content in the composites was 1-2.5 wt. % (table 5).

When using a blowing agent and introducing natural zeolites in PTFE in an amount of 5 wt.%, a decrease in the mass wear rate up to 90 times, a friction coefficient up to 2 times, a temperature in the contact zone up to 2 times compared with the initial PTFE are observed. An increase in wear resistance and a decrease in the coefficient of friction is due to the fact that the oil contained in the pores of the composite is squeezed out from the bulk of the polymer material as it wears during friction. Formation of lubricant layers on the friction surfaces leads to a decrease in the coefficient of friction. Thus, a constant supply of lubricant to the friction zone is provided. However, the resource of the developed composites is limited due to the fact that the amount of absorbed engine oil is small.

4. CONCLUSION

It was found that the most wear-resistant are oil-modified composites obtained by impregnation of porous samples with engine oil, followed by heat treatment. It has been established that hydrocarbon engine oils undergo a number of changes in the volume of the composite during sintering, such as oxidation with the formation of various oxygen-containing compounds. The products of complete and incomplete combustion of engine oil during PCM sintering at 375 ° C play a significant role in the formation of a tribo-resistant structure both in the volume and in the surface layer of the composite.

The reason for the increase in wear resistance is the increase in the adhesive interaction between PCM and the counterbody surface due to the presence of oxygen-containing compounds in the bulk and surface of composites. It contributes to the formation of a polymer layer firmly adhering to the counterbody, which prevents direct contact of sliding surfaces, thereby protecting the

composites from wear. In addition, oxygen-containing compounds, which are characterized by the presence of a mobile hydrogen atom, play the role of wear inhibitors and break the chain of radical oxidation reactions of the polymer material.

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