Research and Prediction of Wear Resistance of Polymer Composites with Mineral Additives

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Abstract—The main regularities of friction and wear of surface layers of composite materials for antifriction and friction purposes have been established. It has been shown that mineral fillers modified with an organic lubricant (travertine, bentonite, tuff, marble, basalt) have a significant effect on the structure and properties of heterochain polymers based on a copolymer of formaldehyde, polyamides, and polyphenylene oxide. The developed composites are characterized by high wear-resistance (1.43–1.9 times), improved strength properties (1.5–2.0 times), and decreased friction coefficient (1.17–1.4 times) compared to the initial polymer materials that extend the opportunities of their application in modern friction units. The main mechanism of fatigue-delamination high-temperature wear of brake friction composite materials with mineral additives is revealed and based on it a physical model of the surface layer destruction is developed. It was established that the performance of these materials under conditions of high-temperature wear is determined by the stress-strain state of thin surface layers, in which tensile and compressive stresses exceeding the ultimate strength at shear act. At the stage of designing brake devices an analytical method to predict the wear resistance of friction linings was developed.

INTRODUCTION

The creation of friction units of machines and mechanisms from polymer composite materials, providing a long service life and minimal wear in the absence of lubrication, makes it possible to increase the reliability, durability, and wear resistance of metal-polymer tribological joints [1, 2]. In this regard, the problem of creating large-scale production of heterochain polymer composites with an improved set of tribological and strength characteristics is becoming increasingly relevant. The study of the laws of the processes of physicochemical modification of polymers with various fillers and the development on this basis of methods for controlling the structure and properties of polymers is of great scientific interest for the targeted creation of new antifriction polymer materials for low-wear and high-tech metal-polymer friction units.

The development of scientific principles for creating friction materials with given tribological parameters and controlled properties based on a comprehensive study of the processes of frictional interaction of materials of friction pairs of brake devices is an urgent task of modern friction materials science, which has great scientific and practical significance. During high-temperature wear of brake friction composites (BFCs), multistage mechanochemical changes, structural and phase transformations, orientation of the working layer in the direction of the sliding path vector, and the presence of secondary structures of the second type on the friction surface are constantly acting factors [3–5]. Under conditions of increasing surface temperature, they irreversibly change the properties of the surface layers of the BFCs and are the main reasons for the decrease in the performance of the friction pair. The resulting wear products mainly have the shape of petals with an uneven surface topography and a molecular structure with the presence of reactive radicals. The mechanism of high-temperature destruction of the surface layers of BFCs is characterized by the accumulation of damage in the subsurface layer, and the process of high-temperature wear of these composites is most fully explained by the concepts of fatigue-delamination destruction of friction surfaces.

Objective—To study the process of physicochemical modification of heterochain polymers with Armenian minerals, their influence on the tribological and strength properties of composites, as well as to identify

the mechanism of high-temperature friction of brake friction materials and develop a methodology for predicting the wear resistance of friction linings of brake devices.

MATERIALS AND METHODS

Antifriction Polymer Composites

To obtain composite materials based on heterochain polymers, Armenian mineral fillers, such as travertine, marble, bentonite, and tuff from various deposits, preheat-treated and modified with fluorine-containing oligomers and fluoroalkanes (FAs), were used as fillers. By modifying FA mineral fillers, an increase in the adhesive properties and plasticity of the composition is achieved, which significantly affects its wear resistance. Heat treatment of fillers was carried out at a temperature of 450−500°C for 8 to 10 h.

Two methods of introducing minerals and FAs into a filled system are considered: by simultaneous mixing of the necessary components in a mixer and a two-stage method, providing for preliminary modification of Armenian minerals with FAs and then introduction into the filled system. The modification consisted in the fact that heat-treated mineral filler powder was mixed in an eccentric vibrating mill together with a lubricant.

The kinetics of the formation of a cross-linked structure during the formation of materials was studied by sol-gel and dilatometric analyses. Hexafluoroacetone hydrate (for SFD), formic acid (for PA6, PA66) and chloroform (for PFO) were used as solvents. Determination of the relative viscosity of the soluble part of the filled materials at a temperature of 200°C (0.5% solution of the polymer in a 95% solvent) and the content of the gel fraction was carried out by extraction with a 95% solvent. Studies of tribochemical processes occurring in the surface layers during friction of the materials under study were carried out using sol-gel and electron paramagnetic resonance (EPR 1301), X-ray photoelectron spectroscopy (XPS), and mass spectrometry (MS-30 spectrometer).

The tribological properties of the materials were assessed on a 2070 SMT-1 friction machine using the "finger-disk" scheme with sample sizes of $\varnothing 10 \times 15$ mm (polymer finger) and $\varnothing 50 \times 14$ mm (steel disk), as well as on an I-47 end-face friction machine according to the "ring-ring" scheme with ring dimensions $\varnothing 22 \times 12$ mm at speeds of 0.25−5.0 m/s and in the load range of 0.1−10.0 MPa. Counterbodies were made of 35, 45 (48−52 HRC), and 1X13 (150−170 HB) steels with roughness $R_a = 2.50-0.20$ μm.

RESULTS AND DISCUSSION

The basic chemical composition of the original and heat-treated mineral filler, for example travertine, as well as some of their characteristics are given in Table 1. It was

Table 1. Chemical composition and characteristics of the original and heat-treated travertine

Indicator	Travertine	
	initial	heat treated
Chemical component:		
CaO	52.80−54.12	54.86−56.18
CO_2	42.24−44.26	40.15−42.08
Specific surface, m^2/g	740	635
Bulk weight, kg/m^3	1578	1346
Dispersity, μm	20−40	25−45

established that when mineral fillers are mixed with FAs together by vibration, the C−C bonds of the FA chain are broken, resulting in the formation of products of the radical type, which is distributed in monolayers over the surface of minerals in the form of a grafted layer, where chlorine atoms are located mainly along its periphery. The developed modified mineral filler (MMF) increased lubrication and adhesion properties. It was shown that the introduction of FAs into the system in a two-stage manner, using an MMF filler, leads to an increase in the thermal stability and wear resistance of the materials under study. This made it possible to create a new type of antifriction materials.

The lubricity of MMF was assessed on an I-47 mechanical friction machine by testing them between two rubbing metal (1X13 steel) surfaces with a stepwise increase in sliding speed [6, 7]. The maximum achievable sliding speed, preceding a sharp increase in the friction coefficient, the so-called "sticking," was chosen as a criterion for lubricity. Analysis of the wettability results of the samples was carried out using the two-liquid method (water and methylene iodide) using an MMI-2 instrumental microscope. The test results showed that the modification of heat-treated FA mineral fillers led to a significant decrease (2.5−3.0 times) in the friction coefficient of the filler itself, amounting to 0.12−0.15 at $V = 0.3$ m/s.

Sol-gel analysis of MMF showed that after joint vibration grinding, part of the FA is not washed off with a solvent (chloroform). With an increase in the amount of FA introduced into the mineral filler, the amount of insoluble FA also increases (Fig. 1). This process continues with the addition of FAs up to 10% for travertine, marble, and bentonite and 15% for tuff and basalt. A further increase in FAs does not lead to a change in the amount of insoluble FAs, which indicates the saturation of mineral fillers with FAs. Dependences similar in nature are observed with heat-treated fillers.

A study of the wettability of samples from MMF allowed establishing that the polar component of the
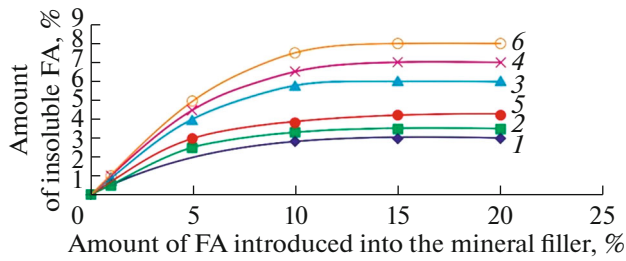
Fig. 1. Sol-gel analysis of FA-modified mineral fillers: (*1*) travertine; (*2*) bentonite; (*3*) marble; (*4*) felsite tuff; (*5*) heat-treated travertine; (*6*) heat-treated felsite tuff.

free surface energy increases 3.5–3.2 times, and after washing FA, 2.2–1.8 times compared to the value characteristic of the original fillers, for example, travertine $(0.48 \times 10^{-3}$ J$/$m$^2)$ and for tuff $(0.35 \times 10^{-3}$ J/m$^2)$. This indicates a special structure of the resulting product, where the polar FA atoms are located at the periphery of the grafted layer.

The results of tribological tests, as previously obtained [8–10], showed the promise and effectiveness of using MMF, leading to a significant reduction in the coefficient of friction and an increase in the wear resistance of compositions based on SFD, PA6, PA66, and PFO. It is known that the formation of a cross-linked polymer structure has a significant effect on tribochemical processes. For this purpose, sol-gel analysis was carried out to determine the relative viscosity of the soluble part and the content of the gel fraction. Studies of samples of original and filled het-

erochain polymers, depending on the filling concentration, indicate that due to the chemical interaction of MMF with the functional groups of heterochain polymers, both an increase in the molecular weight of the materials and the formation of a network of intermolecular bonds are observed, which is confirmed by an increase in the relative viscosity of the soluble part and gel fraction content with increasing filling concentration (Table 2).

The dependences of the relative viscosity of the soluble part, the content of the gel fraction, and the antifriction properties of heterochain polymers on the MMF content were revealed during friction without lubrication ($P_a = 1.91$ MPa, $V = 0.78$ m/s) according to the "shaft–partial liner" scheme on steel 45 (48–52 HRC, $R_a = 1.25$ μm) [10].

The formation of intermolecular chemical bonds in filled heterochain polymers is confirmed by EPR spectroscopy data. Thus, the rotation correlation time of a stable iminoxyl radical (Fig. 2) introduced into filled heterochain polymers SFD, PA6, PA66, and PFO increases by 2.1–1.6 times compared to the starting materials ($\tau_i = 27 \times 10^{-10}$ to 21×10^{-10} s), which may indicate an increase in the rigidity of macromolecules due to the formation of a network structure. Apparently, when MMF is administered in an amount from 5 to 40 wt % into the original polymers, conditions arise for the formation of a stable network of intermolecular bonds, which is also evidenced by a significant increase in the gel fraction.

The developed composite materials have fairly stable tribological properties under the conditions of the

Table 2. Influence of the content of filling on the relative viscosity of the soluble part, the content of the gel fraction, and the antifriction properties of heterochain polymers

Composition, wt %	Relative viscosity, η_{rel}	Gel fraction, wt %	Linear wear intensity, $I \times 10^{-9}$	Friction coefficient, f
SFD	3.5	0	10.29	0.15
SFD + 5% MMF	5.1	17.8	9.8	0.14
SFD + 20% MMF	5.9	38.6	4.2	0.11
SFD + 40% MMF	6.2	48.7	2.2	0.08
PA6	2.8	0	12.2	0.20
PA6 + 5% MMF	4.7	16.2	10.8	0.20
PA6 + 20% MMF	5.6	36.0	8.0	0.17
PA6 + 30% MMF	5.9	42.5	5.2	0.14
PA66	3.1	0	10.5	0.22
PA66 + 5% MMF	4.9	15.6	10.0	0.22
PA66 + 10% MMF	5.5	26.0	7.2	0.18
PA66 + 20% MMF	5.7	34.5	4.8	0.15
PFO	4.1	0	15.0	0.28
PFO + 5% MMF	5.9	14.5	14.2	0.25
PFO + 20% MMF	6.5	27.6	9.8	0.20
PFO + 30% MMF	6.8	36.5	5.4	0.16
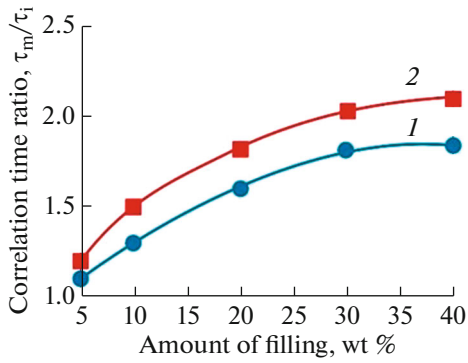
Fig. 2. The dependence of τ_m/τ_i on the content of MMF in polyamide PA6 (*1*) and SFD (*2*), τ_m—correlation time for the modified material; τ_i—correlation time for the initial material.

Fig. 3. Stress state calculation scheme of the BFC working layer.

studied loads, speeds and temperatures, which is mainly explained by the formation of smooth, monolithic, and coherent friction transfer films on the friction surfaces. These materials are recommended in instrument making and mechanical engineering for the manufacturing of plain bearings (bushings, liners), fine-module gears, and sliding guides operating without lubrication at $PV \leq 2.5$ MPa m/s.

Modeling and Calculation of High-Temperature Wear of Brake Friction Composites

When assessing the performance and wear resistance of a friction pair of brake and other friction devices, it is recommended to use the criterion of energy intensity of wear, which, according to the theory of fatigue wear, is expressed by relation [11]:

$$I_W = \frac{\Delta h A_a}{L F_f}, \tag{1}$$

where Δh is the thickness of the worn layer; A_a is the nominal contact area; L is the friction path; and F_f is the friction force.

At the stage of designing and creating BFCs with functional properties, it is desirable to pre-predict thickness Δh of the worn layer. Therefore, it is necessary to develop a method for calculating the worn layer in relation to high-temperature (intensive) wear of friction linings, taking into account the stress-strain state of thin surface layers according to the concepts of fatigue-delamination destruction of friction surfaces.

Taking into account the above, the design scheme can be presented as follows (Fig. 3). Here, under the friction surface of BFCs at depth Δh, at angle β relative to the normal force vector, a crack of length $2a$ is considered, at the edges of which normal σ and tangential τ stresses act (it is assumed that adjacent roughness protrusions of the counterbody do not influence each other during the formation of stressed zones under contact).
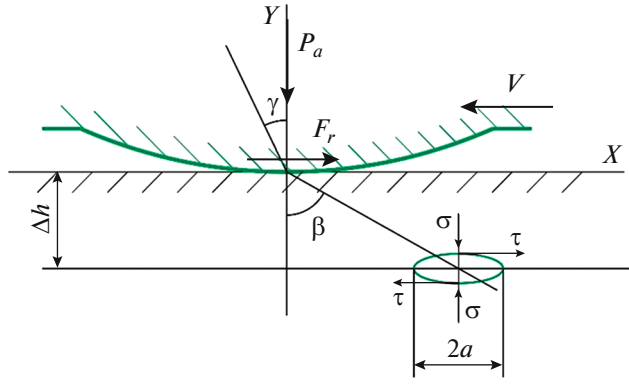
Based on the concept of the delamination mechanism of destruction of friction surfaces [12, 13], we determine these stresses as follows:

$$\sigma = -\frac{2P_a}{\pi \Delta h} \frac{\cos(\gamma - \beta)\cos^3 \beta}{\cos \gamma},$$
$$\tau = -\frac{2P_a}{\pi \Delta h} \frac{\cos(\gamma - \beta)\cos^2 \beta \sin \beta}{\cos \gamma}, \tag{2}$$

where P_a is the specific linear pressure, N/m; γ is the friction angle; and β is the crack angle.

The problem of calculating thickness Δh of the worn layer is reduced to the following:

(1) Determine the laws of stress distribution σ and τ under the contact at depth Δh and identify the coordinates of their maximum values; (2) determine the values of angles β_0 corresponding to the maximum values of stresses σ and τ; (3) taking into account experimental data on the minimum and maximum sizes of intensive wear products, derive a relationship for calculating the thickness Δh at the design stage of brake and other friction devices.

Having expressed $\cos \gamma$ and $\cos \beta$ included in relation (2) through $\tan \gamma$ and $\tan \beta$ and opening the brackets, we transform the formula to form:

$$\sigma = -\frac{2P_a}{\pi \Delta h} \frac{1 + \tan \beta \tan \gamma}{(1 + \tan^2 \beta)^2},$$
$$\tau = -\frac{2P_a}{\pi \Delta h} \frac{1 + \tan \beta \tan \gamma}{(1 + \tan^2 \beta)^2}. \tag{3}$$

Here, $\tan \beta = x/y$ is the relative distance from the contact, and $\tan \gamma$ is the friction coefficient. Let us rewrite Eq. (3) as follows:

$$\frac{\pi \Delta h \sigma}{2P_a} = -\frac{1 + \tan \beta \tan \gamma}{(1 + \tan^2 \beta)^2},$$
$$\frac{\pi \Delta h \tau}{2P_a} = -\frac{1 + \tan \beta \tan \gamma}{(1 + \tan^2 \beta)^2} \tan \beta. \tag{4}$$
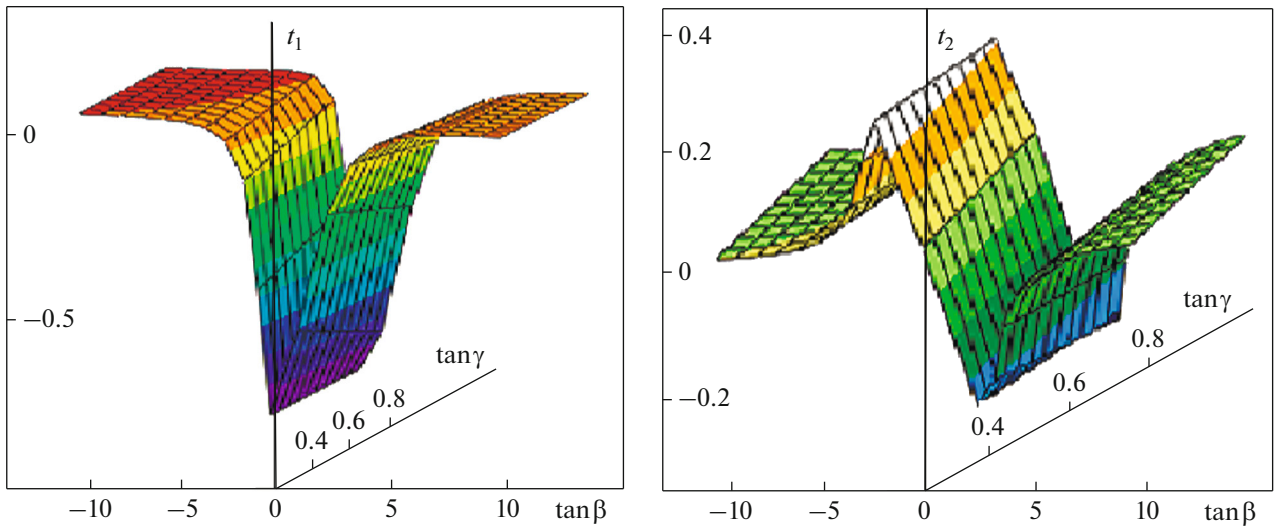
Fig. 4. Results of studies of functions t_1 and t_2.

The left side of Eqs. (4) determines the tension of the frictional contact depending on the depth, starting from the friction surface, and the right side determines the geometry of the location of the subsurface crack. Let us denote $t_1 = \pi \Delta h \sigma / 2 P_a$ and $t_2 = \pi \Delta h \tau / 2 P_a$, which at constant values of Δh will characterize the dependence of σ / P_a and τ / P_a on the geometry of the location of the subsurface crack. The relationships under study will take form:

$$t_1 = -\frac{1 + \tan\beta \tan\gamma}{(1 + \tan^2\beta)^2}, \quad t_2 = -\frac{1 + \tan\beta \tan\gamma}{(1 + \tan^2\beta)^2} \tan\beta. \quad (5)$$

Table 3. Values of functions t_1 and t_2 at different friction coefficients

$\tan\beta$	$\tan\gamma = 0.2$		$\tan\gamma = 0.3$		$\tan\gamma = 0.4$	
	t_1	t_2	t_1	t_2	t_1	t_2
−3.0	−0.004	0.012	−0.001	0.003	−0.002	0.006
−2.5	−0.009	0.020	−0.0048	0.012	−0.003	0.009
−2.0	−0.024	0.040	−0.016	0.032	−0.008	0.016
−1.5	−0.066	0.090	−0.052	0.078	−0.038	0.060
−1.0	−0.200	0.200	−0.175	0.175	−0.150	0.150
−0.5	−0.570	0.280	−0.944	0.270	−0.512	0.256
0	−1	0	−1	0	−1	0
0.5	−0.700	−0.350	−0.736	−0.368	−0.768	−0.384
1.0	−0.300	−0.300	−0.325	−0.325	−0.350	−0.350
1.5	−0.123	−0.180	−0.137	−0.206	−0.150	−0.230
2.0	−0.050	−0.112	−0.064	−0.128	−0.070	−0.140
2.5	−0.029	−0.070	−0.053	−0.080	−0.038	−0.095
3.0	−0.016	−0.040	−0.020	−0.060	−0.022	−0.066

Using the MathCAD 2000 Professional program with friction coefficient $\tan\gamma = 0.2–0.7$ and $\tan\beta = -10$ to 10, functions t_1 and t_2 were calculated and studied (Fig. 4).

It turned out that outside interval −3 to +3 functions t_1 and t_2 take values close to zero, so they should be analyzed only in interval −3 to +3. The results of calculations of functions t_1 and t_2 for this interval are given in Table 3.

It is necessary to note that function t_1 takes on an extreme value at $\tan\beta = 0$, that is, compressive stresses σ take on the greatest values at points located on the axis of vector P_a. Tangential stresses τ take on extreme values approximately at $\tan\beta = |0.5|$, which corresponds to $\beta_0 = 26°$.

Cracking of the working layer under such a stressed state occurs when surface cracks close with a subsurface crack at points where τ takes on a maximum value. This assumption is consistent with the results presented in [13], where, in particular, it is shown that the angle of the direction of propagation of a vertical crack, measured relative to the vertical, varies within 0°−20° and the tendency for a vertical crack to intersect with a horizontal one leads to destruction of the surface layer material through peeling. This interpretation of the stress state makes it possible to determine the location of cracks in the surface layer of the BFC (Fig. 5) and graphically depict the distribution law of stresses σ and τ (functions t_1 and t_2). Here, a subsurface crack is shown at distance Δh from the friction surface. At points m and n, corresponding to extreme values of tangential stresses τ, a crack emerges on the surface or closes with a vertical surface crack, resulting in the formation of lobe wear BFC products.
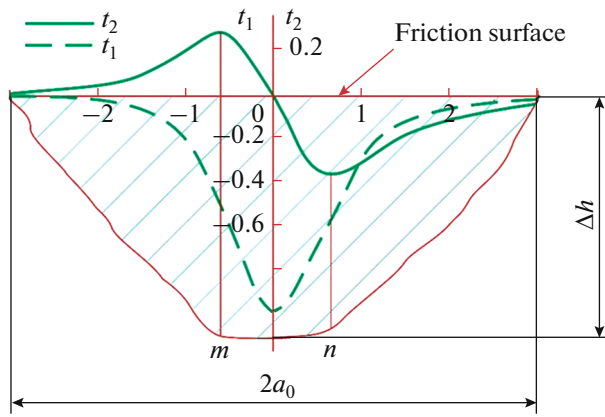
Fig. 5. Location of cracks and laws of stress distribution.

Based on this, thickness Δh can be determined as follows:

$$\Delta h = \frac{mn}{2 \tan \beta_0}, \qquad (6)$$

where β_0 is the angle of location of the subsurface crack corresponding to maximum stress values τ. As a first approximation, we can take $mn = a_0/3$, where a_0 is half the length of the subsurface crack in the contact zone and, therefore, the petal wear products.

The calculated dependence of the thickness of the worn layer in this case will take form:

$$\Delta h = \frac{a_0}{6 \tan \beta_0}. \qquad (7)$$

The value of Δh represents the thickness of the worn working layer along friction path L_o, necessary to remove one layer of wear particles from the friction surface of the friction lining. Substituting into formula (1) the value of the thickness of worn working layer Δh and assuming that along general friction path L (working life of a friction pair of a brake or other friction device) K_0 layers may be subject to wear, to assess the wear rate of friction linings at the design stage of brake devices in in the final form we get:

$$I_W = \frac{K_0 a_0 A_a}{6 L F_f \tan \beta_0} = \frac{H a_0 A_a}{6 \tan \beta_0 \Delta h_m L F_f}. \qquad (8)$$

The value of K_0 for each friction composition depends on the specific values of the operational parameters of the friction pair and the dimensions of

Table 4. Values $2a_0$, Δh, and Δh_m at various BFCs

Type of BFC	$2a_0$, μm	Δh, μm	Δh_m, μm
Combined	30–60	5–10	7.50
Rubber based	80–120	13.3–20	16.65
Resin based	20–30	2.5–3.1	2.8

the lining. It can be preliminarily determined by formula:

$$K_0 = \frac{H}{\Delta h_m}, \qquad (9)$$

where H is the friction lining thickness; Δh_m is the mean value of the worn working layer. The values of $2a_0$, Δh, and Δh_m for the BFC under consideration are presented in Table 4.

Thus, based on the study of the patterns of high-temperature wear of BFCs, it becomes possible to construct a physical model of the destruction of surface layers and develop an analytical technique for predicting the wear resistance of friction linings at the design stage of brake devices. Taking into account the fact that for BFC class materials the thickness of the worn layer varies within the range $\Delta h_m = 2.8$–11.75 μm, with known design parameters of the friction linings, the derived dependence can also be used for a preliminary assessment of the performance of new materials with given tribological parameters and controlled properties.

CONCLUSIONS

The presented results indicate that heat-treated mineral fillers modified with an organic lubricant have a complex effect on the structure and properties of heterochain polymers, increasing their wear resistance by 1.43–1.9 times and thermal-oxidative stability by 1.2–1.8 times, while reducing the friction coefficient by 1.17–1.4 times in a wide temperature range (40–100°C), and also improving the strength characteristics by 1.5–2.0 times compared to the original materials. The results are of great scientific interest for the targeted creation of new wear-resistant materials based on large-scale polymers produced for metal-polymer friction units and increasing the operational parameters of a modern tribological system.

According to the concepts of the fatigue-delamination mechanism of destruction of friction surfaces, a method for calculating the worn layer during high-temperature wear of friction linings was developed, taking into account the stress-strain state of thin surface layers. Based on the proposed physical model of the destruction of the surface layer, it was found that the wear resistance of brake friction materials under high-temperature friction conditions is determined by the stress-strain state of the surface layers in which tensile and compressive stresses exceed the shear strength. The derived dependence for calculating the thickness of the worn layer at the design stage of brake and other friction devices allows for a preliminary assessment of the performance and wear resistance of the developed materials with given tribological parameters.

NOTATION

V	sliding speed
I_W	wear energy intensity
Δh	thickness of worn layer
A_a	nominal contact area
L	friction path
F_f	friction force
P_a	specific linear pressure, N/m
σ	normal stresses
τ	shear stresses
γ	angle of friction
β	crack location angle
β_0	subsurface crack location angle corresponding to maximum values of shear stresses
a_0	half length of subsurface crack in contact zone
K_0	number of worn layers
H	thickness of friction lining
Δh_m	mean value of worn working layer

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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