ESTIMATION OF THE FRICTION COEFFICIENT
OF A NANOSTRUCTURED COMPOSITE COATING

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The frictional-mechanical properties of a thin polymer-ceramic coating obtained by gas-phase impregnation of nanoporous anodic alumina with a fluoropolymer (octafluorocyclobutane) have been investigated. The coefficient of sliding friction of the coating is predicted based on an analysis of contact deformation within the framework of the Winkler elastic foundation hypothesis and a three-phase micromechanical model. It is shown that an acceptable prediction accuracy can be obtained considering the uniaxial strain state of the coating. It was found that, on impregnation by the method of plasmachemical treatment, the relative depth of penetration of the polymer increased almost in proportion to the processing time. The rate and maximum possible depth of penetration of the polymer into nanoscale pores grew with increasing porosity of the alumina substrate.

Introduction

The dominating tendency of modern technology is the use of materials that are optimally "designed" or self-organized at the nanoscale level [1]. Among them, there are structurally ordered thin porous coatings based on anodic (anodized) alumina (AA) in the form of nanocombs and nanobrushes [1-3]. Additional possibilities are opened by impregnation (filling) of AA with polymer materials. As a result, composites with distinctive physicomechanical properties are formed. Thus, it was established experimentally [4] that the combination of anodic alumina with high-molecular compounds having a low shear strength enables one to significantly increase the antifriction characteristics of the initial

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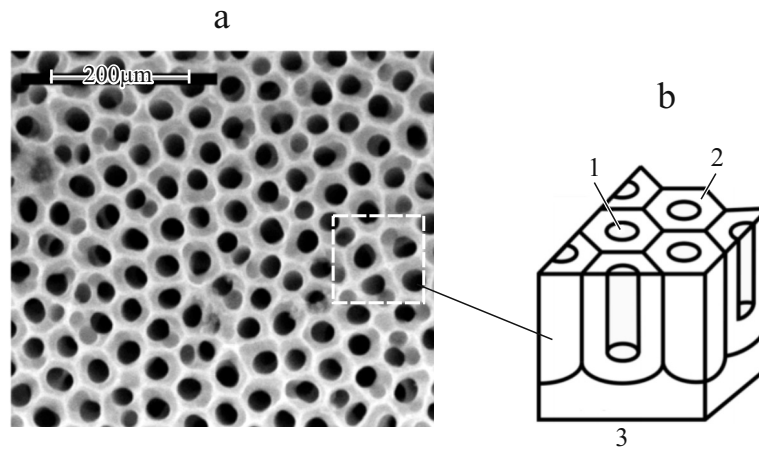*Corresponding author; e-mail: shilko_mpri@mail.ru

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Fig. 1. Microphoto (a) and structural model (b) of a porous anodic alumina film: pore (1); oxide cell (2); aluminum (3).

AA, and the use of polymer piezoelectrics as a filler — to harness the sensory and actuator properties of the functional composites obtained in such a way.

However, the formation laws of antifriction properties of composite coatings by introducing polymers into the porous structure of an anodic alumina film, and especially the possibility of a theoretical prediction of tribotechnical characteristics of these nanostructured coatings, has not been studied adequately in the scientific literature up to the present time.

The purpose of this work is the development of a technique for predicting the friction coefficient of composite nanoporous substrate-polymer filler coatings.

Description of the model used

Determination of elastic characteristics. The ordered structure of an anodic alumina film, together with a schematic of its fragment separated out, is shown on Fig. 1. If its pores are filled (impregnated) by a polymer, its volume fraction c is determined by the simple relation

$$c = \frac{\pi}{2\sqrt{3}} \left(\frac{d}{D} \right)^2 .$$

The material obtained in such a way is a unidirectional composite with the symmetry axis of its elastic characteristics oriented along the normal to the coating surface. Its honeycomb-like hexagonal structure is characterized by three geometric parameters: layer thickness h, pore diameter d, and distance D between the centers of neighboring pores. As already mentioned, it is of interest to consider a contact loading of the given coating. As is known [5], the Winkler hypothesis can be used in solving contact problems for thin elastic layers whose thickness is smaller than the characteristic size of the contact area. If the coating simply lies on the substrate, the stiffness coefficient k_n of Winkler foundation (Winkler coefficient) is determined by the longitudinal (along the reinforcement direction) Young's modulus E_z and Poisson's ratio v_{zx},

$$k_n = \frac{E_z}{h\left(1 - v_{zx}^2\right)}, \tag{1}$$

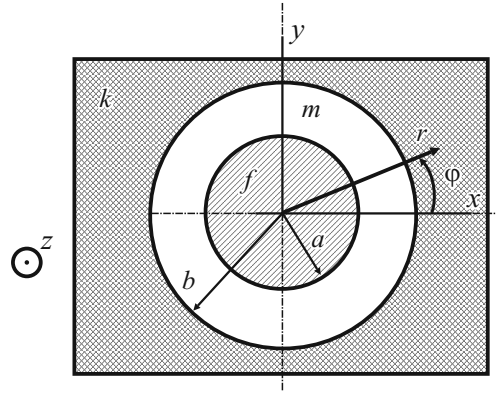where h is coating thickness.

Fig. 2. Schematic of the periodicity cell of a fiber-reinforced composite investigated.

If the coating is bonded adhesively to the substrate, the coefficient k_n is calculated by the formula

$$k_n = \frac{C_{zz}}{h},$$ (2)

where C_{zz} is the axial elastic modulus of the orthotropic composite corresponding to the reinforcement direction.

The effective elastic characteristics of filled coatings are determined on the basis of an elementary reinforcement theory [6] or the Mori–Tanaka model [7]. At the same time, the strict ordering of the structure and the regular shape of repeating structural elements allows one to use the more accurate three-phase model of reinforced composite for this purpose [8]. The corresponding periodicity cell is a compound cylinder of infinite length and radius b imbedded in an elastically deformable medium (Fig. 2).

The inner region of the cylinder with a radius a is the filler (polymer), the outer layer is the matrix (alumina), and the medium surrounding the cell is a composite with desired elastic characteristics. The radii a and b are related to the volume fraction c of filler by the formula

$$c = \left(\frac{a}{b}\right)^2.$$ (3)

The deformation process of the cell is described in the cylindrical coordinate system z, r, φ. The z axis coincides with the central axis of the compound cylinder.

Materials of the matrix and filler are considered isotropic, linear, and elastic. Their basic mechanical characteristics are Young's moduli E^m and E^f and Poison's ratios v^m and v^f. In addition, to characterize the matrix and filler, we will also use the components C_{11} and C_{12} of the tensor of elastic moduli and the shear modulus G of the isotropic material

$$C_{11} = \frac{E(1-v)}{(1-2v)(1+v)}, \quad C_{12} = \frac{Ev}{(1-2v)(1+v)}, \quad G = \frac{E}{2(1+v)}.$$

In determining the effective Young's modulus E_z and Poisson's ratio v_{zx}, we consider a uniaxial stress state of the model. At infinity ($r \to \infty$), the only nonzero component of the macroscopic stress tensor in the compound cylinder is σ_z. The symmetry of the model and the uniaxial stress state allows us to obtain the general solution of elasticity equations for each component of the model

$$u_r^j = A_1^j r + \frac{A_2^j}{r}, \quad u_\varphi^j = 0, \quad u_z^j = \varepsilon_z z.$$ (4)

Here, u_r, u_z, and u_φ are the elastic displacements in the cylindrical system of coordinates; ε_z is the longitudinal strain, which is the same for all components of the three-phase model; A_1 and A_2 are constants determined from boundary conditions. The superscript φ in Eqs. (4) stands for f, m, and k and indicates the corresponding component of the model (see Fig. 2).

From the requirement that displacements at $r = 0$ have to be bounded, it follows that $A_2^f = 0$. The energy criterion of Eshelby homogenization [9] for the loading regime considered is satisfied if $A_2^k = 0$. Then, the radial stress σ_{rr}^k of the composite does not depend on the coordinate r,

$$\sigma_{rr}^k = C_{rr}^k \varepsilon_{rr}^k + C_{rz}^k \varepsilon_{zz}^k + C_{r\varphi}^k \varepsilon_{\varphi\varphi}^k = C_{rr}^k \frac{\partial u_r^k}{\partial r} + C_{rz}^k \varepsilon_z + C_{r\varphi}^k \frac{u_r^k}{r}$$

$$= C_{rr}^k \left(A_1^k - \frac{A_2^k}{r^2} \right) + C_{rz}^k \varepsilon_z + C_{r\varphi}^k \left(A_1^k + \frac{A_2^k}{r^2} \right) = A_1^k \left(C_{rr}^k + C_{r\varphi}^k \right) + C_{rz}^k \varepsilon_z.$$

Here, C_{rr}^k, C_{rz}^k, and $C_{r\varphi}^k$ are components of the tensor of elastic moduli of the orthotropic composite in the cylindrical system of coordinates.

In the uniaxial stress state of the composite, the radial component σ_{rr}^k of the stress tensor is zero at $r \to \infty$, therefore, it is also zero at $r = b$

$$\sigma_{rr}^k \Big|_{r=b} = 0 . \tag{5}$$

On interfaces of the filler–matrix ($r = a$) and matrix–composite ($r = b$), continuity conditions for the radial displacement and stress are fulfilled:

$$u_r^f \Big|_{r=a} = u_r^m \Big|_{r=a}, \quad \sigma_{rr}^f \Big|_{r=a} = \sigma_{rr}^m \Big|_{r=a}, \quad u_r^m \Big|_{r=b} = u_r^k \Big|_{r=b}, \quad \sigma_{rr}^m \Big|_{r=b} = \sigma_{rr}^k \Big|_{r=b}. \tag{6}$$

With account of Eqs. (4) and (5), equalities (6) represent a system of four linear equations for determining the constants A_1^f, A_1^m, A_2^m, and A_1^k. Performing some mathematical transformations, we obtain

$$a_\sigma^m = \frac{A_1^m}{\varepsilon_z} = -\frac{C_{12}^m \left(Z + C_{11}^m \right) + c \left(C_{12}^f - C_{12}^m \right) \left(C_{11}^m - C_{12}^m \right)}{Z \left[C_{11}^m (1+c) + C_{12}^m (1-c) \right] + C_{11}^m \left[C_{11}^m (1-c) + C_{12}^m (1+c) \right]},$$

$$a_\sigma^f = \frac{A_1^f}{\varepsilon_z} = \frac{2a_\sigma^m C_{11}^m - C_{12}^f + C_{12}^m}{Z + C_{11}^m}, \tag{7}$$

$$b_\sigma^m = \frac{A_2^m}{\varepsilon_z b^2} = -c \frac{a_\sigma^m \left(Z - C_{11}^m \right) + C_{12}^f - C_{12}^m}{Z + C_{11}^m}, \quad a_\sigma^k = \frac{A_1^k}{\varepsilon_z} = a_\sigma^m + b_\sigma^m,$$

where, for the sake of brevity, the designation

$$Z = C_{11}^f + C_{12}^f - C_{12}^m \tag{8}$$

has been introduced.

Poisson's ratio ν_{zr} of the composite is defined by the relation

$$\nu_{zr} = -\frac{\varepsilon_{rr}^k}{\varepsilon_{zz}^k} = -\frac{A_1^k}{\varepsilon_z} = -a_\sigma^k . \tag{9}$$

To determine Young's modulus E_z, the axial stress σ_{zz}^k in the composite has to be found. We assume that it coincides with the average stress σ_{zz} in the cross section of the compound cylinder. Then,

$$E_z = \frac{\sigma_{zz}^k}{\varepsilon_{zz}^k} = \frac{1}{\varepsilon_z} \langle \sigma_{zz} \rangle = c \left(C_{11}^f + 2C_{12}^f a_\sigma^f \right) + (1-c) \left(C_{11}^m + 2C_{12}^m a_\sigma^m \right). \tag{10}$$

The elastic modulus C_{zz} is determined in the uniaxial strain state of the composite, when all components of the strain tensor, except $\varepsilon_{zz}^k = \varepsilon_z$, are equal to zero at infinity. In this case, as in the uniaxial stress state, the general solution of elasticity theory equations has form (4). As before, from the requirement that displacements at $r = 0$ have to be zero, it follows that $A_2^f = 0$, but from the energy criterion of homogenization — that $A_2^k = 0$. The equality of the constant A_2^k to zero means that the radial strain ε_{zz}^k does not depend on the coordinate r. Therefore, the constant A_1^k in the uniaxial strain state of the composite is equal to zero and

$$\varepsilon_{rr}^k = \frac{\partial u_r^k}{\partial r} = A_1^k - \frac{A_2^k}{r^2} = A_1^k = 0 .$$

The first three equations of system (6) allow us to determine the nonzero constants A_1^f, A_1^m, and A_2^m. Performing some mathematical transformations, we obtain that

$$a_\varepsilon^m = \frac{A_1^m}{\varepsilon_z} = c \frac{C_{12}^f - C_{12}^m}{Z(1-c) + C_{11}^m(1+c)},$$

$$a_\varepsilon^f = \frac{A_1^f}{\varepsilon_z} = -\frac{1-c}{c} a_\varepsilon^m, \quad b_\varepsilon^m = \frac{A_2^m}{\varepsilon_z b^2} = -a_\varepsilon^m .$$

(11)

As before, we assume that the axial stress is equal to the average stress σ_{zz} in the cross section of the compound cylinder. Then,

$$C_{zz} = \frac{\sigma_{zz}^k}{\varepsilon_{zz}^k} = \frac{1}{\varepsilon_z} \langle \sigma_{zz} \rangle = c\left(C_{11}^f + 2C_{12}^f a_\varepsilon^f \right) + (1-c)\left(C_{11}^m + 2C_{12}^m a_\varepsilon^m \right).$$

(12)

Using the fourth equation of system (6), we find the radial stress and the modulus C_{zr} of the composite:

$$C_{zr} = \frac{\sigma_{rr}^k}{\varepsilon_{zz}^k} = \frac{1}{\varepsilon_z} \sigma_{rr}^m \Big|_{r=b} = C_{12}^m + 2C_{11}^m a_\varepsilon^m .$$

(13)

The characteristics E_z, ν_{zr}, C_{zz}, and C_{zr} of the orthotropic composite are related as

$$C_{zz} - E_z = 2\nu_{zr} C_{zr} .$$

(14)

Account of the nonuniform filling of pores. It is known [4] that, during impregnation of an AA coating with a polymer material, in most cases, the pores are filled only partly. We will characterize the degree of their filling by the coefficient p equal to the ratio between the penetration depth h_p of filler and coating thickness h. The value of the coefficient p varies from 0 (no filler) to 1 (full filling and formation of a continuous polymer phase). At $p < 1$, the coating can be considered as a two-layer system. The characteristics E_z, ν_{zr}, C_{zz}, and C_{zr} of the material of the upper layer are determined by relations (9), (10), (12), and (13). For calculating the effective elastic characteristics E_z^0, ν_{zr}^0, C_{zz}^0, and C_{zr}^0 of the material of the lower layer, the above-mentioned relationships can also be used, but taking into account that $C_{11}^f = C_{12}^f = 0$. Then, considering the partial filling of pores, the characteristics E_z^p, ν_{zr}^p, C_{zz}^p, and C_{zr}^p of the coating, are determined as follows:

$$E_z^p = \frac{E_z E_z^0}{E_z(1-p) + E_z^0 p}, \quad C_{zz}^p = \frac{C_{zz} C_{zz}^0}{C_{zz}(1-p) + C_{zz}^0 p},$$

$$C_{zr}^p = C_{12} p + C_{12}^0(1-p), \quad \nu_{zr}^p = \frac{C_{zz}^p - E_z^p}{2C_{zr}^p}.$$

(15)

Calculation of the friction coefficient

The structure of AA ordered at the nanoscale level allows us to predict the effective friction coefficient μ^k by means of the simplified calculation technique described, e.g., in [10]. In accordance with this technique, the coefficient μ^k is determined as the ratio between the integral value of the shear force F_u on coating surface corresponding to the onset of sliding and the corresponding integral value of the normal reaction N_u. The value of N_u is equal to the integral of the axial stress σ_{zz} over the cross section of the composite cylinder in the three-phase model:

$$N_u = \iint_S \sigma_{zz}\, dS = 2\pi\left(\int_0^a \sigma_{zz}^f\, r\, dr + \int_a^b \sigma_{zz}^m\, r\, dr \right) = \pi a^2\left(C_{11}^f \varepsilon_z + 2C_{12}^f A_1^f \right)$$

$$+\pi\left(b^2 - a^2 \right)\left(C_{11}^m \varepsilon_z + 2C_{12}^m A_1^m \right) = \varepsilon_z \pi b^2\left[c\left(C_{11}^f + 2C_{12}^f a^f \right) + (1-c)\left(C_{11}^m + 2C_{12}^m a^m \right) \right].$$

The constants a^f and a^m for the uniaxial stress state of the composite are determined by relations (7) ($a^f = a_\sigma^f$ and $a^m = a_\sigma^m$), but for the uniaxial strain state — by relations (10) ($a^f = a_\varepsilon^f$ and $a^m = a_\varepsilon^m$). The value of F_u is found by integrating the shear stress τ over the end face of the compound cylinder. For both components of the composite (matrix and filler), this stress satisfies the Coulomb law

$$\mu^f\ \tau^f = \mu^f \sigma_{zz}^f, \quad \tau^m = \mu^m \sigma_{zz}^m,$$

where μ^f and μ^m are friction coefficients of the filler and matrix, respectively.

Hence,

$$F_u = \iint_S \tau\, dS = 2\pi\left(\int_0^a \mu^f \sigma_{zz}^f\, r\, dr + \int_a^b \mu^m \sigma_{zz}^m\, r\, dr \right) = \mu^f\left(C_{11}^f \varepsilon_z + 2C_{12}^f A_1^f \right)\pi a^2$$

$$+\mu^m\left(C_{11}^m \varepsilon_z + 2C_{12}^m A_1^m \right)\pi\left(b^2 - a^2 \right)$$

$$= \varepsilon_z \pi b^2\left[c\mu^f\left(C_{11}^f + 2C_{12}^f a^f \right) + (1-c)\mu^m\left(C_{11}^m + 2C_{12}^m a^m \right) \right].$$

Then, for the effective friction coefficient of the coating, we obtain

$$\mu^k = \frac{F_u}{N_u} = \frac{c\mu^f\left(C_{11}^f + 2C_{12}^f a^f \right) + (1-c)\mu^m\left(C_{11}^m + 2C_{12}^m a^m \right)}{c\left(C_{11}^f + 2C_{12}^f a^f \right) + (1-c)\left(C_{11}^m + 2C_{12}^m a^m \right)}. \tag{16}$$

With account of Eq. (10), the last relation, in the uniaxial stress state of coating, takes the form

$$\mu^k = \frac{1}{E_z}\left[c\mu^f\left(C_{11}^f + 2C_{12}^f a_\sigma^f \right) + (1-c)\mu^m\left(C_{11}^m + 2C_{12}^m a_\sigma^m \right) \right]. \tag{17}$$

For the uniaxial strain state, with account of Eq. (12), we obtain

$$\mu^k = \frac{1}{C_{zz}}\left[c\mu^f\left(C_{11}^f + 2C_{12}^f a_\varepsilon^f \right) + (1-c)\mu^m\left(C_{11}^m + 2C_{12}^m a_\varepsilon^m \right) \right]. \tag{18}$$

In the case of partial filling of pores with polymer, the integral value of the normal reaction N_u is determined by characteristics of both the upper and lower layers of the gradient coating. Consequently, for calculating the effective friction coefficient at $p < 1$, the elastic moduli E_z and C_{zz} in Eqs. (17) and (18) have to be replaced by the quantities E_z^p and C_{zz}^p, respectively. The distribution of the limiting shear stress τ, corresponding to sliding of the body along the coating surface, on the surface of the upper layer of the covering is determined by the distribution of the axial stress σ_{zz} only in this layer. In
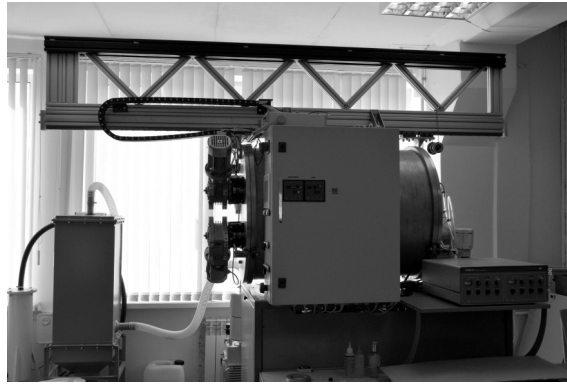
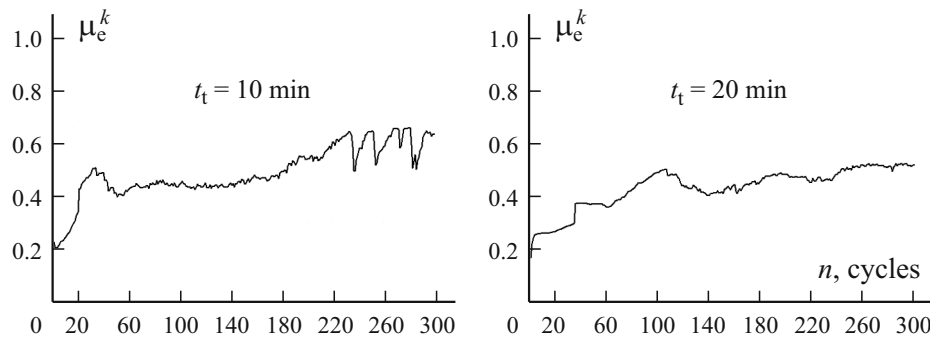Fig. 3. View of the semi-commercial plant for plasma-chemical treatment.

Fig. 4. Friction coefficient μ_e^k of composite coating vs. the number of sliding cycles n of indenter.

this connection, the quantities a^f and a^m in relations (17) и (18), at any $p > 0$, are determined by relations (7) or (10), in which $C_{11}^f \neq 0$ and $C_{12}^f \neq 0$.

Experimental determination of the friction coefficient

Specimens of AA coatings of thickness $h = 70$ μm, with porosities $c_1 = 0.54$ and $c_2 = 0.75$, produced at the Belarusian State University of Informatics and Radioelectronics, were investigated experimentally. The substrate material was aluminum. Impregnation of the coatings with a fluoropolymer (octafluorocyclobutane) was carried out on a semi-commercial plant of plasma-chemical treatment (PCT) developed at the IMMS of the National Academy of Sciences of Belarus [11] (see Fig. 3). Impregnation of the nanosize pores of AA was performed by polymerization in the gas phase.

The friction coefficient of the specimens against steel was measured on a universal MTU-2K7 microtribometer at the tribocenter of the IMMS of the NAS of Belarus, which allows one to measure the tribotechnical characteristics at low loads (from 10 mN to 1 N) with sliding speeds of 0.1-10 mm/s. A 0.1-N load and sliding speed of 0.5 mm/s were chosen. The friction mode was selected such as to register the friction coefficient at the instant of partial failure of the outer polymer layer.

The characteristic experimental relations between the friction coefficient μ_e^k and the number n of test cycles are presented in Fig. 4, where an initial section with a relatively low value of the friction coefficient, μ_0, stands out. This section ends with a sudden increase in μ. The duration of this section increases with processing time t_t. It can be assumed that, in this case, the value μ_0 registered corresponds to the friction process of indenter on the outer polymer layer. The increase in
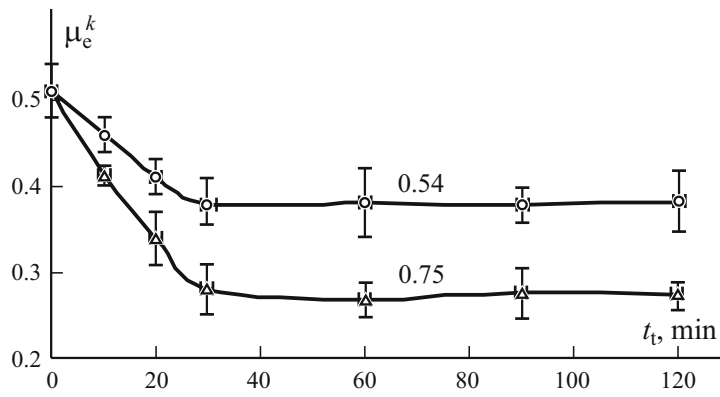
Fig. 5. Quantity μ_e^k vs. duration time t_t of plasma-chemical treatment. The numbers at the curves are the volume fractions c of polymer.
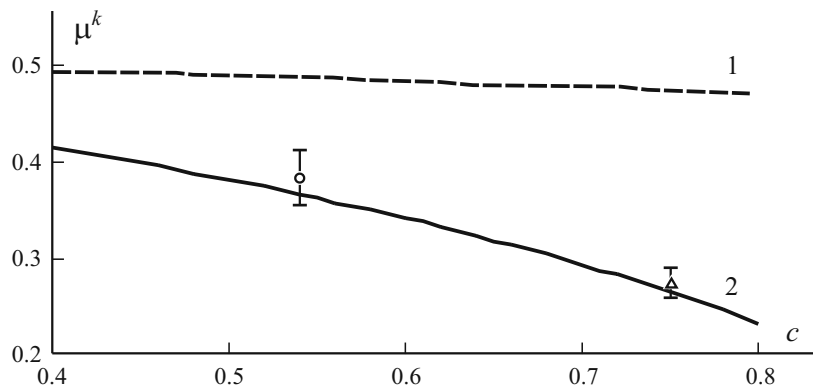

Fig. 6. The effective friction coefficient μ^k of composite coating vs. the volume fraction c of filler: 1 and 2 — calculations by formulas (17) and (18), respectively. Line (1) corresponds to experimental values at t_t = 120 min.
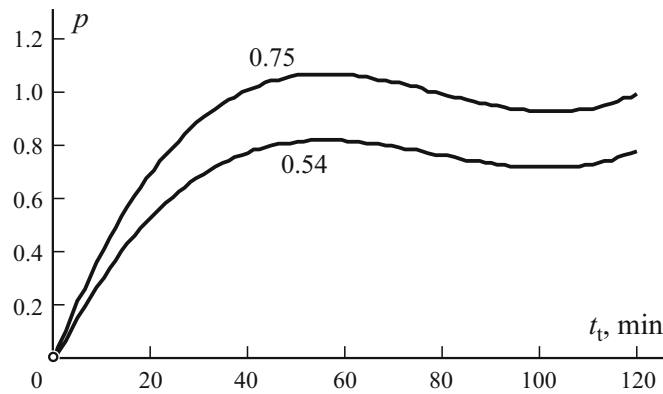

Fig. 7. The coefficient of filling p vs. time t_t. The numbers at the curves are the volume fractions c of polymer filler.

the friction coefficient after the initial section is due to a partial failure of this layer. The steady-state value $\mu_e > \mu_0$ is considered as the experimental friction coefficient of the composite coating.

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The quantity μ_e^k as a function of processing time t_t of specimen is shown in Fig. 5. At $t_t > 30$ min, the value of the coefficient μ_e^k is stabilized, because the degree of pore filling with the polymeric filler increases with time and the coefficient p, introduced previously at the duration $t_t \approx 30$ min, reaches the maximum possible value p_{max}.

Let us assume that $p_{max} = 1$ and compare the steady-state value of the coefficient μ_e (at $t_t = 90$ s) with the calculated estimates obtained by using relations (17) and (18) with initial data for polytetrafluoroethylene $E^f = 300$ MPa, $v^f = 0.493$, and $\mu^f = 0.04$ [12], and the matrix (alumina) phase, $E^m = 140$ GPa and $v^m = 0.32$ [3]. The friction coefficient of alumina was assumed equal to the average experimental value $\mu_e = \mu_e^0 = 0.51$ for an untreated covering.

The results shown in Fig. 6 allow us to conclude that the use of relation (18) for predicting the effective friction coefficient of the composite coating is reasonable. It is seen that the calculated estimates, obtained on the basis of equality (17), are overestimated and do not completely reflect the tendency of variation in the friction coefficient of the composite coating in relation to the volume concentration of the filler. The uniaxial strain state of the composite is more adequate. Relation (18) allows us to estimate the friction coefficient at full filling of pores with polymer ($p = 1$). As already mentioned, the elastic modulus C_{zz} in this expression at $p < 1$ has to be replaced by the quantity C_{zz}^p (15).

Let us find the values of the coefficient of filling p at which the value of the coefficient μ^k calculated in this way coincides with the corresponding average experimental value μ_e^k. From the data presented in Fig. 7, it follows that the coefficient p at $t_t < 30$ min increases practically linearly with processing time. In addition, the maximum value of the coefficient of filling, p_{max}, increases with volume content c of the filler.

Conclusions

The methods of micromechanics of composites can be used for predicting the frictional-mechanical properties of thin nanocomposite (polymer-ceramic) coatings obtained by the gas-phase impregnation of porous anodic alumina with a monomer. The highly ordered hexagonal structure of alumina allows one to consider this coating as a unidirectional composite with the symmetry axis of elastic characteristics oriented along the normal to the coating surface. Verification of the calculation technique proposed for determining the friction coefficient of coating showed an acceptable prediction accuracy on the assumption that the strain state of the composite is uniaxial. It was found that relative penetration depth of polymer during impregnation grew practically proportionally to the time of plasma-chemical treatment. The speed and the maximum possible penetration depth of polymer into nanoscale pores increased with increasing porosity of the initial coating.

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