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Formation and Prediction of the Properties of Ion-Plasma Diamond-Like Coatings under Nitrogen Stabilization

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Abstract—One of the options for solving the scientific and applied problem of the predicted formation of ion-plasma coating tribological characteristics is presented. The problem is solved by creating and analyzing a carbon coating database. The object of research in this work is ion-plasma diamond-like coatings (DLCs) deposited on a steel substrate. It is shown that the use of nitrogen instead of hydrogen to stabilize carbon coatings not only ensures stable thicknesses of DLCs at the level of 1.0–1.5 μm, but also serves as an important and convenient technological parameter for regulating the tribological coating characteristics during deposition. Based on the predicted and experimental values of friction coefficient μ and data on sample path length L, the intervals of optimal values of technological parameters %N and λ are determined. The studied ion-plasma DLCs, obtained according to the established optimal application modes, can be recommended for application under friction conditions equivalent to the tribological tests carried out at friction load $F \approx 10$ N.

Keywords: vacuum ion-plasma technology, diamond-like coatings (DLCs), coating microstructure, mechanical properties, sliding friction tests, tribological properties, machine learning, neural network algorithms

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INTRODUCTION

Vacuum ion-plasma technology for the formation of coatings, reinforced layers or implantation cannot be called completely new. Despite many years of history, in-depth research, and scientific achievements [1–5], ion plasma coatings rub through the production sector. To date, their successful implementation has been noted in aircraft manufacturing, heat and power engineering, as well as tool production. However, in the field of mechanical engineering, for example, to increase the wear resistance of contact surfaces of friction units, such coatings are not yet widely used. This is explained not only by the relatively high cost of technological processes that use vacuum, or the limited size of the vacuum chambers of ion-plasma installations, but also by a large number of physical and technological parameters that control the coating quality. Only among arc process parameters, the composition and quality of the cathodes, their quantity and electromagnetic parameters of the evaporation mode of each cathode (with a complex coating chemical composition), bias voltage, magnetic separation mode, pressure of the working and reaction gas, substrate temperature, deposition angle, etc., are of great importance. An excessive number of descriptors does not allow for reliable unification of deposition control, minimizing the number of variable parameters and significantly complicates modes optimization. In addition, the plasma state in the ion-plasma working chamber is realized by converting a glow arc discharge into a non-self-sustained discharge. Such plasma is characterized by the fluctuation nature of the emerging unstable states, which can be caused by the most unexpected factors, such as, for example, defective cathodes or chemical incompatibility of their elements, poisoning of targets, instability of electrical parameters, the presence of light elements deposited on the chamber walls, etc. In this regard, a sustainable improvement in the quality of coatings and the stability of the formation of a given tribological properties remains an urgent task, the solution of which this work is aimed at.

Objective—Establishing the dependence of tribological properties on the parameters of ion-plasma technology varied during the deposition of diamond-like coatings (DLCs) on a steel substrate. Analysis of the obtained data using neural network algorithms to optimize the parameter space and reliably predict the tribological coating properties. A feature of the DLCs under study is the stabilization of their thickness and structure by introducing nitrogen atoms instead of the traditionally used hydrogen.

MATERIALS AND METHODS

To apply ion-plasma DLCs, a BRV600 vacuum unit (manufactured by BelRosVak, Minsk, Belarus) was used, in which graphite was evaporated by laser. Cylindrical cathodes were made of VP-6 powder graphite. Since the system is initially multi-parameter, in order to reduce the range of experiments, some of the parameters were taken as fixed. The following characteristics were adopted as such parameters during the coating process on the BRV600 installation remained unchanged during the entire experimental work: spark gap voltage, 300 V; laser radiation energy, 600-700 mJ; laser frequency, 10 Hz; graphite cathode scanning speed, 1 mm/s; pressure in the working chamber $P = 0.012–0.080$ Pa; substrate temperature <250°C; coating time $t = 14–20$ min. The following were chosen as variable parameters, the study of the influence and optimization of which this work is devoted to: the current of induction coils (solenoids) λ and amount of nitrogen supplied to the chamber %N. Parameter λ regulates the intensity of the deposited carbon flow, the variation range of its experimental values was $\lambda = 1–5$ A. The %N parameter is determined as a percentage by the gas supply regulator at the installation, its variation range was %N = 1–5. It should be noted that in vacuum technologies the use of working gas, in particular nitrogen, is usually characterized by the value of partial pressure P_N. However, for the BRV600 installation, the empirical relationship between %N and P_N is nonlinear, and at low concentrations of the working gas (nitrogen), in practice and in experiments, the %N parameter is used as a control process parameter due to the possibility of its smooth adjustment.

The scientific interest of studying the influence of the %N parameter on the properties of DLCs is in the fact that nitrogen in this work was used as a modifier of the carbon coating structure instead of traditional hydrogen. Without the use of modifiers in tetrahedral amorphous carbon (ta-C) coatings with a content of sp^3 bonds over 70% at a thickness exceeding ~500 nm, spontaneous cracking begins [6]. The use of nitrogen makes it possible to stabilize the thickness of coatings to 3–4 μm or more due to an increase in the number of phonon modes available for excitation in the carbon film structure and the formation of amorphous carbon nitride (a-CN_x) [7].

As substrates in the work, polished samples made of 40CrNi2Mo tempered steel were used, common in mechanical engineering, with a sorbite structure and surface relief characteristics $R_a \leq 0.12$ μm and $R_z \leq 0.6$ μm. The choice of steel for the substrate was dictated by the prospect of using DLC coatings to increase the wear resistance of contact surfaces of loaded friction units in machine-building units [8, 9].

A significant difference in the mechanical properties of the hard coating and the plastic substrate can negatively affect the adhesion of the coatings, so the work also used variants of DLCs with a Ti or TiN sublayer.

The scope of the studies carried out on DLCs included scanning electron microscopy (SEM), energy dispersive X-ray microanalysis (EDA), X-ray photoelectron spectroscopy (XPS), as well as determination of the physical and mechanical characteristics of the samples using continuous indentation and tribological tests on a friction machine.

The microstructure, surface condition, and fine structure of the coatings were studied by SEM methods using a double-beam ZEISS Crossbeam 340 installation, which combines high-resolution electron microscopy (up to 2 nm) with the possibility of etching and preparing cross-sections on the sample surface being studied directly in a vacuum chamber microscope using a focused ion beam (FIB). The microscope is also equipped with a EDA detector model X-Max 50N (Oxford Instruments) to determine the elemental composition of coatings.

To determine the proportion of sp^2/sp^3 electronic configurations in the carbon coatings, which gives grounds to classify the coatings as DLCs, as well as to improve the accuracy of data on the elemental and phase surface composition, experimental studies were carried out using the SPECS surface analysis system (Germany) using XPS. An AlK_α monochromatic X-ray line with an energy of 1486.6 eV was chosen as the exciting radiation. The energy resolution of the analyzer at constant transmission energy was 0.45 eV at the Ag3$d_{3/2}$ line. The vacuum when taking spectra was maintained at a level of 1×10^{-10} mbar. The sp^2/sp^3 ratio in the synthesized films was determined using differential C(KVV) Auger electron spectra. For quantitative assessments, the C(KVV) Auger graphite spectra with fraction content $sp^2 = 1$, and diamond spectra, where fraction content $sp^2 = 0$, which are commonly used to identify the form of carbon atom hybridization [10].

To study the physical and mechanical properties of the samples on the nano- and microscale, a Nanotest 600 measuring platform was used. Elastic modulus E and hardness H were determined using the continuous indentation method. Test conditions and processing of the data were carried out in accordance with GOST 8.748–2011. For statistical data processing, the recommendations of GOST R 50779.25–2005 and GOST R ISO 16269-4–2017 were used. Based on the results of indentation of H and E, the ratios of H/E and H^3/E^2 were also calculated, which characterize the resistance

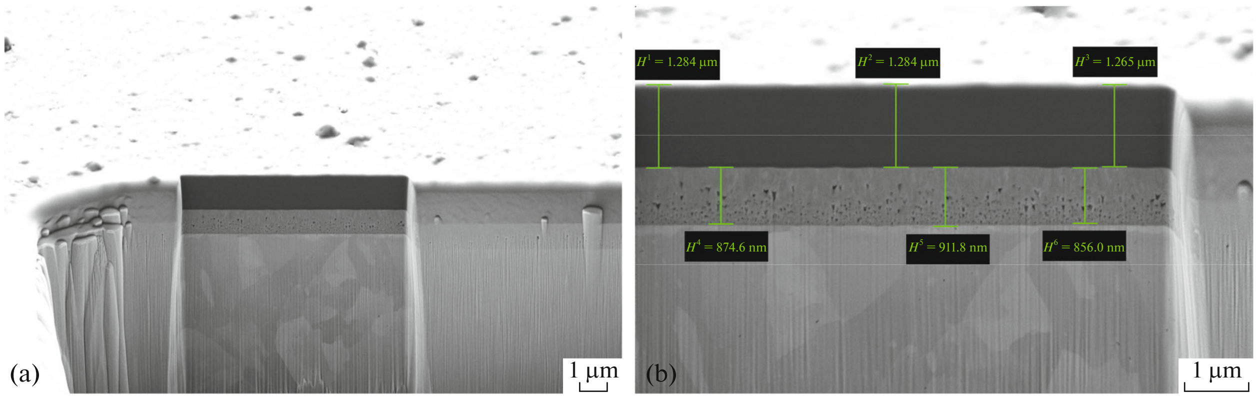

Fig. 1. Structure of a nitrogen-stabilized DLC with a Ti sublayer (SEM): (a) general view of the coating surface and a FIB cross section; (b) thickness markers of the carbon and titanium layers.

of the material to elastic and plastic deformation, respectively [3, p. 608].

Tribological coating tests were carried out on a TRB friction machine (Anton Paar Tritec) in accordance with DIN 50324–1992 and ASTM G99–2017. A pin-to-plate test circuit was used, either for alternating plate movement (coated sample) at a frequency of 10 Hz and an amplitude of 800 μm, or for moving along a circle with a diameter of 6 mm. The normal force on the pin varied discretely and amounted to 1, 5, and 10 N. The counter sample was a ball with a diameter of 6.35 mm fixed in a pin made of cermet (hard alloy) WC–Co. Due to the fact that the ball is stationary in the pin, the tests are classified as sliding friction. The main tribological parameters determined are the coefficient of friction μ and the length of path (friction path) L, traversed by the sample before the coating destruction and measured in meters.

Using the above research methods based on varying the above technological application parameters, a database of our own experimental data on the resulting DLCs was formed. This paper presents the results of the analysis of the created database.

RESULTS AND DISCUSSION

The structure and distribution of elements in the cross section, typical for the coatings under study, are shown in Figs. 1 and 2. The used analysis methods did not reveal the presence of any phases or granularity of the carbon coatings. Their structure is homogeneous (Fig. 1b), the surface quality is high, with poorly developed relief and a small number of artifacts (Fig. 1a). In the carbon coating of the samples that formed the database, the nitrogen content did not exceed 15–20 at %.

The homogeneity of the DLC structure, the nitrogen content, its thickness (significantly exceeding the critical value of ~500 nm), the absence of warping and cracks in the coating, allow us to characterize the resulting coatings as nitrogen stabilized. The role of nitrogen, apparently, comes down to modifying the amorphous carbon structure of the a-C type in the form of a spatial "fullerene-like" microstructure with curved and intersecting basal planes of the a-CN_x structure type [11, 12]. Changing the coatings structure with the introduction of nitrogen certainly affects the properties. However, studying the structure at such a deep structural level requires the use of subtle physical methods of analysis using simulation (for example, molecular dynamics methods), which is beyond the scope of the present work.

The generalized research results of the obtained carbon coatings using the database as a whole, carried out using the XPS method (sampling was carried out from survey spectra in the energy range of the 1s lines of each element), showed the content in the coating: 72.2–92.4 at % C and 2.0–16.5 at % N. At the same time, 5.6–12.0 at % oxygen were found in the surface nanometric coating layer, the content of which rapidly decreased with the coating depth. The ratio of carbon electron configuration in the coatings in the database as a whole was sp^2/sp^3 = 0.1/0.9, which allows us to classify the coatings under study as DLCs.

A correlation between the mechanical and tribological properties of the coatings is not always observed. During friction, its degree depends on the wear mechanism, tribological test methods, the state of the coating–substrate system and other, less important factors. Therefore, a detailed analysis of the generated database on the mechanical properties of DLCs is not provided in this work. We will confine ourselves only to the generalized results given in Table 1, as well as the level of predicted mechanical properties of the coatings achieved with optimal values of varied technological parameters λ = 3.0–3.8 A and %N = 5–8:

$$H \geq 18\text{–}20 \text{ GPa}, \quad E \geq 250 \text{ GPa},$$
$$H/E \geq 0.07, \quad H^3/E^2 \geq 0.08 \text{ GPa}.$$

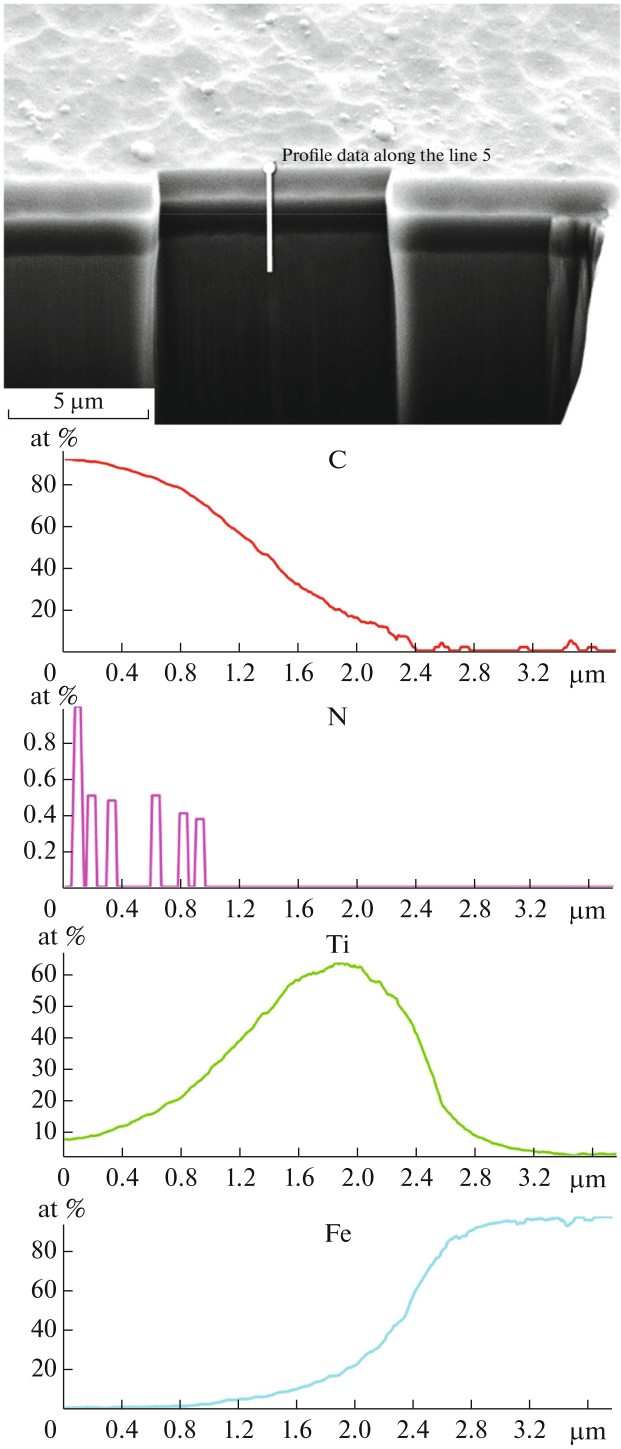

Fig. 2. Distribution of the main elements along the cut line (the line shown in the electronic image: from the surface of the DLC, indicated by a dot, into the sample depth) in a coating similar to Fig. 1: DLC stabilized with nitrogen, with a Ti sublayer.

To reliably determine and analyze the tribological properties of coatings intended for use in loaded friction pairs, it is necessary to clarify a methodological issue, the magnitude of the load when tested on a friction machine. For this purpose, Fig. 3 shows the dependences of the average values of tribological characteristics μ and L on friction load F for the entire database as a whole.

As can be seen from Fig. 3, the spread of the values of μ and L significantly exceeds the confidence interval, especially at low loads $F \leq 5$ N. In view of this circumstance, an analysis of the experimental database on parameter μ was carried out using neural network and machine learning algorithms.

The initial dataset consisted of the results of 58 experiments on the application of DLCs of which 36 rows were filled in for friction coefficient μ. The input parameters of the model included amount of nitrogen %N supplied to the chamber and current λ supplied to the induction coils (A). The output (target) parameters of the model included the values of μ obtained at different load values F equal to 1, 5, and 10 N, and denoted in the model as *CoatMu1*, *CoatMu5*, *CoatMu10*, respectively. To approximate the dependence of friction coefficient μ on technological parameters %N and λ three algorithms were used: the linear Ridge algorithm (ridge regression), the ExtraTrees algorithm (randomized trees), and a fully connected feed-forward neural network (multilayer perceptron, MLP). During training cross-validation was used to correctly assess the quality of model prediction, which was carried out using the method of single exclusion of sample elements (LeaveOneOut cross-validation) due to the relatively small size of the original dataset. Before models training, the data was normalized.

As a regularity criterion, the value of the coefficient of determination R^2 was used, which is determined as follows. Let y_i and f_i ($i = 1, ..., n$) be the experimental and predicted values of the unknown function (friction coefficient μ), and m be the average value of all y_i, then the value of R^2 is calculated as:

$$R^2 score = 1 - \frac{\sum (y_i - f_i)^2}{\sum (y_i - m)^2}.$$

The maximum value of $R^2 = 1$ corresponds to ideally accurate prediction quality. In practice, R^2 can take zero or even negative values if the data is random noise or contains large outliers, or for a poorly trained model [13, 14].

Without going into the mathematical and procedural details of the performed analysis, let us present its useful results. Fig. 4 presents scatter plots constructed by means of the Ridge and ExtraTrees algorithms for each experiment at corresponding load F, which show the predicted values of the $CoatMu_{\text{predicted}}$ friction coefficient and the $CoatMu_{\text{original}}$ values obtained in the experiment. The algorithms were trained on raw data without filtering. The R^2 metric for unfiltered data was close to zero for both algorithms. However, most of the points in Fig. 4, are

Table 1. Average mechanical characteristics of materials used in the work

Material	Mechanical characteristics			
	hardness *H*, GPa	elastic modulus *E*, GPa	H/E	H^3/E^2
Substrate—40CrNi2Mo steel:				
— with sorbite structure	2.5	200	0.0125	0.00039
— with martensite structure	5.2	200	0.026	0.00352
DLC	20 ± 9	250 ± 70	0.068 ± 0.029	0.076 ± 0.031

located along the "prediction = experiment" line, while in the remaining graphs of Fig. 4, the points are close to the constant prediction line. This indicates that the ExtraTrees ensemble model identified higher correlations in the original experimental data than the Ridge algorithm model. Therefore, further work was carried out to improve the quality of prediction in the form of filtering out those experiments in which the ExtraTrees algorithm shows the greatest error. The result obtained after data filtering is shown in Fig. 5. After removing the five experiments with the largest error, the quality of ExtraTrees prediction for the *CoatMu*10 trait increased significantly, reaching R^2 = 0.57. However, filtering did not lead to improved predictions in the Ridge model. For two other target features, *CoatMu*1 and *CoatMu*5, data filtering in the ExtraTrees model resulted in a less noticeable improvement in quality, to R^2 = 0.1 and R^2 = 0.22, respectively. The results indicate that the quality of the prediction ultimately turns out to be higher, the higher the load when measuring the friction coefficient. This is explained by the fact that during tribological tests on a friction machine at low loads, the counterbody (pin), moving along the surface of a coated sample (plate, disk), turns out to be sensitive to uneven surface topography. An "oscillation effect" (repulsion, vibration jumps) of the pin occurs when moving along the relief contour, which leads to a large scatter of μ values. Under heavy loads, crushing, deformation, smoothing, or chipping of surface relief irregularities (artifacts, drip and crystallization coating defects, etc.) occurs under the action of a moving pin (counterbody). At the same time, its movement is carried out more smoothly, due to which the experimental values of μ have less scatter, and the accuracy of the predicted values of μ increases.

Neural networks (MLP) were also trained on both unfiltered and filtered data. In all tests, MLP demonstrated prediction quality similar to the ExtraTrees model. The maximum prediction quality of the MLP algorithm was also achieved for the *CoatMu*10 feature on filtered data and amounted to R^2 = 0.5, which is only slightly inferior to the best prediction quality of the ExtraTrees algorithm (R^2 = 0.57).

Thus, of the results available in the dataset (in the database), only the *CoatMu*10 character data pool is trustworthy. Only character predictions using the ExtraTrees and MLP algorithms be considered reliable. The predictions of the ExtraTrees and MLP models trained on filtered data are shown in Fig. 6 in

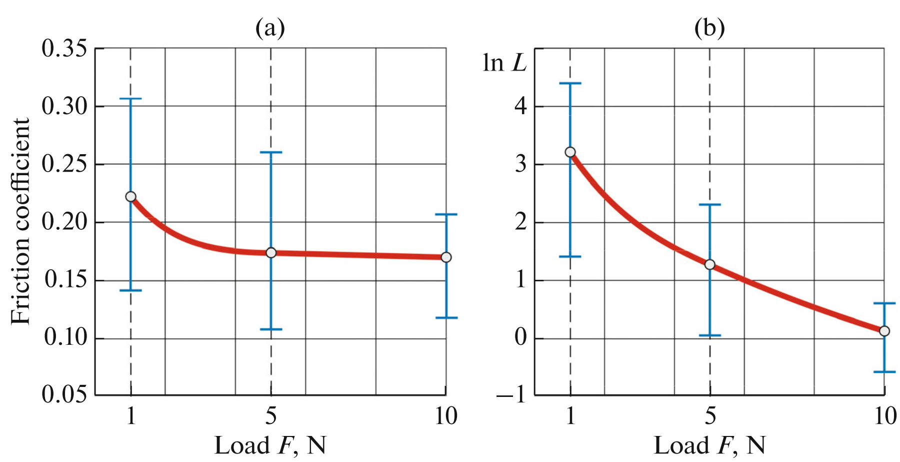

Fig. 3. Experimental dependences of the change in friction coefficient μ (a) and path length *L* (b) traversed by the sample before coating failure on load *F* during tribological tests.

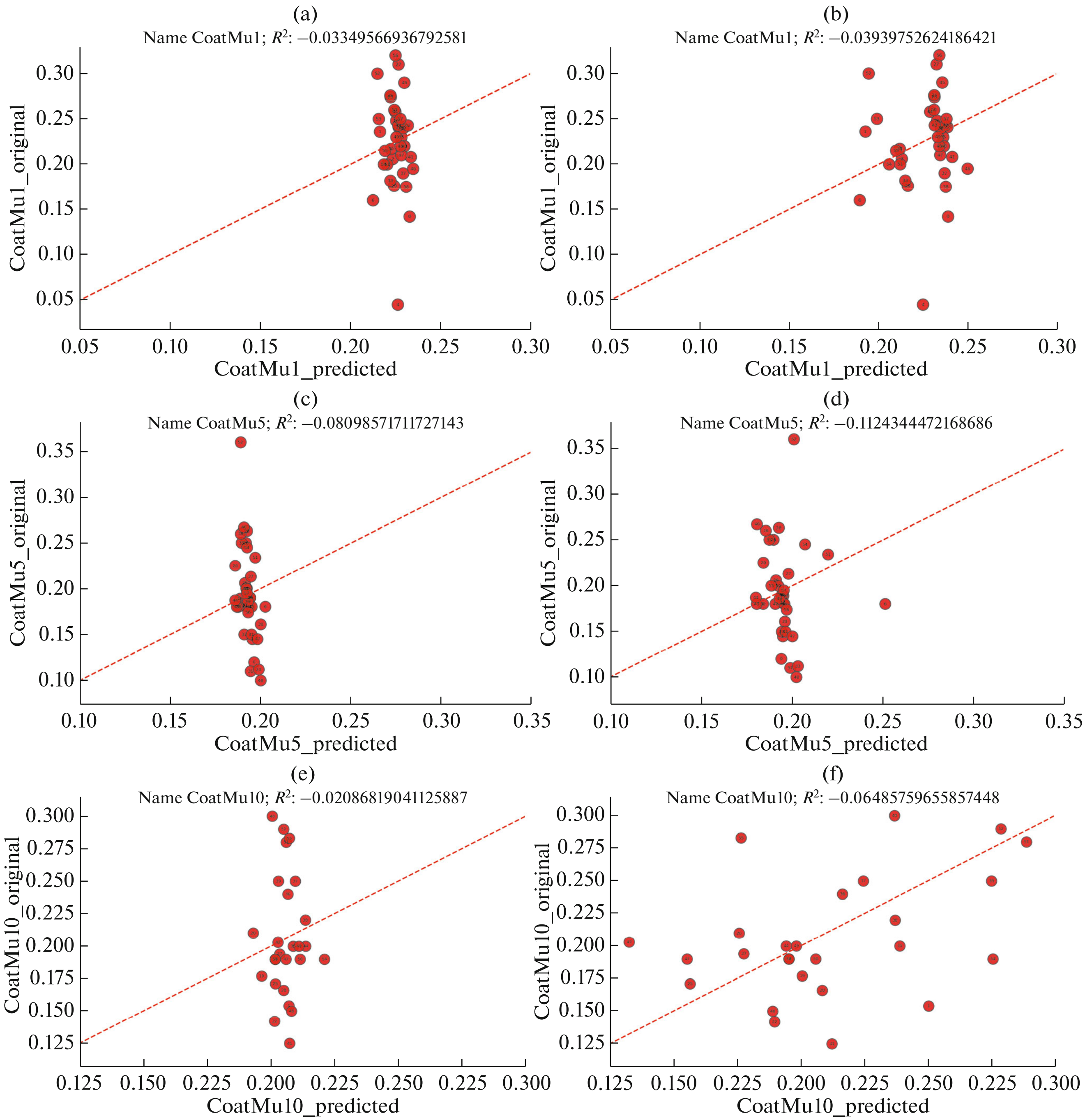

Fig. 4. Scatter plots of unfiltered friction coefficient data constructed for the Ridge regression algorithm (a, c, e) and for the Extra Trees ensemble method (b, d, f) under friction loads $F = 1$ N (a, b), 5 N (c, d), 10 N (e, f).

the form of heat (color) maps constructed on the %N–λ process parameter plane. The constructed color maps highlight zones of optimal values of parameters %N and λ, which predict the lowest values of friction coefficient μ at friction load $F = 10$ N.

To validate the modeling results, experimental one-parameter dependences of μ on each of the varied technological parameters %N and λ were constructed. They are presented in Fig. 7. From a comparison of model (Fig. 6) and experimental (Fig. 7) data, it follows that the correspondence between them is observed in the region of small parameter values from the studied variation intervals, and only for the ExtraTrees model in the region %N = 5–6% and λ = 1.5–2.2 A give de facto exact agreement with the values of μ at the level of 0.17–0.19, both according to

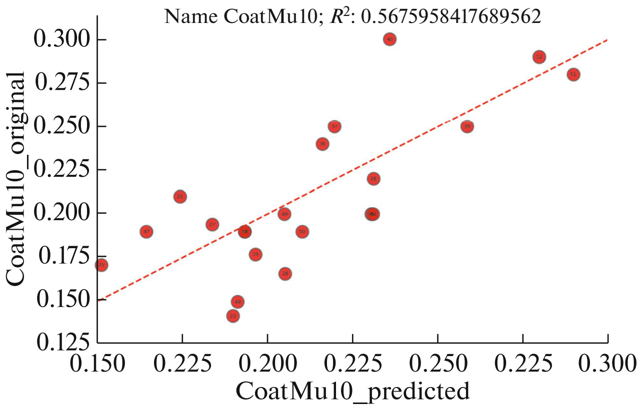

Fig. 5. Scatter plot of the friction coefficient values, built for the Extra Trees algorithm at friction load $F = 10$ N after data filtering.

model predictions and in the experiment. The MLP neural network model predicts a higher level of friction coefficient $\mu = 0.20–0.22$ than is obtained in the experiment. During modeling and optimization of parameters, two more conditions should be taken into account: firstly, when the coil current is $\lambda < 1.5$ A, the plasma discharge in the working chamber of the vacuum installation becomes unstable. Secondly, the friction coefficient values of $\mu \geq 0.20$ do not meet the tribological requirements for DLCs.

With allowance for these conditions, it can be stated that the ExtraTrees algorithm with data filtering, in contrast to the other studied Ridge and MLP algorithms, provides the required accuracy in predicting the tribological characteristics of DLCs for friction loads at the level of $F \approx 10$ N.

With regard to another studied tribotechnical characteristic of coatings, the path length of samples L under load F, then, first of all, it should be noted that when analyzing the generated experimental database, it was not possible to identify any stable dependences of the influence of the %N parameter on the path length of samples with DLCs. The experimental dependence $\ln L = f(\lambda)$, constructed on the basis of the DLC database, demonstrates the presence of a clearly defined maximum at $\lambda = 1.8–2.5$ A, which makes it possible to optimize coating application modes according to parameter λ (current) of induction coils. In addition, the experimental dependences $\ln L = f(\lambda)$ and $\ln L = f(F)$ are approximated with high accuracy using the least squares method by third- and second-order polynomials, respectively (these data are not presented in this work). This made it possible to construct a topological surface of sample path length L (the length of the friction path before the coating is destroyed) on the plane of parameters $F–\lambda$, that is, a graph of the function $\ln L = f(F, \lambda)$, which is shown in Fig. 8.

The intervals of the true values of quantities F and λ, defining the plane of parameters in Fig. 8, are $F = 1–11$ N and $\lambda = 1–5$ A. When constructing the topological surface, for the purpose of maximum clarity, they were converted into discrete code values i and j, respectively, which are shown in Fig. 8 along F- and λ-axes. The encoding algorithm is as follows: $F = 1 + 0.5i$, where $i = 0–20$; $\lambda = 1 + 0.2j$, where $j = 0–20$. Along the vertical axis in Fig. 8 the true values of $\ln L$ are shown, where path length L is expressed in meters.

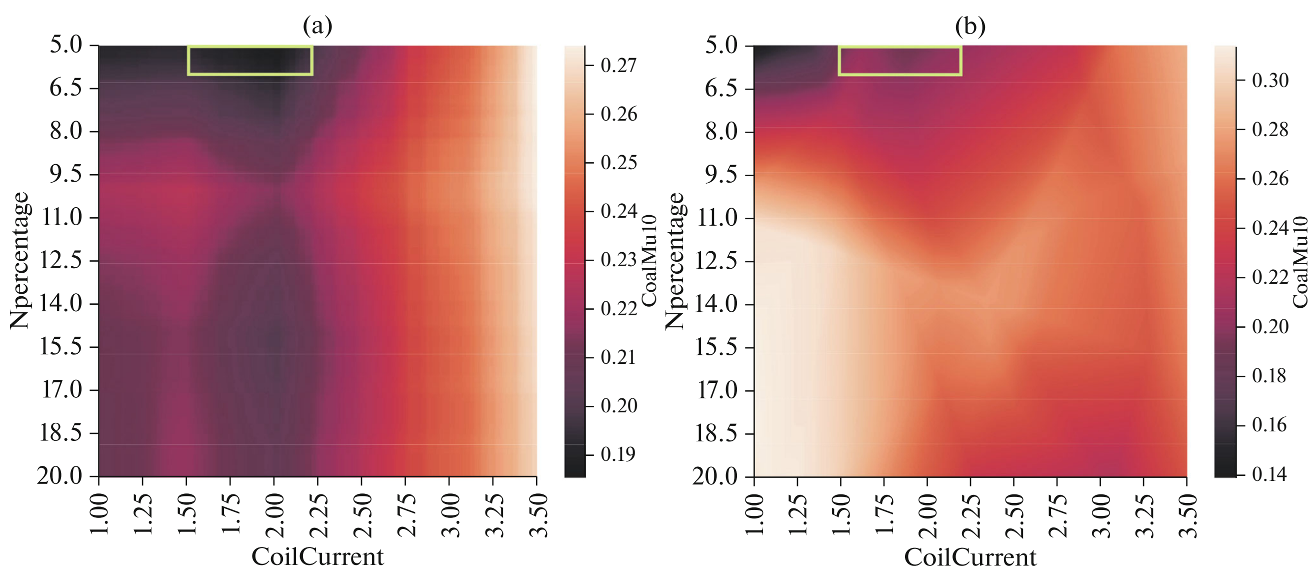

Fig. 6. Two-parameter color maps of the friction coefficient μ, constructed on the %N—λ parameter plane using the trained Extra Trees algorithm (a) and the trained MLP neural network (b) under friction load $F = 10$ N.

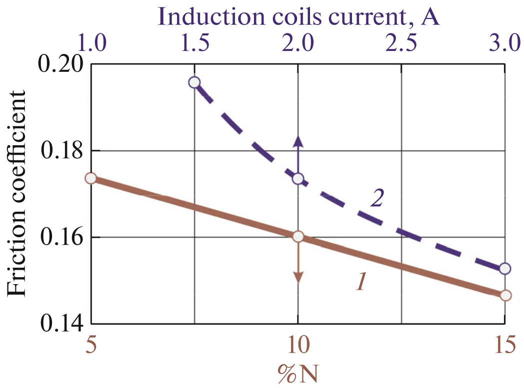

Fig. 7. Experimental dependences of friction coefficient μ on parameters %N (*1*) and λ (*2*) for steel samples with DLCs at friction load $F = 10$ N.

The change in the length of friction track L depending on load F in Fig. 8 is characterized by the same reasons as the change in friction coefficient μ: at small loads $F \leq 5$ N, due to the "oscillation effect" of the friction machine pin, the surface wear is very small. Therefore, with the chosen scheme of tribological tests, only the results obtained at relatively large loads ($F = 10$ N) are worthy of attention, moreover since the nature of dependence $\ln L = f(\lambda)$ (curve with a maximum) when load F changes is preserved. The stability of this dependence allows us to reliably determine the intervals of optimal values of technological parameters %N = 5.5 ± 0.5% and λ = 2.0 ± 0.2 A, based on both analytical (predicted) and experimental values of friction coefficient μ and sample path length L.

CONCLUSIONS

The results of the performed research show that the use of nitrogen to stabilize carbon coatings instead of hydrogen ensures not only stable thickness of DLCs at the level of 1.0–1.5 microns, but also serves as an important and convenient process parameter for regulating the tribological characteristics of the coating during its application.

Despite the dominance of the diamond fraction of electron carbon configurations in the studied coatings ($sp^2/sp^3 = 0.1/0.9$ in the database as a whole), the values of friction coefficient μ of the synthesized nitrogen-stabilized DLCs are inferior to diamond friction coefficient μ and at optimal application modes are at the level μ = 0.17–0.19. On the other hand, the obtained values of μ of synthesized nitrogen-stabilized DLCs (a-C:N) approximately correspond to the μ values of amorphous hydrogen-stabilized coatings (a-C:H) [3, 15]. This also indicates the feasibility of using nitrogen stabilization of DLCs instead of explosive hydrogen.

The experimental dependences of parameters F, %N, and λ on the value of friction coefficient μ established in the work have the following physical meaning. An increase in the intensity of the ionic flux, which is regulated by the current of induction coils λ during coating application, and an increase in load F during friction tests contribute to the coating compaction, reducing its defectiveness and smoothing the relief, which ultimately reduces mechanical friction component μ. Increasing the proportion of nitrogen chemical bonds in the coating (%N parameter) reduces the number of uncompensated atomic carbon

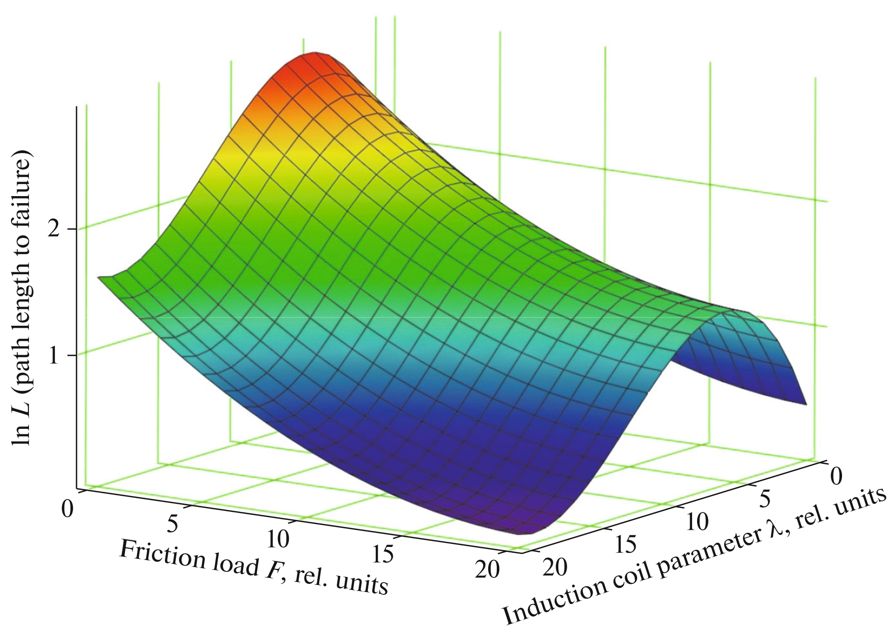

Fig. 8. Topological surface of change in path length L depending on parameters λ and F.

bonds in the coating surface layer, which reduces the adhesion component of friction coefficient μ, which contributes to the adhesion of the coating to the counterbody during friction. The dependences of path length L during tribological tests, which characterize the wear resistance of coatings, either have a complex curve shape with a maximum (dependence $\ln L = f(\lambda)$), or are not identified at all from the available database (dependence $\ln L = f(\%N)$). This makes it very difficult to determine their physical significance.

DLCs with a titanium sublayer, obtained on the substrate of 40CrNi2Mo steel using optimal application modes with values of technological parameters $\%N = 5.5 \pm 0.5\%$ and $\lambda = 2.0 \pm 0.2$ A, can be recommended for practical use under friction conditions equivalent to the tribological tests carried out at a friction load $F \approx 10$ N.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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