

## Surface Modification of Diamond-Like Carbon Coatings to Control over Run-In Processes in Friction Pair

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**Abstract**—The effect of the nanostructure and chemical composition of thin Mo–Se–C films on the run-in of the diamond-like carbon coating (a-C) and the steel counterbody during ball-on-disk sliding test is studied. At the final stage of the deposition of the a-C coating on the steel substrate, a flow of atoms from the MoSe<sub>2</sub> target was added to the flow of atoms being deposited. The influence of the air humidity on the selection of optimum structure and chemical composition of the film, which provide a fairly low (<0.07) coefficient of friction during the entire period of the run-in stage, is considered. In order to explain the effect of the modifying films, analysis of the specific features of the friction-induced changes in the composition of the films during wear debris formation has been carried out using Raman spectroscopy.

**Keywords:** diamond-like coatings, pulsed laser deposition, run-in, coefficient of friction, nanostructured thin films

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### INTRODUCTION

Amorphous diamond-like carbon coatings (a-C) are characterized by a wide set of original mechanical, physical, and chemical properties; of these properties, a low coefficient of friction, and high hardness and wear resistance, as well as chemical inertness, are especially important for the practical use of these coatings in tribology. These coatings have found wide commercial application all over the world to enhance the efficiency of tools and members, especially in automotive industry. A fairly comprehensive description of all possible fields of application of a-C coatings can be found in [1].

The a-C coatings are generally characterized as wear-resistant solid-lubricating coatings, which can provide a very low coefficient of friction (down to 0.001 for hydrogen-containing a-C coatings under sliding in vacuum). However, a specific feature of the behavior of the a-C coatings under dry sliding in air is a fairly high initial value of the coefficient of friction, which slowly decreases due to the run-in of the surfaces in contact. The amplitude of changes in the coefficient of friction and the duration of run-in depend on both the properties of the coating, such as the proportion of  $sp^2/sp^3$  bonds, the density, and the concentration of alloying impurities, especially hydrogen, and on conditions of friction, i.e., the load, the air humidity, and the nature of the counterbody [2, 3]. It is commonly believed that, in the course of run-in, a tribofilm, which provides a decrease in the force of friction,

is formed on the surfaces in contact [4]. The formation of this film is accompanied by a slight wear of the a-C coating and the counterbody, and the chemical composition of this tribofilm is determined by the wear products.

For practical application of a-C coatings to modify friction pairs, it seems important to accelerate the process of triboadaptation and minimize the wear of the members of a friction pair at this stage. From the scientific point of view, it is a topical issue to identify the mechanisms of the formation of the tribofilm and to search for ways to control this formation. One of the possible methods for controlling the run-in stage involves the deposition of a special thin film to reduce the coefficient of friction and facilitate the formation of the tribofilm with an optimum composition. In the course of the operation of the friction pair, the chemical elements contained in the modifying film should be removed from the tribofilm and replaced with the elements contained in the materials of the friction members. The role of this modifying film can be played by a film that contains a solid-lubricating component based on transition-metal dichalcogenides (TMDs). Coatings that contain TMDs have a very low coefficient of friction under sliding in relatively dry air. However, the surfaces of TMD-based and a-C materials are very inert, which can hamper the good adhesion of a TMD film to an a-C coating. Therefore, the possibility of changing the chemical properties of the TMD film by alloying it with carbon should be inves-

tigated [5]. It is also of interest to study the behavior of a multilayer modifying film, which contains nanosized TMD and a-C layers. It is important to determine the dependence of the run-in process, which occurs with the participation of the TMD-containing film, on the air humidity.

**The aim of this work** was to investigate the possibility of reducing the coefficient of friction at the initial stage (the run-in stage) of the sliding of a steel counterbody against a diamond-like coating by the preliminary deposition of a thin film based on solid-lubricating molybdenum diselenide ( $\text{MoSe}_2$ ) on that coating, as well as to study specific features of the friction and wear of the a-C coatings with the participation of the modifying films of various structures and chemical compositions.

## EXPERIMENTAL

a-C coatings and  $\text{MoSe}_x$ -containing modifying films were produced using pulsed laser deposition, which involved the standard arrangement and an antidrip screen [6]. The substrates were polished 95Kh18 steel plates. Laser radiation was supplied to the vacuum chamber and scanned over the target holder using an automated device, which operated following a special program. Two targets were placed on the holder, one made of synthesized  $\text{MoSe}_2$  and the other of graphite. The a-C coating was initially deposited from the graphite target following the standard procedure. After the  $\sim 0.5\text{-}\mu\text{m}$ -thick coating was deposited, the laser beam was directed to the  $\text{MoSe}_2$  target or fairly rapidly switched from one target to the other so that the amount of the substance deposited in a single cycle of target irradiation not to exceed a single monolayer. In some cases, a disk screen was placed ahead of the  $\text{MoSe}_2$  target, which prevented the deposition of molybdenum nanoparticles and large drops. In order to enhance the efficiency of deposition in the shadow zone, argon was supplied to the chamber until the pressure of 2 Pa was attained. In some experiments, the negative bias voltage of  $-200\text{ V}$  was supplied to the substrate in order to initiate the ion bombardment of the film being deposited, which was necessary to increase the density of the film.

When producing the modifying films, the duration of the irradiation of the  $\text{MoSe}_2$  and graphite targets was varied in such a way as to change the atomic concentration of carbon in the Mo–Se–C composite film from 0 to  $\sim 75\%$ . In the case of vacuum deposition (without using the screen), the ratio  $x = \text{Se}/\text{Mo}$  was less than the stoichiometric value; however, with increasing concentration of carbon the concentration of selenium grew and the ratio increased from 1.5 to 1.9. When the screen was used, the ratio  $x$  in the films substantially exceeded the stoichiometric value and reached the value  $x \sim 4$ , but with increasing concentra-

tion of carbon and under the conditions of ion bombardment the ratio  $x$  decreased to the value of  $\sim 3$ .

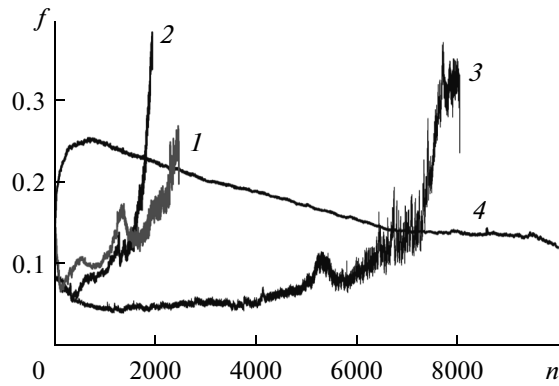
In addition to the composite films, multilayer films were produced that consisted of alternate  $\text{MoSe}_3$  and a-C layers. The layers were  $\sim 25$ , 8, and 3 nm thick. The total thickness of all modifying films was 100–200 nm.

The targets were irradiated using nanosecond laser pulses with an energy density of  $\sim 10\text{ J}/\text{cm}^2$ . The main characteristics of the a-C coatings produced using this energy density to irradiate the graphite target were studied elsewhere [7]. During the deposition of all coatings, the temperature of the substrate was equal to the room temperature.

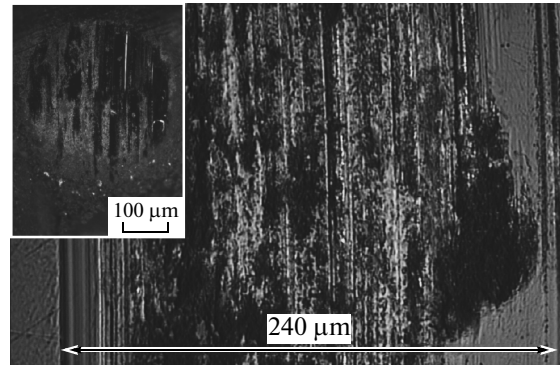
The thickness and chemical composition of the films were studied using Rutherford backscattering spectroscopy of helium ions. Tribotests of the coatings were carried out using a Tribometer (CSM Instruments), which used the stationary ball–rotating disk (coated substrate) arrangement. The tribotests were performed under dry conditions in air with reduced ( $\sim 40\%$ ) and increased (70%) relative humidity. A ball 3 mm in diameter was made of 100Cr6 steel. The load applied to the ball was 1 N. The velocity of sliding was selected to be equal to 10 cm/s. Trial tests had shown that the modifying films were completely worn out in a few thousands of friction cycles; therefore, the total duration of the tests was limited by the value of  $10^4$  cycles. After the tribotests, the surfaces of the substrate and the steel ball were examined using optical microscopy and profilometry, which made it possible to determine the wear rates of the coating and the counterbody. Analysis of the surfaces of the friction tracks was carried out before and after the ultrasonic washing of the specimens. The phase composition of the films before and after the tribotests was examined using Raman spectroscopy at a wavelength of 473 nm implemented in an NTEGRA Spectra spectrometer. The diameter of the laser probe did not exceed  $0.2\text{ }\mu\text{m}$ .

## RESULTS AND DISCUSSION

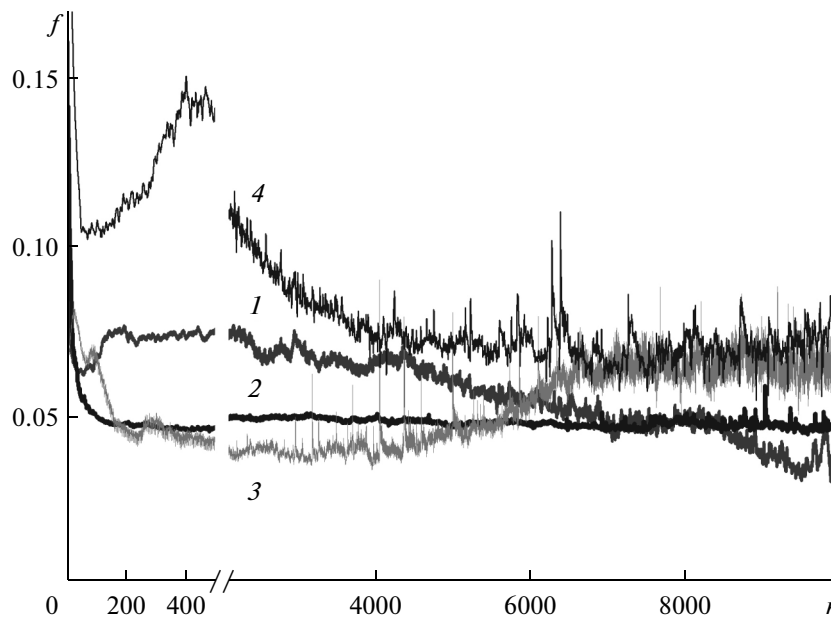
Figure 1 shows the results of measuring the coefficient of friction during the tests of the multilayer films on the a-C coating carried out at the air humidity of 40%. It can be seen that during sliding against the film with the fairly thick (8 and 25 nm) layers the wavelike dependence of the coefficient of friction is observed, which is related to the successive wear of the  $\text{MoSe}_3$  and a-C layers. When the thickness of the layers is reduced to 3 nm, the coefficient of friction at the initial test stage substantially decreases. However, the complete wear of this multilayer film leads to the sharp rise in the coefficient of friction, followed by the severe wear of the a-C coating and the counterbody. The width of the friction track exceeds  $240\text{ }\mu\text{m}$ , and the signs of oxidation are observed on the surface of the track (Fig. 2). It can be assumed that during the sliding test of the multilayer films hard a-C nanoparticles



**Fig. 1.** Dependences of coefficient of friction on number of cycles in tribotests of a-C coating covered with multilayer modifying MoSe<sub>3</sub>/a-C films with different thickness of layers: (1) 25, (2) 8, and (3) 3 nm and (4) original a-C coating.



**Fig. 2.** Images of friction track and (inset, dark field) zone of contact on counterface after tribotest of multilayer MoSe<sub>3</sub>/a-C film with 3-nm-thick layers on a-C coating over  $8 \times 10^3$  cycles.

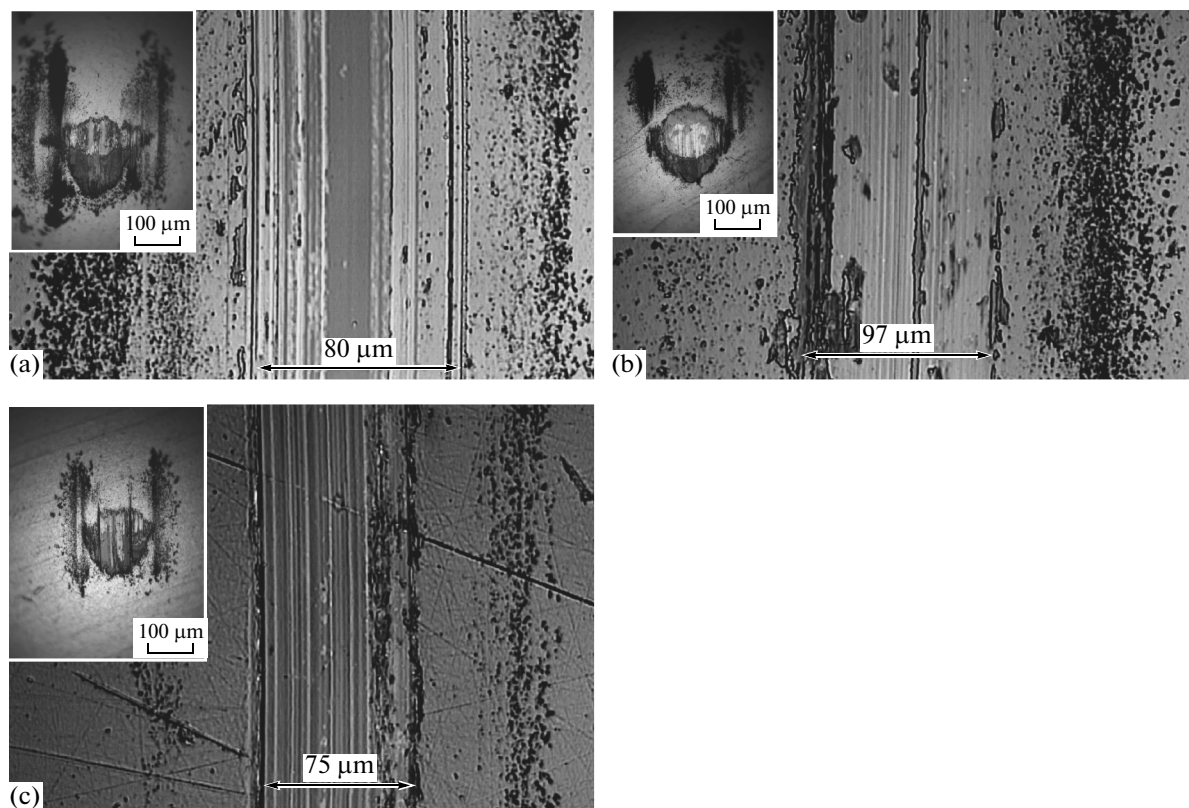


**Fig. 3.** Dependences of coefficient of friction on number of cycles in tribotests of a-C coating covered with various modifying films in dry air: (1) MoSe<sub>1.5</sub>, (2) MoSe<sub>1.7</sub>-C (25%), (3) MoSe<sub>3</sub>-C (50%), and (4) MoSe<sub>4</sub>-C (25%).

could be formed due to the cracking of the hard a-C layers; those particles could have abrasive impact and hampered the formation of a continuous tribofilm with an optimum composition.

The behavior of the composite Mo–Se–C films during their sliding against the counterbody is governed by the concentration of the elements and the air humidity. Figures 3 and 4 show the results of the tribotests carried out in dry air. The typical initial value of the coefficient of friction for the films of this type is 0.15–0.25. The coefficient of friction of the MoSe<sub>1.7</sub>-C (25%) and MoSe<sub>3</sub>-C (50%) films decreases to ~0.05 in about ten friction cycles. We note that the

films that contain over 50% of carbon atoms have the negative effect on run-in since their use leads to the noticeable increase in the coefficient of friction (results are not presented). The most efficient run-in is observed when using a film of pure molybdenum diselenide. It can be seen in Fig. 4a that the MoSe<sub>1.5</sub> film is completely removed at the run-in stage; however, this does not result in wear of the a-C coating, but leads to a slight wear of the counterbody. The wear factor for the ball determined using the method described in [8] does not exceed  $W \sim 1.4 \times 10^{-8} \text{ mm}^3/(\text{N m})$ . Examination of the nanostructure of these films has shown that they contain spherical molybdenum parti-



**Fig. 4.** Images of friction track and (insets) zone of contact on counterface after tribotests of a-C coating covered with various modifying films in dry air over  $10^4$  cycles: (1)  $\text{MoSe}_{1.5}$ , (2)  $\text{MoSe}_{1.7}\text{-C}$  (25%), and (3)  $\text{MoSe}_3\text{-C}$  (50%).

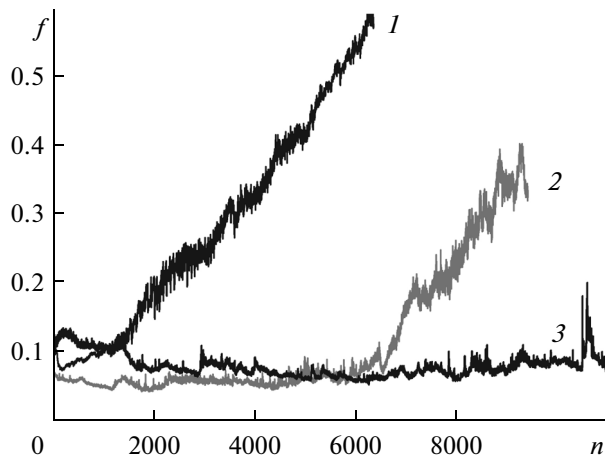
cles up to  $\sim 50$  nm in size [9]. Apparently, these nanoparticles had an effect on the development of run-in.

The deposition of the composite  $\text{MoSe}_{1.7}\text{-C}$  (25%) and  $\text{MoSe}_3\text{-C}$  (50%) films also leads to the noticeable decrease in the coefficient of friction (Fig. 3). However, in this case, the friction track has the nonuniform surface (Figs. 4b and 4c), which is indicative of the fairly slight wear of the a-C coating. The wear factor for the counterbody increases to  $W \sim 2.5 \times 10^{-8} \text{ mm}^3/(\text{N m})$ . We note that increasing concentration of carbon in the Mo–Se–C films reduces the efficiency of the sticking of the wear particles to the counterface and improves the efficiency of their removal from the surface of the specimen. As a result, the productive influence of the thin  $\text{MoSe}_3\text{-C}$  (50%) film on the coefficient of friction is retained during the shorter period ( $5 \times 10^3$  cycles) as compared with that of the other films that contain the smaller amounts of carbon. Subsequently, the friction contact apparently occurred between the counterface and the a-C coating.

A comparison of the results of the tribotests of the  $\text{MoSe}_3\text{-C}$  (50%) films deposited on the substrate with the applied bias voltage using the screen, i.e., under the conditions of ion bombardment, and the  $\text{MoSe}_4\text{-C}$  (25%) films produced without applying the bias voltage has shown that the excessively high concentra-

tion of Se in the  $\text{MoSe}_x$  phase vs. the stoichiometric concentration deteriorates the tribological characteristics of the Mo–Se–C films (Fig. 3). At the initial stage of the tribotests of the  $\text{MoSe}_4\text{-C}$  (25%) films, they are rapidly removed from the friction track, which provides the almost perfect contact of the counterface with the a-C coating. In this case, the coefficient of friction increases to 0.15. Subsequently, the coefficient of friction fairly rapidly decreases to 0.07, but this does not eliminate the wear of the counterbody. The wear factor for the counterbody in pair with the composite  $\text{MoSe}_4\text{-C}$  (25%) film is  $W \sim 3 \times 10^{-8} \text{ mm}^3/(\text{N m})$ . We note that, despite the rapid wear of these films (in  $\sim 400$  cycles), they have generally a noticeable effect on the subsequent sliding of the counterbody against the a-C coating. When the tribotests are carried out under similar conditions, but in the absence of the solid-lubricating films, the wear factor for the steel counterbody in a pair with the a-C coating can reach  $W \sim 1.3 \times 10^{-6} \text{ mm}^3/(\text{N m})$  [2].

The results of the tribotests performed at the increased air humidity have shown that the  $\text{MoSe}_{1.5}$  films have the negative effect on the run-in of the a-C coating (Fig. 5), which can be explained by the specific feature of the behavior of the TMD-containing materials in the oxidizing environment. The low friction characteristics of TMD-containing materials



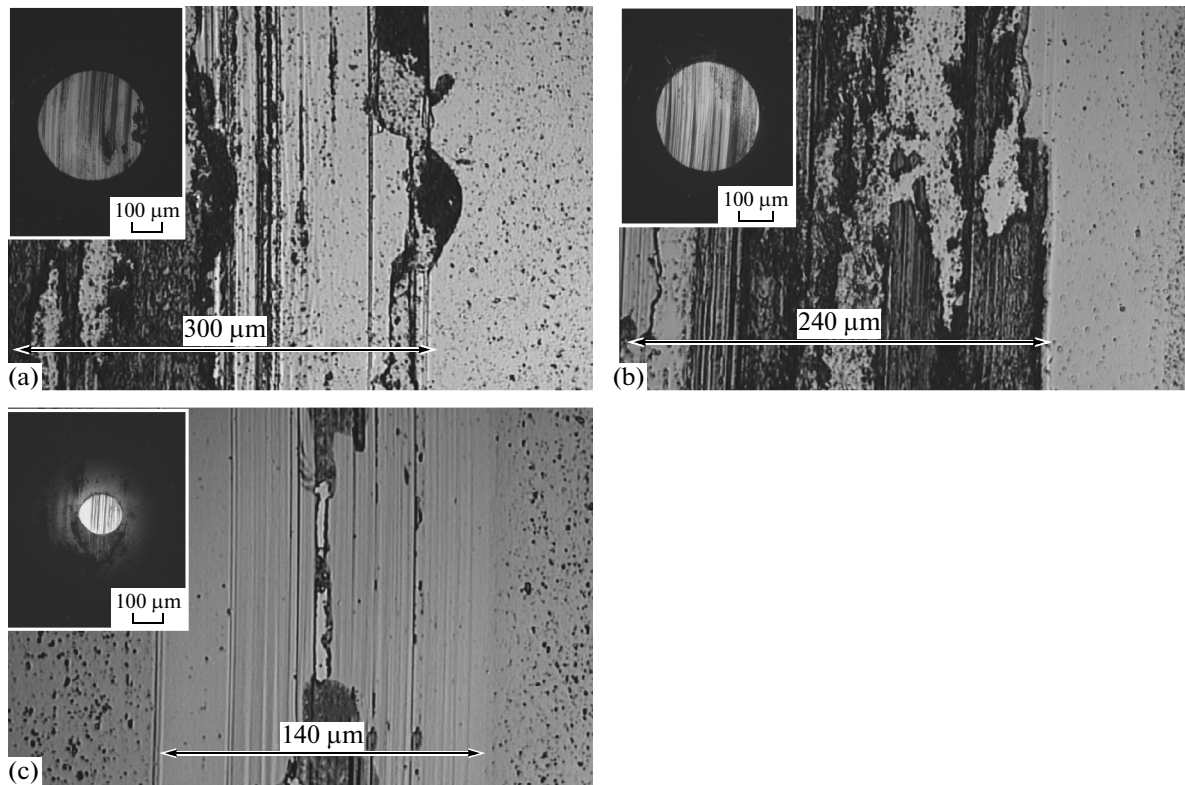
**Fig. 5.** Dependences of coefficient of friction on number of cycles in tribotests of a-C coating covered with various modifying films in humid air: (1)  $\text{MoSe}_{1.5}$ , (2)  $\text{MoSe}_{1.7}\text{-C}$  (25%), and (3)  $\text{MoSe}_3\text{-C}$  (50%).

substantially deteriorate when oxidizing processes occur, which results in the destruction of the layered structure of the TMD-containing films and in the formation of hard molybdenum oxides. As a result, the presence of the  $\text{MoSe}_{1.5}$  films leads to the severe abra-

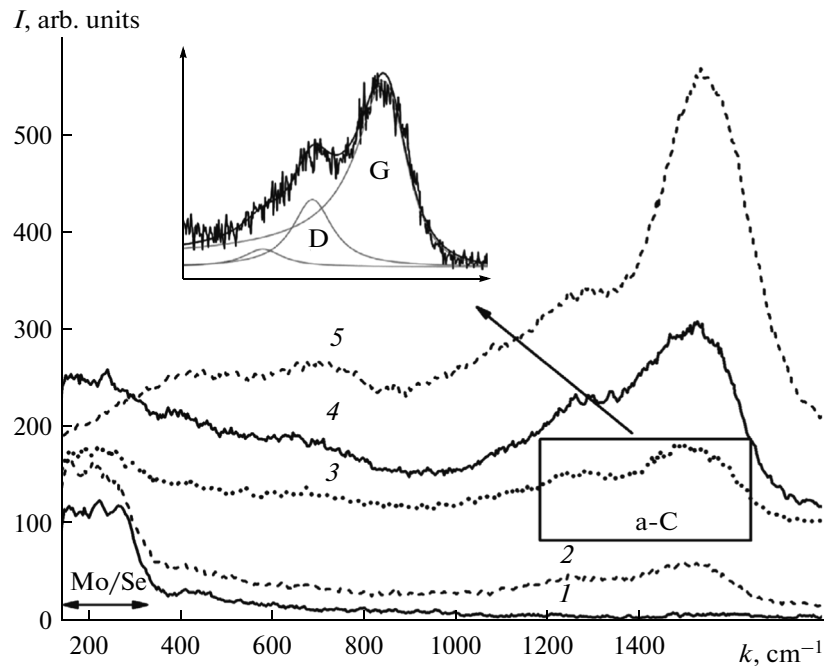
sive wear of the a-C coating, which propagates almost to the steel substrate (Fig. 6a).

Joint use of the TMD phase and carbon substantially changes the tribological characteristics of the Mo-Se-C films deposited on the a-C coating. The results of the tribotests presented in Fig. 5 have shown that increasing the concentration of carbon to ~50% results in the fairly low coefficient of friction (~0.15) at the initial test stage (~1500 cycles) and the ever lower coefficient of friction (0.08) during steady sliding. In the course of  $10^4$  sliding cycles, the composite  $\text{MoSe}_3\text{-C}$  (50%) film is completely removed from the friction track, and the friction contact occurs between the steel counterface and the a-C coating. The wear factor for the counterbody is  $W \sim 7.3 \times 10^{-8} \text{ mm}^3/(\text{N m})$ . Considering that the tribological characteristics of the a-C coatings depend only slightly on the relative air humidity in the range of 40–70%, the somewhat increased wear factor for the counterbody (compared to that obtained under dry friction) should be attributed to the effect of water vapor on the behavior of the TMD phase in the composite coatings.

The Raman spectroscopy data have shown that phase separation and the formation of nanosized  $\text{MoSe}_x$  and a-C clusters occur already at the lowest concentration of carbon (25 at %) in the Mo-Se-C



**Fig. 6.** Images of friction track and (insets) zone of contact on counterface after tribotests of a-C coating covered with various modifying films in humid air: (1)  $\text{MoSe}_{1.5}$ , (2)  $\text{MoSe}_{1.7}\text{-C}$  (25%), and (3)  $\text{MoSe}_3\text{-C}$  (50%).



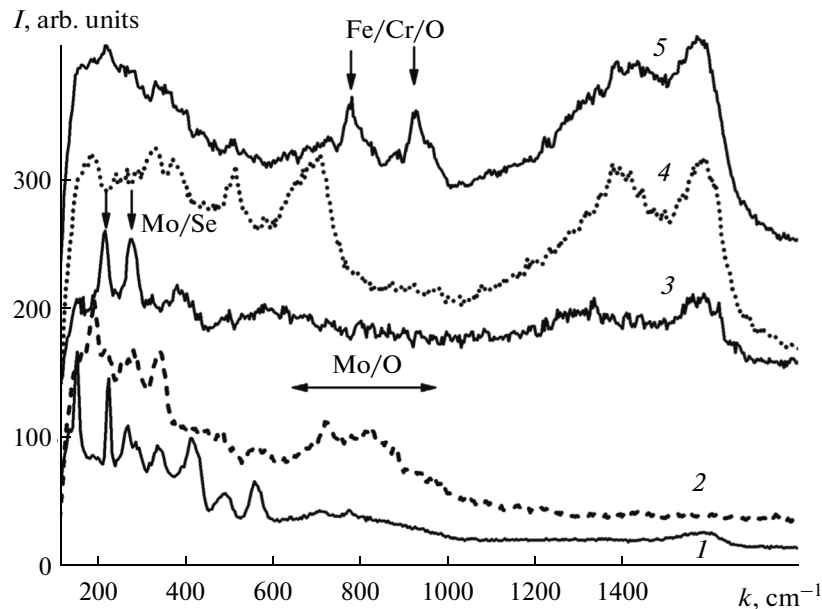
**Fig. 7.** Raman spectra for various modifying films: (1)  $\text{MoSe}_{1.5}$ , (2)  $\text{MoSe}_{1.7}\text{-C}$  (25%), (3)  $\text{MoSe}_3\text{-C}$  (50%), (4)  $\text{MoSe}_{1.9}\text{-C}$  (75%), and (5) pure a-C coating. Inset shows decomposition of spectrum of carbon into G and D lines.

films implemented in the course of their deposition (Fig. 7). The properties of the resulted phases are determined by the concentration of carbon in the film. In the pure  $\text{MoSe}_{1.5}$  film, local ordering of the atomic packing occurs, followed by the formation of “layered”  $\text{MoSe}_2$  clusters, while the alloying of this film with carbon leads to the complete amorphization of the  $\text{MoSe}_x$  phase. The ordering of the atomic packing in the  $\text{MoSe}_2$  nanoclusters is confirmed by the presence of the relatively narrow lines in the Raman spectrum (Fig. 7, spectrum 1) at 230 and 280  $\text{cm}^{-1}$  [10]. The Raman spectrum of the completely amorphized  $\text{MoSe}_x$  phase is presented by a largely broadened band in the range of 150–300  $\text{cm}^{-1}$ .

The properties of the a-C phase in the composite films were determined using the results of the decomposition of the Raman spectra of carbon (the range 1100–1700  $\text{cm}^{-1}$ ) into the components G and D; using the parameters of those lines, such as the wavenumber and the relative intensity, the proportion of graphite ( $sp^2$  hybridization) and diamond ( $sp^3$  hybridization) bonds was found [11]. In addition to the G and D lines located at 1535 and 1270  $\text{cm}^{-1}$ , respectively, the extra line appears in the decomposition of the spectrum in the vicinity of 1090  $\text{cm}^{-1}$ ; the origin of this line is still unclear. Analysis of the Raman spectra has shown that the concentration of the  $sp^3$  bonds in the composite films with the smallest amount of carbon does not exceed 17% and the size of the graphite

clusters is  $\sim 0.8$  nm. Increasing concentration of carbon leads to the growth in the concentration of the  $sp^3$  bonds to 20% (at 75 at % of C) and to the decrease in the size of the graphite clusters in the amorphous a-C matrix to 0.5 nm. The increase in the concentration of carbon results also to a growth in the mechanical stresses in the composite film.

The specific features of the nanostructure of the modifying films, which was produced during their deposition, have a pronounced effect on the formation of wear debris at the run-in stage. Figure 8 shows the Raman spectrum of the wear debris retained on the friction track after the tribotests of the a-C coatings covered with various modifying films, which were carried out in humid air, as well as the Raman spectrum of the original a-C coating. For comparison, the spectrum recorded for synthesized  $\text{MoSe}_2$ , which was subjected to heating by a high-energy laser beam used in Raman spectroscopy, is presented. The friction of the films that contain up to 50 at % of carbon results in the crystallization of the  $\text{MoSe}_x$  phase, which manifests as the appearance of the fairly narrow peaks in the corresponding wavenumber range. The positions of these peaks correlate to some extent with the positions of the peaks in the spectrum of the synthesized  $\text{MoSe}_2$  specimen subjected to heating in air. The shift in the positions of the peaks in the spectrum of the thin-film specimen toward lower wavenumbers (220 and 280  $\text{cm}^{-1}$ ) relative to those observed in the spectrum of the synthesized specimen can be attributed to the



**Fig. 8.** Raman spectra for (1) synthesized  $\text{MoSe}_2$  and wear debris resulted from tribotests of a-C coating covered with various modifying films: (2)  $\text{MoSe}_{1.5}$ , (3)  $\text{MoSe}_3\text{-C}$  (50%), (4)  $\text{MoSe}_{1.9}\text{-C}$  (75%), and (5) pure a-C coating.

small size of the nanocrystalline  $\text{MoSe}_2$  particles contained in the wear debris. The Raman spectrum of the wear debris of the  $\text{MoSe}_{1.5}$  film also contains the lines that can correspond to the molybdenum oxides.

Friction also results in the modification of the a-C nanophase, which manifests as its graphitization. This is confirmed by the character of the variations in the parameters of the G and D lines. The wear of the original a-C coating leads to more pronounced graphitization than the wear of the composite films that contain  $\leq 50$  at % of carbon. In the wear debris of the a-C coating, the traces of the oxides of the metals contained in the material of the counterbody have also been found.

The specific feature of the Raman spectrum of the wear debris of the composite  $\text{MoSe}_3\text{-C}$  (50%) films is the absence of discernible signs of the formation of molybdenum oxides, as well as iron and chromium oxides. This is indicative of the fact that the phase composition and structure of the modifying  $\text{MoSe}_3\text{-C}$  (50%) film turn out to be optimum from the viewpoint of the formation of the tribofilm, which contains two solid-lubricating nanophases, i.e.,  $\text{MoSe}_2$  and graphite; one of these phases is responsible for the low coefficient of friction in dry air, and the other provides the low coefficient of friction in humid air.

## CONCLUSIONS

Thin films that contain carbon and molybdenum diselenide have the pronounced effect on the run-in processes that occur during the sliding of the steel counterbody against the a-C coating on the steel substrate. The character of these processes largely depends on the

structure and chemical composition of the films. The use of multilayer films that consist of nanosized  $\text{MoSe}_2$  and a-C layers can lead to severe wear of the friction members since their fracture is accompanied by the formation of the hard diamond-like carbon nanoparticles, which result in abrasive wear.

The effect of the composite Mo–Se–C films on run-in is related to the friction-induced transformations of their structure. In dry air, the most productive effect is observed when using the  $\text{MoSe}_{1.5}$  film, which provides a low ( $\sim 0.07$ ) initial coefficient of friction; upon removal of the film, i.e., when the contact of the counterface with the a-C coating occurs, this value of the coefficient of friction changes only slightly. In this case, the lowest wear factor for the steel counterbody has been obtained. At increased air humidity, the  $\text{MoSe}_{1.5}$  film undergoes accelerated wear and causes the wear of the a-C coating because of the formation of molybdenum oxides in the tribofilm.

The oxidizing process is substantially retarded in the case of sliding of the counterbody against the composite film that contains 50 at % of carbon. The friction of this film results in the formation of a two-phase tribofilm, which contains molybdenum diselenide nanoparticles with a layered structure and nanosized graphite. During sliding of the counterbody against this composite film, the coefficient of friction depends on the air humidity only slightly and is equal to 0.04–0.07. Moreover, under all conditions of testing of this modifying film, fairly slight wear of the coating and counterbody is observed.

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## NOTATION

$x$	ratio of atomic concentrations Se/Mo in composite coatings
$f$	coefficient of friction
$n$	number of cycles of sliding of steel ball against coating in tribotests
$I$	intensity of signal in Raman spectra
$k$	wavenumber, and
$W$	wear factor

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