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Mono- and Multimolecular Langmuir–Blodgett Films as Protective Coatings for Friction Surfaces

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Abstract—Tribological properties of monomolecular Langmuir–Blodgett films formed from behenic acid, dimethyl octadecylmalonate, 2,4-heneicosanedione, and its copper complex were studied and compared with those of a self-arranged monolayer of octadecyltrichlorosilane. In the micrometer contact range, in friction of a modified silicon surface with a steel ball (indentor), the monomolecular Langmuir–Blodgett films under consideration surpass in the wear resistance the chemisorbed monomolecular octadecyltrichlorosilane film by a factor of 40. The stability of Lamgmuir–Blodgett films in friction correlates with the density of molecular packing on the surface. The friction and wear of Langmuir–Blodgett films are accounted for by the difference in the density of molecular packing of the films under consideration, and also by the transfer of the film substance between the friction surfaces.

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The reliability of operation of contact systems, e.g., in microelectromechanical devices or units for data recording and storage, is determined by conglutination, friction, and wear of contacting components [1–3]. One of promising approaches improving the stability of contacting surfaces is the use of molecular films as protective coatings, thanks to their high uniformity and low friction coefficient [4, 5]. Langmuir monolayers are examples of perfect, close-packed molecular structures on a water surface; their transfer onto a solid surface by the Langmuir–Blodgett method [6] yields homogeneous, defect-free mono- and multilayer films. According to [7, 8], Langmuir–Blodgett films are characterized by low wear resistance caused by high compressibility of the monolayer and low adhesion of the films to the surface at high loads. In this connection, much greater attention was given to testing as protective layers both polymer coatings and covalently grafted self-arranged monolayers based on alkylsilanes [4, 9]. Indeed, polymers are good adhesives, because they undergo plastic and reversible deformation at friction. However, good adhesives have high friction coefficients. Therefore, it was suggested [9–11] to use as boundary lubricants self-arranged monolayers based on chlorosilanes, which are characterized by a highly ordered structure and by strong covalent binding of functional groups of the molecules in the self-arranged monolayer with the silicon surface. It was reported [9] that antifriction properties of self-arranged monolayers are largely determined by their molecular structure (chain length, molecular packing, structure of functional groups). Unfortunately, chemisorbed molecules of self-arranged monolayes, having low friction coefficient, often show poor resistance to mechanical action, which is caused by cleavage of covalent bonds in the course of deformation. One of ways to improve the wear resistance of protective coatings is to prepare composite multilayer polymer systems [12, 13]. Such nanostructured films withstood high local pressure, which is caused by the hardness of their surface, and showed higher wear resistance compared to a classical self-arranged octadecyltrichlorosilane monolayer.

At the same time, Briscoe and Evans [13] reported that, at a low normal load, the wear resistance of the surface coated with Langmuir–Blodgett films based on fatty acids increased by several orders of magnitude relative to the unmodified surface. Higher loads caused physical desorption of the Langmui–Blodgett films from the surface, because of their mechanical and thermal instability [14]. On the other hand, Zhavnenko et al. [15] showed that mobile surfactant molecules in a Langmui–Blodgett film can show high wear resistance, being responsible both for reversible elastic strains and for the energy dissipation due to melting and solidification of the monolayer at friction.

In [7, 16], the tribological properties of modifying films were examined by atomic-force microscopy (AFM). It should be noted, however, that the majority

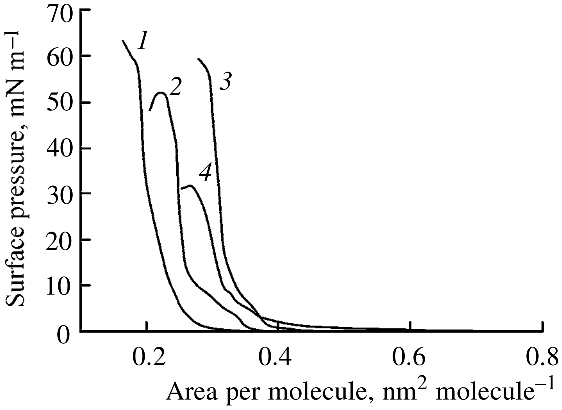

Fig. 1. π–*A* isotherms for (*1*) behenic acid, (*2*) 2,4-heneicosanedione, (*3*) $(HD)_2Cu$, and (*4*) diethyl octadecylmalonate on the water surface. Langmuir–Blodgett monolayers of these surfactants were transferred onto a silicon surface by horizontal deposition at a surface pressure of 30 mN m^{-1}. Isotherm 3 was obtained for $(HD)_2Cu$, but the calculation was made per 2,4-heneicosanedione molecule.

of studies were performed under simplified conditions (friction by an AFM needle of a film on an atomically smooth surface in the nanometer range). Apparently, to improve the tribological characteristics of monomolecular organic films, it is necessary to study their behavior under real friction conditions (geometry and pressure of contact) in a microsystem.

In this study, with the aim to assess the possibility of using Langmuir–Blodgett films as protective boundary layers, we examined the tribological behavior of four different monomolecular Langmuir–Blodgett films in contact of friction surfaces of micrometer size. Langmuir–Blodgett monolayers of various amphiphilic molecules [behenic acid, 2,4-heneicosanedione (HD), its copper complex $(HD)_2Cu$, and diethyl octadecylmalonate] were formed on the silicon surface using the horizontal deposition method. The choice of monomolecular 2,4-heneicosanedione and $(HD)_2Cu$ films as protective coatings is caused by the chemical affinity of the polar groups of their molecules both for the silicon surface and for the surface of a steel ball-indentor, i.e., for both hydrophilic and hydrophobic surfaces [17]. The monolayer of behenic acid as model compound was used for comparison.

Langmuir films on a water surface. The isotherms of the surface pressure vs. area per molecule (π–*A* isotherms) for behenic acid, 2,4-heneicosanedione, $(HD)_2Cu$, and diethyl octadecylmalonate (Fig. 1) show that these substances are good surfactants, and their Langmuir monolayers can be transferred onto a solid surface. In all cases, we recorded a plateau on the isotherms, suggesting a phase transition from a liquid stretched film to a condensed film with vertical orientation of hydrocarbon tails in the monolayer. The minimal area per molecule in the close-packed state (A_0, nm^2) was estimated by extrapolation of the linear part of the π–*A* isotherm for a condensed film of each compound to the zero surface pressure. We found that the quantities A_0 for films of behenic acid, diethyl octadecylmalonate, 2,4-heneicosanedione, and $(HD)_2Cu$ (per 2,4-heneicosanedione molecule) on the aqueous subphase are 0.20, 0.31, 0.27, and 0.33, respectively. That is, the presence of copper ions in the structure of $(HD)_2Cu$ leads to the least dense molecular packing in the Langmuir film.

Examination of film mophology by atomic-force microscopy and Auger electron spectroscopy. We showed previously by atomic-force microscopy [17] that horizontal deposition of monolayers of behenic acid, 2,4-heneicosanedione, and $(HD)_2Cu$ onto a hydrophilic silicon surface yields smooth and uniform monomolecular films. In so doing, the thickness of the condensed solid monomolecular 2,4-heneicosanedione film estimated by AFM analysis of the surface section profile was 2.7 nm [17], suggesting the vertical orientation of molecules in the monolayer. However, the height of an artificial rectangular defect formed by scanning of the film section by the microscope needle acting on the surface with a force of ~10 nN (Fig. 2a) was two times smaller. Apparently, the film subjected to the action of the AFM neelde remains on the surface but becomes denser, which suggests relatively strong interaction of polar molecular heads with the silicon surface.

When studying the surface morphology, we revealed no significant difference between the surface patterns for behenic acid, 2,4-heneicosanedione, and $(HD)_2Cu$. At the same time, the surface of the film of diethyl octadecylmalonate, prepared at a pressure of 30 mN m^{-1}, had a multilayered structure characteristic of collapsed monolayers (Fig. 2b). The difference between the content of elements in the intact part of the film and in the wear track was manifested in the Auger electron spectra of films of behenic acid, 2,4-heneicosanedione, and $(HD)_2Cu$ (Figs. 3a, 3b, respectively). According to the data obtained, in the course of friction the amount of carbon on the surface decreases, which confirms the removal of organic molecules from the friction zone. An increase in the oxygen content and appearance of iron signals are indicative of oxidation processes occurring on the silicon surface and of slight damage of the indentor.

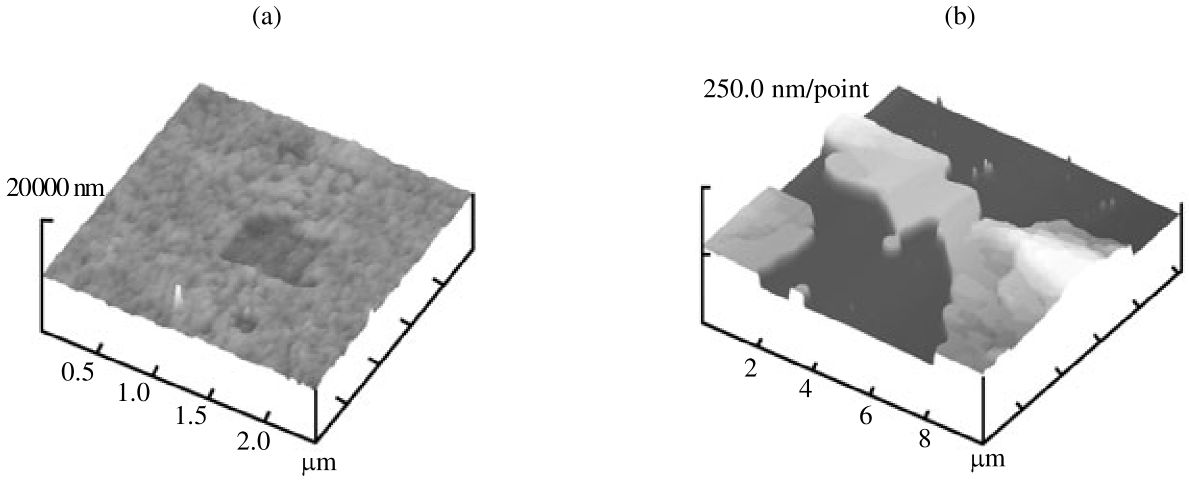

Fig. 2. AFM patterns of the surface of a monolayer of (a) 2,4-heneicosanedione and (b) diethyl octadecylmalonate. Monolayers were formed on the silicon surface by horizontal deposition at a surface pressure of 30 $mN\,m^{-1}$.

Tribological measurements. Monomolecular octadecyltrichlorosilane (OTS) film on silicon was chosen as a model system. The wetting angle (~104°) of the octadecyltrichlorosilane film suggests acceptable quality of the monomolecular octadecyltrichlorosilane coating. Indeed, the surface roughness estimated by AFM did not exceed 0.4 nm. The results of tribological studies showed that the Si/SiO_2 surface modified with a monolayer of octadecyltrichlorosilane had a low friction coefficient and was more resistant to mechanical actions than the surface of unmodified silica with the natural oxide layer (Fig. 4). A sharp increase in the friction forces, indicative of the coating failure, occurred after 200–400 sliding cycles at a normal load of 0.3 N. Thus, apparently, under the experimental conditions the silicon surface modified with octadecyltrichlorosilane is insufficiently resistant to friction. This may be due to incomplete binding of octadecyltrichlorosilane molecules with the surface, because of uncontrollable hydrolysis of octadecyltrichlorosilane molecules in the reaction mixture in the course of surface modification. Indeed, the presence of trace amounts of moisture in the solution can cause partial hydrolysis of the Si–Cl groups, which will lead to cross-linking of octadecyltrichlorosilane molecules with each other, and not only with the support. As a result, a nonuniform polysiloxane islet film can be formed from octadecyltrichlorosilane molecules, as in [18]. It should also be noted that a more stable monolayer of anhydrous solvent, which is more stable under the friction conditions at a relatively low load (0.3 N) [19], can be formed on the Si/SiO_2 surface. Nevertheless, in the latter case the octadecyltrichlorosilane monolayer also failed after 500 sliding cycles at a load of 1.8 N [20]. Application of a multimolecular Langmuir–Blodgett film onto an octadecyltrichlorosilane monolayer enhanced the wear resistance of silicon (Fig. 4). Apparently, the Langmuir–Blodgett

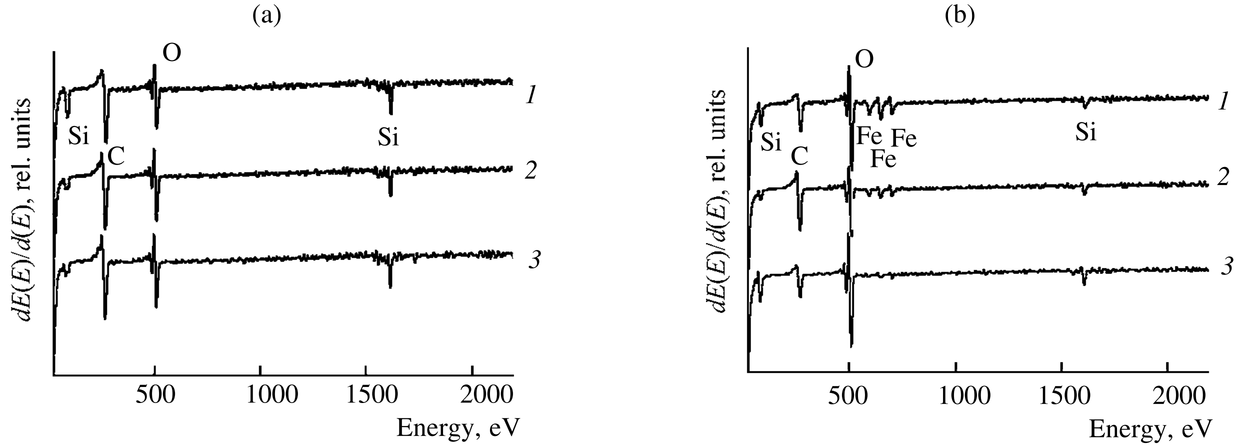

Fig. 3. Auger electron spectra of the surface of the (a) intact and (b) worn areas of films of (*1*) behenic acid, (*2*) 2,4-heneicosanedione, and (*3*) $(HD)_2Cu$.

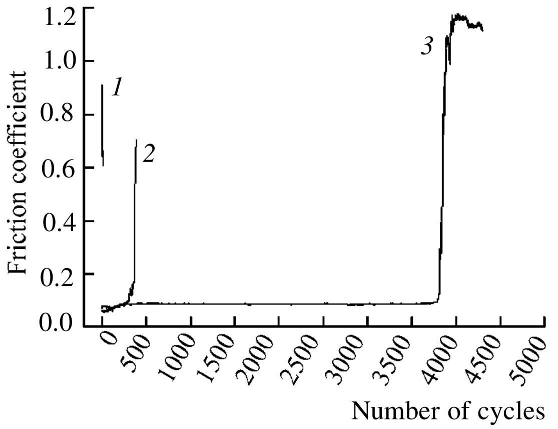

Fig. 4. Variation of the friction coefficient at a relatively low applied load (0.3 N) depending on the surface nature: (*1*) unmodified silicon surface with a natural oxide layer, Si/SiO_2; (*2*) Si/SiO_2/OTS surface; and (*3*) Si/SiO_2/OTS + five $(HD)_2Cu$ bilayers.

film acts as a lubricant and prevents the Si/SiO_2/OTS surface from failure.

We examined the wear resistance of the Si/SiO_2 surface modified with monolayer Langmuir–Blodgett films of behenic acid, 2,4-heneicosanedione, and $(HD)_2Cu$, and also with a Langmuir–Blodgett film of diethyl octadecylmalonate, having a multilayer structure according to AFM (Fig. 2b). Before isolation, all amphiphilic monolayers were compressed to a constant surface pressure of 30 mN m^{-1} to ensure similar “starting” conditions when revealing the effect of molecular packing on the film properties. Owing to high symmetry of the structure, behenic acid molecules occupy the smallest area (per molecule). It could be expected that the behenic acid molecules in the film would have the most compact packing compared to the other surfactants. Examination of the surface by Auger electron spectroscopy confirmed the expected initial chemical composition of the surfaces under consideration (Fig. 3b). The surface mainly consisted of carbon, whereas oxygen and silicon prevailed at a larger depth. We assign the oxygen signal from the near-surface layer to a layer of silicon dioxide (thickness approximately 1–2 nm [8]), because in the whole bulk of the amphiphilic monolayer (the thickness of behenic acid monolayer is approximately 2.7 nm [21]) the carboxy groups account for only 3% of the layer thickness. It should also be noted that the amount of carbon in the behenic acid film is somewhat higher than in films of the other compounds studied, which correlates with the higher molecular density of behenic acid film compared to the other compounds and coincides with the conclusions following from the π–A isotherms.

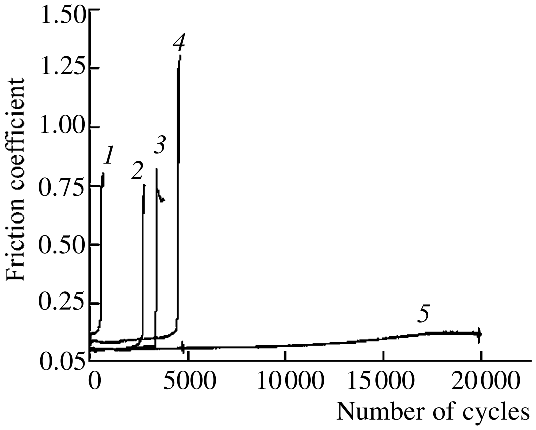

Fig. 5. Friction coefficient as a function of the number of sliding cycles. Film of (*1*) octadecyltrichlorosilane, (*2*) 2,4-heneicosanedione, (*3*) $(HD)_2Cu$, (*4*) behenic acid, and (*5*) diethyl octadecylmalonate; load 1.8 N.

At a normal load of 0.3 N, Langmuir–Blodgett films of behenic acid, 2,4-heneicosanedione, $(HD)_2Cu$, and diethyl octadecylmalonate, as well as the octadecyltrichlorosilane film, showed considerably better microtribological properties compared to the unmodified silicon surface. The Lamgmuir–Blodgett films of the compounds under consideration showed no surface failure and no appreciable increase in the friction after 20000 cycles, which was the maximal number of cycles in our experiments (7.5 h), whereas unmodified silicon failed virtually immediately after starting the test. The behenic acid film showed more stable behavior in the course of friction, taking into account that 2,4-heneicosane and $(HD)_2Cu$ films showed a slight increase in the friction coefficient to a value of 0.16, followed by its decrease to a value comparable with that for the behenic acid film. A considerable difference in the wear resistance between the Langmuir–Blodgett films under consideration and the octadecyltrichlorosilane film appeared at a higher load. At a normal load of 1.8 N, the octadecyltrichlorosilane film failed after 400–600 sliding cycles, whereas the $(HD)_2Cu$, 2,4-heneicosane, and behenic acid films were stable for 2700, 3300, and 4400 cycles, respectively (Fig. 5). Indeed, the silicon surface coated with a monomolecular Langmuir–Blodgett film appeared to be approximately 100 times more resistant to wear compared to the chemisorbed self-arranged monomolecular film of octadecyltrichlorosilane.

The film of diethyl octadecylmalonate did not fail under a load of 1.8 N even after 20000 sliding cycles (Fig. 5). It seems most probable that the extremely high stability of this film is caused by “self-healing” of defects arising in the course of friction, due to movement of molecules between the friction surfaces (lubrication effect), taking into account the multilayer structure of the film. Indeed, the friction coefficient was only slightly different for Langmuir–Blodgett films of diethyl octadecylmalonate of various thicknesses and was 0.026 for the monolayer and 0.20 for the multilayer. The stronger adhesion interaction of the monomolecular layer of diethyl octadecylmalonate with the surface, compared to the layers of the other compounds under consideration, may be due to structural features of this compound, whose molecules exhibit more pronounced orientation and dispersion interactions compared to behenic acid molecules.

Apparently, the tribological behavior of Langmuir–Blodgett films is affected both by the density of molecular packing and by the nature of polar groups in the molecules. In particular, Houston et al. [22, 23] reported that the friction coefficient of a molecular film is inversely proportional to the chain length, because of intermolecular interactions. The closest-packed monolayer shows the best friction characteristics because of concerted motion of molecules [24]. Analysis of the π–A isotherms (Fig. 1) and Auger electron spectra (Fig. 3) shows that the density of molecular packing decreases in the order behenic acid > 2,4-heneicosanedione > $(HD)_2Cu$. Therefore, it could be expected that behenic acid molecule would show the highest stability in friction (Fig. 5), especially at a higher contact pressure, compared to looser 2,4-heneicosanedione films. Strong interaction between the hydrocarbon tails of molecules in a close-packed behenic acid film apparently decelerates the desorption of molecules from the test surface. Probably, the close-packed monolayer of behenic acid under high loads is characterized by restricted motion of individual chains in the monolayer, i.e., behaves as a single whole. As a result, the monolayer resists the destroying effect of friction for a relatively long time (Fig. 5).

A slight decrease in the friction coefficient of Langmuir–Blodgett films in the initial step of the experiment (Fig. 5) may be due to reorientation of molecules, in particular, to a change in the angle of their inclination relative to the surface, and also to the movement of polar heads over the surface, caused by an external action. Furthermore, a part of molecules can reversibly move between the friction surfaces, changing the geometry of the experiment. This is impossible with chemisorbed films with a self-arranged monolayer, covalently cross-linked with the surface, which cannot move over the surface. Thus, only in the case of physically sorbed molecules (such as, e.g., behenic acid, 2,4-heneicosanedione, and diethyl octadecylmalonate) the defects can be "self-healed" owing to redistribution of the material between sliding surfaces and to redistribution of excess energy due to melting–solidification processes protecting the monolayer from burning in excess of the released heat.

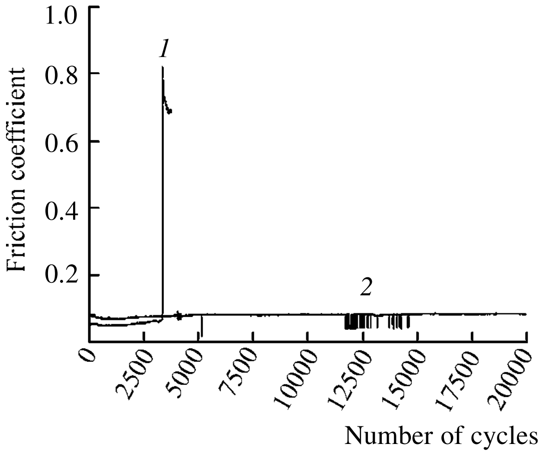

Fig. 6. Measurement of the tribological properties of friction contact between (*1*) 2,4-heneicosanedione monolayer on the Si/SiO_2 surface and a steel indentor, and (*2*) 2,4-heneicosanedione monolayer on the Si/SiO_2 surface and 2,4-heneicosanedione monolayer on a steel indentor.

To check the idea of possible movement of molecules between the friction surfaces, we additionally coated the steel indentor with a layer of 2,4-heneicosanedione molecules adsorbed on the metal surface from solution in chloroform. This system appeared to be extremely stable, because failure was not detected even at the highest possible load (1.8 N) after more than 20000 sliding cycles (Fig. 6). Apparently, molecules of this surface-active compound served as both a protective coating and a lubricant.

Thus, our results show that monolayers of physically sorbed long-chain molecules are sometimes more effective as protective coatings in friction than chemisorbed monolayers. It should also be emphasized that the nature of the support also strongly affects the stability of the monomolecular film. Indeed, in contrast to the silicon surface, the monolayer of behenic acid on mica was unstable, as in [21].

Thus, systematic tribological tests showed that the presence of a Langmuir–Blodgett film on the surface of monomolecular octadecyltrichlorosilane film enhanced the stability of the film in the course of friction. In the case of Langmuir–Blodgett films on the hydrophilic silicon surface, the first deposited monolayer was actually more resistant to the mechanical effect of the indentor, compared to the chemisorbed monolayer of octadecyltrichlorosilane molecules. The contact between the monomolecular films of the same nature on both friction surfaces considerably enhanced the wear resistance of the silicon surface. The results obtained show that monomolecular films on the silicon surface decrease the surface energy of the system and the friction coefficient and increase the lifetime of the friction surfaces owing to modification of the surface with a physically sorbed Langmuir–Blodgett monolayer as a result of "self-healing" of the structural defects in the course of friction.

In the micrometer range, the Langmuir–Blodgett films under consideration showed stably high resistance to friction and enhanced resistance to wear of the silicon surface compared to the already used organic monomolecular film of octadecyltrichlorosilane. The degree of molecular packing is critical for tribological tests of Langmuir–Blodgett films. Apparently, the movement of the monolayer molecules from the test surface to the surface of indentor can be responsible in part for the enhancing effect of the Langmuir–Blodgett films on the wear resistance of the surface. Better understanding of the friction processes and mechanism of the wear of Langmuir–Blodgett films would make it possible to control, to some extent, the chemical structure of molecules with the aim of their use as molecular lubricants in microsystems.

EXPERIMENTAL

Behenic acid $C_{21}H_{43}COOH$ and solvents (hexane, chloroform, carbon tetrachloride) from Aldrich were used without additional purification. Dimethyl octadecylmalonate $C_{18}H_{37}CH(COOCH_3)_2$ was prepared from dimethyl malonate and octadecyl bromide in the presence of sodium alcoholate [25]; 2,4-heneicosadione $C_{17}H_{35}C(O)CH_2C(O)CH_3$ was prepared by the procedure suggested in [26]. Copper heneicosanedionate $[(HD)_2Cu]$ $[C_{17}H_{35}C(O)CHC(O)CH_3]_2Cu$ was prepared according to [27] by extraction of copper ions from a copper chloride solution with 2,4-heneicosanedione molecules. Supports made of polished (100) silicon wafers were hydrophilized by heating in an $H_2O : NH_4OH : H_2O_2$ mixture (volume ratio 7 : 4 : 1) at 320 K for 15 min. After hydrophilization, the silicon wafers were repeatedly washed with double-distilled water and dried in a nitrogen flow. The isotherms (π–A, i.e., surface pressue–area per molecule) were recorded with a Langmuir bath interfaced with a computer [17] at a monolayer compression rate of 0.2–0.3 nm^2 $molecule^{-1}$ min^{-1} and subphase temperature of 290 K. The monolayers of the compounds under consideration were prepared from their solutions in chloroform (0.5 mM) on the surface of double-distilled water (pH 5.3–5.5) or of aqueous copper chloride solution (0.5 mM). The monomolecular films were deposited on a silicon support by the horizontal deposition method [28] at a surface pressure of 30 mN m^{-1} and lowering of the water surface level at a rate of 3–5 cm h^{-1}. The hydrophilic silicon wafers were hydrophobized by keeping them in a 5 mM solution of octadecyltrichlorosilane in hexadecane and carbon tetrachloride (5 : 1 by volume) for 15 min. Then the hydrophobic plates were successively washed with hexadecane, carbon tetrachloride, ethanol, and distilled water.

The elemental composition of the initial and spent monomolecular coatings was determined by Auger electron spectroscopy (PHI-670 spectroscope) in the impact emission field with an accelerating voltage of 5 kV and a current of 0.0211 μA. The working potential in etching with argon ions was 1 kV. At such working conditions, the etching rate was 0.5 nm min^{-1} on the SiO_2 surface (calibration surface).

The film morphology was examined by atomic force microscopy. The images were obtained with a Nanoscope IIIa device (Veeco, the United States) calibrated with a D scanner in the contact mode. We used contact 100- and 200-μm Nanoprobe cantilevers made of Si_3N_4 with elasticity constants of 0.12 and 0.36 N m^{-1}. The needle acted on the specimen in the contact mode with a force of several nanonewtons.

The film stability was studied with a reciprocating microtribometer [29]. As indentor we used a steel ball 3 mm in diameter with a polished surface (roughness less than 10 nm within an area of 2 × 2 μm). The indentor was cleaned by ultrasonic treatment in isopropanol and acetone (15 min in each solvent) and dried in a nitrogen flow. Then it was fixed in a holder and brought into forced oscillations relative to the flat sample at an applied load of 0.3 or 1.8 N. The sliding velocity was 4.42 mm s^{-1} at a path of 3 mm. To perform experiments with the modified indentor, some of steel balls were coated with a monolayer of 2,4-heneicosanedione by immersing the indentor in a 0.5 mM solution of 2,4-heneicosanedione in chloroform for 20 h.

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