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Effect of a Multilayer Structure and Lubrication on the Tribological Properties of Coatings of Fe–W Alloys

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Abstract—The methods for expanding the use of nanocrystalline electrolytic Fe–W alloys by creating a multilayer structure and using oil lubrication were studied. It was shown that lubricants can reduce the coefficient of friction and oxygen penetration into the sliding pairs, thus enhancing the wear resistance behaviour of Fe– W coatings as compared to that at dry friction, when the surface tribo-oxidation dominates. The electrodeposition of multilayer Fe–W/Cu coatings from a single bath was shown to be feasible. The tribological and mechanical properties of such coatings were investigated. The multilayer structure was found to improve the wear resistance characteristics of the coatings even at dry friction and at a relatively high normal load of 10 N.

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INTRODUCTION

The induced codeposition of metals of the iron group with refractory metals (W and Mo) [1–3] from citrate solutions can be used as an effective method for obtaining coatings that have improved corrosion and tribological characteristics [4–8]. Recently, such coatings have been regarded to be an alternative to electrolytic chromium coatings owning to the fact that the ecological conditions of their obtainment are safer as compared to those of electrolytic chromium from toxic electrolytes [9].

Such coatings are primarily amorphous (nanocrystalline) [3, 10–14], which is a peculiarity of Fe–W alloys. Their mechanical properties at deposition from various electrolytes in the range of current densities of 10–350 mA/cm^2 and deposition temperatures of 40–90°C [3, 7, 9, 10, 14] were studied previously. Maximum values of the microhardness were obtained for the coatings from a citrate-ammonia electrolyte deposited at 70°C.

Materials known as multilayer coatings have also been an object of intensive studies lately. Such materials, which consist of alternating nanoscale layers of various metals and alloys, have improved physicomechanical, optical, electric, magnetic, and magneto-optical properties as compared with the traditional alloys [15–20]. Multilayer coatings of micrometric and nanometric sizes [23–28] may be produced by electrodeposition.

This study is aimed at a comparative examination of the mechanical and tribological properties of coatings made of iron-tunglten alloys and chromium at their friction with a lubricant, and Fe–W/Cu coatings at dry friction.

EXPERIMENTAL

The Fe–W and Fe–W/Cu alloys were deposited from electrolytes A and B.

(A) The electrodeposition of Fe–W coatings was performed from an electrolyte of the following composition, (g/l): iron sulfate $FeSO_4 \cdot 7H_2O$)—55; sodium wolframate ($Na_2WO_4 \cdot 2H_2O$)—132; sodium citrate ($Na_3C_6H_5O_7$)—112; citric acid ($C_6H_8O_7$)—33. The pH of the obtained solution was adjust to 7.6–7.8 using ammonia. The current density was 1–5 A/dm^2.

(B) The electrodeposition of the Fe–W/Cu coatings was performed from a single bath (electrolyte A) with the addition of copper sulfate ($CuSO_4 \cdot 5H_2O$) with a concentration less (by weight) by 100 times than that of iron sulfate. The pH of the obtained solution was adjust to a value of 7. The deposition was carried out from a cell with unseparated anode and cathode spaces under galvanostatic conditions at 70°C. A stainless steel plate served as the anode. The thickness of the coatings varied in the range of ~8–15 μm depending on the current density of the deposition.

The Fe–W and Fe–W/Cu coatings were deposited onto substrates of two types. In order to study the mechanical and triblogical characteristics, the electrodeposition of the coatings was performed on mechanically polished St3 steel. For the polarization measurements, copper electrodes were used as the substrate. The substrate was degreased in an ultrasonic bath with acetone followed by degreasing in water with a soft detergent and then rinsed prior to the electrodeposition. After that, the substrate was applied to a nickel underlayer from a nickel-plating electrolyte that contained $NiCl_2 \cdot 6H_2O$ 240 g/l + HCl 80 g/l for 1 min.

Polarization curves were obtained from the B electrolyte in a three-electrode cell with an unseparated anode and cathode space on a Parstat 2273 potentiostat. A saturated silver-chloride electrode served as the reference electrode in relation to which the potential values were measured. The polarization curves were read at a potential sweep of 2 mV/s on a copper wire cathode. On the basis of the obtained polarization curves (Fig. 1), the optimal currents of the deposition for copper were also calculated for the Fe–W alloys from the limiting current of the diffusion. The polarization curve of the deposition was used to experimentally select a galvanostatic mode of the deposition. The electrodepositon of the multilayer coatings was performed in the pulse-galvanostatic mode (table). In all of the experiments, the current density in the electrodeposition of the alloys and copper was constant. After the period of the electrodeposition of the copper (t_{Cu}) and the alloy (t_{FeW}), there was a pause (t_{pause}) (Fig. 2, table).

The roughness of the surfaces of the deposited coatings was studied using (WYKO NT 3300) noncontact white light interferometry. The same methods were applied for the determination of the extent of wear volume after fretting tests on the coatings. The hardnesss was defined on a Nano-Hardness Tester, CSM.

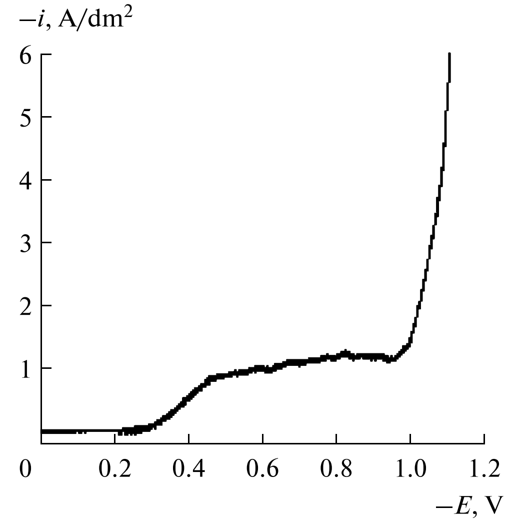

Fig. 1. Polarization curve of the electrodeposition of the Fe–W/Cu alloy that was obtained on a plate electrode from electrolyte B.

Testing of the Coatings at Dry Friction

The friction and wear of the electrolytic coatings that were deposited on the St3 steel were estimated based on the dry bi-directional ball-on-flat sliding tests (fretting mode 1). The electroplated multilayer coatings were tested under the following conditions: the normal force was 10 N, the amplitude of the displacement of the counterbody was 200 μm, the frequency of the reciprocal motion was 5 Hz, and the number of cycles was 50000. The coatings were exposed to friction at oscillation against a fixed counterbody, which was a 10-mm-diameter corundum ball with an elasticity modulus of 300 GPa. All of the tests were performed at an environmental temperature of 23 ± 2°C and 50% relative humidity. The coatings were prepared for the friction tests: they were degreased in acetone and ethanol and dried. After the testing, the coatings were cleansed in an ultrasonic bath with ethanol to remove wear debris prior to examining the wear profiles.

Testing the Coatings for Wear Resistance in the Presence of Oils

The tests were performed on an MVPD-1KPI laboratory facility according to the methods of [29]. The wear test conditions are: upper immobile cylindrical specimen (of 10.04 mm diameter with a 30-mm-long working part) coated with Fe–W on the flat surface of

Conditions of the electrodeposition and the roughness of the multilayer coatings

Deposition mode Fe–W/Cu	t_{Cu}, s	i_{Cu}, A/dm^2	t_{Fe-W}, s	t_{pause}, s	i_{Fe-W}, A/dm^2	Thickness of the layers, nm	R_a, μm
1	45		6.8	19.5		~10	0.98
2	180	0.1	34	10	2	~50	2.01
3	360		68	10		~100	0.09

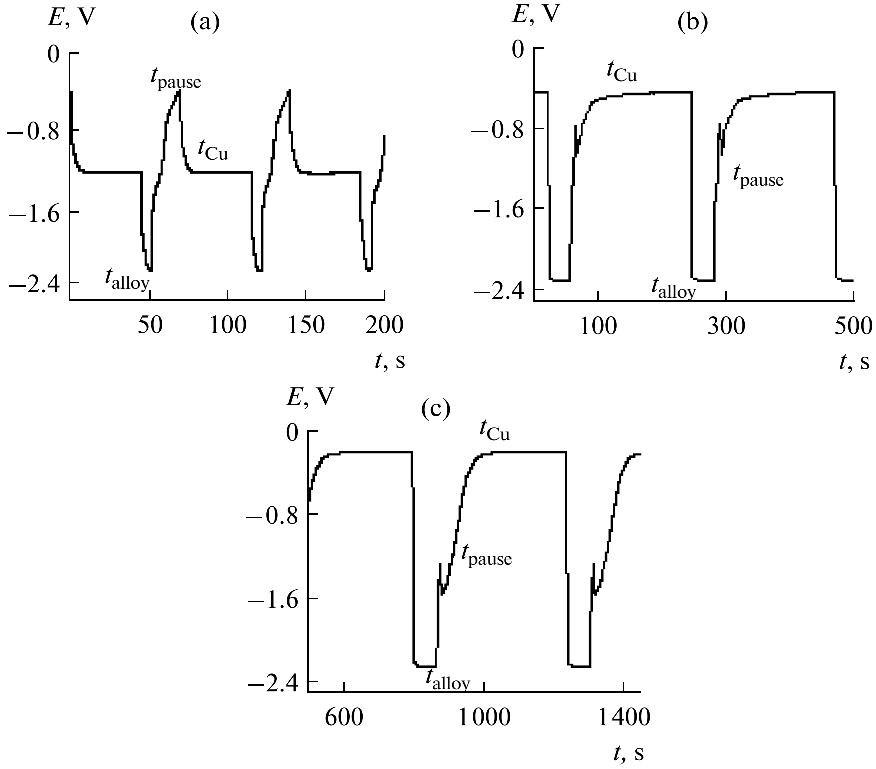

Fig. 2. Typical curves of the dependency of the potential on the time at the pulse-galvanostatic electrodeposition of Fe–W/Cu coatings in modes *1–3*. The potential is indicated without an ohmic component.

the lower specimen of quenched 45 steel (the HRC was 46–47, and the sizes were 110 × 50 mm), which was performed a reciprocal motion (at a frequency of 280 double strokes/min with a 100-mm peak-to-peak displacement amplitude) under a load of 300 N. The friction couples were lubricated with M-10 G_2K oil. The oil was supplied by a programed control mini-feeder, which ensured the accuracy of the oil expenditure and the synchronism of its being fed into the zone of the friction. The linear wear of the Fe–W specimens and the electrolytic chromium at the end of the testing was determined with regard to the average width of the wear area using a PMT-3 microscope.

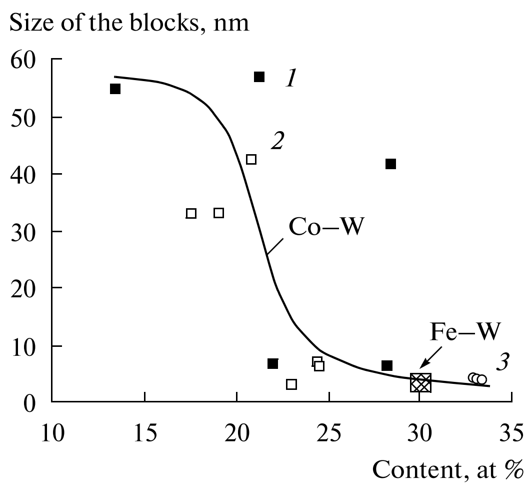

Fig. 3. Effect of the W concentration in the Co–W and Fe–W alloys on the size of the blocks (of a grain) that were electrodeposited at direct (*DC*) and pulse (*PC*) currents of the electrodeposition. (*1*) *DC*, pH 6.7; (*2*) *PC*, pH 6.7; (*3*) *DC*/*PC*, pH 8.

For comparison, the tribological characteristics of the Fe–W coatings that were deposited from the A electrolyte at a current density of 1 A/dm^2 and those from the chromium electrolyte (the standard electrolyte) at 55 A/dm^2 were determined.

RESULTS AND DISCUSSION

The Wear Study of the Coatings in the Presence of Oils

It was shown previously [30] that electrodeposited iron–tungsten alloys can be obtained in a nanocrystal state. Thus, at a tungsten concentration in the coating above 22–25% (at.), its grain size becomes less than 10 nm, which must lead to the change of the tribological and mechanical properties (Fig. 3 and 4 [31]). Indeed, such a small size of the grain enhances the hardness of the coatings, and its magnitude becomes comparable to (or even higher than) that of chromium coatings.

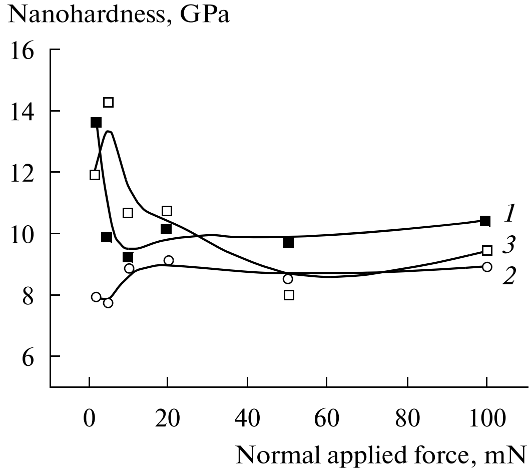

Fig. 4. Nanohardness that was measured at various loads of the indenter of the Fe–W and Co–W alloys and the Cr coatings; (*1*) Fe–W , (*2*) Co–W, and (*3*) Cr.

However, as the tribological tests of the iron-tungsten coatings showed, at the dry friction and a load of 2 N, such coatings undergo tribooxidation during the friction process (Fig. 5), which naturally interferes with their application. The Fe–W coatings undergo a high degree of wear (a darker color implies a larger depth in the image) resulting from the oxidation of the surface [30].

In order to expand the possibilities of using these alloys, we carried out investigations of the Fe–W – based alloys at their friction in the presence of oils, along with the variant of the multilayer coatings with copper.

One of the ways to decrease the wear is lubrication. The character of the behavior of the friction couples in the presence of oils greatly depends on the ability of the oil to penetrate into the area of the wear and remain there. The presence of oils substantially decreases oxygen's access to the area of contact in comparison with the degree of wear without lubrication, and this fact is found to be of special importance in the case of iron-based coatings [32].

In the context of the aforementioned, we studied the behavior of the Fe–W coatings in the presence of oils with a steel 45 counterbody. Indeed, the improvement of the tribological properties of the iron-tungsten coatings at friction in the presence of oils was noted. The friction coefficient decreases in the presence of oils to indicate the penetration of the lubricant into the contact area (Fig. 6). However, the friction coefficient that was measured is not so low as the one of the chromium coatings. This may be accounted for by the fact that the oxides that increase the friction coefficient [32] appear even in the presence of oils in the case of the Fe–W coatings, which affects the wear characteristics (Fig. 7).

It is seen that the depth of the wear of the Fe–W alloys deposited at various current densities is larger than that of the chromium coating (Fig. 7). Moreover, the Fe–W coatings deposited at higher current densities are characterized by a larger depth of wear, which is attributed to the somewhat higher tungsten content at the deposition at low current densities. Thus, even in the presence of oils at friction, the wear for the Fe–W coatings remains substantial, resulting, obviously, both from the choice of the oils for the given friction couple and the high load, which prevents reaching the required hydrodynamic mode at the given conditions of loading.

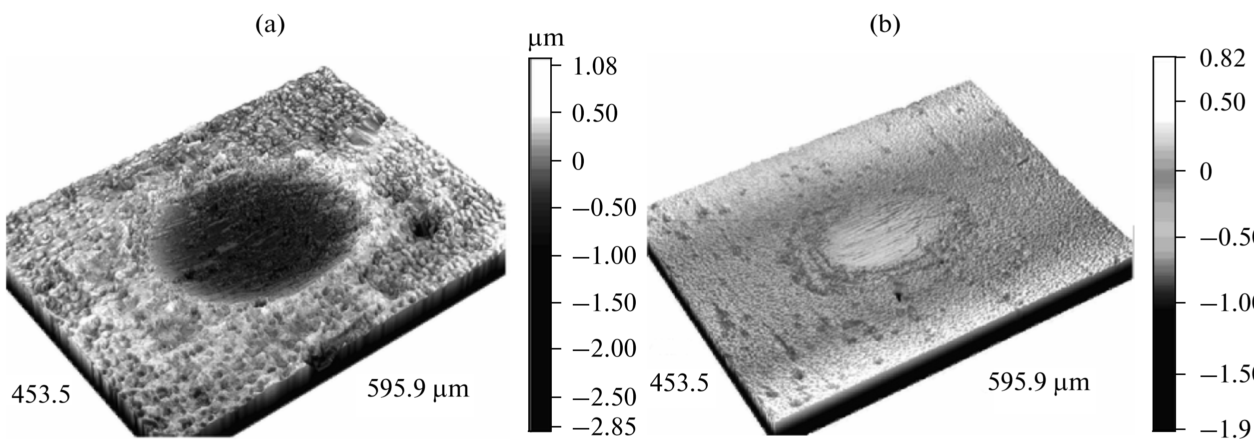

Fig. 5. The 3D images after the dry friction at 2 N and 10000 cycles of the electroplated coatings: (a) Fe–W (26 at % of W); (b) Co–W (24 at % of W).

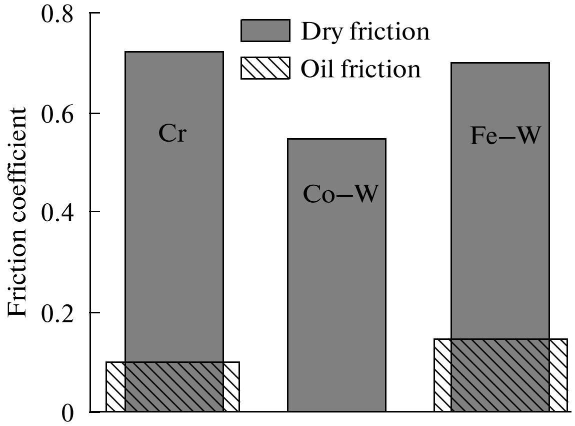

Fig. 6. Friction coefficient of the electroplated Fe–W, Co–W, and chromium coatings in the presence of oil.

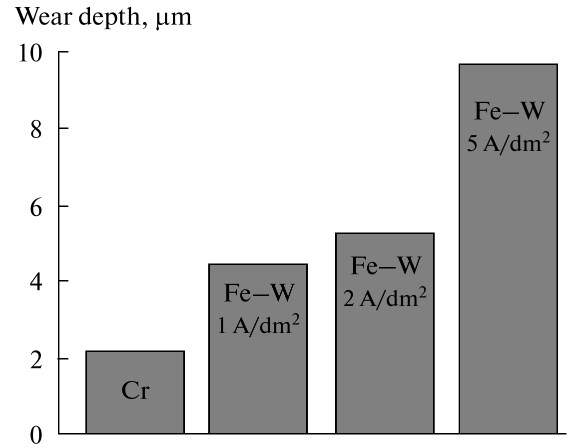

Fig. 7. Depth of wear of the electroplated Fe–W coatings at different current densities and coatings of electrolytic Cr. The friction tests were performed in the presence of oil and at a load of 300 N (after 21000 cycles).

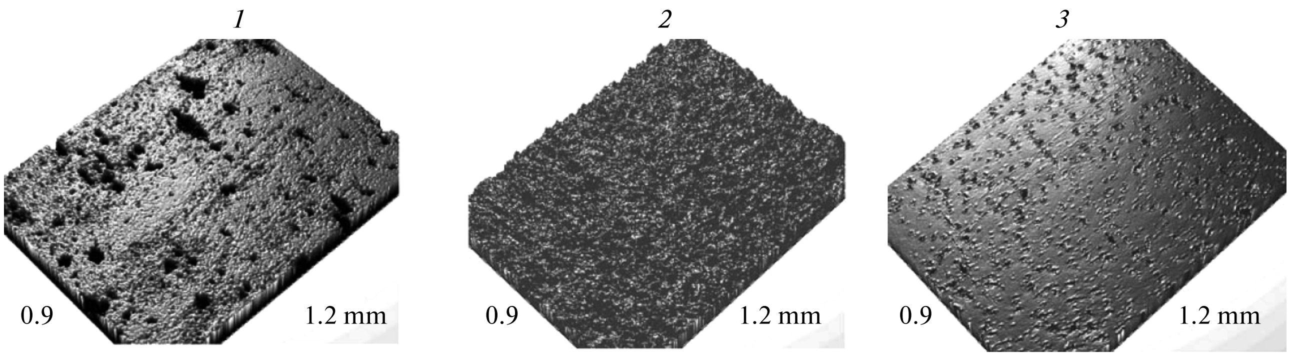

Fig. 8. The 3D images of the surfaces of the Fe–W/Cu multilayer coatings for modes 1–3 of the electrolysis and a layer's thickness in the multilayer coating, nm: 10 (*1*), 50 (*2*), and 100 (*3*).

Wear Study of the Fe–W/Cu Coatings at Dry Friction

The analysis of the tribological properties of the obtained multilayer Fe–W/Cu coatings was compared with electrodeposited Fe–W coating at 1 A/dm^2.

The study of the surfaces of the multilayer electrodeposited coatings showed that the roughness attains considerable values (Fig. 8 and the table), which is evidently specified by the nonuniformity of the obtained layers and the growth of the sizes of the grains at the deposition of the layers having a small thickness. In this case, high roughness is observed for individual layers with a thickness less than 100 nm. The finest roughness is observed at 100 nm, which is probably correlated with the more uniform deposition of the copper layers and the iron-tungsten alloy.

The sufficiently high roughness and nonuniformity of the coatings certainly must affect the tribological and mechanical properties. Thus, at the dry friction tests of the multilayer coatings at 10 N and 50000 cycles, the obtained wear track depend on the initial roughness of the coatings (Fig. 9). On the average, the wear depth at a given load was ~6 μm in 10-nm-thick layers, which is practically identical to the value of the wear depth for pure iron-tungsten coatings that were deposited at 1 A/dm^2. Note that, for the coatings having high roughness (as for instance in the case of a 50-nm-thick layer), one can hardly talk of the accuracy of the value of the wear depth (Fig. 9). Nevertheless, the layers with a thickness of less than 50 nm are found to not show any special improvement of the wear characteristics, which are correlated with the tribooxidation of the iron-tungsten coatings at dry friction.

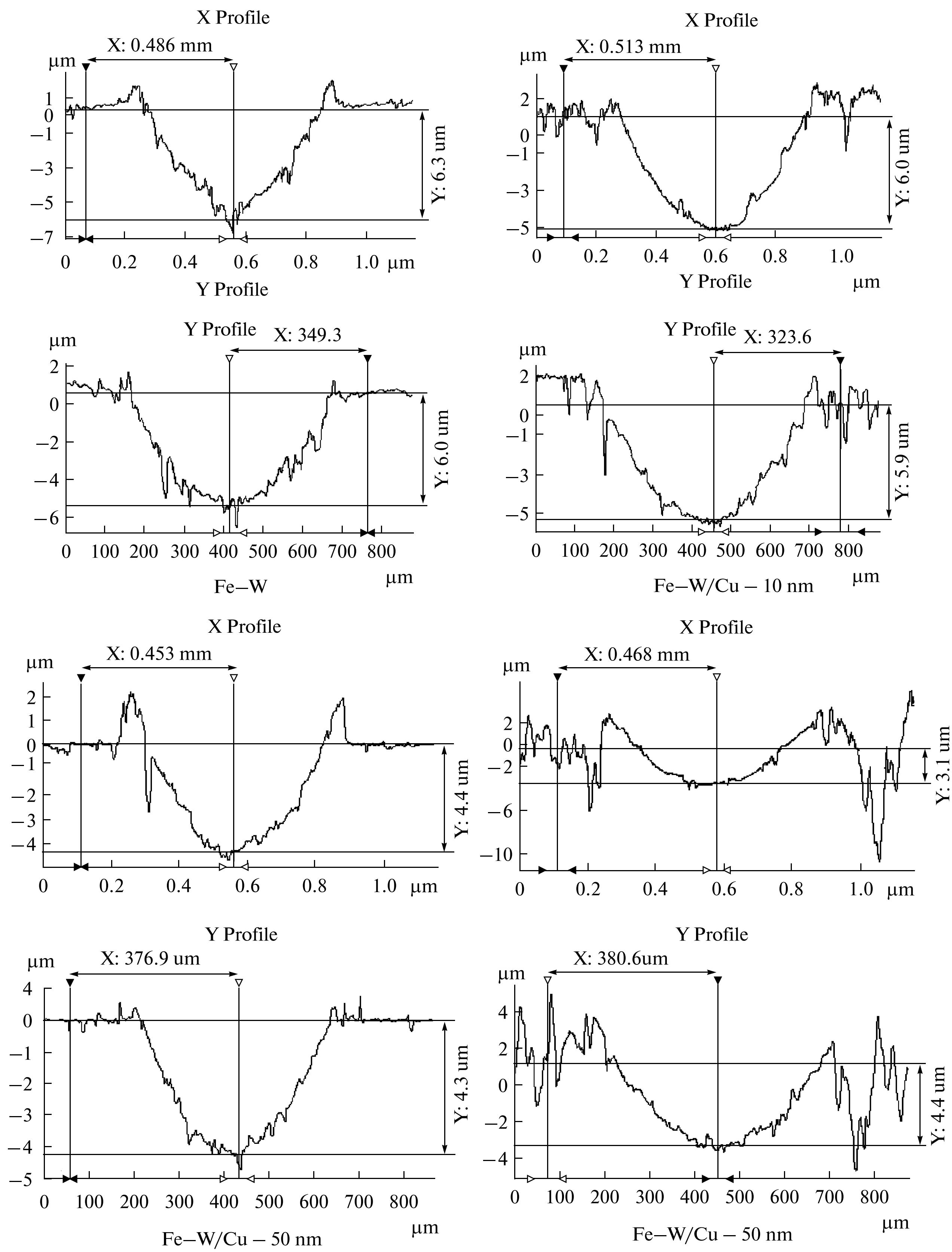

Fig. 9. Profiles of the multilayer coatings after the wear as measured in various directions for deposition modes 1–3 (table) and the Fe–W coating.

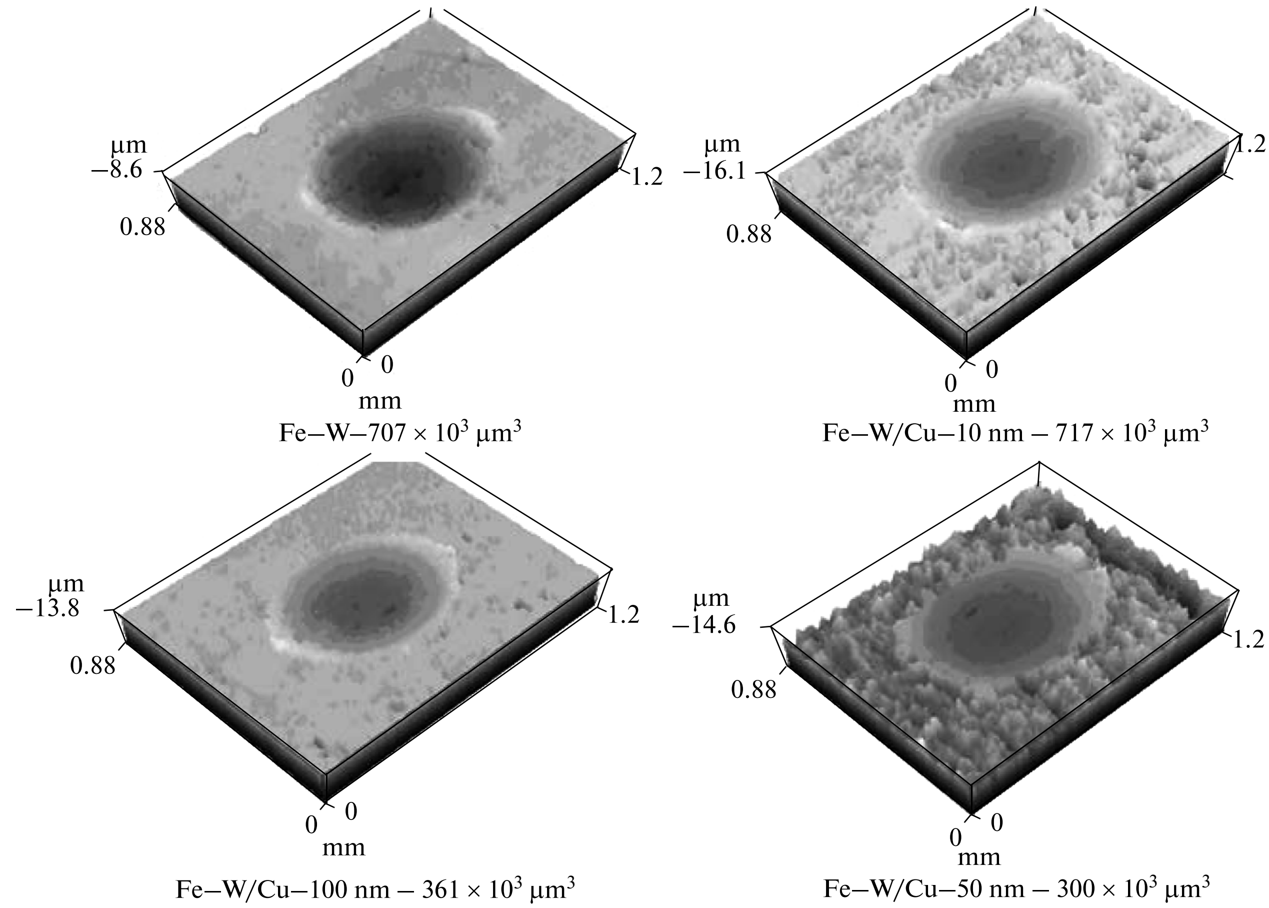

Fig. 10. The 3D images of the surfaces of the multilayer coatings and pure Fe–W after the tests for wear.

Thus, the volume of wear for the multilayer coatings with 10-nm-thick individual layers equals the wear for pure iron-tungsten coatings (Fig. 10). A decrease of the volume of the wear (Fig. 10) occurs at an increase of the thickness of the ingle layers with the minimum wear for 50-nm-thick layers.

The change of the friction coefficient of the multilayer coatings obtained at 10 N and an appropriate number of cycles is shown in Fig. 11. As is seen, for the specimen that was deposited with ~50-nm-thick layers, a certain decrease in the friction coefficient is observed as compared to the other coatings. This fact correlates with the lower value of the wear obtained for these coatings.

Hardness Measurement

The study of the mechanical properties, in particular, the hardness of the deposited multilayer coatings, was carried out using the multicycle method (MCM) on a Nano Indentation Tester. This method makes it possible to estimate the effect of various loads of the indenter (at different depths of penetration) on the hardness of the coatings. In this case, the hardness variation of a coating may be estimated in one area of the coating according to the thickness of the electrodeposit, instead of a few areas when using monoindentation. Figure 12 shows the results of the MCM study of the nanohardness of the multilayer coatings that were deposited in 1–3 modes of electrolysis (table). Copper is found to abruptly decrease the hardness of the multilayer coatings, particularly of those with 10-nm-thickness. This relates to the hardness obtained for the 50-nm-thick layers to a lesser degree. The values of the hardness of these coatings are practically identical to the hardness of the Fe–W coatings (Fig. 4). Note that high roughness makes it difficult to estimate the hardness. The data in Fig. 12 are presented for a more uniform area (Fig. 13a): the hardness estimation is found to be impossible over more rough areas (Fig. 13b).

CONCLUSIONS

The results of this study show the principle possibility for expanding the use of nanocrystalline Fe–W electroplating alloys. The friction coefficient and the penetration of oxygen to the friction surface are found to decreased at friction in the presence of lubricants. This makes it possible to improve the wear character-

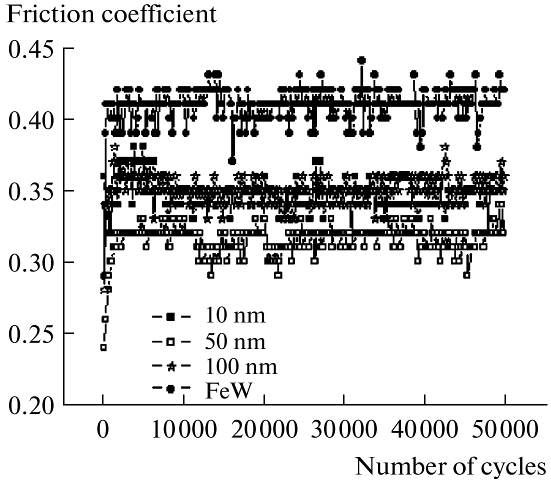

Fig. 11. Dependence of the friction coefficient on the number of cycles at dry friction using the method of "a ball along a plane" at 10 N for the Fe–W/Cu multilayer coatings and the Fe–W coatings.

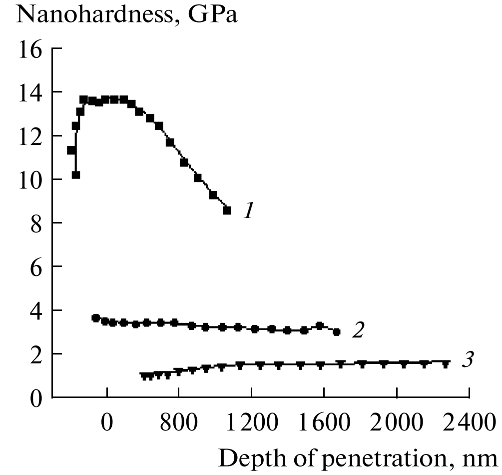

Fig. 12. Hardness measurements using a multicycle method (MCM) for modes *1–3* of the deposition of coatings (table) with the thickness of the layer, nm, being as follows: (*1*) 50; (*2*) 100; (*3*) 10. See the text for other details.

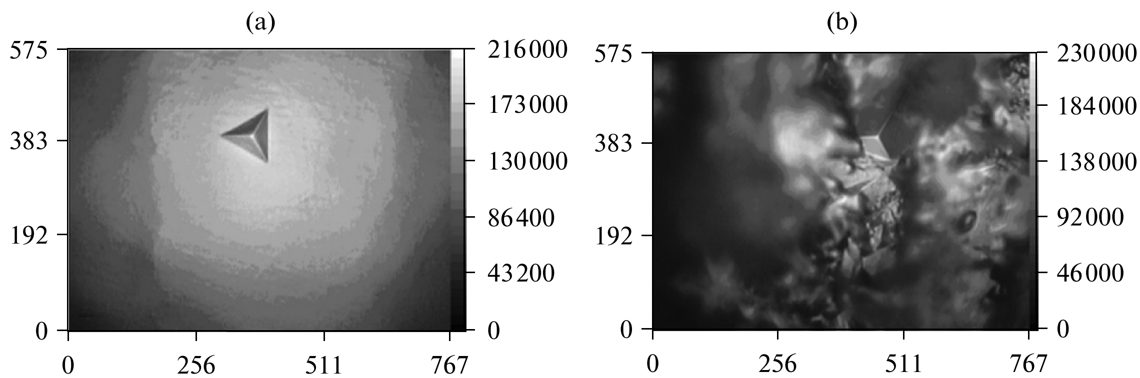

Fig. 13. Microstructure of the prints and adjacent zones after the indentation of the multilayer coatings using the MCM: (a, b) electrolysis mode 2 (prints in various areas of the coating).

istics of the Fe–W coatings versus the dry friction, when the surface tribooxidation is predominat.

The study of the tribological and mechanical properties of the multilayer Fe–W/Cu coatings by means of electrodeposition from a single bath leads to the improvement of the wear characteristics of the coatings even at dry friction and at a sufficiently high load of 10 N. The hardness of these coatings can be high in the case of the ~50-nm-thick individual layers, nevertheless of the copper presence.

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