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Research Article

Corrosion and Mechanical Properties of the Fe-W-Wo_2 and Fe-Mo-MoO_2 Nanocomposites

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Analyzing of composition electrolytic coatings' application for the metal surface protection is considered. It is established that using different components for coatings' modification gives possibility to obtain surfaces with expanding exploitation properties, in particular, with improved wearing and anticorrosion resistance. The new approach for protecting details which are made from cast irons by obtaining two kinds of composition coatings from binary alloys iron-molybdenum and iron-tungsten is proposed. It is found that the modification of iron by refractory metals up to 37 wt. % leads to a noticeable change in the microstructure of the coatings' surface. It is established that the incorporation of refractory metals into the iron matrix is a good way to increase the microhardness of the surface by 2.5–3.5 times and rising of the wear resistance by 40%, as well as decreasing the friction coefficient by 3-4 times in comparison with the cast iron substrate. The research results can be used for surfaces hardening and protection in different industries.

1. Introduction

Treatment technologies with the application of thin-film coatings, in particular electrochemical, are quite common in the industry of high-tech countries [1–3]. Recently, considerable attention in the development of renovation, hardening, and protection of surfaces of parts has been paid to the processes of formation of electrolytic coatings based on iron triad metals alloyed with additional components [4]. The combination of valuable properties of alloying components makes it possible to obtain coatings with enhanced corrosion resistance, microhardness, and wear resistance on cast iron parts [5–7].

To deposit the Fe-W alloy with tungsten content from 30 to 60% of the citrate-ammonia electrolyte with the pH 8 was proposed [8]. The electrodeposition was carried out at the cathode current density of $5\,A/dm^2$ and the electrolyte temperature of 70°C. The current efficiency (CE) was in the interval from 20 to 28%. In the study by Porto et al. [9], Fe-W alloys with tungsten concentration of 39–69.6 wt. % were obtained at temperature of 60°C and direct current density of $50\,mA/cm^2$ from ammonia-citrate electrolytes based on Fe (III). It was shown in [10] ammonia-citrate baths to possess the required buffer capacity for the electrodeposition of nanocrystalline and smooth Co-W and Fe-W. A qualitative study of the mechanical behavior of electrodeposited Co-W and Fe-W alloys was performed by different methods.

Authors of the study [11] have established that iron and molybdenum are included in the metallic form into the Fe-Mo deposits obtained from ammonia-citrate. Smoother and crack-free Fe-Mo coatings with molybdenum content of 54 wt. % were electrodeposited from a highly saturated ammonium acetate bath at current density of $30\,mA/cm^2$ [10]. It was considered that the coatings to be catalysts for the hydrogen evolution reaction. The composition and morphology of Fe-Mo alloys electrodeposited from a pyrophosphate bath using the AC mode were studied in [11]. However, in these works, no attention was paid to the study of Fe-Mo electrolytic alloys' corrosion and mechanical properties. The step mechanism of Fe-Mo alloys' electrodeposition from an alkaline solution containing Fe (III) ions and sorbitol was established [12]. The incorporation of molybdenum (IV) oxide into the coatings was shown.

In the study by Grgur et al. [13], commercial electrolytic baths (Ni, Cr, Cu, and Zn) were observed to obtain the composite coatings based on the metallic matrix incorporated with the dispersed phase—an insoluble solid with nanometric particle size. Some theoretical models of the electrodeposition of composite coatings considering the adsorption and electrophoretic migration of particles before the incorporation were given in [14]. The composite Fe/ZrO_2 coatings with increased microhardness were obtained from the iron electroplating baths containing the particles of zirconia stabilized by 3 mol% yttria [15]. Nanocrystalline Fe-W alloy and Fe-W/Al_2O_3 composite coatings with submicrosized alumina particles have been obtained by electrodeposition from Fe(III)-based electrolyte with the aim to produce a novel corrosion and wear resistant material [16]. It was found that the wear rate decreases by a factor of 10 as compared to Fe-W in presence of 12 vol% of Al_2O_3 in deposits, but alumina particles slightly increase the corrosion resistance of the coatings. All above investigations were made in suspension electrolytes which need additional procedure for stabilization, and thus, the service life of such electrolytes is limited.

The goal of this work is to obtain Fe-W-WO_2 and Fe-Mo-MoO_2 nanocomposite coatings from stable electrolytes and to study their mechanical properties and corrosion behavior.

2. Materials and Experimental Methods

Electrochemical coatings by nanocomposites were applied on the substrate made of gray cast iron (GCI) widely used for manufacturing piston rings and other machine parts. Coatings with an alloy of iron-molybdenum or iron-tungsten were deposited at a temperature of 18–40°C from a complex electrolyte of the composition, mol/dm^3: iron (III) sulfate—0.1–0.15; sodium molybdate—0.06–0.08 (sodium tungstate—0.04–0.06); sodium citrate—0.2–0.3; boric acid—0.1; the pH was in the range of 3.0–4.5 [17, 18].

The coatings were formed in two modes: by direct current (dc) with varying current density i in the range of 2.5–6.5 A/dm^2, and by unipolar pulse current (pc) with an amplitude of 2.5–8.5 A/dm^2 with a pulse duration t_{on} of 5 ms and pause time t_{off} of 20 ms [19, 20]. The dc polarization was carried out with a stabilized dc source of the B5-49 series. Pulse electrolysis and determination of the corrosion behavior of the coatings were carried out using a PI-50-1.1 potentiostat with a PR-8 programmer.

The chemical composition of the coating surface was studied on an INCA Energy 350 energy dispersive spectrometer. The coating composition (wt.%) is calculated in terms of the metal components of the alloy, and the content of nonmetallic adsorbed impurities (oxygen) was taken into account when assessing the surface topography. The surface morphology of the coatings was studied using a ZEISS EVO 40XVP microscope.

The corrosion rate for coated samples was defined using the method of polarization resistance [21] in the environment of different composition: 0.001 M NaOH (pH 10) against the background of 1 M Na_2SO_4; 0.001 M H_2SO_4 (pH 3) against the background of 1 M Na_2SO_4; 3% NaCl solution (pH 7). To define the corrosion resistance, the polarization measurements were taken in the potentiostatic mode at the potential scanning rate within 2 mv/s. In the article, the values of the corrosion potential are indicated relative to a standard hydrogen electrode (SHE). The corrosion current density i_{cor} and the corrosion potential E_{cor} were defined using the graphic method at the point of intersection of the linear sections of the anode and cathode polarization dependences in the semilogarithmic coordinates of $\lg i - \Delta E$. The depth index of corrosion k_h was calculated based on i_{cor} as in [22].

The adhesion quality of coatings with the substrate material was studied by polishing using pastes based on chromium oxide, bending at an angle of 90°, and heating to a temperature of 150–200°C, followed by cooling in air. The microhardness of the coatings was determined on a PMT-3 microhardness meter with a load of 50–100 g. The investigations were carried out after the coatings were endured during 24 hours following the application. The thickness of coatings taken for the studies ranged from 25–30 μm.

The tribo-technical properties of coatings on gray cast iron were evaluated by the friction coefficient f_{fr}. In addition, wear resistance was also determined during tests on a 2070 SMT-1 serial friction machine with gradual loading of conjugated samples from 0.2 kN to 0.8 kN according to the "piston ring-cylinder liner" scheme and a reciprocating friction machine.

3. Results and Discussion

3.1. Composition and Surface Morphology of Composites. An influence of the electrolysis mode on the character of the alloy surface can visually be traced for Fe-Mo(W) coatings deposited onto the substrates made of gray cast iron. At the dc mode (Figures 1(a) and 1(c)), a very inhomogeneous surface is formed with a high quantity of the burrs of an irregular shape. The content of molybdenum or tungsten is higher on coating hills in comparison with the entire surface. And, the concentration of oxygen is in the range of 18–21 wt. %; this allows us to consider coatings as composite Fe-Mo(W)-MO_x where M is Mo or W, respectively. The formation of incompletely reduced oxides of refractory metals in situ in the cathode process follows from the mechanisms of electrodeposition of alloys proposed in [23]. In addition, their incorporation into the Fe-Mo(W) matrix is described in [12].

The use of pulse electrolysis (Figure 1(b)) contributes to the leveling of surface relief; the number of burrs is considerably decreased, and the coating becomes a little bit brighter. The coatings contain 5 wt. % more refractory metal and less oxygen at the level of 10–12 wt. %. It should be noted

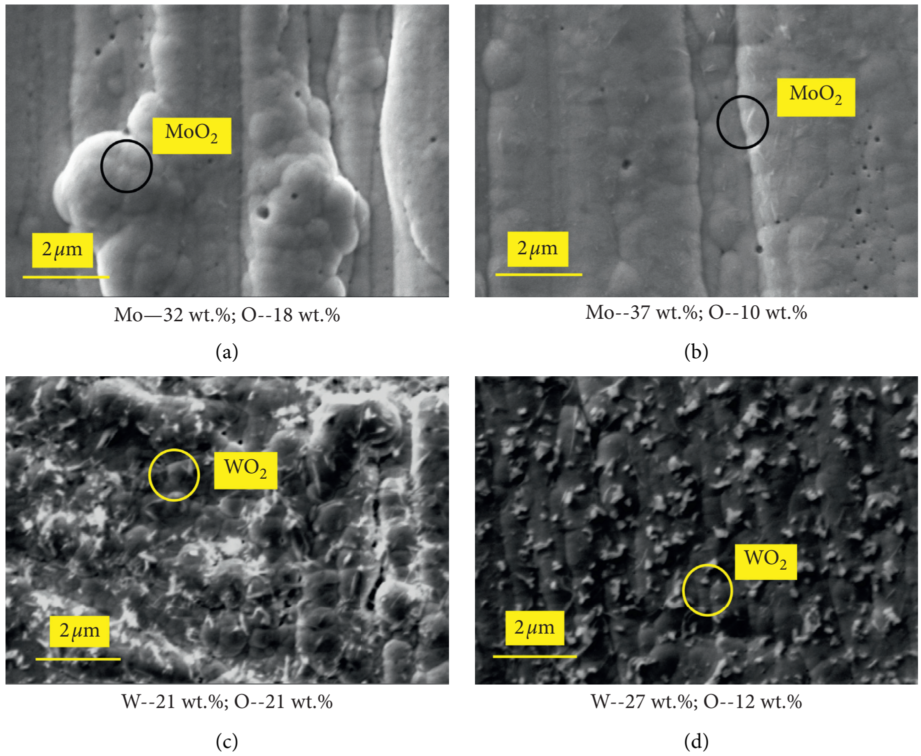

FIGURE 1: The surface morphology of the Fe-Mo (a, b) and Fe-W (c, d) coatings deposited on the cast iron at dc (i) = 3.5 A/dm^2 (a, c); at pc (i) = 5.5 A/dm^2; $(t)_{on}/(t)_{off}$ = 5/20 ms (b, d).

that the surface of molybdenum-containing composites is globular spheroids up to 100 nm alternate with islands of agglomerates of larger size up to 1-2 μm. Tungsten-containing composites are finely crystalline, with crystallite sizes being smaller compared to Fe-Mo based coatings. Considering the obtained data, we can draw a conclusion that more uniform and lower porosity Fe-Mo(W) based coatings with higher molybdenum (tungsten) content deposited by the pc mode will have more perfect functional properties.

3.2. Corrosion Resistance. The studies [23, 24] showed that the corrosion of iron-based coatings with refractory metals proceeds mainly with hydrogen depolarization in the acidic solution due to the presence of iron, while in neutral and alkaline environments, it proceeds with the oxygen depolarization.

As was established, the pc deposition mode enables the formation of composite coatings Fe-W-WO$_2$ and Fe-Mo-MoO$_2$ with a more perfect surface morphology and a higher content of alloying components that contributes to an increase in the corrosion resistance of coatings obtained through the pulse electrolysis. These assumptions are totally confirmed by the corrosion indices of the substrate material and those of Fe-Mo(W) based composites formed on cast iron (Table 1).

It should be noted that Fe-Mo alloy coatings have a high corrosion resistance in the corrosive environment of a different acidity independently of deposition conditions as compared with Fe-W. Of particular note is the increase in the corrosion resistance of these alloy composites in acidic environments and in the presence of Cl$^-$, which is ensured by the presence in coatings of the alloying component (Mo), which increases both the resistance to pitting corrosion and the tendency to passivation.

3.3. Microhardness and Tribotechnical Properties of Coatings. The use of citrate Fe (III)-based electrolytes and the electrolysis conducted at recommended pH and current density enable the formation of uniform coatings that have a high adhesion to the substrate material with a minimum content of unwanted admixtures that can degrade the hardness indices of electrolytic alloys. The microhardness of Fe-Mo-MoO$_2$ composite coatings deposited by the dc mode is 4 times higher in comparison to that of the substrate material (gray iron cast) (Figure 2(a)). Such phenomena may be explained by the coatings' composition and the specific features of morphology formed during the electrodeposition. The presence of refractory components in alloys contributes to the amorphization of the coating surface, which leads to an increase in microhardness. In addition, the incorporation of molybdenum and tungsten oxides into a metal matrix during electro crystallization also has a positive effect on microhardness indicators. It is obvious that the electrolysis mode has an effect on the microhardness of coatings.

Hence, the iron and molybdenum alloy composites that were applied in the pulse electrolysis mode are characterized by higher microhardness indices (Figure 2(a)) due to a

Table 1: The corrosion indices of the cast iron, and Fe-W-WO_2 and Fe-Mo-MoO_2 coatings deposited by different modes.

pH of test medium	Cast iron		dc mode				pc mode			
			Fe-Mo-MoO_2		Fe-W-WO_2		Fe-Mo-MoO_2		Fe-W-WO_2	
	E_{cor} (V)	K_h (mm/year)	E_{cor} (V)	K_h (mm/year)	E_{cor} (V)	K_h (mm/year)	E_{cor} (V)	K_h (mm/year)	E_{cor} (V)	K_h (mm/year)
3	−0.34	1.980	−0.30	0.038	−0.22	0.045	−0.31	0.030	−0.24	0.040
7	−0.55	1.150	−0.47	0.040	−0.33	0.042	−0.49	0.035	−0.35	0.039
10	−0.35	0.300	−0.58	0.028	−0.03	0.032	−0.60	0.021	−0.037	0.028

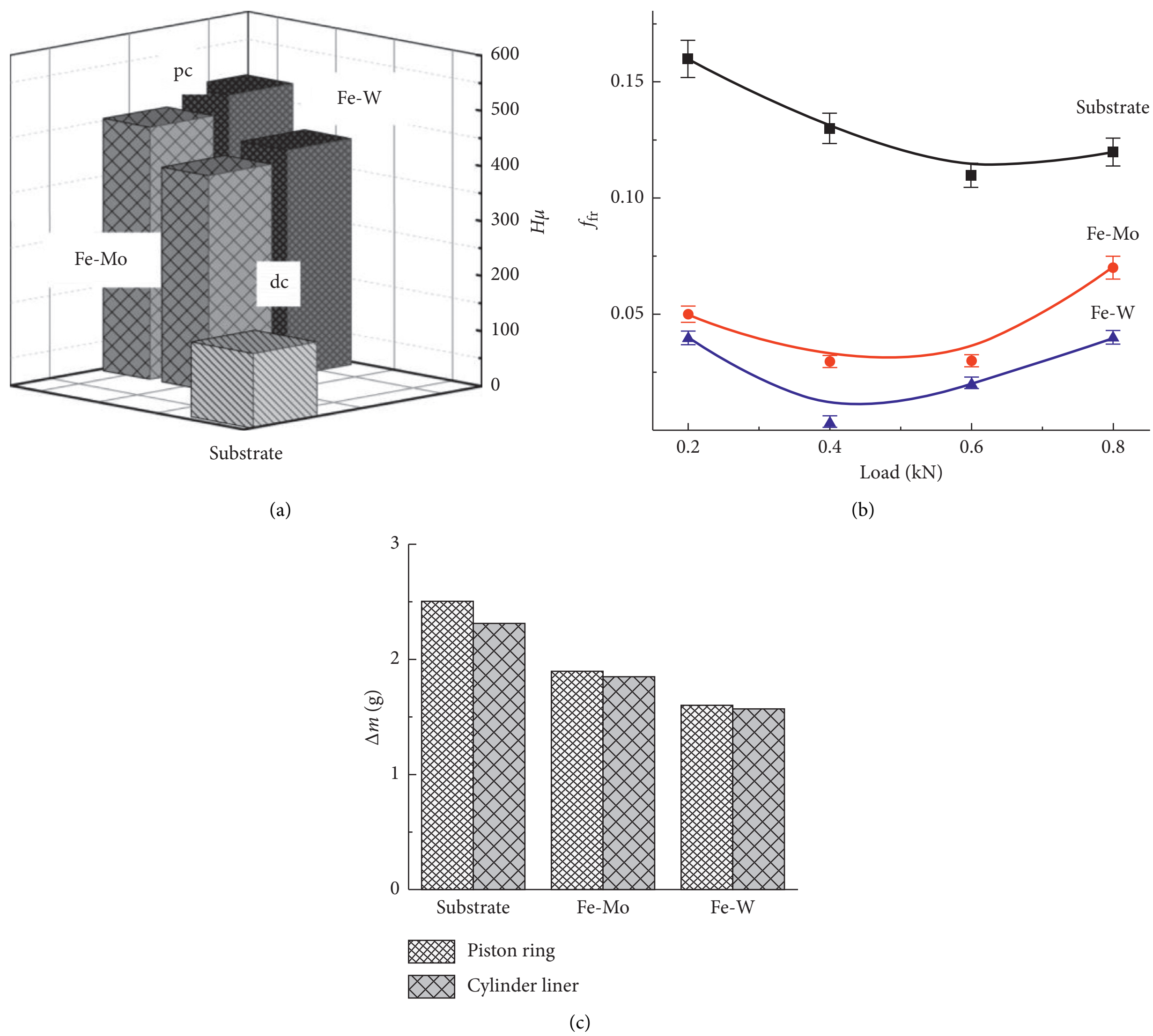

Figure 2: Mechanical characteristics of Fe-Mo(W) based composites: (a) microhardness; (b) friction coefficient; (c) wear resistance.

higher content of alloying components, more perfect surface relief, and a lower number of adsorbed admixtures as was established for similar coatings [20, 25–29].

At the same time, Fe-W-WO_2 composites are characterized by a higher microhardness in comparison with molybdenum-containing ones due to the presence of tungsten as well as a higher content of oxide hardening phase in the coatings, as evidenced by the higher oxygen content in the tungsten-containing thin films [30, 31]. The results obtained for the microhardness indicators of coatings containing tungsten and molybdenum correlate with the data obtained for similar coatings by other researchers [32], which confirms the correctness of the studies performed.

The studies of the dependence of the friction coefficient and the disposition of appropriate materials to scuffing at the stepwise loading showed that the f_{fr} value of studied coatings is 3.0 to 4.0 times lower in comparison to that of cast iron (Figure 2(b)) and it is indicative of the high antifriction properties of formed coatings. In addition, the specific features of morphology cathode deposits and their porosity are the additional factors of the improvement of antifriction properties due to the additional confinement of lubricant materials in the cavities and pores of the coatings.

It is established that the electrolytic composite coatings Fe-Mo(W)-MO_2 obtained in the proposed electrolysis modes have high adhesion to the substrate and retain it during mechanical (bending, polishing, and cross section with subsequent grinding) and temperature influences (heating up to 150–200°C).

The wear resistance of samples with coatings was evaluated by the change in mass Δm on the testing samples of a conjugate friction pair “piston ring-cylinder liner” coupling (Figure 2(c)). A decrease in the mass of samples indicates the wear of the surface of the parts and can serve as a quantitative characteristic of the wear resistance of the material. Investigations on a serial machine 2070 SMT-1 with a step load of conjugated samples from 0.2 kN to 0.8 kN according to the “piston ring-cylinder liner” scheme showed that the wear resistance of the surface of cast iron with Fe-Mo(W)-Mo(W)O_2 coatings exceeds 1.7-1.8 times the wear resistance of cast iron samples (Figure 2(c)). The research results confirm that the wear resistance of tungsten-containing composites, like other mechanical properties, is higher than that of composites based on molybdenum (Figure 2).

Hence, a set of the corrosive characteristics and the physical and mechanical properties of galvanic composite coatings based on the Fe-Mo(W) alloys allows us to view them as the promising materials for the technologies used for the hardening of the parts made of cast iron and low-carbon steel, and these can also be used by the maintenance services to repair and upgrade worn out surfaces that are operated in the environment of a different corrosiveness.

4. Conclusions

The composite coatings Fe-W-WO_2 and Fe-Mo-MoO_2 were deposited on the cast iron substrate by different electrolysis modes from citrate Fe(III)-based bath. The hardening phase of refractory metal oxides is formed directly in the cathode process and is included in the alloy matrix, which helps to increase the uniformity of the distribution of components over the thickness of the coating and its surface.

The use of pulse current electrolysis allows obtaining more uniform coatings enriched with alloying components with a smaller amount of adsorbed nonmetallic impurities.

Composite Fe-Mo-MoO_2 has a higher corrosion resistance due to the chemical stability of molybdenum and its oxides in environments of various aggressiveness including chloride-containing solutions.

The physic-mechanical properties such as microhardness, friction coefficient, and wear resistance of Fe-W-WO_2 composites surpass not only the base material but also the molybdenum-containing coatings.

Combination of higher strength characteristics and increased corrosion resistance of composite coatings Fe-W-WO_2 and Fe-Mo-MoO_2 in comparison with cast iron allows us to consider them as promising materials in the technology of surface hardening and restoration of worn surfaces of parts.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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