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PROPERTIES OF COATINGS PRODUCED BY HVOF SPRAYING OF COMPOSITE POWDERS BASED ON AMORPHIZING FeMoNiG13 ALLOY

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ABSTRACT

Applying the method of HVOF spraying with the use of mechanically-alloyed powders based on the amorphizing FeMoNiCrB alloy with the additions of (Ti, Cr)C and $FeTiO_3$ compounds, the coatings with amorphous crystalline heterophase structure were produced. The coatings of FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ systems have a dense fine-grained structure with a porosity of 2.4 and 1.2 % and a hardness of 5510 ± 250 and 4410 ± 190 MPa, respectively. The study of corrosion resistance and resistance to fretting corrosion of the developed coatings was conducted. It is shown that the use of composite powders based on FeMoNiCrB alloy with the addition of (Ti, Cr)C and $FeTiO_3$ compounds as spraying materials allows increasing the protective properties of the coatings compared to the coating of FeMoNiCrB alloy. It was found, that in the case of producing composite FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ coatings on the steel base St3, the corrosion resistance of the specimens in the solutions of 3 % NaCl, 10 % H_2SO_4 and 10 % KOH is increased by 7.3, 9; 3.9, 5.3 and 9.5, 9.7 times, respectively. FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ coatings have a fairly high resistance to fretting corrosion, which is 4.6 and 5.8 times higher than the resistance of titanium OT4-1 alloy. The obtained results indicate the prospect of using HVOF spraying of the developed FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ coatings to strengthen and restore surfaces operating in aggressive environments and those subjected to fretting corrosion.

KEYWORDS: HVOF spraying, amorphous phase, amorphous iron-based alloy, composition coating, corrosion resistance, fretting corrosion

INTRODUCTION

Compared to crystalline materials, materials with an amorphous structure as a result of absence of the grain boundaries and dislocations have such properties as high strength, high elasticity limit, wear and corrosion resistance [1]. Among the systems of amorphous metallic materials iron-based alloys are of great interest for application in industry, not only owing to their properties, but also due to relatively low cost and availability of the material [2]. More over, the lower cost or absence of Ni and Co in amorphizing iron-base alloy makes application of these alloys more cost-effective than that of standard nickel- and cobalt-based materials (such as self-fluxing alloys, Ni–Cr–B–Si, stellite alloys, etc.).

Application of amorphizing Fe-alloys, as materials for thermal spraying of coatings allows forming layers with amorphous and nanocrystalline surface on the part surface, which have protective properties. Coatings based on amorphous Fe-alloys are produced using the methods of plasma [3, 4], supersonic plasma [5], electric arc [6], detonation [7, 8], HVOF and HVAF [9, 10] processes. The produced coatings are characterised by a combination of hardness, corrosion and wear resistance, ability to absorb neutrons and hydrophobicity, making them rather promising for application in different areas (defense, nuclear, oil and gas and other industries) [11].

Protective properties can be further improved by adding a small (up to 30 vol.%) quantity of ceramic particles (for instance, WC/Co, B_4C, TiN, Al_2O_3, ZrO_2, etc.) to the coating. Composite coatings of FeSiBCrMo–TiN, FeCSiBPCrMoAl–B_4C, FeCrMoCB–WC/Co, produced by plasma and HVOF spraying processes, have not only higher hardness, but also self-lubrication ability, demonstrating 2–5 times higher wear resistance compared to single-phase coatings [12–14]. It is shown [15] that addition of 20 wt.% ZrO to plasma coating based on FeCrMoCB increases the coating wear resistance two times under the conditions of sliding friction: its addition to plasma coating based on FCrMnBSi alloy improves the thermal protection properties [16]. Addition of 20 wt.% Al_2O_3 to a coating of FeCrMoCBY alloy spray-deposited by HVOF process, improves the wear and corrosion resistance of the coatings in 3.55 % NaCl environment by 2–3 times [17].

The objective of the work was investigation of the corrosion resistance and fretting corrosion resistance of composite coatings produced by high-velocity oxygen flame (HVOF) spraying by powders based on amorphizing FeMoNiCrB alloy, with addition of (Ti, Cr)C and $FeTiO_3$ hardening compounds to their composition.

EXPERIMENTAL MATERIALS AND PROCEDURES

Used as materials for HVOF spraying of coatings was powder of amorphizing FeMoNicrB alloy and composite cermet powders based on FeMoNiCrB alloy with strengthening additives of double carbide (Ti, Cr)C and iron titanate $FeTiO_2$. Selection of strengthening components of the studied coatings was based on their corrosion resistance and tribological characteristics [18, 19]. Composite powders were produced by mechanical alloying in Activator 2SL planetary mill of powder mixtures, which consists of FeMoNiCrB alloy and additive of one of the following compounds: (Ti,Cr)C (solid solution of 24 vol.% Cr_3C_2 in TiC) and $FeTiO_3$ (ilmenite). Content of strengthening components in the powder mixture was selected on the base of the recommendations on development of cermet coatings based on amorphous Fe-alloys, in keeping with which the optimal content of strengthening phases in cermet coating is 10–30 vol.% [12–17, 20]. Mechanical alloying (MA) resulted in formation of amorphous-nanocrystalline cermet powders based on oversaturated solid solution of Fe(Ni, Cr) with additives of strengthening phases. A detailed description of the process of mechanical alloying of powders of these compositions is given in work [12]. Table 1 gives the characteristic of powders, used in this work for HVOF-coatings.

HVOF spraying of coatings was performed in UVShGPN-M1 unit using the following technology parameters [22]: propane-butane pressure — 4 atm, oxygen pressure — 7 atm, air pressure — 6 atm, nitrogen pressure — 5 atm, spraying distance of 120 mm. To increase the strength of adhesion to the steel substrate FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ cermet coatings were sprayed on NiCr sublayer (50–100 μm thickness), which was deposited by electric arc spraying process.

The structure of the coatings was studied by metallographic methods (NEOPHOT 32 microscope, fitted with an attachment for digital photography of SIGE-TA model); microdurometric analysis of the coatings was performed in microhardness meter PMT-3 at 50 g load on the indenter. X-ray diffraction analysis (XRD) was performed in DRON-3 diffractometer in CuK_α-radiation with graphite monochromator at step displacement of 0.1° and 4c exposure time in each point 4c with further computer processing of the derived digital data. Phases identification was conducted using PSTM data base. Coating porosity was determined on metallographic sections by the method of image analysis (standard) ASTM B-276 using Image-Pro Plus.

Corrosion resistance of coatings deposited on samples from St3 500 μm thick were studied by potentiostatic method in P-5827M potentiometer at sweep rate of 2 mV/s in the solutions of 10 % H_2SO_4, 3 % NaCl and in 10 % KOH. These electrolyte were selected for studying the influence of the nature of the aggressive environment (different kind of anions) on the corrosion processes. Chlorine-silver electrode was used as the electrode, with platinum as an auxiliary electrode. Experimental values were used to plot the cathode and anode polarisation curves in the following coordinates: $E = E = f(\lg i)$, where E_c is the corrosion potential, V; i_c is the current, A/cm^2 [23, 24]. The corrosion current and corrosion potential were determined by the graphic method by the polarisation curves by extrapolation of tafel areas of the cathode and anode curves to $E = E_c$. Corrosion current values were used to calculate the massometric (K_w) and depth (K_d) corrosion values were calculated by formulas of [23]:

$$K_w = \frac{i \cdot A \cdot 1000}{n \cdot F},$$

where K_w is the weight index of corrosion, g/m^2·h; I is the corrosion current, A/cm^2; A is the atomic weight of metal, g/mol, (for iron and steel $A = 56$); n is the valence of metal ion, which moved into the solution (for iron $n = 2$); F is the Faradey number, 26.8 A·h/mole:

Table 1. Characteristics of powders for HVOF spraying of coatings

Composition, wt.%	Particle size, μm	Method of producing	Phase composition
FeMoNiCrB (36.2Fe–29.9Mo–23.6Ni–7.6Cr–2.7B)	<40	Melt atomisation by nitrogen	Fe(Ni, Cr), Fe_2B, Mo_2FeB_2, CrB_2 solid solution
77FeMoNiCrB–23(Ti, Cr)C	<40	MA 1.5 h	Fe(Ni, Cr), Mo_2FeB_2, (Ti, Cr)C, TiC, Cr_3C_2 solid solution, amorphous phase
75FeMoNiCrB–10$FeTiO_3$	<40	MA 1.5 h	Fe(Ni, Cr), Mo_2FeB_2, $FeTiO_3$ solid solution, amorphous phase

$$K_i = K_w \frac{8.76}{\rho},$$

where K_i is the depth index of corrosion, mm/y; ρ is the metal density, g/cm^3 (ρ_{Fe} = 7.85 g/cm^3); 8.76 is the conversion factor for transition from the weight index of corrosion to calculation per 1 hour to depth index to 1 year, calculated from the number of hours per year (24 h × 365 = 8760 h) and divided by 1000.

Protective effect of coatings was evaluated using the corrosion deceleration coefficient γ:

$$\gamma = \frac{K_w(s)}{K_w(c)},$$

where γ is the corrosion deceleration coefficient; K_w(s) and K_w(c) are the weight indices of corrosion rate of steel and of coated steels in corrosive environments (g/m^2·h).

Degree of protection from corrosion Z (%) was calculated by the following formula:

$$Z = \frac{K_w(s) - K_w(c)}{K_w(s)} \cdot 100\ \%.$$

Comparative characteristic of corrosion resistance was derived using a ten-point scale of assessment, based on application of the depth index of corrosion (K_d) [23].

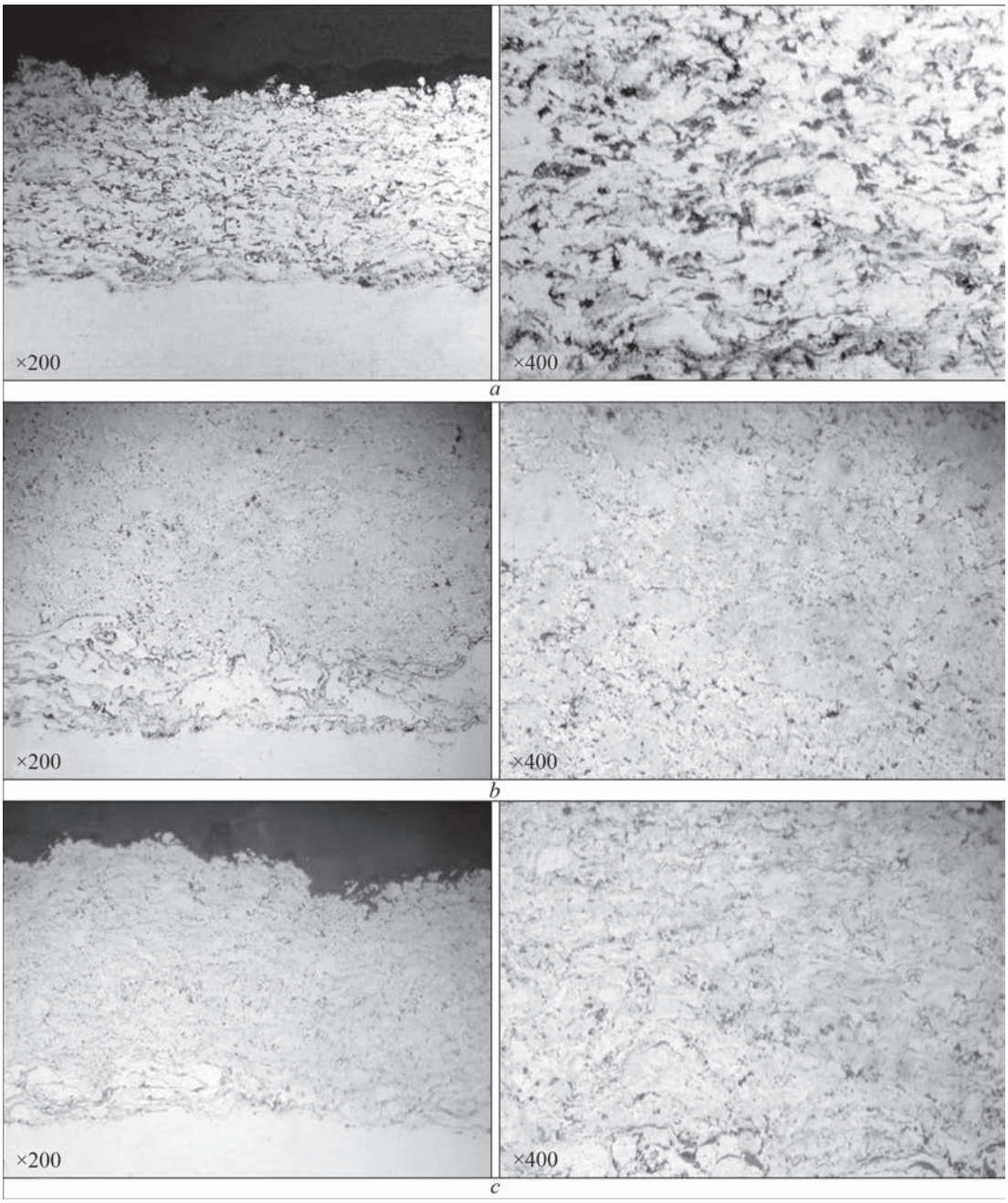

Figure 1. Microstructure of coatings produced by HVOF: *a* — FeNiCrMoB; *b* — FeNiCrMoB — (Ti, Cr)C; *c* — FeNiCrMoB–$FeTiO_3$

Testing for evaluation of comparative wear resistance of the studied coatings was conducted under the conditions of fretting-corrosion wear. Coating thickness was 500 μm, spraying was followed by machining with preservation of 300 μm thickness, including the underlayer.

Conditions of vibrocyclic loading were as follows: amplitude of relative vibrational displacement of the samples A = 120 μm, specific load on the sample contact surfaces P = 20 MPa, vibration frequency f = 25 Hz, number of cycles of sample vibrational displacement (test base) $n = 5 \cdot 10^5$ cycles. Samples were tested in air at temperature T = 293 K. In each friction pair the samples with the studied coating were stationary, and countersamples from steel 45 quenched to the hardness of *HRC* ~ 48–50 were mobile. OT4-1 titanium alloy was the basic variant for wear-resistant comparison. Sample wear was determined by the linear method after testing.

INVESTIGATION RESULTS AND THEIR DISCUSSION

Investigations of the microstructure of HVOF coatings (Figure 1) showed that dense coatings of uniform thickness form at spraying of powders both from FeMoNiCrB alloy and FeMoNiCrB–(Ti, Cr)C and FeNiCrMoB–$FeTiO_3$ composite powders. Porosity and microhardness of coatings FeMoNiCrB, FeMoNiCrB–(Ti, Cr)C and FeNiCrMoB–$FeTiO_3$ are equal to 3.4, 2.4, 1.2 % and 4390, 5510 and 4410 MPa, respectively. Coatings have a fine-grained structure typical for HVOF, which is formed from completely molten particles, which spread and solidified, and partially deformed particles, having an oval or close to the spherical shape. Interlayers of a dark-gray colour are observed on the boundaries of partially deformed particles, which is a result of interaction of the powder particles with oxygen and formation of oxide layers during spraying.

XRD analysis (Figure 2) revealed that multiphase coatings with an amorphous-crystalline structure form as a result of HVOF of powders based on FeMoNiCrB alloy.

Phase composition of the produced coatings is somewhat different from that of the initial powders. At spraying of crystalline FeMoNiCrB powder its partial amorphisation takes place, which is evidenced by the presence of a halo from the amorphous phase in the roentgenograph (Figure 2, *a*). At spraying of composite powders the interaction of the initial FeMoNiCrB alloy with the strengthening additives takes place with formation of Fe_2Ti phase in FeNiCrMoB–(Ti, Cr)C coating (Figure 2, *b*) and of NiTi phase in FeNiCrMoB–$FeTiO_3$ (Figure 2, *c*). Iron oxides are present in all the coatings, and in FeMoNiCrB–(Ti, Cr)C and FeNiCrMoB–$FeTiO_3$ composite coatings they are found in a small quantity. It should be noted that due to formation of hard phase of titanium carbonitiride in FeNiCrMoB–(Ti, Cr)C coating it has a somewhat higher (by 1100 MPa FeNiCrMoB) microhardness, compared to FeNiCrMoB coatings.

The characteristic of HVOF coatings based on FeMoNiCrB alloy is given in Table 2.

Investigations of the kinetics of electrode potential of FeMoNiCrB-based coatings showed that the stationary potential value is stabilised after 15–40 min.

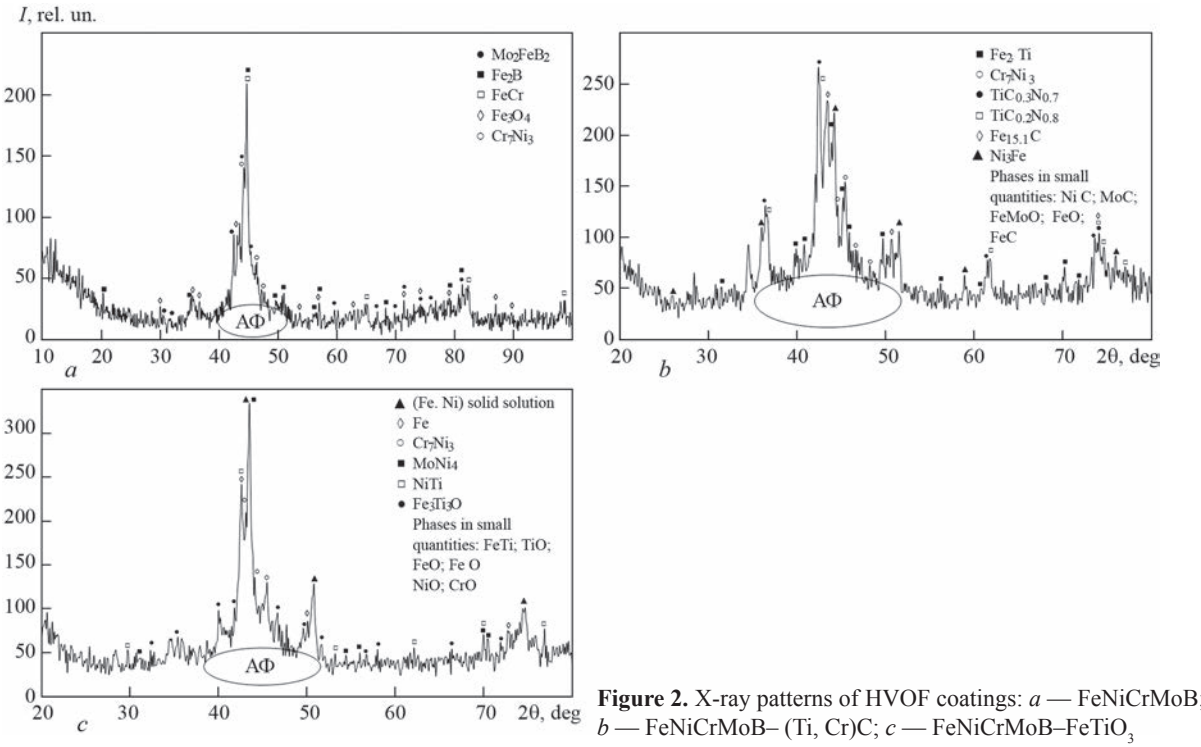

Figure 2. X-ray patterns of HVOF coatings: *a* — FeNiCrMoB; *b* — FeNiCrMoB– (Ti, Cr)C; *c* — FeNiCrMoB–$FeTiO_3$

Table 2. Characteristics of coatings based on FeMoNiCrB alloy, produced by the method of HVOF spraying

Coating material	Porosity, %	Microhardness $HV_{0.05}$, MPa	Phase composition
FeMoNiCrB	3.4 ± 0.7	4390 ± 290	Mo_2FeB_2; Fe_2B; FeCr; Fe_3O_4; Cr_7Ni_3 amorphous phase
FeMoNiCrB–(Ti, Cr)C	2.4 ± 0.4	5510 ± 250	Fe_2Ti; Cr_7Ni_3; $TiC_{0.3}N_{0.7}$; $TiC_{0.2}N_{0.8}$; $Fe_{15.1}C$; Ni_3Fe; amorphous phase; phases in small quantities: Ni_3C; MoC; $FeMoO_4$; FeO; FeC_8
FeMoNiCrB–$FeTiO_3$	1.2 ± 0.3	4410 ± 190	Solid solution (Fe, Ni); Cr_7Ni_3; $MoNi_4$; NiTi; Fe_3Ti_3O; amorphous phase; phases in small quantities: FeTi; TiO; FeO; Fe_3O_4; NiO; CrO

Table 3. Results of electrochemical tests of HVOF-coatings

Coating	3 % NaCl			10 % H_2SO_4			10 % KOH		
	$E_{??}$, V	E_c, V	i_c, A/cm^2	$E_{??}$, V	E_c, V	i_c, A/cm^2	$E_{??}$, V	E_c, C	i_c, A/cm^2
FeMoNiCrB	–0.43	–0.44	$6.5\cdot10^{-4}$	–0.2	–0.18	$5.8\cdot10^{-5}$	–0.44	–0.4	$5\cdot10^{-4}$
FeMoNiCrB–(TiCr)C	–0.38	–0.34	$4.3\cdot10^{-6}$	–0.06	–0.04	$4.2\cdot10^{-5}$	–0.50	–0.48	$4.2\cdot10^{-6}$
FeMoNiCrB–$FeTiO_3$	–0.34	–0.3	$2.9\cdot10^{-6}$	–0.06	–0.05	$2.5\cdot10^{-5}$	–0.56	–0.53	$4\cdot10^{-6}$
Uncoated St3 sample	–0.52	–0.5	$2\cdot10^{-5}$	–0.24	–0.22	$3\cdot10^{-3}$	–0.6	–0.58	$4\cdot10^{-5}$

On the coating surface the corrosion potential shifts to the positive side, compared to St3 sample, corrosion current decreases by one-two orders, and deceleration of both the cathode and the anode process takes place (Figure 3). In 10 % H_2SO_4 solution a lowering of hydrogen overvoltage occurs on the coating surface, in 3 % NaCl solution and 10 % KOH solution inhibition of oxygen reduction process takes place (Figure 3). Irrespective of the coating composition, in 3 % NaCl solution the cathode and anode polarisation curves have practically the same shape, in 10 % H_2SO_4 solution and in 10 % KOH solution passive areas appear on anode polarisation curves. No pittings or crevices are observed on the surface of coated samples after exposure to these solutions. After cleaning the surfaces become darker which is indicative of anodic dissolution process.

Analysis of the obtained results shows that for all the studied coatings the corrosion current in 3 % NaCl (Ph 7) and in 10 % KOH (pH 11) is by an order of magnitude higher than that in 10 % H_2SO_4 (pH 2–3) solution, i.e. the corrosion rate correlates with the solution pH. The corrosion resistance is also influenced by a change in the nature of depolarisation of the corrosion process (from hydrogen in the sulphate acid solution to predominantly oxygen one in 3 % solution of sodium chloride and in alkali solution).

Electrochemical studies in 3 % NaCl solution showed that in the coated samples the corrosion current decreases by an order compared to uncoated St3 samples (from $2\cdot10^{-5}$ for steel to $2.9–6.5\cdot10^{-6}$ A/cm^2 for coatings) (Table 3). Absence of passivation in 3 % NaCl solution can be due to the fact that this solution is aggressive with a high Cl^-, in the presence of which gradual driving of oxygen from the protective film on the electrode surface takes place, and by the impossibility of passive film formation. In 10 % H_2SO_4 solution on the coating surface, the corrosion potential shifts to passive state region, corrosion current decreases by two orders ($3\cdot10^{-3}$ for steel to $2.5–5.8\cdot10^{-5}$ A/cm^2) for coatings compared to uncoated St3 samples. Investigations of coatings in 10 % KOH solution showed that, compared to uncoated

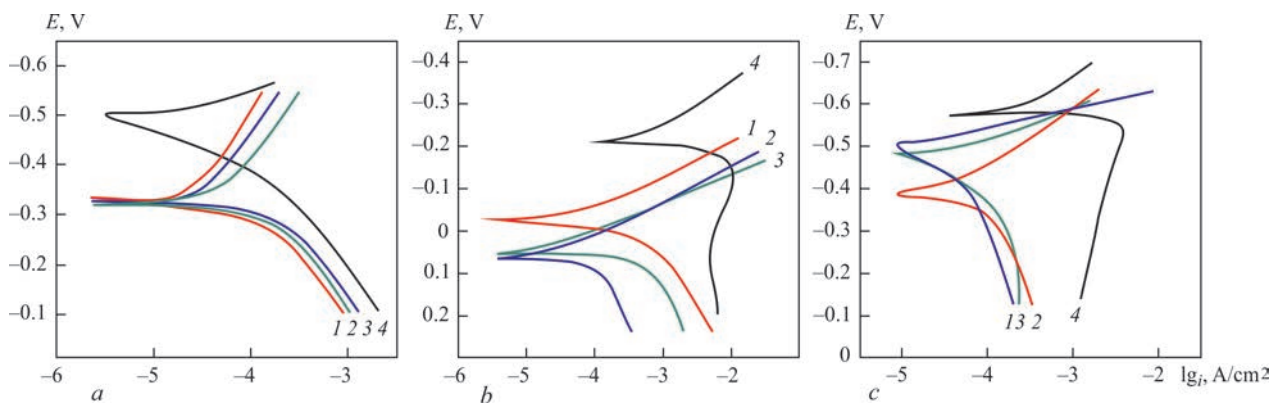

Figure 3. Polarisation curves of HVOF coatings in 3 % NaCl solution (*a*), in 10 % H_2SO_4 solution (*b*); in 10 % KOH solution (*c*)

Table 4. Corrosion resistance of HVOF-coatings in 3 % NaCl solution

Coating	K_w, g/m²·h	K_c, mm/year	γ	Z, %	Corrosion resistance rating
FeMoNiCrB	0.052	0.05	5.8	82.7	4
FeMoNiCrB–(TiCr)C	0.036	0.04	7.3	86.3	4
FeMoNiCrB–$FeTiO_3$	0.031	0.032	9.0	89.2	4
Uncoated St3 sample	–	0.293	–	–	6

Table 5. Corrosion resistance of HVOF-coatings in 10 % H_2SO_4 solution

Coating	K_w, g/m²·h	K_c, mm/year	γ	Z, %	Corrosion resistance rating
FeMoNiCrB	0.18	0.16	3.6	72.2	6
FeMoNiCrB–(TiCr)C	0.16	0.15	3.9	74.3	6
FeMoNiCrB–$FeTiO_3$	0.12	0.11	5.3	81.1	6
Uncoated St3 sample	–	0.59	–	–	7

Table 6. Corrosion resistance of HVOF-coatings in 10 % KOH solution

Coating	K_w, g/m²·h	K_c, mm/year	γ	Z, %	Corrosion resistance rating
FeMoNiCrB	0.05	0.056	8.2	87.8	4
FeMoNiCrB–(TiCr)C	0.044	0.048	9.5	89.4	4
FeMoNiCrB–$FeTiO_3$	0.042	0.047	9.7	89.6	4
Uncoated St3 sample	–	0.34	–	–	6

St3 samples, the corrosion current decreases by an order of magnitude (from $4 \cdot 10^{-5}$ for steel to $4–5 \cdot 10^{-6}$ A/cm² for coatings).

It was found that at application of composite powders with additives of (Ti, Cr)C and $FeTiO_3$ the corrosion resistance in the studied solutions increases 1.2–2.3 times, compared to a coating from FeMoNiCrB. The highest corrosion resistance is found in FeMoNiCrB–$FeTiO_3$ coating that may be related to its low porosity and presence of NiTi compounds and Fe_3Ti_3O complex oxide in its phase composition, which increase the corrosion resistance (Table 2).

Values of corrosion currents, calculated from polarisation curves, allowed calculation of the weight and depth indices of corrosion (Tables 4–6).

By ten point scale of corrosion resistance [24] amorphous coatings based on FeMoNiCrB alloy in 3 % NaCl solution and in 10 % KOH solution have resistance point 4 and belong to the "resistant" group, in 10 % H_2SO_4 solution they have resistance point 6 and belong to "lower resistance" group.

Conducted electrochemical testing showed that at deposition of composition coatings based on FeMoNiCrB, the rate of St3 corrosion in 3 % NaCl solution, in 10 % H_2SO_4 solution and in 10 % KOH solution decreases by 7.3–9, 3.9–5.3 and 9.5–9.7 times, respectively. Degree of protection here is equal to 86.3–89.2, 74.3–81.1 and 89.4–89.6 %, respectively.

Results of the conducted investigations of HVOF coatings based on FeMoNiCrB alloy are indicative of their corrosion resistance in 3 % NaCl solution at the level of VT-6 alloy ($i_c \sim 10^{-6}$ A/cm²) [25].

Results of investigations of the coatings for fretting corrosion resistance (Figure 4) showed that wear resistance of HVOF coatings from FeMoNiCrB, FeNiCrMoB–(Ti, Cr)C and FeNiCrMoB–$FeTiO_3$ is higher than that of OT4-1 titanium alloy by 3.3, 4.6 and 5.8 times, respectively. Such a high wear resistance of the

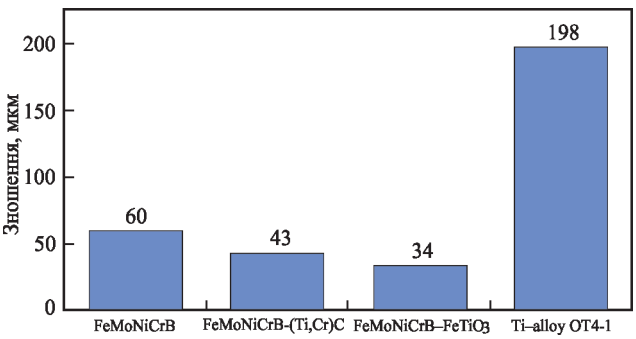

Figure 4. Results of investigations of HVOF-coatings for fretting corrosion resistance

coatings is achieved due to the specific structure of the coatings of heterogeneous type, where hard inclusions are uniformly distributed in a softer ductile matrix. Under the conditions of fretting corrosion and under the impact of high specific loads in service the ductile matrix is easily transferred to the connected surface, protects it from damage and promoted stress relaxation [26]. Presence of dispersed hard inclusions that take the main force load, results in the increase of fatigue fracture resistance of contacting materials.

In keeping with the results of the conducted investigations, the most fretting-resistant is FeNiCrMoB–$FeTiO_3$ coating, and the sequences of increase of HVOF coating resistance are as follows: FeMoNiCrB → FeNiCrMoB–(Ti, Cr)C → FeNiCrMoB–$FeTiO_3$. Such a regularity is, obviously, related to the relative pore content in the coatings, as porosity decreases the coatings resistance under the conditions of friction without lubricants, considering that pores are stress raisers and microcrack nuclei.

Proceeding from the conducted investigations of protective properties, the produced composite amorphizing HVOF coatings based on FeMoNiCrB alloy can be proposed to improve the durability of the parts of machines and mechanisms, exposed to aggressive environments and prone to fracture at fretting-corrosion, in particular, for protection of titanium parts of aviation equipment.

CONCLUSIONS

The method of high-velocity oxygen flame spraying of mechanically-alloyed powders based on amorphizing FeMoNiCrB alloy with additives of (Ti, Cr)C and $FeTiO_3$ compounds was used to produce dense heterogeneous coatings with not more than 3 % porosity. Results of phase analysis of FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ coatings demonstrate their amorphous-crystalline structure.

Corrosion resistance of thermally-sprayed composite coatings based on FeMoNiCrB alloy measured in the solutions of 3 % NaCl, 10 % H_2SO_4 and 10 % KOH, by the deep corrosion indices exceeds St3 steel durability 7.3–9; 3.9–5.3 and 9.5–9.7 times, respectively, with the values of 0.032–0.04; 0.11–0.15 and 0.047–0.048 mm/y. By the index of corrosion current the obtained amorphous coatings have corrosion durability at the level of titanium alloy VT-6 ($i_c \sim 10^{-6}$ A/cm^2).

Determination of fretting-corrosion resistance of the coatings showed that wear resistance of FeMoNiCrB–(Ti, Cr)C and FeMoNiCrB–$FeTiO_3$ cermet coatings is 4.6 and 5.8 times higher that that of OT4-1 titanium alloy, making these coatings promising at application for strengthening the parts of aviation equipment components, prone to fretting-corrosion.

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CONFLICT OF INTEREST
The Authors declare no conflict of interest

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