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INVESTIGATION OF PROCESS OF FORMATION OF STRUCTURE AND PROPERTIES IN MAGNETRON NANOLAYER FeAl-COATINGS

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The work is dedicated to investigation of process of formation of Fe-Al coating with regulated composition on substrates of 08Kh18N10T steel at mutual magnetron sputtering of composite Al + Fe target with heated above the Curie point (768 °C) insert of St.3 and aluminum target. Application of a system of cyclic substrate movement in the active zones of magnetron operation allowed forming a nanolayer structure of coatings with Al — 1.3–1.9 and Fe — 1.6 nm nanolayer thickness. The coatings were investigated using Auger spectrometry, X-ray diffraction and microindentation. It is determined that 3 μm FeAl-coatings containing 39.6 and 54.6 at.% of Al are an ordered B2 — FeAl phase consisting of 0.135–0.173 and 0.293–0.335 μm size grains, formed from nanocrystallites of 7 and 22 nm, respectively. 17 Ref., 4 Tables, 6 Figures.

Keywords: *magnetron sputtering, nanolayer structure, FeAl-coatings, regulated layer*

One of the tasks of surface engineering at modern stage of equipment development is a development of new coatings and technologies for their deposition ensuring protection of the parts and assemblies of equipment under conditions of elevating operating temperatures and mechanical loads, effect of aggressive media. In this connection, FeAl intermetallics, being characterized with high heat-resistance at 600–1000 °C, corrosion resistance in aggressive sulfur-containing media and wear, represent themselves a perspective material for development of new protective coatings. The technologies of coating deposition based on iron aluminides using plasma, electric arc, high-velocity plasma arc, detonation spraying [1, 2], allow forming FeAl-coatings of 100–300 μm thickness. Another field for producing FeAl-coatings is the vacuum methods, namely cathode sputtering [3], ion spraying [4], electron-beam evaporation [5], pulse laser deposition [6–8] and magnetron sputtering [9–12]. The magnetron sputtering method has gained the widest distribution.

Magnetron sputtering is widely used due to its capability to provide formation of coatings with thicknesses from nanometers to microns by means of condensation from neutral or ionized atoms at relatively low temperature with dense nanocrystalline structure [9]. Deposition of such coatings can be made using different technological schemes. There is an experience of application of magnetron sputtering system with the targets from FeAl alloy, produced by melting and casting in vacuum of pure Fe and Al metals [10]. The cheaper variant is receiving of FeAl coatings of various compositions by sputtering of compound target consisting of aluminum base and changeable iron disks of different diameters [11]. It should be noted that low iron deposition velocity was obtained at that. It is related with the fact that a magnetic flow, formed with magnetron magnetic system, is shunted by magnetic insert. This provokes distortion of distribution of transverse component of magnetic induction and its maximum displacement out of the insert limits. A variant of simultaneous sputtering of two targets from aluminum and iron is characterized by higher possibility for producing FeAl-coating of any composition. The multi-layer Al (4nm)/Fe(3.7 nm) magnetic films of 140 nm thickness were deposited on rotating silicon substrates with iron deposition velocity 9 nm/min [12]. The X-ray diffraction investigations showed that these films have bad crystallinity and do not contain reliable characteristics of ordered B2 phase.

Present work is dedicated to investigation of process of FeAl-coating formation with regulated composition at simultaneous magnetron sputtering of two targets.

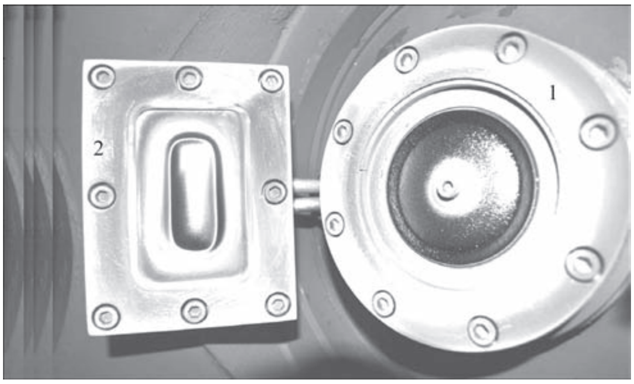

Figure 1. Positioning of magnetrons in vacuum chamber of VU-1BS unit: 1 — magnetron 1 with compound target Al + Fe (d = 88 mm); 2 — magnetron 2 with aluminum target of 80×50 mm size

Procedures of experiment and investigations. FeAl-coatings were deposited using modernized vacuum unit VU-1BS, which was equipped with direct current magnetron sputtering module consisting of two magnetrons (Figure 1). A compound Al + Fe target consisting of water-cooled aluminum part (d = = 88 mm, δ = 10 mm) and heat-insulated from it insert (d = 65 mm, δ = 3 mm) of low-carbon steel St.3 was installed on the magnetron 1. A magnetron discharge was excited at P_1 = 850 W, with cold insert at the periphery of aluminum part of the target, as well as maximum value of transverse constituent of magnetic induction $B_{\text{tr.max}}$ of magnetron 1 magnetic field. It provides fast heating of the insert via its end to the temperature above the Curie point (768 °C for iron) that provokes simultaneous displacement of $B_{\text{tr.max}}$ and discharge on insert surface. As a result the insert became a source of constant deposition velocity of iron atoms (Figure 2). Magnetron 2 with aluminum target of 80×50 mm size was used for receiving regulated aluminum constituent of FeAl-coating at power variation in P_2 = 0.35–1.4 kW range.

An auxiliary magnetic system of the same structure as a magnetron magnetic system was installed in front of magnetron 1 at 100 mm distance from it. Its magnetic field formed a consistent configuration with a field of magnetron 1 magnetic system (central and external poles of the auxiliary magnetic system have intensity of magnetization opposite in relation to poles of the magnetron magnetic system). This provided increase of current density of ion cleaning of substrate and enhancement of effect of plasma ions of magnetron discharge on a surface of growing coating that promoted rise of coating adhesion.

Glass substrates (65×30×4 mm) were used for initial experiments on investigation of the process of formation of FeAl-coating layers at different power of magnetrons. Choice of the glass substrates was stipulated by the necessity to perform accurate measurements of coating thickness with the help of profilograph — profilometer of AI Model 252 type. Specimens of steel 08Kh18N10T of 65×30×0.5 mm size were used as base substrates.

Figure 2. Compound target Al + Fe with insert (d = 64 mm, δ = = 3 mm) of steel St.3

The substrate was moved in sequence in relation to the magnetrons crossing the zones of Al and Fe atom flows, being formed on sputtered targets *1* and *2* with 0.3 m/s velocity. The distance between the targets and substrate made 55 and 70 mm, respectively (Figure 3). Deposition of FeAl-coating took place in cyclic mode with formation of single layers at substrate passing through the zones of Al and Fe atom flows. Thickness of the single layers depends on speed of rotation of holder with fastened substrate and deposition velocity of coating components (Fe and Al).

Thickness of the single layers makes 1.6 nm for Fe and 1.3–1.9 nm for Al under conditions of 0.3 m/s speed of substrate linear movement and deposition velocities v_{Fe} = 24nm/min and v_{Al} = 20–29 nm/min.

The next procedure was developed for calculation of content of aluminum in FeAl-coatings using the deposition velocities of iron and aluminum.

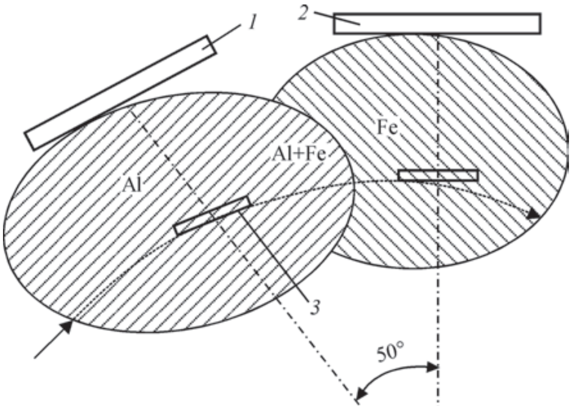

Figure 3. Scheme of deposition of magnetron FeAl-coating: *1* — aluminum target; *2* — St.3 heated insert of compound Al + Fe target; *3* — position of substrate relative to magnetrons in rotation with 15 rpm rate

Content of aluminum in FeAl-coatings can be presented by expression:

$$C_{\mathrm{Al}} = \frac{Q_{\mathrm{Al}}}{Q_{\mathrm{Al}} + Q_{\mathrm{Fe}}} \cdot 100\ \%,\ \text{wt.\%}, \qquad (1)$$

where Q_{Al} and Q_{Fe} are the weight of aluminum and iron composing the coating, which are simultaneously deposited using magnetrons *2* and *1*, respectively.

Values of Q_{Al} and Q_{Fe} are equal:

$$Q_{\mathrm{Al}} = \rho_{\mathrm{Al}} v_{\mathrm{Al}} ts;\ Q_{\mathrm{Fe}} = \rho_{\mathrm{Fe}} v_{\mathrm{Fe}} ts, \qquad (2)$$

where ρ_{Al} and ρ_{Fe} are the densities of deposited aluminum and iron, g/cm^3; v_{Al} and v_{Fe} are the deposition velocities of aluminum and iron, nm/min; t is the time of deposition, min; s is the area of specimen surface, cm^2.

Following the expressions (1) and (2)

$$C_{\mathrm{Al}} = \frac{\rho_{\mathrm{Al}} v_{\mathrm{Al}} ts}{\rho_{\mathrm{Al}} v_{\mathrm{Al}} ts + \rho_{\mathrm{Fe}} v_{\mathrm{Fe}} ts} \cdot 100\ \% = \frac{\rho_{\mathrm{Al}} v_{\mathrm{Al}}}{\rho_{\mathrm{Al}} v_{\mathrm{Aa}} + \rho_{\mathrm{Fe}} v_{\mathrm{Fe}}} \cdot 100\ \%,\ \text{wt.\%}. \qquad (3)$$

Transformation of expression (3) generates formula (4) for calculation of deposition velocity of aluminum v_{Al} on set values of aluminum content C_{Al} and deposition velocity of iron v_{Fe}.

$$v_{\mathrm{Al}} = \frac{C_{\mathrm{Al}} v_{\mathrm{Fe}} \rho_{\mathrm{Fe}}}{(100\ \% - C_{\mathrm{Al}}) \rho_{\mathrm{Al}}},\ \text{nm / min}. \qquad (4)$$

The next experiments were carried out for determination of ρ_{Al} and ρ_{Fe} of received coatings. Coatings of iron and aluminum were deposited on glass substrates at magnetron power $P_1 = 830$ W and $P_2 = 1350$ W. The profilograph-profilometer was used for determination of thickness of these coatings and calculation of deposition velocities $v_{\mathrm{Fe}} = 24$ nm/min and $v_{\mathrm{Al}} = 38$ nm/min. At these values of P_1 and P_2 and corresponding velocities v_{Fe}, v_{Al} the coatings of iron and aluminum were deposited on substrate of steel 08Kh18N10T of 18 cm^2 area without its ion cleaning. The substrates were weighed on VLR-200 scales before and after coating deposition (weighing error ±0.00012 g) and weight increment of Fe and Al was determined: $\Delta_{\mathrm{Fe}} = 0.01145$ g, 0.01205 g ($\delta = 1000$ nm), $\Delta_{\mathrm{Al}} = 0.0118$ g, 0.0121 g ($\delta = 2440$ nm). Coating densities, i.e. $\rho_{\mathrm{Fe}} = 6.97$ g/cm^3 and $\rho_{\mathrm{Al}} = 2.7$ g/cm^3 were calculated on average values of received values $\Delta_{\mathrm{Fe}} = 0.01205$ and $\Delta_{\mathrm{Al}} = 0.01195$ g.

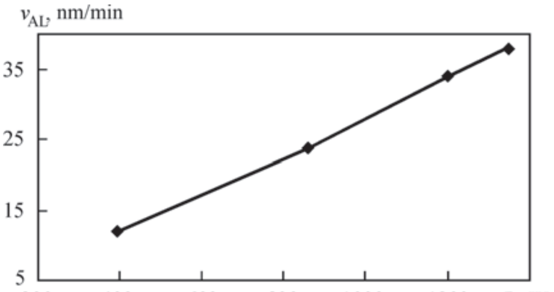

Figure 4. Dependence of aluminum deposition velocity on magnetron *2* power

Density of iron coatings has appeared to be less than standard volumetric density 7.86 per 0.89 g/cm^3 (11.3 %). This difference can depend on series of factors effecting vacuum coating formation. Thus, work [9] indicates that density of vacuum films is related with such characteristics as porosity, size of block structure, microstresses and can differ from volumetric density by up to 13 % value. Angle of incidence of sputtered atoms flow can vary in deposition of the coatings on moving substrate that can also influence the density of formed coating [14].

The following is received using $\rho_{\mathrm{Fe}} = 6.97$ g/cm^3, $\rho_{\mathrm{Al}} = 2.7$ g/cm^3 and constant value $v_{\mathrm{Fe}} = 24$ nm/min applied in all experiments.

$$v_{\mathrm{Al}} = \frac{61{,}92 C_{\mathrm{Al}}}{100\ \% - C_{\mathrm{Al}}},\ \text{nm / min}. \qquad (5)$$

The dependence v_{Al} (P_2) was received (Figure 4) for determination of magnetron *2* power on calculated v_{Al}.

The process of coating formation on surface of steel 08Kh18N10T substrates consisted of the following stages:

- before positioning in a vacuum chamber the substrate was cleaned in ultrasonic pool in acetone medium, and then in spirit;
- heating of substrate in vacuum chamber at $p = 5.0 \cdot 10^{-4}$ Pa, $T = 150$ °C, $t = 20$ min;
- ion cleaning of fixed substrate surface located between magnetron *1* and auxiliary magnetic system at pressure in the chamber $p_{\mathrm{Ar}} = 1.3$ Pa, $U = 1100$ V, current density $j = 3.5$ mA/cm^2, $t = 20$ min;
- excitation of magnetron discharge at a periphery of aluminum part of compound target of magnetron *1*, heating of steel insert to temperature above the Curie point with further movement of discharge over insert surface at $p_{\mathrm{Ar}} = 1.3$ Pa, $P = 0.9$ kW, $t = 5$–6 min;
- deposition of Fe sublayer on moving substrate at $p_{\mathrm{Ar}} = 0.3$ Pa, $P_1 = 830$ W, $U_{\mathrm{disp}} = -1100$ V, $T = 300$ °C, $t = 10$ min, $\delta = 240$ nm;
- deposition of FeAl-coating on moving substrate at magnetron *2* switching-on at $p_{\mathrm{Ar}} = 0.3$ Pa, $U_{\mathrm{disp}} = 0$ V, $T = 150$–200 °C, $\delta = 3$ μm.

Investigation of content and structure of coatings was carried out using Auger spectrometer JUMP 9500 F. X-ray phase analysis of the coatings was performed with the help of diffractometer Philips X'Pert – MRD based on Cu-$K_\alpha 1$ radiation (wave length $\lambda = 0.15405980$ nm). Diffraction spectra were recorded by scanning in step-by-step mode, scanning step made 0.025 °, time of setting in a point made 1 s. Qualitative phase composition was determined using ICDD, data base PDF-2 Release 2012.

Determination of mechanical characteristics of coatings and friction coefficient was performed employing micronanoindentor «Micron-Gamma» and friction machine «Micron-tribo» [15, 16]. Values of hardness and elasticity modulus in indentation were calculated automatically on ISO 14577-1:2002 standard [17].

Table 1. Parameters of deposition of FeAl-coating on moving substrates of steel 08Kh18N10T and measured values of aluminum content in coatings

Specimen	P_1, W	V_{Fe}, nm/min	P_2, W	v_{Al}, nm/min	C^{calc}_{Al},		C^{meas}_{Al},		Deviation value $C^{meas}_{Al} - C^{calc}_{Al}$	
					wt.%	at.%	wt.%	at.%	wt.%	at.%
Fe40Al	830	24	1040	29	32	49.6	36.74	54.6	4.74	5.0
Fe55Al	830	24	700	19.6	24	38.6	24.05	39.6	0.05	1.0

Results and their discussion. Investigations of process of FeAl-coating production was based on 47.0–52.7 at.% of Al concentration range, where B2–FeAl phase is formed, and coatings with 37–49 at.% of Al concentration, which have experimentally determined high heat-resistance at 600 – 1000 °C, including in aggressive gaseous media [10].

The values of aluminum concentration in the coating C^{calc}_{Al} = 36 and 24 wt.%, formula (5) and dependence $\nu_{Al}(P_2)$ were used for determination of aluminum deposition velocities and corresponding magnetron *2* powers, and production of coatings of 3 μm thickness on specimens *1* and *2*. During coating deposition the substrate successively passes through flows of aluminum and iron sputtered atoms (see Figure 3). Close location of the targets (distance between their centers 105 mm) and fast movement of the substrate promoted active mixing of deposited on its surface atoms of Al and Fe and generation at each turn FeAl nanolayers, which form FeAl-coating along its whole thickness (Table 1). It should be noted that Fe/Al-coating of alternating nanolayers of iron and aluminum can be obtained at lager distance between the magnetrons of indicated system design and installation of a screen between them. Table 1 shows that calculated concentration of aluminum differs from measured by 1–5 at.% of Al that can be explained by the following reasons:

- during selection of C_{Al} and ν_{Al} calculation procedure it was assumed that FeAl-coating consists only of iron and aluminum. However, JUMP 9500 F spectrometer registered in the vacuum coating specimens the constant additives (oxygen, carbon) reaching in sum up to 5 wt.%;
- another reason lies in quick change of erosion zone of aluminum target in process of coating formation. Carried investigations on sputtering of 4 mm thick target at P_2 = 1000 W showed that typical V-shaped erosion zone with *3* mm depth limit is formed in it at electric energy consumption 4.7 kW·h. It is determined that proportionality of $v_{Al}(P_2)$ dependence was preserved at electric energy consumption up to 2 kW·h. However, deepening of the erosion zone promotes gradual decrease of deposition velocity at constant discharge power. Thus, at zone depth 3 mm the measured deposition velocity of aluminum was less than calculated on curve $v_{Al}(P_2)$ (Figure 3) by 20.3 %. Decrease of aluminum deposition velocity is obviously related with formation of V-shaped surface of erosion zone that provokes increase of intensity of ionizing collisions in plasma of magnetron discharge, constriction of plasma area, growth of density of positive ions of Ar^+ as well as Al^+ ions of sputtered aluminum. As a result portion of ionized atoms of aluminum rises in the general flow of ions bombarding the target. They come back to the target, deposit on the surface of erosion zone and do not reach the substrate.

Figure 5 shows radiographs of coatings on specimens *1* and *2*.

The radiographs contain clear reflections (110) and (200) of FeAl phase, that indicate that the structure of coating on the specimens is the ordered B2–FeAl phase with body-centered crystalline lattice having the following parameters: Fe55Al — a_1 = 0.29097 nm, Fe40Al — a_2 = 0.29090 nm. The Scherrer formula was used for calculation of the dimension of coherent scattering region (CSR) for crystals forming the coating: Fe55Al — D_1 = 7 nm, Fe40Al — D_2 = 22 nm.

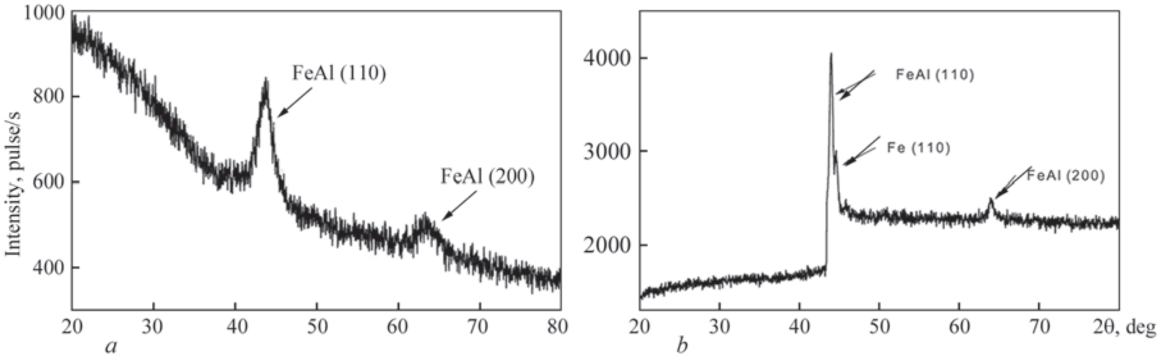

Figure 5. Radiographs of FeAl-coating with different Al content: *a* — FeAl-coating with 54.6 at.% of Al (specimen Fe55Al); *b* — FeAl-coating with 39.6 at.% of Al (specimen Fe40Al)

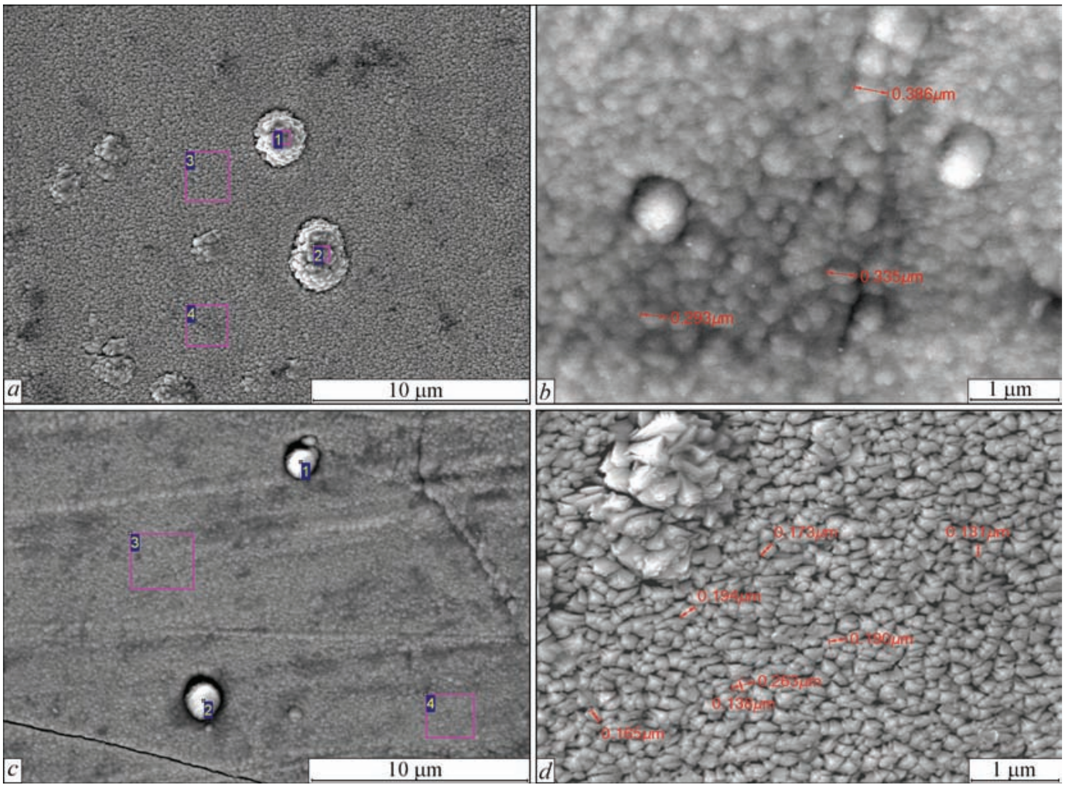

Figure 6. Microstructure of surface of magnetron FeAl-coatings of 3 mm thickness deposited on substrates of steel 08Kh18N10T with different Al content: *a*, *b* — Fe55Al; *c*, *d* — Fe40Al

Reflection Fe (110) is also registered on Fe40Al radiograph. It can be related with the fact that content of aluminum in this coating is less than the lower boundary of content range, in which B2–FeAl phase is formed in 47–52.7 at.% of Al.

Figure 6 and Tables 2 and 3 present the data and photos from scanning electron microscope (SEM) of the structure of coating surface on Fe55Al and Fe-40Al specimens and compositions of these coatings at different areas of the specimens.

Analysis of received data shows formation of dense, pore-free crystalline structure of the coatings on the surfaces of both specimens of steel 08Kh18N10T. Grain sizes in these coatings depend on Al content and make 0.293–0.335 μm (Fe55Al) at 54.6 at.% of Al and 0.131–0.173 μm at 3.9 at.% of Al (Fe40Al).

Small content of chromium (C_{Cr} = 0.42–0.78 at.%) is found in the content of specimen coatings that can be related with insignificant sputtering of the frame made of steel 08Kh19N10T, with the help of which aluminum target is fixed to magnetron *2*.

Surfaces of FeAl-coating specimens include separate convex polycrystalline formations of 1.7 and 3.2 μm diameter, in which content of aluminum comparing with nearby area is more by 1.2–1.8 at.%.

It is apparently related with appearance of microarcs in the process of aluminum target sputtering that has stipulated generation of micron particles of aluminum, which were deposited on the surface of formed coating and was overgrown by this coating.

Mechanical characteristics of the coatings, determined by microindentation method, are presented in Table 4.

The results of measurement of mechanical properties of magnetron FeAl-coatings, made with the help of micronanoindentor «Micron-Gamma» showed that the value of their contact elasticity modulus rises with decrease of Al content and makes 204 GPa for Fe40Al coating in comparison with 188 GPa in Fe55Al coating. Values of hardness in both coatings differ insignificantly (14 and 13 GPa), but data of specified hardness, effecting coating wear resistance, are higher in Fe55Al coating (0.074 in contrast to 0.064 in Fe40Al coating).

Value of coefficient of dry friction in «coating–diamond indentor pair» with rounded radius

Table 2. Composition of FeAl-coating in different areas of Fe-55Al (at.%)

Spectrum	Al	Cr	Fe
1	54.72	0.42	44.87
2	55.37	0.78	43.85
3	54.01	0.78	45.21
4	54.14	0.77	45.08

Table 3. Composition of FeAl-coating in different areas of Fe-40Al (at.%)

Spectrum	Al	Cr	Fe
1	40.42	0.47	59.11
2	39.39	0.83	59.79
3	38.52	1.60	59.88
4	39.92	0.64	59.45

Table 4. Mechanical characteristics of FeAl-coating on substrates of steel 08Kh18N10T

Specimen	Hardness *H*, GPa	Contact elasticity modulus E^*, GPa	Specified hardness H/E^*, relative units	Friction coefficient *f* at loading on indentor, g		
				225	375	525
Fe55Al	14	188	0.074	0.11	0.12	0.12
Fe40Al	13	204	0.064	0.11	0.12	0.12

0.12 mmwith slip velocity 12 mm/s at 225–525 g loading made 0.11–0.12.

Conclusions

1. Microcrystalline FeAl-coatings of 3 μm thickness, formed from nanocrystallines with the size depending on coating composition and equal 7 nm in Fe54.6Al at.% coating and 22 nm in Fe39.6Al at.% coating, were received using the method of simultaneous direct current magnetron sputtering of compound target Al + Fe on moving substrates of steel 08Kh18N10T. The target consists of heated above the Curie point (768 °C) insert of steel St.3 ($d_{\text{insert}} = 0.75d_{\text{Al parts of target}}$) and aluminum target.

2. Developed system of magnetron sputtering of FeAl-coatings with the device for cyclic movement of substrate in zones of Al and Fe atom flows, being generated on used Al and Al + Fe targets, allows forming the nanolayer structure of FeAl-coating with thickness of single layers of 1.6 nm and iron 1.3–1.9 nm aluminum. This provides formation of FeAl phase without additional heat treatment by annealing at 600–1000 °C.

3. Calculation-experimental procedure was developed for regulation of aluminum content in FeAl-coating in 40–55 at.% of Al range by means of variation of power of magnetron discharge at aluminum target with constant power of sputtering of St.3 insert of compound target.

4. The results of examination of FeAl-coatings using Auger spectrometry and X-ray diffraction methods show that they are ordered B2–FeAl phase at content of 39.6 and 54.6 at. % of Al in them, grain size of which rises with increase of Al content in FeAl-coating, making 0.135-0.173 μm in the case of Fe40Al coating and 0.293–0.335 μm for Fe55Al coating.

5. Values of contact modulus of elasticity of FeAl-coatings, determined with microindentation method, depend on Al content and make 188 GPa in Fe55Al coating and 204 GPa in Fe40Al coating. Hardness values of received FeAl-coatings are 13–14 GPa, that of specified hardness make 0.064–0.074. Coefficient of dry friction of FeAl-coating in pair with diamond indentor made 0.1–0.12.

6. Application of developed method of magnetron deposition of FeAl-coating allows forming heat-resistant coatings with increased resistance to oxidation and scaling as well as multilayer soft magnetic FeAl-coatings with high intensity of magnetization in weak magnetic fields, which can be used in magnetic protection devices.

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