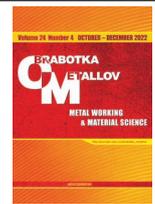




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Deposition of titanium silicide on stainless steel AISI 304 surface

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ABSTRACT

Introduction. Metal-ceramic coatings based on titanium silicide are promising for protecting stainless steel *AISI 304* from wear, corrosion and high-temperature oxidation. **Purpose of the work:** to investigate the stainless steel *AISI 304* surface layer structure after electrospark deposition in a mixture of titanium granules with silicon powder, and to study oxidation resistance, corrosion resistance and tribotechnical properties of the obtained coatings. **Research methodology.** *Fe-Ti-Si* coatings on the stainless steel *AISI 304* samples were obtained by electrospark machining with a non-localized electrode consisting of titanium granules and 2.6-6 vol.% mixture of titanium and crystalline silicon powders. **Results and discussion:** it is shown that a stable positive gain of the cathode is observed when the proportion of silicon in the powder mixture does not exceed 32 vol.%. The phase composition of the coatings includes: a solid solution of chromium in iron, titanium silicide Ti_3Si_3 , titanium and silicon, which is confirmed by the energy dispersion analysis data. The microhardness of *Fe-Ti-Si* coatings ranges from 10.05 to 12.86 GPa, which is 5-6 times higher than that of uncoated steel *AISI 304*. The coefficient of friction of the coatings is about 20% lower compared to steel *AISI 304* and hovers around 0.71-0.73. Wear tests in dry sliding mode show that *Fe-Ti-Si* coatings can increase the wear resistance of steel *AISI 304* up to 6 times. The oxidation resistance of the coatings at a temperature of 900 °C is 7-12 times higher as compared to steel *AISI 304*. The conducted studies have shown that new electrospark *Fe-Ti-Si* coatings can increase corrosion resistance, oxidation resistance, microhardness, as well as reduce the coefficient of friction and wear rate of the stainless steel *AISI 304* surface.

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Introduction

Austenitic stainless steel *AISI304* is used in the chemical and aerospace industries, atomics, medicine and other fields, due to its high corrosion resistance [1]. However, it is highly susceptible to wear due to its relatively low hardness (~200 HV) [2]. In addition, *AISI304* steel is also subjected to pitting corrosion in electrolytes and begins to oxidize actively at temperatures above 800 °C in air [3]. The application of hard and anticorrosive coatings on the surface of *AISI304* stainless steel is designed to reduce these disadvantages and expand its scope of application.

Metal-ceramic (*MC*) materials are a kind of metal matrix composites, and combine in its composition a metal matrix reinforced with ceramic particles. Ceramic phases provide high hardness, and a relatively soft matrix holds the ceramics and gives such a composition high crack resistance and strength. *MC* materials are highly resistant to abrasion [4]. There are two ways to obtain a *MC* structure: the introduction of ceramic particles into a metal matrix or the crystallization of solid phases from the melt [5].

MC coatings attract a lot of attention of researchers because of its high hardness, wear resistance and corrosion resistance [6]. One of the promising reinforcing compounds is titanium silicide Ti_5Si_3 , which has a high melting point, high-temperature resistance and oxidation resistance [7, 8]. Thus, in [9] it was shown that the coating with Ti_5Si_3 was preserved under cyclic oxidation conditions at 900 °C for 1,000 hours. Due to the strong covalent atomic bond, Ti_5Si_3 has high hardness and is stable under conditions of abrasive and adhesive wear [10].

Earlier, we showed the possibility of single-stage coating based on titanium silicide Ti_5Si_3 by electro-spark deposition (*ESD*) of a titanium alloy with a non-localized anode made of titanium granules with the addition of crystalline silicon powder [11]. The deposited coatings had advanced high-temperature resistance at 900 °C and high wear resistance. It was shown that the Ti_5Si_3 phase is formed by the interaction of silicon with titanium melt in the discharge microbath. Moreover, this interaction can occur both on the surface of the granules and on the titanium cathode. In the case of processing steel *AISI304*, only the first option can be implemented, so the transfer of *Ti-Si* material from titanium granules to the stainless steel surface plays a key role. As is known, the *ESD* is based on the phenomenon of polar material transfer from the anode to the cathode. It consists in the release of very hot microparticles of the electrode material into the melt microbath at the cathode, mixing of these materials and rapid solidification after the discharge [12]. Polar transfer is observed if the erosion of the anode exceeds the erosion of the cathode during the *ESD* process. The proportion of anode elements in the coating and its thickness depend on the polarity degree. The polarity criterion positively correlates with the thermophysical properties of the materials of the electrode pair; however, it is influenced by many factors [13]. Therefore, the establishment of polar transfer and its degree is achieved empirically. The purpose of this work is to study the structure of *AISI304* stainless steel surface layer after *ESD* in a mixture of titanium granules with silicon powder, and to study the high-temperature resistance, corrosion and tribotechnical properties of the deposited coatings.

Materials and methods

Preliminary experiments have shown that in the case of an anode mixture of titanium granules with silicon powder, a positive cathode weight gain was not observed. To achieve a positive cathode weight gain, titanium powder was gradually added to the anode mixture, since it has better electrical conductivity compared to silicon. Titanium particles act as contact bridges among silicon particles, reducing the resistance of the system. The content of titanium powder in the mixture was gradually increased until a stable of the cathode weight gain began to be observed. So, the silicon concentration in the powder mixture was 31.6 vol.%. Granules from titanium alloy *VTI-00* and powder mixture in various ratios (Table 1) were poured into a metal container connected to the positive output of the pulse generator; therefore, the granules acted as an anode. The silicon powder had an average particle size of 10 μm. The coatings were applied to a stainless steel *AISI304* substrate in the form of a cylinder with a diameter of 12 mm and a height of 10 mm (Table 2). The substrate was connected to the negative of pulse generator. The *IMES-40* pulse generator

Table 1

Composition of anode mixtures, designations and characteristics of coatings

Designation of samples	Composition of powder mixture, vol.%		Charge content in the anode mixture, vol.%	The content of <i>ERTi-1</i> granules, vol.%	Thickness, μm
	<i>Si</i>	<i>Ti</i>			
<i>Si2.6</i>	31.6	68.4	2.633	97.367	24.8 ± 7.9
<i>Si6.0</i>			6.048	93.952	21.7 ± 11.2

Table 2

Chemical composition of *AISI304* steel

Element	<i>Fe</i>	<i>Cr</i>	<i>Ni</i>	<i>Mn</i>	<i>Cu</i>	<i>P</i>	<i>C</i>	<i>S</i>
Concentration, wt. %	66.374	18	8	≤ 2	≤ 1	≤ 0.045	≤ 0.03	≤ 0.03

generated discharge rectangular current pulses with an amplitude of 110 A, at a voltage of 30 V, with a duration of 100 μs and a period of 1,000 μs . Argon was filled into the working volume of the container at a rate of 10 L/min to prevent titanium nitriding. The setup for electrospark deposition of coatings with a non-localized electrode is described in detail in [14, 15].

The structure of the coatings was studied using a *Vega 3 LMH* (Tescan, Czech Republic) and an *X-max 80* energy dispersive spectrometer (*EDS*) (Oxford Instruments, UK). The phase composition of the coatings was determined using a *DRON-7* X-ray diffractometer in *Cu-K α* radiation. The microhardness of the coatings was measured on a *PMT-3M* hardness tester at a load of 0.5 N according to the Vickers method. The wear resistance and coefficient of friction of the coatings were studied according to the *ASTM G99 – 17* using “pin-on-disk” scheme. The tests were carried out in dry sliding mode using a counterbody in the form of a disk made of high-speed steel *M45* at speed 0.47 ms^{-1} at a load of 10 N for 600 s. Polarization tests were carried out in a three-electrode cell after 30 minutes of holding samples in a 3.5% *NaCl* solution under natural aeration conditions at room temperature until a stationary value of the corrosion potential was established. Scanning was performed using a *P-2X* potentiostat (Elins, Russia) at a rate of 10 $\text{mV}\cdot\text{s}^{-1}$ in the range of $-1.5 - 0.5$ V. The contact area of the samples with the electrolyte solution was 1 cm^2 . The counter electrode was a paired *ETP-02* platinum electrode, the reference electrode was a standard silver chloride electrode, and the coated samples and *AISI304* steel served as the working electrode.

The test for cyclic high-temperature resistance was carried out in a furnace at a temperature of 900 $^{\circ}\text{C}$. The total testing time was 100 hours. The samples were kept at a given temperature and after some time intervals (~ 6 hours) were removed and cooled in a desiccator to room temperature. During the test, all samples were placed in a corundum crucible to account for the mass of exfoliated oxides. The weight change of all samples was measured using a laboratory balance with an accuracy of 0.1 mg.

Results and discussion

The coatings were deposited within 180 s, since the mass of the substrate began to decrease during further processing due to the accumulation of defects and the onset of the threshold of brittle fracture of the coating, which is characteristic of *ESD* [16]. The X-ray analysis of the coatings showed the presence of ferrochrome (*Fe-Cr*) and hexagonal titanium (αTi) phases forming the coating matrix (Fig. 1). Titanium silicide Ti_5Si_3 and silicon act as a reinforcing ceramic. The Ti_5Si_3 phase is formed during the interaction of silicon with titanium melt on the granule's surface, which is accompanied by heat release ($\Delta H_{298}^{\circ} = -581.2$ kJ/mol), according to reaction 1:



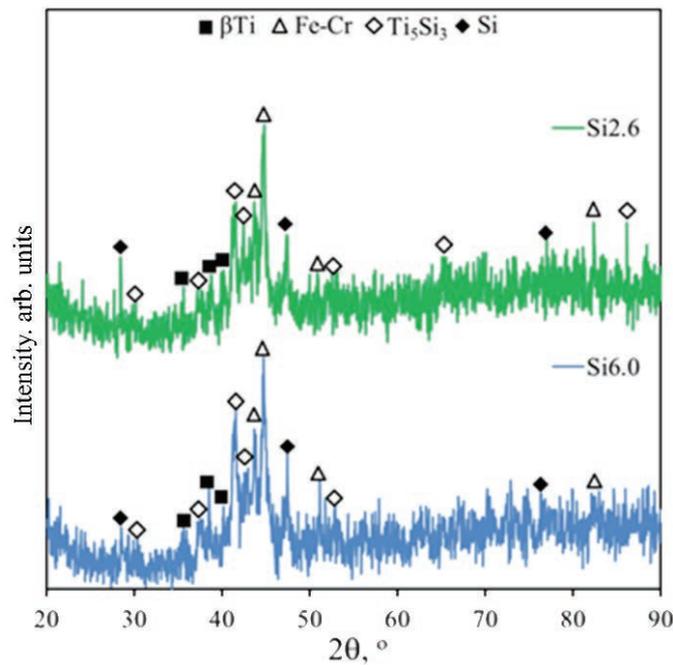


Fig. 1. X-ray diffraction patterns of deposited coatings

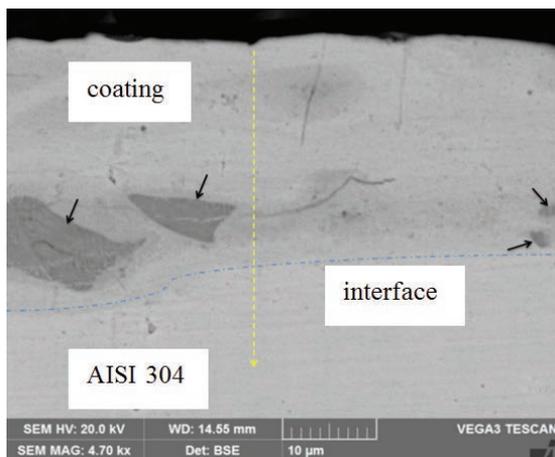
Characteristically, silicon reflections are observed in the X-ray spectrum of the coatings, but there are no iron silicides. This may indicate unfavorable conditions for the formation of ferrosilicon under conditions of a low-voltage electric discharge. This also explains the halo visible in the diffraction patterns of the coatings in the angle range 2θ 35 – 50°, which indicates the presence of an amorphous phase in the coatings. As consequence, according to the results of X-ray phase analysis, it is impossible to judge reliably the effect of silicon concentration in the anode mixture on the content of titanium silicide in coatings.

Figure 2, a shows a cross section image of the *Si2.6* coating. The coating has a darker shade compared to the substrate due to the enrichment with silicon and titanium, which have a lower atomic weight compared to *AISI304* steel elements. There are no clear boundaries and longitudinal cracks between the deposited layer and the substrate, which indicates good adhesion of the *Fe-Ti-Si* coating to steel *AISI304*. According to the *EDS* data (Fig. 2, b), the composition of the coating is dominated by iron and chromium from the substrate, which corresponds to the of X-ray data (Fig. 1). The concentrations of titanium and silicon dissolved in the coating matrix ranged from 5 to 20 at.%. There are dark inclusions in the coating structure (Fig. 2, c). According to its *EDS* analysis, the ratio of titanium to silicon is 49.3 to 31.8, which corresponds to titanium silicide Ti_5Si_3 (Fig. 2, d). The microstructure of inclusions is represented by columnar crystallites, which corresponds to the microstructure of *Ti-Si* coatings on a titanium alloy [11]. With an increase in the powder charge concentration in the anode mixture from 2.6 to 6 vol.%, the average coating thickness decreased from 24.8 to 21.7 μm (Table 1).

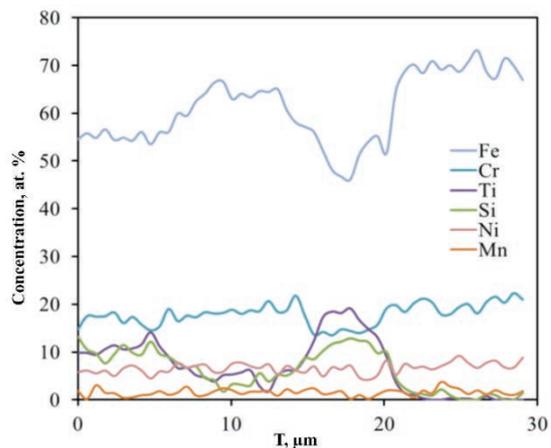
Figure 3 shows polarization diagrams of *Fe-Ti-Si* coatings and *AISI304* steel in 3.5 % *NaCl* solution at room temperature. Based on these data, the corrosion current density I_{corr} , the corrosion potential E_{corr} and the polarization resistance R_p were calculated (Table 3). R_p was calculated using the simplified expression (2):

$$R_p = \frac{b_a b_c}{2.303 I_{corr} (b_a + b_c)}, \quad (2)$$

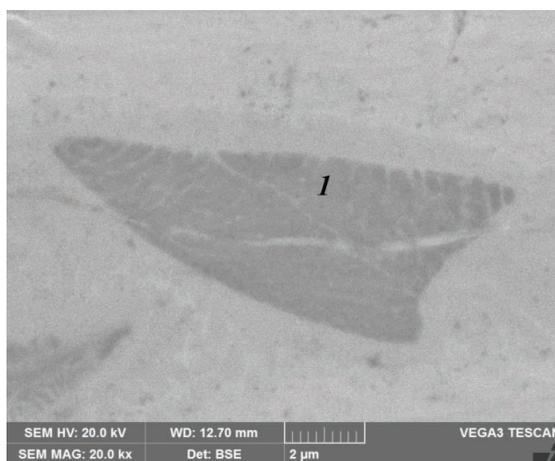
where b_a and b_c are the slopes of the Tafel section of the anode and cathode curves, respectively. It follows from Table 3 that the corrosion potentials were similar for both coatings and significantly greater than for *AISI304* stainless steel. This suggests that *Fe-Ti-Si* coatings can reduce the activity of the stainless steel surface to spontaneous corrosion. The corrosion current density of the coatings was 1.8 to 2.1 times lower than that of *AISI304* stainless steel (Table 3). The *Si2.6* sample showed the highest corrosion potential and



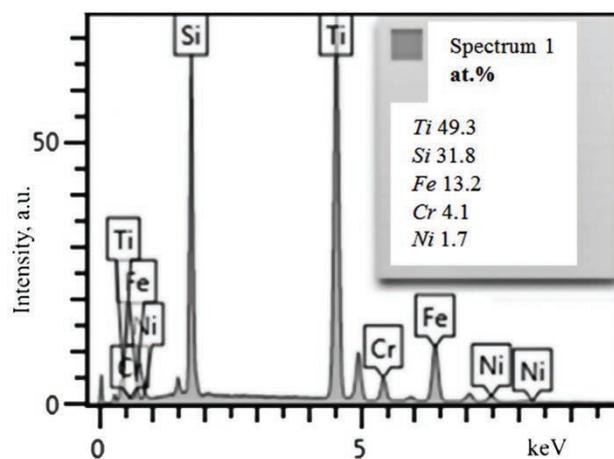
a



b



c



d

Fig. 2. SEM image of the cross-section of the Si_{2.6} coating in the back scattered electrons mode at magnifications of 4.7X (a) and 20X (c); EDS distribution of elements over the coating depth (b) and EDS spectrum of point 1 (d). The black arrows indicate inclusions of titanium silicide

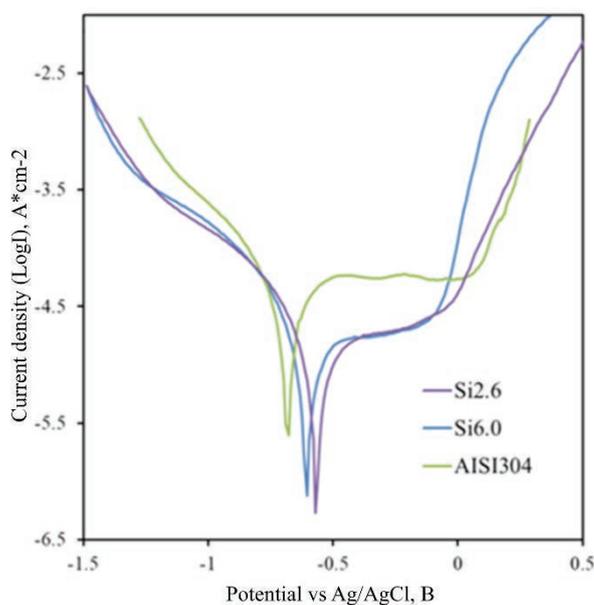


Fig. 3. Tafel polarization diagrams of coatings compared to AISI304 stainless steel

Table 3

Corrosion parameters of coatings calculated from the slopes of polarization curves

Parameters	Samples		
	<i>AISI304</i>	<i>Si2.6</i>	<i>Si6.0</i>
E_{corr}, V	-0.68	-0.57	-0.603
$I_{corr}, \mu A$	27.5	13.1	15.1
$R_p, k\Omega$	2.5	10.1	10.7

the lowest corrosion current density. The calculated polarization resistance of the deposited coatings was four times higher than that of the original *AISI304* steel. Despite the high corrosion resistance of *AISI304* steel, due to the high chromium content (Table 2), it can be concluded that the use of electrospark *Fe-Ti-Si* coatings can significantly improve its anti-corrosion properties.

Figure 4, *a* shows the average values of microhardness measured on the surface of the coatings. The hardness of the coatings decreased from 12.86 to 10.05 GPa with the growth of powder charge in the anode mixture. Thus, the application of *Fe-Ti-Si* coatings can significantly increase the surface hardness of *AISI304* steel (1.9 GPa). The high hardness of the coatings is primarily due to the presence of the Ti_5Si_3 phase, which hardness is 9.5 GPa [17, 18]. The higher hardness of the deposited coatings is explained by the structure refinement up to the amorphous state, due to the high cooling rates of the material after the completion of the discharge during *ESD* [19].

Figure 4, *b* shows the dynamics of the friction coefficient of the coatings compared to stainless steel *AISI304*. It follows from this that the application of *Ti-Si* coatings makes it possible to reduce the friction coefficient of *AISI304* steel by 20 % from 0.9 to 0.73. With an increase in the powder content in the anode mixture, the average values of the friction coefficient slightly decreased from 0.73 to 0.71. With an increase in the silicon content in the anode mixture, the average values of coating wear rate increased from $1.07 \cdot 10^{-6}$ to $1.45 \cdot 10^{-6} \text{ mm}^3/\text{Nm}$, which is consistent with the hardness data (Fig. 4, *a*). In general, the application of *Fe-Ti-Si* coatings makes it possible to increase the wear resistance of the *AISI304* steel surface from 4.6 to 6.2 times.

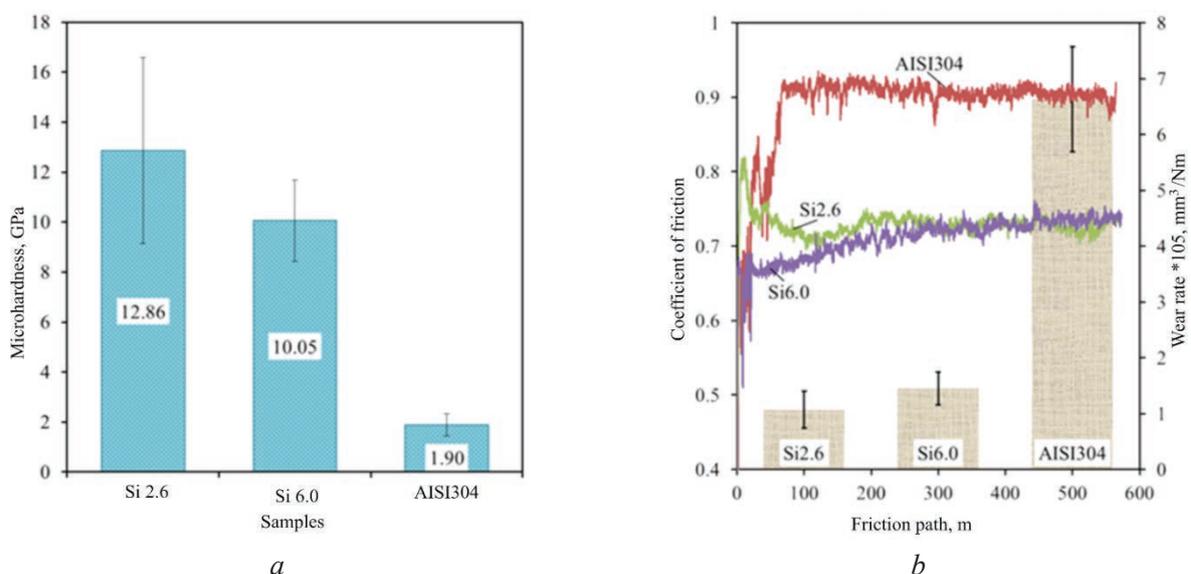

 Fig. 4. Microhardness (*a*), coefficient of friction and wear rate at a load of 10 N (*b*)

Figure 5, *a* shows the change in the mass of samples with *Fe-Ti-Si* coatings and *AISI304* steel at a temperature of 900 °C. The weight gain of samples with coatings for 100 hours of testing ranged from 23.3 to 37.9 g/m². The smallest weight gain was observed for the *Si2.6* sample, which also showed the best corrosion resistance. Coated specimens were damaged by corrosion in 7–12 times less than *AISI304* steel. The weight gain is due to the fixation of oxygen on the surface of the samples in the form of oxides of iron, titanium and chromium in the modifications of hematite, rutile and iron (II) chromite $FeCr_2O_4$ (Fig. 5, *b*). In contrast to *AISI304* steel, on X-ray patterns of the coating's surface, the reflections of ferrochrome, which is resistant to oxidation, are observed. The high oxidation resistance of coated samples is due to the limited contact of oxygen with the substrate, primarily due to the Ti_5Si_3 phase, which is resistant to oxidation at temperatures up to 1,000 °C. This is explained by the formation of a thin silicon dioxide barrier layer on the surface of Ti_5Si_3 particles [20].

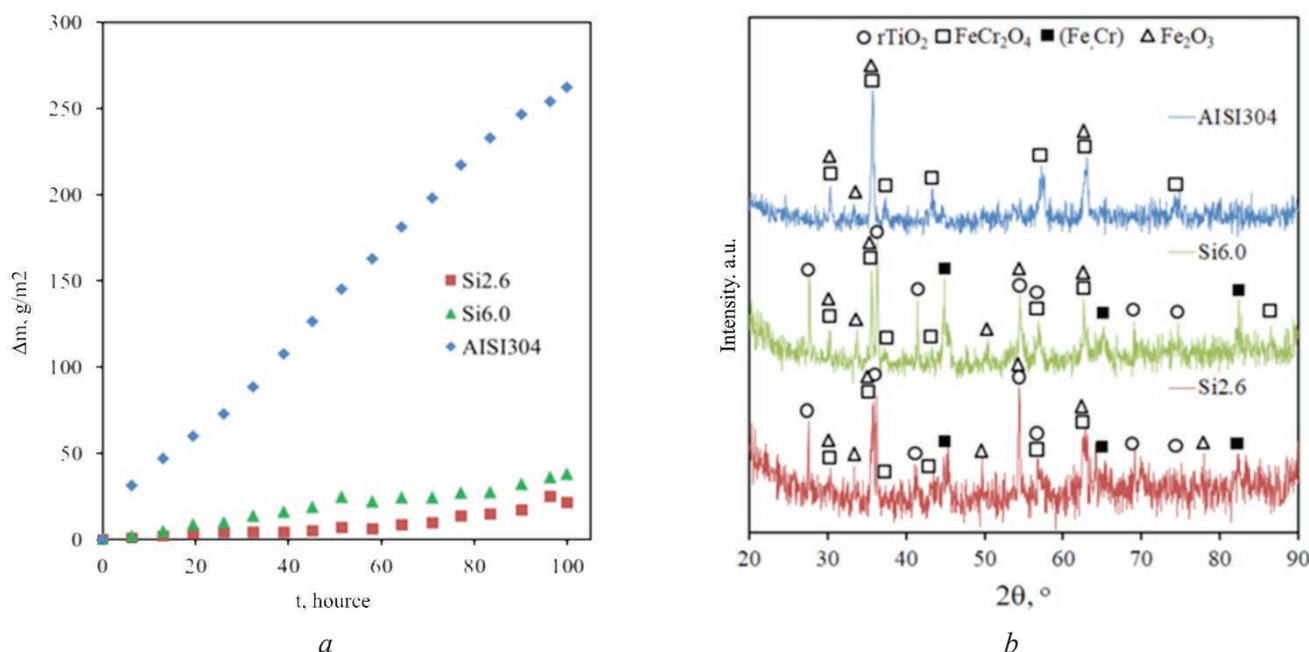


Fig. 5. High-temperature oxidation resistance of samples at a temperature of 900 °C in air (*a*) and X-ray patterns of its surface after high-temperature oxidation resistance test (*b*)

Conclusions

A technique is proposed for obtaining *Fe-Ti-Si* coatings by electrospark treatment of stainless steel *AISI304* with an anode consisting of titanium granules and 2.6 – 6 vol.% of a mixture of titanium and crystalline silicon powders. It is shown that a stable positive weight gain of the cathode is observed when the fraction of silicon in the mixture of powders does not exceed 32%. The phase composition of the coatings included: a solid solution of chromium in iron, titanium silicide Ti_5Si_3 , titanium, and silicon, which is confirmed by the energy dispersive analysis data. Titanium silicide Ti_5Si_3 is present in the coatings as separate inclusions. The thickness of the coatings ranged from 21.7 to 24.8 μm. The conducted studies have shown that *Fe-Ti-Si* coatings, prepared by a new method of electrospark deposition with a non-localized electrode with silicon and titanium powders, can increase corrosion resistance, oxidation resistance and hardness, as well as reduce the friction coefficient and wear of the *AISI304* stainless steel surface.

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Conflicts of Interest

The authors declare no conflict of interest.

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