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STRUCTURE AND PROPERTIES OF HYBRID COATINGS OF Al_2O_3 / Cr / TiN APPLIED TO A STEEL SUBSTRATE

Annotation. This article analyzes both our own results and the results obtained by Al_2O_3 / Cr / TiN with a high-current electron beam. The dependence of the structural-phase state, microhardness distribution along the depth of the surface layer and wear of the surface of powder coatings on the method of their preparation and the characteristics of the irradiated material was discovered. The results of studies of the physical and mechanical properties of titanium alloys after surface modification with electron beams are presented. The peculiarities of the formation of the surface relief and the structural and phase states of steel-based powder coatings modified by SEP and pulsed plasma flows by other authors are noted. The results of modification of hybrid coatings based on .

Key words: electrochemical corrosion, hybrid coatings, modification, surface, elemental analysis, lattice parameters.

Introduction. Currently, protective coatings on parts of electrochemical and chemical equipment are of interest. It is known that certain types of processing, such as ion implantation, ion- assisted deposition of thin films, electron beam processing, coating using high-speed plasma jets, etc. [1-3]. They cannot directly lead to the desired effect. Therefore, in recent years, combined processing methods have been used to solve applied problems, which make it possible to solve important problems in modern materials science.

On the other hand, it is known that such parts, such as acid pump blades, require high adhesion of the coating to the surface of the part, low porosity and the presence of passivating elements such as Cr , Ti , etc.

The article analyzes both our own results and the results obtained by other authors. The results of modification of hybrid coatings based on stainless steel 321 Al_2O_3 / Cr / TiN with a high-current electron beam (HCEB) are considered. The peculiarities of the formation of the surface relief and the structural and phase states of steel-based powder coatings modified by FEB and pulsed plasma flows are noted. Thus, the purpose of this work was to create multicomponent combined coatings based on Ti – Al , N / Ti – N / Al_2O_3 on a steel substrate, and to study their structure and physical and mechanical properties.

Experiment details. Three series of samples were used: 1st series – initial Al_2O_3 / Cr / TiN coating without irradiation; 2nd series treatment with high-current electron beam (HEC), current density 20 mA partial melting of the coating; 3rd series – processing of SEP with penetration of the coating and part of the substrate (current density 35 mA).

Protective coatings were prepared on thin samples (0.3 mm thick) of austenitic 321 stainless steel (composition 18 wt % Cr ; 9 wt% Ni ; 1 wt% Ti , balance Fe). The technique for applying protective hybrid coatings was as follows. Using a high-speed pulsed plasma jet on the Impulse-5 installation, a base aluminum oxide powder coating (α- $Al_2O_{3\ powder}$) with a thickness of 45 to 65 µm was applied. When applying coatings, the operating parameters of the installation were as follows: detonation frequency (number of pulses) - 4 Hz, electrical energy consumption for each plasma pulse

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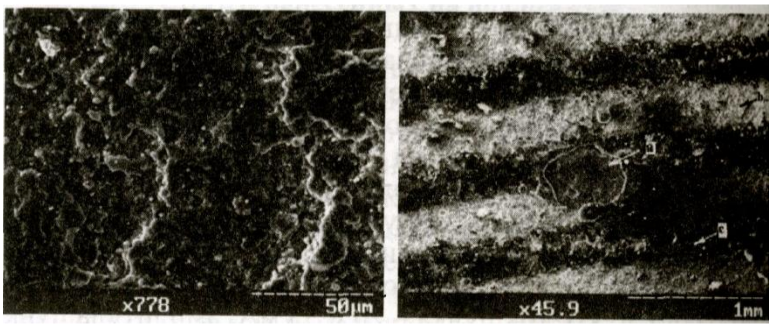
was (2.5-3.5) 10^3 J, distance from the plasmatron nozzle exit to the substrate - 40 см, spot diameter – 33 мм. To improve the corrosion properties of the surface in a solution of sulfuric acid, a layer of titanium nitride (TiN) with a thickness of 1.2 to 2 microns was applied to the $Al_2O_{3\ coating}$ in a vacuum arc source of the "Bulat - 3T" type (manufactured in Tomsk) . To analyze the elemental composition, we used backscattering of protons with an initial energy of 1.55 MeV at the Sokol accelerator. A study was also carried out using an electron scanning Auger spectrometer. The depth distribution of elements was obtained by ion sputtering of hybrid coating layers. Ar^+ ions with energies of 3.5 keV were used for sputtering . The surface study was carried out on a scanning electron microscope REMMA 102. Microanalysis was carried out on an X-ray spectrometer with an attached WDS -2, made on the basis of a semiconductor Si (Li) detector. The phase composition was analyzed by X-ray diffraction analysis using a DRON-2 installation in CuK_2 radiation (Tomsk, Russia).

For mechanical tests, tests were carried out for hardness, adhesion and corrosion resistance in sulfuric acid at temperatures of (200 - 400) ^{0}C. [1].

The results and their discussion.

Analysis of the results obtained indicates a high roughness, since films of Cr and TiN , condemned in a vacuum , repeated the relief of the coating obtained using a pulsed plasma jet (Fig. 1.a).



a b

Fig1. Surface morphology of Al_2O_3 / Cr / TiN hybrid coatings deposited using a combined method: a – initial state before electron irradiation (series No. 1);

b – after electron irradiation (series No. 2)

Note also that the formation of the "droplet" fraction, which is visible on the surface of the hybrid coating (Fig. 1.b), is due to the non-segregation deposition of TiN and Cr . Irradiation of hybrid SEP coatings leads to the formation of a "stripe" structure on the surface (alternating light (initial state of the coating) and dark (surface melted by an electron beam) stripes). On these strips, arrows indicate the places where microanalysis was carried out.

In Fig.2. Spectra taken in different areas of the coating surface are presented.

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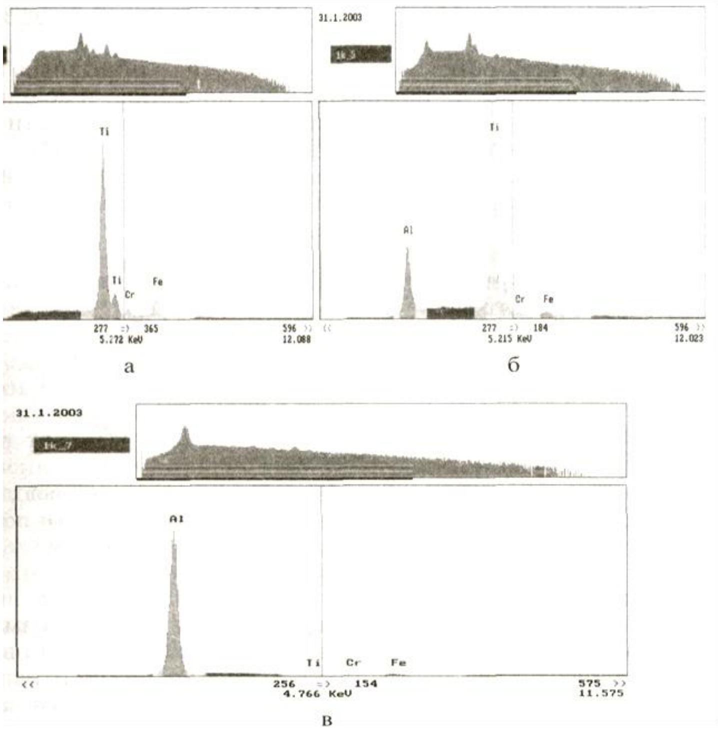
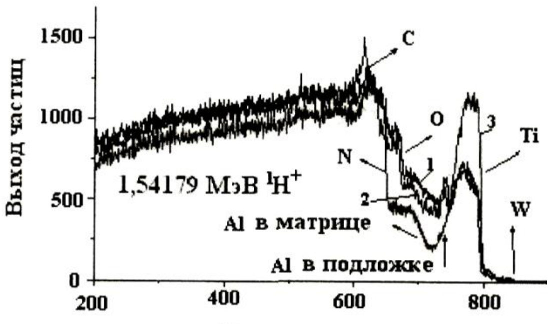


Fig2. Spectra of elemental analysis of the composition of hybrid coatings, obtained at the points indicated in Fig. 1.a - the region of the droplet fraction at the bottom of the surface crater; b – light area of the coating, not melted by the SEP; c – dark area of the coating, melted by the SEP.

The spectra obtained on round inclusions (areas of the droplet fraction) contain the following elements: Ti , Cr and Fe (see Fig. 2.a). The predominant element is titanium. It was also found that the concentration of these elements in the region of the molten drop at different points is not constant.

The elemental composition of hybrid coatings was studied in more detail using proton backscattering (BS). The interpretation of the spectrum is shown in Fig. 1.3 (presence of Al , Ti , O and N in the hybrid coatings on the surface after irradiation with HEC).



Channel number

Fig3. Energy spectrum of backscattering of protons with an initial energy of 1.55 MeV, obtained from the surface of hybrid coatings: 1 – initial state; 2 – series No. 2; 3 – episode No. 3.

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The results of the OR analysis also indicate an increase in the oxygen concentration in the coating, right up to the very surface of the samples, after irradiation with the SEP. In the surface layers of the coating without SEP treatment, the oxygen concentration is significantly lower. According to the data obtained, in coatings after electron beam melting there is a decrease in Ti content with depth (Figure 4) and at the same time its distribution profile broadens.

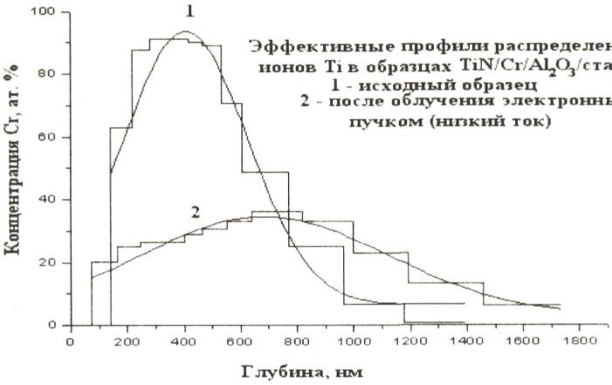

Fig4. Effective depth distribution profiles of Ti ions (profile calculated from the spectrum of OR protons using the DVBS code)

Ti concentration distribution and the Gaussian curve, the effective diffusion coefficient of titanium was calculated ($2.4 \cdot 10^{-8}$ cm ^{2}/sec).

The same samples were used to study the phase composition of Al_2O_3 / Cr / TiN hybrid coatings . The results obtained indicate that the main element of the substrate matrix is γ- Fe with a lattice parameter of 3.592 Å. XRD analysis, which provides an integral characteristic of a layer several micrometers thick, indicates that the hybrid coating is a multiphase compound. Along with the main phase of α - $Al_2O_{3\ powder}$, the presence of γ- Al_2O_3 , β- Al_2O_3 , TiN and Cr is observed . The percentage of phases in the coatings was assessed under two processing modes in Fig. 5.

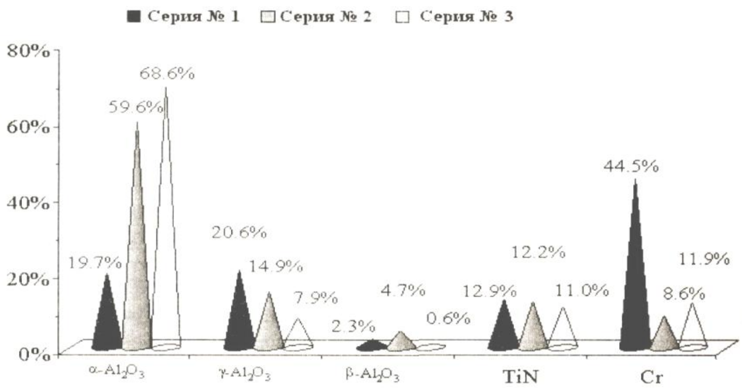

Fig5. Results of calculation of the percentage of phases on the surface of hybrid coatings.

The results of the calculations are presented.

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It can be seen that modification of the Al_2O_3 / Cr / TiN coating with the help of an EBW leads to polymer transformations γ→α and β→α in Al_2O_3 . Based on the assessments carried out, it was concluded that surface modification using SEP has a significant effect on the lattice parameters of the components of the coating elements.

Changes in the lattice parameters of α- $Al_2O_{3\ \text{were detected}}$, which are associated with the formation of residual compressive stresses: series No. 1 (without irradiation of the SEP) – a(α- Al_2O_3) = 4.773 Å, c(α- Al_2O_3) = 13.581 Å, s/a = 2.87 Å; series No. 2 (irradiation of SEP in light mode) a(α- Al_2O_3) = 4.77 Å; c(α- Al_2O_3) = 12.906 Å; s/a = 2.72; series No. 3 (irradiation with SEP in hard mode) - a(α- Al_2O_3) = 4.767 Å, s(α- Al_2O_3) = 12.875 Å, s/a = 2.71 Å. [2.4].

In the initial state (before irradiation with HEP), for example, the lattice parameter of titanium nitride is 4.264 Å, and after exposure to HEP it is 4.221 Å.

After processing the SEP with complete penetration of the combined coating, the lattice parameter is 4.247 Å. Changes in the lattice parameter Cr were detected (series 1-a(Cr) = 2.879 Å; series 2.3 – a(Cr) = 2.868 Å).

In Table 1. Corrosion test data is presented . The decrease in corrosion potential E , as well as the decrease in corrosion currents τ_{cor} and passivation τ_{pas} in the case of electronic exposure to the Al_2O_3 / Cr / Ti / N coating , indicates good corrosion resistance in sulfuric acid of the hybrid coating. It can be assumed that this increase in resistance and corrosion is associated with a decrease in through pores in Al_2O_3 with an increase in the thickness of the coating due to TiN and Cr and their mixing with the ceramic layer:

Table 1

Electrochemical Corrosion Data

No.	Sample	E (mV)	τ_{core} (mA)	τ_{pass} (mV)	E_{race} (mV)	E (mV)
1	Series 1 (Al_2O_3 + TiN)	-425	3.8	1.15	1015	1012
2	Series 2 (Al_2O_3 + TiN)	-360	0.15	0.14	970	935
3	Series 3 (Al_2O_3 + TiN)	-265	0.95	0.5	985	970

Conclusions. Thus, it can be concluded that there is an increase in hardness after FEB treatment. The hardness of the coating without electron irradiation ranged from 1.3 10^4 N/mm ^{2} to 1.42 10^4 N/mm ^{2} and after irradiation in soft mode (series 2) ≈ 1.6 10^4 N /mm ^{2} in hard mode (series 3) = 1.6 10^4 ±0.8 10^4 N/mm ^{2} .

The application of hybrid coatings from Al_2O_3 / Cr / TiN by subsequent processing of SEP in two modes leads to a change in the phase composition of the coating , mass transfer of elements, in particular Ti (microanalysis results), as well as to a significant change in service characteristics, such as adhesion, hardness and corrosion resistance in sulfuric acid.

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It is shown that the physicomechanical and chemical properties of modified materials are largely determined by the structure, phase and elemental composition of combined and hybrid coatings obtained using modern advances in ion beam and ion plasma.

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