PROPERTIES OF Al_2O_3 + Cr + TiN COATINGS AFTER ELECTRON BEAM TREATMENT

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Morphology of surface, element and phase composition of the Al_2O_3 + Cr + TiN composite coatings deposited by the combined methods using the pulse-plasma and vacuum-arc technologies with subsequent electron beam treatment were examined by scanning electron microscopy with microanalysis, back-scattered protons and X-ray diffraction analysis. It is shown that exposure to the high-power flow of electrons causes a change in structure, composition and properties of the composite coatings.

Keywords: *composite coating, pulse-plasma, vacuum-arc and electron beam treatment, electron exposure, structure and phase composition, X-ray diffraction analysis, surface modification*

Formation of protective coatings on thin (0.2–0.5 mm) walls of components of atomic, electrochemical and chemical industries is of interest at present time. These components are manufactures as a rule from stainless steels of 18-10 type or special alloys. For example, operation of such components as blades of acid pumps requires a high adhesion of coating with the component surface, insignificant closed porosity and presence on the surface of passivating elements such as chromium, aluminum, titanium etc. A pulse-plasma coating technology [1, 2] fulfils these requirements.

Protective composite coatings were made on thin (0.3 mm) samples from stainless steel in the following way. A base coating from aluminum oxide of around 45–60 μm thickness was deposited on Impuls-5 unit with the help of high-velocity plasma jet. α-Al_2O_3 powder of 27–56 μm in size was used as an initial powder.

The following modes were used for coating deposition: consumption of combustible mixture components 2 m^3/h at frequency of detonation initiation 4 Hz; consumption of electric energy per each pulse of plasma — 2500–3500 J; length of powder jet limited by barrel — 0.35 m, distance 0.04 m; diameter of a coating spot deposited per one pulse — 0.033 m.

It is shown in study [1] that the coating consisted of up to 8 % of Al_2O_3 γ-phase, amorphous phases and the rest was α-phase. Microhardness of the layer made up to 13000 MPa. Adhesion of the Al_2O_3 powder coating with a steel substrate, determined by glue procedure and scribing methods, made 40–60 MPa.

A layer of chromium of around 0.5 μm thickness and the following layer of coating from titanium nitride of 1.2–2.0 μm thickness were deposited on Al_2O_3 coating for improving corrosion properties. The coating was deposited using vacuum-arc source Bulat-5M. After that the surface was treated by a high-current electron beam (HEB) on U-212 unit [3]. Energy density of the HEB was sufficient for full fusion of the composite coating and partial fusion of the substrate layer (accelerating voltage was 30 kV, beam current — 20 mA, amplitude of beam oscillation — 15 mm, rate of surface scanning — 50 m/h (series No.1), 30 (series No.2), 3–15 (series No.3).

The peculiarities of HEB treatment lied in that the beam diameter made 0.3 mm at scanning pitch 0.9 mm. This provided formation of a streaky macrostructure on the surface, when strips with melted coating alternated with strips of non-melted coating.

Surface investigation was performed on the scanning electron microscope REM-MA-102. Analysis of obtained results (Figure 1) is evidence of surface roughness. Sufficiently low roughness of Al_2O_3 initial surface (Figure 1, *a*) is indicated. The roughness increases (Figure 1, *b*) after HEB treatment of composite coating, particularly, in the area of melted coating. HEB-treated zone contained an area of titanium drop, deposited in the vacuum-arc source Bulat-5M (Figure 1, *b*). It is to be a crater of 0.5 mm diameter at the bottom of which presence of round shape melted inclusions is observed.

Element analysis was carried out on an X-ray spectrometer made on the basis of semi-conducting Li–Si detector. The spectra, obtained on round shape inclusions of the crater bottom, contain such elements as titanium, chromium and iron, among which the titanium is dominant. It is also determined that the concentration of these elements in different points of the crater bottom is not constant, i.e. the thickness of drop fractions is also different. A presence of small aluminum concentrations was found on the clean areas of crater surface. The results obtained in areas of the coating non-melted by HEB, were the following, wt.%: 13.9 Al, 48.2 Ti, 0.03 Cr and 0.42 Fe. The coating areas after HEB fusion contain, wt.%: 55.18 Al, 0.519 Ti, 0.2 Cr and 0.83 Fe.

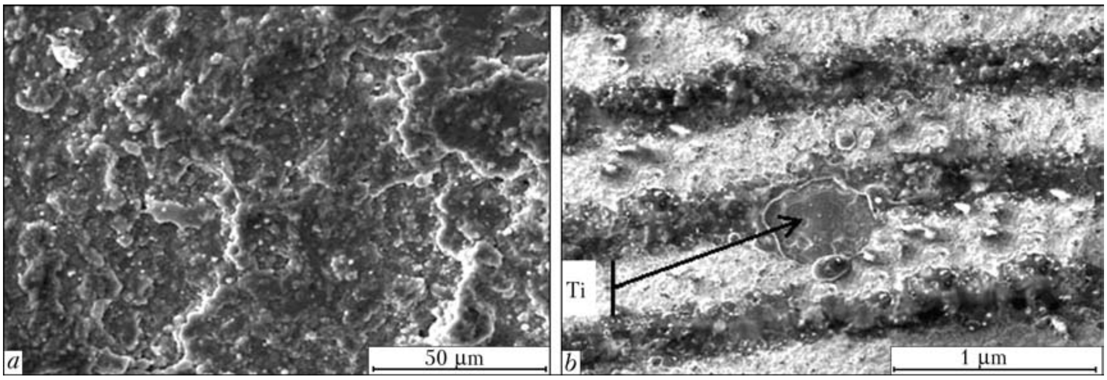

Figure 1. Microstructure of surface of the composite coatings: *a* — Al_2O_3 coating after pulse-plasma deposition; *b* — Al_2O_3 + Cr + + TiN coating after HEB treatment (series No.2)

Additional element analysis of the composite coatings was carried out by back-scattered (BS) protons method on the accelerator «Sokol» of the National Science Center «Kharkov Institute of Physics and Technology». Identification of the spectrum showed the presence of aluminum, titanium, oxygen and nitrogen on the surface of coatings in the samples subjected to fusion. BS-analysis showed an increase of oxygen concentration along whole thickness of the composite coating after HEB treatment. The concentration of oxygen is significantly lower without HEB treatment in near-surface layers of the coating. Reduction in depth of titanium content and simultaneous widening of distribution profiles are observed in the composite coatings after electron beam melting according to obtained results. An effective coefficient of titanium diffusion around $2.4\cdot10^{-8}$ cm^2/s was obtained taking into account diffusion theory and good matching of form of distribution of titanium concentration with the Gaussian curve.

Study of phase composition of the substrate for Al_2O_3 coating and surface of composite coatings Al_2O_3 + Cr + TiN were made on X-ray diffractometer DRON-2.0 in CuK_α-irradiation. Obtained results indicate the fact that the main element of substrate matrix is γ-Fe (fcc) with lattice parameter 0.3592 nm. It was determined with the help of X-ray diffraction analysis that the coating surface is to be a multiphase combination. Presence of such phases as γ-Al_2O_3, β-Al_2O_3, TiN and Cr is observed simultaneously with the main phase of corundum powder.

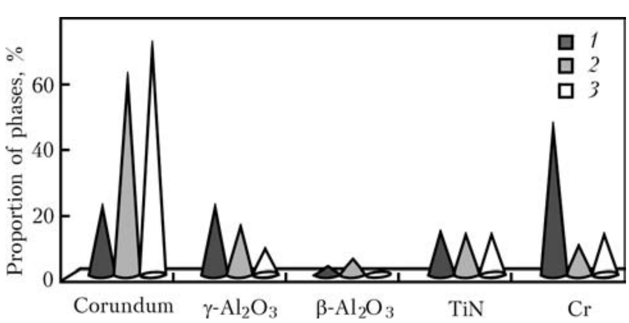

Figure 2. Calculation results of percent proportion of phases of the composite coatings of series Nos. 1–3 (*1*–*3*)

Evaluation of percent proportion of phases in the coatings with somewhat changed modes of modification (Figure 2) was carried out. Analysis of data is evidence of the fact that modes of coating and energy of HEB treatment have significant influence on the phase composition of coating material. Most probably that, in particular, the high-energy pulse-plasma deposition of coatings and electron beam treatment are the reason of occurrence of polymorphic transformations of $\gamma \rightarrow \alpha$ and $\beta \rightarrow \alpha$ type in Al_2O_3. Increase of concentration of the initial composition of α-Al_2O_3 depositing powder is observed at increase of time of electron beam treatment.

Hardening of Al_2O_3 particles with a speed not more than 10^7 K/s in air [4] is required for preserving γ-phase, nucleuses of which were formed in a melt. α-phase can be obtained in air if the particles are cooled with the speed of less than $5\cdot10^4$ K/s.

Based on calculations carried out a conclusion was made about significant influence of HEB modification of surface on a change of parameters of lattice of surface constituent elements (series No.1: a = = 0.447 nm, c = 0.136 nm, c/a = 2.87; series No.2: a = 0.477 nm, c = 0.129 nm, c/a = 2.72; series No.3: a = 0.477 nm, c = 0.129 nm, c/a = 2.71).

The parameter of nitride titanium lattice equals 0.426 nm in the surface initial state. It makes 0.422 nm after surface treatment with low dose of HEB. Size of titanium nitride lattice makes 0.425 nm in the composite coatings after surface treatment with high dose of HEB. The results obtained for lattice parameters of chromium sublayer were the following: for series Nos 1–3 a = 0.288, 0.287 and 0.288 nm, respectively.

Thus, aluminum, titanium, chromium, nitrogen and oxygen are the main constituents of the surface. Initial material of the coating from aluminum oxide powder undergoes a series of phase transformations [4, 5] after composite coating. The X-ray diffraction analysis showed the presence of aluminum oxide in the form of three modifications (α-, β-, γ-Al_2O_3) in the coating: chromium and titanium nitride.

Modification of the composite coating with the help of HEB results in recovery of corundum phase and increases the coefficient of titanium diffusion. The material of composite coating after HEB melting has higher density and consists of high temperature phases of aluminum oxide alloyed by titanium and chromium. It is expected that this coating will have increased corrosion properties under operation in active high temperature media.

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NEWS

THREE IN ONE

A welding multisystem (chopper) providing three welding processes (MIG/MAG, TIG and MMA) is proposed by SELMA company.

VD-320 KS welding multisystem is designed:

• for semi-automatic gas-shielded welding (MIG/MAG-DC mode) by 1.0–1.6 mm diameter steel wire using wire feed mechanism manufactured by OJSC SELMA Company as well as for semi-automatic welding of aluminum and its alloys in argon atmosphere by 1.2 mm diameter OK 18.01, OK 18.04, AMg-5 wires in 130–180 A current range at arc voltage of 19–24 V;

• for non-consumable electrode argon arc welding at direct current (TIG-DC mode) at completing with BU-TIG control unit;

• for consumable covered electrode arc welding of products from carbon and alloyed steels (MMA-DC mode).

Main advantages:

• low consumption of energy in comparison with traditional welding sources for 300 A;

• rectifier has a built-in block for reduction of open-circuit voltage increasing safety during performance of welding operations in MMA mode;

• possibility of carrying out of three welding processes: MIG/MAG-DC, TIG-DC, MMA-DC;

• smooth adjustment of welding current;

• digital display of welding current and voltage;

• adjustment of short-circuit current in MMA mode;

• adjustment of time of «Hot start» for providing stable arc initiation in MMA mode;

• possibility of connection of a remote-control station for adjustment of welding current in MMA mode;

• availability of thermal protection from overloading;

• availability of socket (36 V) for a gas heater connection;

• state-of-the-art element base;

• small weight and overall dimensions in comparison with traditional welding sources for 300 A.