ALTERAÇÕES ESTRUTURAIS E DE FASE NAS CAMADAS DE SUPERFÍCIE DA COBERTURA MULTICOMPONENTE RESISTENTE AO CALOR DO REVESTIMENTO DE ÍONS DE PLASMA NI-CR-AL-Y APÓS MODIFICAÇÃO POR FEIXES DE ELÉTRONS DE CORRENTE FORTE COM DURAÇÃO DE MICROSSEGUNDOS**STRUCTURAL AND PHASE CHANGES OF THE SURFACE LAYERS OF HEAT-RESISTANT MULTICOMPONENT COATING OF ION-PLASMA COATING Ni-Cr-Al-Y AFTER MODIFICATION BY HIGH-CURRENT ELECTRON BEAMS MICROSECOND DURATION****СТРУКТУРНО-ФАЗОВЫЕ ИЗМЕНЕНИЯ ПОВЕРХНОСТНЫХ СЛОЕВ ЖАРОСТОЙКОГО МНОГОКОМПОНЕНТНОГО ПОКРЫТИЯ ИОННО-ПЛАЗМЕННОГО ПОКРЫТИЯ NI-CR-AL-Y ПОСЛЕ МОДИФИЦИРОВАНИЯ СИЛЬНОТОЧНЫМИ ЭЛЕКТРОННЫМИ ПУЧКАМИ МИКРОСЕКУНДНОЙ ДЛИТЕЛЬНОСТИ**

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RESUMO

O desenvolvimento da engenharia aeroespacial e da engenharia mecânica depende diretamente do desenvolvimento de novos materiais metálicos e tecnologias avançadas. O problema de criação dos materiais e seus tipos de processamento para aumentar o nível de propriedades operacionais é relevante em relação à complicação e à rigidez das condições de trabalho das tecnologias modernas. Uma das tarefas mais importantes da construção de aeronaves modernas é aumentar as propriedades operacionais da camada superficial. O objetivo do artigo é elucidar a influência de feixes de elétrons de corrente forte com duração de microssegundos nas alterações estruturais e de fase nas camadas de superfície da cobertura multicomponente resistente ao calor do revestimento de íons de plasma Ni-Cr-Al-Y sob várias condições. Utilizando um complexo de métodos de pesquisa metalofísica, os estados físico-químico e de fase estrutural da camada superficial foram estudados antes e após a modificação das amostras. Essas amostras foram revestidas com revestimentos de plasma de íons condensados resistentes ao calor de três composições diferentes usando nove feixes de elétrons de alta corrente em nove modos com diferentes valores de energia eletrônica e número de pulsos no intervalo selecionado de energia eletrônica. Uma análise das mudanças na fase estrutural que ocorrem durante a modificação foi realizada. Amostras cilíndricas de alvos feitos de liga de níquel resistente ao calor ZhS36 revestida com revestimento multicomponente condensado por íon-plasma SDP-2 + VSDP-16. Essas amostras foram usadas de acordo com a tecnologia serial, ambas com subsequente modificação usando feixes de elétrons de alta corrente e sem modificação. Verificou-se que o cromo no estado inicial está desigualmente distribuído: o cromo está presente nas partículas, a matriz é empobrecida no cromo. Os resultados do estudo podem ser úteis para os cientistas que estudam as propriedades do revestimento de íons de plasma multicomponente resistente ao calor Ni-Cr-Al-Y e o efeito de feixes de elétrons de corrente forte nele, bem como para a fabricação de materiais mais resistentes ao calor em engenharia aeroespacial e engenharia mecânica.

Palavras-chave: *irradiação de feixe de elétrons de corrente forte, revestimentos de íões de plasma condensado, estado estruturais e de fase, estudo de microestrutura.*

ABSTRACT

The development of aerospace engineering and mechanical engineering directly depends on the development of new metal materials and advanced technologies. The problem of creating materials and their types of processing to increase the level of operational properties is relevant in connection with the complication and tightening of working conditions of modern technologies. One of the most important tasks of contemporary aircraft construction is to increase the operational properties of the surface layer. The purpose of the article is to elucidate the effect of high-current electron beams of microsecond duration on changes in the surface layers of the heat-resistant multicomponent ion-plasma coating Ni-Cr-Al-Y under various conditions. Using a complex of metallophysical research methods, the physicochemical and structural-phase states of the surface layer were studied before and after modification of the samples. These samples were coated with heat-resistant condensed ion-plasma coatings of three different compositions using nine high-current electron beams in 9 modes with different values of electron energy and number of pulses in the selected interval of electron energy. An analysis of the structural phase changes occurring during modification was carried out. Cylindrical samples of targets made of heat-resistant nickel alloy ZhS36 coated with ion-plasma condensed multicomponent coating SDP-2 + VSDP-16. These samples were used according to serial technology, both with subsequent modification using high-current electron beams and without modification. It was found that chromium in the initial state is unevenly distributed: chromium is present in the particles; the matrix is depleted in chromium. The research results can be useful for scientists to study the properties of heat-resistant multicomponent ion-plasma coatings Ni-Cr-Al-Y and the effect of high-current electron beams on it, as well as for the manufacture of more heat-resistant materials in aerospace engineering and mechanical engineering.

Keywords: *high-current electron beam irradiation, condensed ion-plasma coatings, structural-phase state, microstructure study.*

АННОТАЦИЯ

Развитие аэрокосмической техники и машиностроения напрямую зависит от разработки новых металлических материалов и передовых технологий. Проблема создания материалов и их видов обработки для повышения уровня эксплуатационных свойств, является актуальной в связи с усложнением и ужесточением условий труда современных технологий. Одной из важнейших задач современной авиации является повышение эксплуатационных свойств поверхностного слоя. Цель статьи является исследование влияния высокопоточных электронных пучков микросекундной длительности на изменения поверхностных слоев термостойкого многокомпонентного ионно-плазменного покрытия Ni-Cr-Al-Y в различных условиях. Были проведены исследования структурно-фазового состояния цилиндрических образцов из жаропрочного никелевого сплава ZhS36 с нанесенным ионно-плазменным конденсированным многокомпонентным покрытием SDP-2 + VSDP-16 как с последующей модификацией с использованием высокопоточных электронных пучков по различным режимам облучения, так и без модификации. Было обнаружено, что хром в исходном состоянии распределен неравномерно: хром присутствует в частицах, матрица обеднена хромом. Результаты исследования могут быть полезными для ученых, исследующих свойства термостойкого многокомпонентного ионно-плазменного покрытия Ni-Cr-Al-Y и влияние высокопоточных электронных пучков на него, а также для изготовления более термостойких материалов в аэрокосмической технике и машиностроении.

Keywords: *высокопоточное электронно-лучевое облучение, конденсированные ионно-плазменные покрытия, структурно-фазовое состояние, исследование микроструктуры.*

1. INTRODUCTION:

The development of aerospace engineering, including aviation weapons, as well as other branches of engineering, is mostly provided by the development of new metal materials and advanced technologies (Kablova, 2006; Terentyev *et al.*, 2012; Galoyan *et al.*, 2014; Muboyajyan and Budinovsky, 2017; Nesterov, 2018). With ever-increasing complication and toughening of working conditions of modern technology, the problem of creating materials and their processing types, which along with high

strength, increase the level of performance properties has become extremely urgent (Tishkov and Firsanov, 2011; Baranov *et al.*, 2007; Kimura *et al.*, 2016; Abraimov, 2017; Korsmik *et al.*, 2018; Bogdan *et al.*, 2019). It is known that the most loaded and expensive parts and assemblies are parts and assemblies of gas turbine flow path (first of all, the blades and disks of the compressor and turbine), thus, increasing the operational properties of the surface layer has become the most important task of modern aircraft manufacturing (Voyevodin *et al.*, 2017; Yurishcheva *et al.*, 2018; Sapozhkov and

Burakova, 2018; Rechenko *et al.*, 2019; Imashuku and Wagatsuma, 2019; Tarellyk *et al.*, 2019).

The solution to this problem is attempted using several approaches: development of promising, highly alloyed, polycrystalline and single crystal alloys; modernization of methods for manufacture, molding, and processing of products and blanks; development of new ways of surface treatment of parts and applying various protective coatings to their surface, including coatings from nanomaterials (Muboyajyan *et al.*, 2012; Matveev *et al.*, 2013; Ovchinnikov and Yakimov, 2016; Gadlov *et al.*, 2017; Shaidurova *et al.*, 2017; Buckhurst and Kaspersen, 2017; Batsikadze *et al.*, 2017). Besides, intensive research is being performed on the creation of all-metal discs with blades, and these technologies have already found widespread application at leading enterprises in the aviation industry, both in several European countries and in the USA.

At present, special attention is paid to the development of high-intensity methods of surface engineering of engineering parts of a fairly wide range, as well as their extremely rapid implementation of technological processes created on their basis in the industry. One of the demanded tools for surface engineering can be using high-current pulsed electron beams (HCPEB) of microsecond duration to modify the surface and near-surface layers of parts (Novikov *et al.*, 2010; Uglov *et al.*, 2011; Shulov *et al.*, 2012; Gromov *et al.*, 2013; Shulov *et al.*, 2014; Shulov *et al.*, 2015). The study of the effect of HCPEB irradiation on the surface of the layers of the heat-resistant multicomponent ion-plasma coating Ni-Cr-Al-Y under various conditions, as well as the analysis of structural-phase changes occurring upon modification, were the objectives of this work. Also, cylindrical target samples of heat-resistant nickel alloy ZhS36 coated with ion-plasma condensed multicomponent coating SDP-2 + VSDP-16 were the objects of research.

2. MATERIALS AND METHODS:

Cylindrical target samples of heat-resistant nickel alloy ZhS36 coated with ion-plasma condensed multicomponent coating SDP-2 + VSDP-16 were used with serial technology as with subsequent modification using high-current electron beams (samples under conv. No. 3 and No. 4), and without modification (about samples per conv. No. 1) (Paykin *et al.*, 2008; Bytsenko *et al.*, 2015; Bytsenko *et al.*, 2017a). Then, irradiation was carried out on a unique complex automated electron-beam installation "RITM-SP" in 9 modes

with electron energies in the range from 20 to 25 keV, namely 20, 22 and 25 keV with the number of pulses 5, 10 and 20, respectively each value of the electron energy, followed by stabilizing heat treatment (Hsu *et al.*, 2016; Kudryashov *et al.*, 2016; Shmorgun *et al.*, 2016; Tarasenko *et al.*, 2016; Shmorgun *et al.*, 2017a; Min *et al.*, 2017; Shmorgun *et al.*, 2017b; Sinitsyn *et al.*, 2017; Fähsing *et al.*, 2017; Zakirov *et al.*, 2017; Kozyr *et al.*, 2018; Blinkov *et al.*, 2018; Rechenko *et al.*, 2018; Kashapova *et al.*, 2018; Guchenko *et al.*, 2019).

Complex research of the physicochemical and structural phase states was carried out using the following methods (Bytsenko *et al.*, 2016; Bytsenko *et al.*, 2017b): X-ray diffraction analysis to determine the level of residual stresses and phase composition of surface layers on Rigaku D / MAX-2500 diffractometer. The diffraction patterns were registered in monochromatized Cu K alpha radiation in the Bragg-Brentano geometry. The scanning range in the range of angles 2θ 10 degrees - was 100 degrees. The diffraction patterns were decoded using the specialized Jade 5 program and the PDF-2 database. The work was carried out following MM 1.595-17-222-2004. The calculation of residual stress values was performed according to the $\sin^2\psi$ method. Stresses were determined automatically according to 10 values of the angle ψ in the range of angles from 0 degrees to 40 degrees from angular position by approximating the function of Gauss; study of the surface condition using laser confocal microscope LEXT OLS 3100 with subsequent processing of the obtained images using software; raster electron microscopy. This research method was performed using VERIOS 460 scanning electron microscope with an X-MAX 80 energy dispersive X-ray microanalyzer, quantitative X-ray micro spectral analysis (MRSA) to determine the local quantitative and qualitative composition of the material of the surface and subsurface layers before and after modification using HCPEB.

3. RESULTS AND DISCUSSION:

After modifying the target surface according to the selected modes during visual inspection, it was established that, depending on the irradiation mode, the samples have different surface: with high electron energy and a large number of pulses, the surface is shiny, when irradiated with lower-energy electrons and with fewer vibrations, the surface of the samples matte.

An analysis of the microstructure of the

coating after surface modification (by optical microscopy) revealed the presence of a large number of chips of the modified layer when the surface was irradiated with electrons with an energy of 25 keV, regardless of the number of pulses. The thickness of the modified layer increases with an increasing number of pulses: at 5 pulses, the thickness of the modified layer is ~ 5 μm ; at 20 pulses, it is ~ 10 μm . When the surface is irradiated with electrons with energies of 22 and 20 keV, shearing of the modified layer occurs at number of pulses of 20. The thickness of the modified layer at 22 keV was ~ 10 μm , at 20 keV - ~ 5 μm . When the electron energy was 22 keV and 20 keV and the number of pulses was 10, the modified layer had a thickness of about 5 μm . At the number of pulses 5, the modified layer has thickness of less than 5 μm , and at an electron energy of 20 keV, the surface was altered by fragments (there were areas, especially in depressions on the surface of samples where there was no layer). Based on performed studies, the mode was chosen: electron energy - 20 keV, and several pulses was 10.

The study of the surface of samples after irradiation using a laser confocal microscope showed a significant decrease in surface roughness of the samples compared to the original samples (without modification). It should be mentioned that for the selected irradiation mode and final heat treatment, only single cracks were observed, i.e., this mode is optimal and allows to level technological defects during coating that was present on the original samples (the presence of droplet phase, pores, etc.) Figure 1). To assess microstructure on samples of ZhS32 single-crystal alloy with multicomponent coating SDP2 + VSDP16 before and after obtaining high-current beams (HCPEV), the level of residual stresses was estimated. To establish a phase composition of both the initial coating and the modified coating, a qualitative phase analysis was performed using x-ray diffraction analysis.

In the diffractogram of the sample (conv. No. 1) with the initial coating, the main phase is the β phase (NiAl) of cubic modification with a lattice period $a = 0.286$ nm. The second phase of gamma-prim (Ni_3Al) with a lattice period $a = 0.357$ nm and chromium carbide Cr_{23}C_6 . Also, the diffraction pattern contains chromium carbide (Cr_3C_2), the intensity of diffraction lines of this phase is weak and traces of tungsten oxide (WO) (Figure 2). From the diffractogram of the irradiated sample (conv. No. 3), it can be seen that the main phase is the β phase (NiAl) with a lattice period $a = 0.286$ nm. There are also weak diffraction lines in

insufficient numbers for unambiguous identification (Figure 3a). The diffraction pattern of another irradiated sample (conv. No. 4) contains the β phase (NiAl) with a lattice period $a = 0.286$ nm and weak diffraction lines of γ 'phase (Ni_3Al) with a lattice period $a = 0.357$ nm and a Ni_3 phase Y (Figure 3b).

As can be seen from the data presented after irradiation, the structure of the surface layer is represented mainly by the β phase, which determines the heat resistance of the coating. Extra straining (OH) in all samples is tensile: for the specimen Conv. No. 1 - $+384 \pm 89$ MPa, for the sample Conv. No. 3 - $+853 \pm 112$ MPa, for the sample Conv. No. 4 - $+424 \pm 62.5$ MPa. The level of OH in the original sample Conv. No. 1 and the irradiated sample is quite close (taking into account possible deviations), and the OH value in sample No. 3 is much higher than in the other two samples (conv. No. 1 and conv. No. 4). The presence of high level of residual tensile straining is associated with presence of defects before irradiation (the presence of a droplet phase, pores, and low cohesion of individual particles and adhesion of the coating to the substrate — in this case, the first coating layer — SDP2) acts as a substrate, as well as defects formed during irradiation of the crater and peeling of the coating due to poor adhesion of dusty coating with the substrate. The data obtained are presented in Table 1. Single cracks are observed in all coatings, which is associated with the presence of tensile stresses after treatment with high-current electron beams. The increase in electron energy and some pulses leads to an increase in the number of cracks in the coating.

The fact of the peeling of the coating after irradiation of the sample Conv. No. 3 was discovered after ultrasonic washing (petroleum ether, 10 min) immediately after irradiation. Then, a study was made of the microstructure of the coating material and X-ray spectral analysis (MRSA) of the surface-modified layer of irradiated samples, as well as samples without irradiation, using optical and scanning electron microscopy (SEM). Studies have shown that after YCPEB irradiation there was a modified layer, and it represented a slightly etched surface layer with depth of 3 to 5 μm , the microstructure of which was mainly represented by beta phase, the layer was homogeneous, had no defects in the form of pores, and there were no laminations with discontinuities. When studying the microstructure of the surface layer using SEM, it was found that modified layer consists of three zones: the first is external (from 2 to 3 μm), the second is

intermediate (from 0.4 to 0.6 μm) and 3rd (from 0.6-0.8 μm) - the zone of thermal influence, transitional to the bulk of the substrate (Figure 4).

The distribution of alloying elements in the coating was studied by X-ray mapping, which made it possible to determine the spatial distribution of alloying elements. Analysis of distribution maps of chemical elements demonstrates that nickel and aluminum are evenly distributed in the coating. Chromium in the initial state is unevenly distributed: chromium is present in particles; the matrix is depleted in chromium. After irradiation, the surface layer (irradiated layer) has a uniform distribution of all the above elements (Figure 5).

4. CONCLUSIONS:

The comprehensive approach to the analysis of physicochemical and structural-phase states of the material of the samples before and after irradiation with high-current pulsed electron beams using methods of analytical microscopy and x-ray diffraction analysis was implemented. X-ray diffraction analysis helped to determine the level of residual stresses and phase composition of surface layers on the Rigaku D / MAX-2500 diffractometer. Analytical microscopy was used in obtaining images to assess the local quantitative and qualitative structure of the materials. Based on the research, the optimal mode for modifying heat-resistant multicomponent condensed ion-plasma coating SDP2 + VSDP16 (the electron energy was 20 keV, the number of pulses was 10) was chosen. It was established that, when irradiated with high-current electron beams, a modified layer with a depth of up to 5 μm was obtained, consisting of three zones: external, intermediate, and heat-affected zone, which transitions into the bulk of substrate.

The dependence of the irradiation regime on the surface of the samples was found. With high electron energy and a large number of pulses, the surface is shiny; when irradiated with lower-energy electrons and fewer pulses, the surface of the samples is dull. It was concluded that the number of microcircuits of the modified layer upon irradiation of the surface by electrons and its independence on the number of pulses. The pattern was observed in the following: the thickness of the modified layer increases with increasing number of pulses. During X-ray diffraction analysis, the structure of the modified layer was found to be represented mainly by the β phase (NiAl), and there were also weak diffraction lines of γ 'phase (Ni₃Al).

Studies have shown that the presence of a high level of residual tensile stress is associated with the presence of defects before irradiation acting as a substrate, and defects formed during irradiation of the crater and peeling of the coating. It was found that the modified layer consists of three zones: external, intermediate, and heat-affected zone, which passes into the bulk of the substrate. When conducting spatial mapping on samples in the initial state (without irradiation), it was established that nickel and aluminum were evenly distributed in the coating. Chromium was unevenly distributed, it was present in the particles, and the matrix was depleted in chromium. After irradiation, the surface layer had a uniform distribution of all of the above elements.

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Figure 1. The surface of the samples coated with SDP 2 + VSDP 16: a) - the original sample conv. No. 1; b) the sample after irradiation conv. No. 3

Figure 2. Diffraction spectrum and stroke - diagram of initial sample No. 1

Figure 3. Diffraction spectra and bar charts of irradiated samples with SDP2 + VSDP16 coating, irradiation mode $E = 20$ keV, number of pulses 10: a) sample conv. No. 3 and b) sample conv. No. 4

Table 1. Results of measuring residual stresses in the samples before and after irradiation

No.	Conventional sample number	condition	The value of residual stresses level, MPa
1	1	Initial state	+384 ± 89
2	3	After irradiation according to E = 20 keV mode, number of pulses 10	+853 ± 112
3	4	After irradiation according to E = 20 keV mode, number of pulses 10	+424 ± 62.5