

MODEL COMPOUND MIXTURES FOR STUDYING THE MAIN TRENDS OF VOLATILE ORGANIC COMPOUNDS CONVERSION IN PROCESSES OF AIR CLEANING BY PULSED DISCHARGES*

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Pulsed discharges of atmospheric pressure are widely used to clean the air from ecotoxic volatile organic compounds (VOC). These discharges are the source of a non-equilibrium low-temperature plasma. The use of such type of plasma allows to carry out processes without substantial heating of the air flows, that promises energy savings. However, due to the wide variety of chemical properties of VOCs, they are removed with different efficiencies and different mechanisms. In addition, a large variety of the discharges and experimental realizations under study does not allow to compare their energy efficiency correctly. It can be said that there is a need to develop a unified approach for the study of air purification processes from VOC vapors and their energy efficiency. Previously, a method of standard mixtures was proposed, which allows to estimate the qualitative parameters of the plasma (relative reactivity of the components) and the energy parameters of the processes [1,2]. The main idea of the method is to use specially selected mixtures of components and gas media to identify the main laws and processes taking place in the air stream processed by the plasma. There is a reason to believe that this approach can be recognized as universal.

This report provides an overview of the development of this method over the past 2 years. A base part of the experimental setup, a high-voltage generator was used, which formed a voltage pulse of negative polarity in air with a current amplitude of 200 - 400 A; voltage pulse amplitude of 100 - 120 kV; voltage pulse full width at half maximum of 15 - 30 ns; pulse energy of 0.4 - 0.6 J and pulse repetition frequency was 10 Hz. For an additional comparison, widely studied compounds were used. The following groups of model mixtures in various gaseous media based on N₂ and O₂ were selected:

1. **Model mixture (I)** of components: hexane (C₆H₁₄), toluene (C₆H₅CH₃), acetone (CH₃COCH₃), ethyl acetate (CH₃COOC₂H₅), butyl acetate (CH₃COOC₄H₉) allows to access the relative reactivity of components of well-known solvents and to identify the main channels for their removal, as well as to estimate the energy yield of the removal process.

2. **Model mixture (II)** of components: carbon tetrachloride (CCl₄), chloroform (CHCl₃), methylene chloride (CH₂Cl₂), dichloroethane (ClCH₂CH₂Cl) makes it possible to assess the relative reactivity of halogen-containing components and to identify the main channels for their removal.

3. **Model components (III)**: styrene (C₆H₅CHCH₂), MMA (CH₂CCH₃COOCH₃), TCE (CCl₂CHCl) and PCE (CCl₂CCl₂) as unsaturated compounds both in the individual state and in combination with separate components of mixtures **I** and **II** (toluene and CCl₄) for comparison.

The use of mixtures **I** and **II** in gas media with different oxygen content showed a low contribution of processes involving oxygen and ozone into the removal process. Moreover, oxygen serves as a deactivator of active plasma particles. On the other hand, components from list **III**: styrene and MMA are efficiently removed by ozone, therefore O₂ increases efficiency. PCE and TCE are removed by mechanisms both with the participation of oxygen and ozone, and by mechanisms with the participation of active forms of nitrogen. Thus, it has been shown that reactive oxygen species, including ozone, are the main active reactants in the case of unsaturated compounds (styrene and MMA and partly for TCE and PCE), but ineffective at removing many other components.

Obviously, these model mixtures and conditions can be used to compare various types of discharges. The found regularities will be useful for the development of new air purification technologies.

REFERENCES

- [1] I. E. Filatov, V. V. Uvarin, D. L. Kuznetsov // Technical Physics. – 2018. – Vol. 63. – No 5. – P. 680-688.
- [2] I. E. Filatov, V. V. Uvarin, D. L. Kuznetsov // Journal of Physics: Conference Series. – 2019. – Vol. 1147. – No. 1. – P. 012122.

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THE USE OF MAGNETRON SPUTTERING TO SYNTHESIS BORIDE NEUTRON-ABSORBING COATINGS*

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When storing spent nuclear fuel, the presence of α -radioactive nuclides can lead to the appearance of neutrons in the (α, n) – reaction. These neutrons can cause the regeneration of a part of the fuel. In this connection, it is necessary to take measures to reduce the risk of a radiation accident. For the manufacture of protective containers used neutron-absorbing constructional materials [1].

As the cost of bulk doping with absorbing elements and related technological difficulties grows, the use of neutron-absorbing coatings becomes most preferable. The most widespread coatings of amorphous boron carbide [2]. We have proposed the idea of using coatings with the B-Ti system for neutron absorption. In the previous work, the effectiveness of this approach was shown [3].

This paper presents the results of experiments on the magnetron sputtering of titanium boride coatings on the surface of structural stainless steel. Used magnetron sputtering system DC VUP-5M. For sputtering, a composite cathode target Ti + B₄C was used. As a plasma-forming gas was used Ar, the residual pressure of which was $\sim 2 \times 10^{-3}$ Pa. The voltage applied to the cathode target varied from 200 to 600 V, with a current of up to 70 A. The resulting coating was a two-phase titanium boride compound: the matrix is Ti₂B₅ (~ 70%), implantation phase - TiB₁₂ (particles with a diameter of up to 0.5 μ m). At the maximum applied voltage within 30 minutes, a coating ~ 370 nm thick was formed. The coating has a columnar structure characteristic of magnetron deposition (Figure 1).

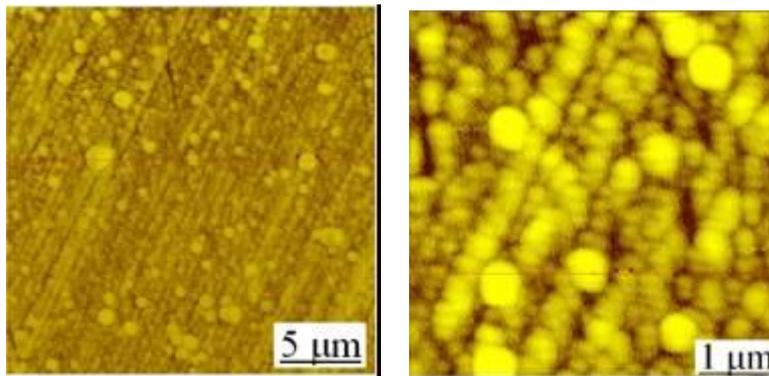


Fig. 1. Scanned (SPM) image of titanium boride coating surface

REFERENCES

- [1] Licensing Requirements for the Independent Storage of Spent Nuclear Fuel, High-Level Radioactive Waste and Reactor-Related Greater Than Class C Waste– Energy, 10 CFR Part 72, 2006.
- [2] E. S. Dvilis, O. L. Khasanov, V. N. Gulbin and act. // The Journal of The Minerals, Metals & Materials Society (TMS). – 2016. – Volume 68. – № 3. 908-919.
- [3] A.S. Larionov, L.V. Chekushina, E.E. Suslov// Materials Science Forum. – 2019. – 945. – 660-664.

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STRUCTURE OF AN ELECTRO-EXPLOSIVE COATING OF THE ZNO-AG SYSTEM*

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The analysis of the results obtained shows that the formed coating is a homogeneous composite material. According to the structural morphology and the etching contrast, the forming coating consists of a light silver matrix and dark ZnO inclusions with dimensions varying from 0.3 to 0.5 μm . The elemental composition of the coating was analyzed by X-ray microanalysis methods. Analyzing the results presented it can be noted that the concentrations of copper, zinc and silver in the coating vary slightly in its thickness. This fact also indicates the structural homogeneity of the coating obtained.

Atomic force microscopy was performed in the coating layer located at a distance of 10 μm from the coating surface, as well as at the interface between the coating and the copper substrate. Since the electro-explosive coating is formed by a silver matrix and ZnO powder particles located in it, small particles of ZnO powder can be crumbled out of the matrix during the preparation of thin sections. In this case, pores, i.e. dark areas 30 to 100 nm deep and 2 to 5 nm wide are formed at the site of fallen particles. ZnO particles are dispersed to 2 ... 5 nm in the process of an electric explosion during the formation of a pulsed plasma jet of products of the electric explosion of conductors.

Separate large particles of various shapes with sizes ranging from 10 to 15 nm are also detected. These ZnO particles do not crumble out of the silver matrix during the preparation of the thin section; they are sharply highlighted in color, i.e. they are lighter than the matrix. They are randomly located in a silver matrix. Large particles have a complex structure. They are composed of spheres (globules) with a diameter of 2 to 5 nm (these are small spherical particles described above). The ratio of the silver matrix, large and small particles of ZnO powder is 0.6: 0.15: 0.25. Taking into consideration the fact that large ZnO particles consist of smaller globular ZnO particles then the ratio of the silver matrix and the inclusions of ZnO powder is 0.6: 0.4. This ratio is proportional to the content of ZnO powder and silver foil used for electro-explosive coating. The average roughness of the surface profile of the ZnO-Ag system coating is 100 nm.

Thus, it was possible to identify an important structural element - the ZnO globule, i.e. a spherical particle with a diameter of 2 to 5 nm. There is a multi-level hierarchical structure of the ZnO-Ag coating system, which is based on uniform spherical ZnO particles with a diameter of 2 to 5 nm. The fact that the ZnO inclusions, located in the silver matrix are made of a single structural unit is a very important argument in favor of the fractal mechanism for the formation of an electro-explosive coating. Such particles constitute the first hierarchical level of the structure of the electro-explosive coating of the ZnO-Ag system. The second hierarchical level consists of globules - large particles of various shapes with sizes ranging from 10 to 15 nm, which, in turn, form the sediment of micron-sized particles of irregular shape, detected by scanning electron microscopy.

At the boundary between the coating and the copper substrate there are visible dark depressions ranging in size from 10 to 15 nm. Aforementioned large ZnO particles crumbled out of them. In addition, surface periodic structures appear at the coating/substrate boundary in a silver matrix. The secant held perpendicular to these structural formations suggests that the wavelength in them is on average 3 nm. The structures are the residual nanorelief of the surface. After the end of the impact of a pulsed plasma jet of products of the electrical explosion of conductors on a substrate and cooling the surface, the induced relief is fixed in the form of surface periodic structures. They can be formed due to evaporation, melting of the surface and displacement of the melt by excess vapor pressure, thermocapillary phenomena and thermochemical reactions, thermal deformations, the emergence and development of various instabilities such as Rayleigh-Taylor, Kelvin-Helmholtz, Marangoni and others. In general, the phenomenon is universal and is an example of self-organization in a system with no initially selected directions and structures. The energy regimes for obtaining surface periodic structures correspond to heating the material to a temperature approximately equal to the melting point (lower limit), but not higher than the temperature of developed evaporation. This regime was used in electro-explosive coating in the present work. Surface profilometry showed that the roughness parameter of the electro-explosive coating of the ZnO-Ag system is 73 nm. In this case, the maximum profile protrusion reaches 536.85 nm, and the depth - 497.5 nm.

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TI-ZR COATINGS FORMED ON THE TITANIUM IMPLANT SURFACE BY THE ELECTROEXPLOSIVE METHOD*

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According to the morphology and etching contrast, the formed coating can be divided into two sublayers. Near the interface, sublayer 1 has a columnar structure, and sublayer 2 has a dendritic structure. It can be assumed that sublayer 2 resulted from melting and subsequent high-rate crystallization of the surface layer of the substrate (titanium-based alloy) initiated by an incident plasma flow formed by an electric explosion of the titanium foil with the zirconium powder placed on it. Sublayer 1 is the actual coating of the Ti-Zr system.

The X-ray microanalysis of the elemental composition at points reveals atoms of other elements along with titanium and zirconium atoms. It is clearly seen that along with the elements characteristic of the titanium-based alloy, carbon and oxygen atoms are present in the coating. Carbon atoms are detected only in the surface layer, and oxygen atoms are present throughout the coating. Based on the results of the elemental analysis, it can be assumed that the formed coating is multiphase and should contain, along with the Ti-Zr alloy, carbide and oxide phases.

The performed investigation revealed the presence of three phases in the surface layer: the main is the α -modification of the TiZr alloy (81.3 % by volume), the fraction of zirconium oxide ZrO (9.5% by volume) and titanium carbide TiC are significantly smaller (9.2 % by volume). Thus, the X-ray diffraction analysis results are in good agreement with the X-ray microanalysis results. The presence of oxide and carbide phases in the surface layer of the coating is obviously due to technical vacuum of the working chamber of the electroexplosive alloying installation and the use of a graphite electrode.

The defect substructure of the coating was studied by transmission electron microscopy of thin foils. Foils were prepared by ion sputtering of plates cut from the specimen bulk in the cross section of the coating. It is clearly seen that the surface layer of the coating has a nanocrystalline structure whose crystallites vary in size from 20 to 100 nm. The underlying layer up to 30 μm in thickness has a submicrocrystalline structure. The size of the crystallites forming this sublayer varies from 200 to 450 nm. The layer located at a greater distance from the coating surface has a bimodal structure. Along with crystallites 200–300 nm in size, it has crystallites tens of nanometers in size. As the coating-substrate interface is approached, the relative content of nanosized crystallites increases. Based on the X-ray microanalysis results on the elemental composition of the coating, it can be assumed that nanoscale crystallites are oxide phases based on titanium and zirconium.

The microstructures show that the grains of one phase (appearing dark-grey) are surrounded by the continuous or discontinuous layers of another phase (appearing light -grey). This phenomenon is intimately connected with the so-called complete and incomplete wetting of grain boundaries by the second solid phase both in titanium as well as in the zirconium-based alloys.

The Ti-Zr coating formed by electric explosion had the following parameters (opposite to the wear resistance of the material): wear parameter $5.5 \cdot 10^{-4} \text{ mm}^3/\text{N}\cdot\text{m}$, friction coefficient 0.572, hardness $3730 \pm 0.495 \text{ MPa}$, and Young's modulus $73.8 \pm 6.19 \text{ GPa}$. The uncoated specimen had the wear parameter $6.5 \cdot 10^{-4} \text{ mm}^3/\text{N}\cdot\text{m}$, friction coefficient 0.376, hardness $3630 \pm 260 \text{ MPa}$, and Young's modulus $84.3 \pm 7.62 \text{ GPa}$. Formation of a Ti-Zr coating is accompanied by an insignificant (by 18%) decrease in the wear parameter (an increase in the wear resistance) of the surface layer, a 1.5-fold increase in the friction coefficient, a slight (by 3%) increase in hardness, and a decrease in Young's modulus by 14%.

The electroexplosive method is used to form a Ti-Zr coating with a thickness of at least 50 μm on the surface of the dental implant made of titanium-based alloy. The coating is found to be multielement and multiphase. It was shown that along with the Ti-Zr-based solid solution, carbide and oxide phases are present in the coating. It was found that the coating formed by the electroexplosive method has a submicro-nanocrystalline structure. It was revealed that the formation of a Ti-Zr coating is accompanied (relative to the uncoated substrate) by a slight (18%) decrease in the wear parameter (increase in the wear resistance) of the surface layer, a 1.5-fold increase in the friction coefficient, a slight (3%) increase in hardness, and a decrease in Young's modulus by 14%.

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DEVELOPMENT OF ENVIRONMENT FRIENDLY TECHNOLOGY OF GENERATION OF ELECTROEROSION-RESISTANT COMPOSITE COATINGS FOR SWITCHES OF HIGH-POWER ELECTRIC LINES, WHICH COMBINES ELECTRO-EXPLOSIVE SPRAYING AND ELECTRON-ION-PLASMA MODIFICATION*

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One of the components of Russian national security until 2020 (approved by Presidential Decree No. 537 of 12.05.2009) is to ensure energy and environmental security. Enhancing fire safety, reliability and profitability of electrical installations and, in particular, their electrical contacts is one of the priority areas for ensuring energy security. Thus, development of new materials for electrical contacts is an important problem. Promising methods for formation of such coatings include electrospray deposition by pulsed multiphase plasma jets. In that respect, the problem seems to be relevant.

Promising direction of development of method of electrospray coating of composite materials is modification of coatings by high-intensity electron beams. Formation of nonequilibrium structural-phase states in surface layer during electron-beam irradiation in submillisecond exposure time range is determined by ultra-high rates of heating (up to 10^6 K/s) of thin surface layer of material (10-4-10-3mm) to the melting point and formation of limiting gradients of temperature (up to 107 - 108 K/m), which ensure cooling of surface layer due to heat transfer to the bulk of material at 10^4 - 10^6 K/s. Compared with high power ion beams, which can also be used to modify surface of materials, low-energy (<30 keV) dense electron beams are generated with significantly higher efficiency (more than 90%) in frequency-pulse ($\sim 10\text{s}^{-1}$) mode with less (by an order of magnitude) accelerating voltages and do not require creation of special radiation protection as soon as the accompanying X-ray radiation is shielded by the walls of working vacuum chamber. High energy efficiency, higher homogeneity of energy density along the flow cross-section, good pulse reproducibility and high frequency of pulses advantageously distinguish pulsed electron beams from pulsed low-temperature plasma flows, for potential use of both for technological purposes. International priority in development of pulsed electron-beam devices based on plasma cathodes, including surface treatment of materials, belongs to the Institute of High Current Electronics of Siberian Branch of the Russian Academy of Sciences (ISE SB RAS). At present, the ISE SB RAS has the most up-to-date complex of research equipment for pulsed electron-beam irradiation of materials in a wide (including unexplored) range of values of irradiation parameters – “UNIKUUM” - the set of unique electrophysical installations for effective electron-ion-plasma modification of surface of materials and products. Having an international priority in development of pulsed electron-beam devices based on plasma cathodes, high professionalism, wide access to modern analytical equipment, significant amount of positive results of preliminary studies on the claimed subject, the team of project executors believes that the results obtained in the work will be original and provide novelty of the world level.

Present work will be carried out within the general direction of development of scientific research and practical developments - surface protection by spraying coatings using concentrated energy flows. The purpose of the work is formation of electroerosion-resistant coatings by electrospray coating method and subsequent electron-beam mixing (including using nitriding electroexplosive coatings to form nitrides to harden the surface coating layer), to study their structure, phase composition and properties. Coatings will be investigated using the following equipment: optical microscope, scanning electron microscope, transmission electron microscope, X-ray diffractometer, optical interferometer. Coatings for wear resistance and electroerosion resistance, nanohardness and Young's modulus will be tested. As a result of the project, physical nature of formation of structure and properties of electroexplosive electro-erosion-resistant composite coatings of Ag-Ni, Ag-Cd, Ag-C and Ag-Co systems will be established after electron-beam mixing, including nitriding of electric explosive coatings. The results obtained in this project will serve as a stimulus for further research in the field of electric explosive spraying and electron-beam mixing of electroerosion-resistant coatings.

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SYNTHESIS OF C-N POWDER MATERIALS BY ARC DISCHARGE PLASMA*

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Carbon nitride C_3N_4 is binary compound of carbon and nitrogen. There are seven different phases of C_3N_4 : α - C_3N_4 , β - C_3N_4 , cubic C_3N_4 , pseudocubic C_3N_4 , g-h-triazine, g-h-heptazine and g-o-triazine. Among them, β - C_3N_4 crystalline phase has similar hardness/low compressibility to that of diamond. However, today more and more attention is paid to the hexagonal or so-called graphite-like carbon nitride h- C_3N_4 (g- C_3N_4) [1]. This material are currently being studied for a wide range of applications, such as main catalyst in hydrogen photocatalysis [2], as a coating for implants in biomedicine [3], as a precursor for synthesis of superhard phases of C-N system [4].

One of the methods to obtain the g- C_3N_4 is electric arc discharge method [5,6]. Today this method is developing in the direction of vacuumless synthesis. In this regard, an attempt to synthesize crystalline C-N powder materials by atmospheric arc discharge plasma has been done.

Figure 1 shows typical current and voltage waveforms taken by oscilloscope during the experiment.

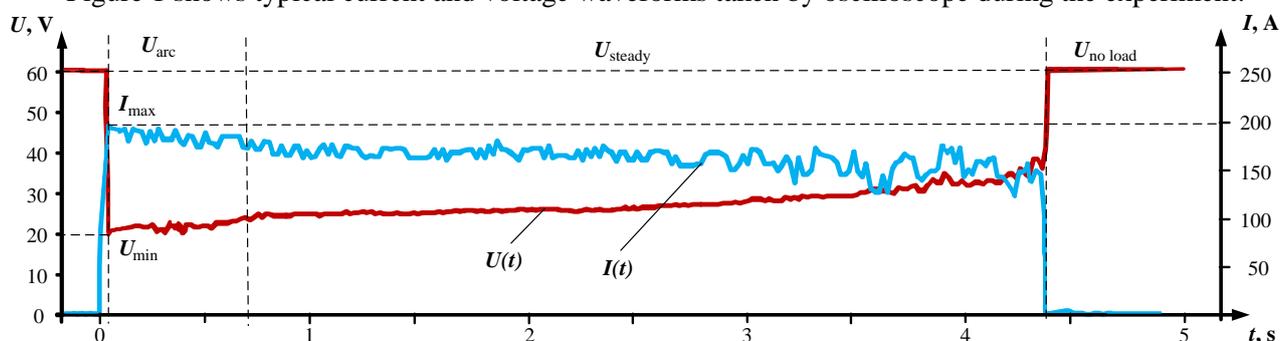


Fig. 1. Typical current and voltage waveforms taken during the experiment

As it can be seen from the fig.1, at the initial time voltage on the electrodes is equal to the no-load voltage of the power supply $U \approx 60$ V, the discharge circuit current is zero. Then, at the moment of discharge initiation, the voltage drops to the minimum value of $U \approx 20$ V, respectively, the current increases to the maximum value of $I \approx 200$ A. After the discharge gap is formed, the voltage and current of the system stabilize to steady-state values $U \approx 30$ V and $I \approx 160$ A, respectively. After the end of the arc discharge, at the time $t = 4.4$ s, the current value drops to zero, the voltage is restored to its initial value, equal to $U \approx 60$ V. It should be noticed that in the time interval from 0.1 s. to 4.4 s. the voltage increases by $\Delta U \approx 8$ V. This is explained by anode evaporation, therefore, anode length decreases, and the discharge gap size increases.

Based on the data from Fig. 1, the dependences of power and energy on time were obtained. Power is maintained in the range of 0.2-4.4 s. Moreover, the $P(t)$ waveform is similar to the $I(t)$. The average power is equal to 4 kW during the experiment. In turn, the power provides energy release in the system, equal to 21 kJ for 4.4 s.

Thus, in this paper, the changing of experiment parameters, such as arc current, voltage, power and energy, obtained during the synthesis of C-N powder materials, was analyzed.

REFERENCES

- [1] J. Wen, J. Xie, X. Chen, X. Li // Applied Surface Science. – 2017. – 391. P. 72–123.
- [2] J. Zhang, F. Guo, X. Wang // Advanced functional materials. – 2013. – 23. – № 23. P. 3008–3014.
- [3] F.Z. Cui, D.J. Li. // Surface and coating technology. – 2000. – 131. P. 481–487.
- [4] J. Liu, T. Sekine, T. Kobayashi // Solid state communications. – 2000. – 137. P. 21–25.
- [5] X.Q. Meng, Z.H. Zhang, H.X. Guo, et al. // Solid state communications. – 1998. – 107. – № 2. P. 75–78.
- [6] A.Y. Pak, G.Y. Mamontov // Technical Physics Letters. – 2019. – 44. – № 7. P. 615–617.

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CHARACTERIZATION OF THE α -Al₂O₃ COATINGS DEPOSITED BY REACTIVE EVAPORATION IN ANODIC ARC UNDER HIGH-CURRENT ION ASSISTANCE*

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Reactive anodic evaporation of Al in a discharge with a self-heating hollow cathode provides high ($\sim 4.5 \mu\text{m/h}$) deposition rates of Al₂O₃ coatings. For the crystallization of coatings during the deposition process at a low temperature, intensive ion assistance [1] is necessary, moreover for the formation of the α -phase of Al₂O₃ the ion energy must be within the narrow range [2].

Stable growth of the α -phase at 640 °C was ensured by using low-energy ($\sim 50 \text{ eV}$) high-current (up to 15 mA/cm²) ion assistance under conditions of reduced degree of ionization of evaporated Al and increased degree of oxygen dissociation. To increase the ion current density and the degree of oxygen dissociation, a hollow anode was used parallel with the anode-crucible. An oxygen flow and a large part (up to 36 A) of the electron current pass through the hollow anode aperture thus ensures the effective interaction of oxygen with energetic electrons.

α -Al₂O₃ coatings were obtained, which are characterized by the presence of a dense surface layer with a structure corresponding to zone 3 of the Thornton diagram [3] and the underlying column layer (Fig. 1). The structural-phase state of the Al₂O₃ coatings, their adhesive strength and surface roughness were investigated.

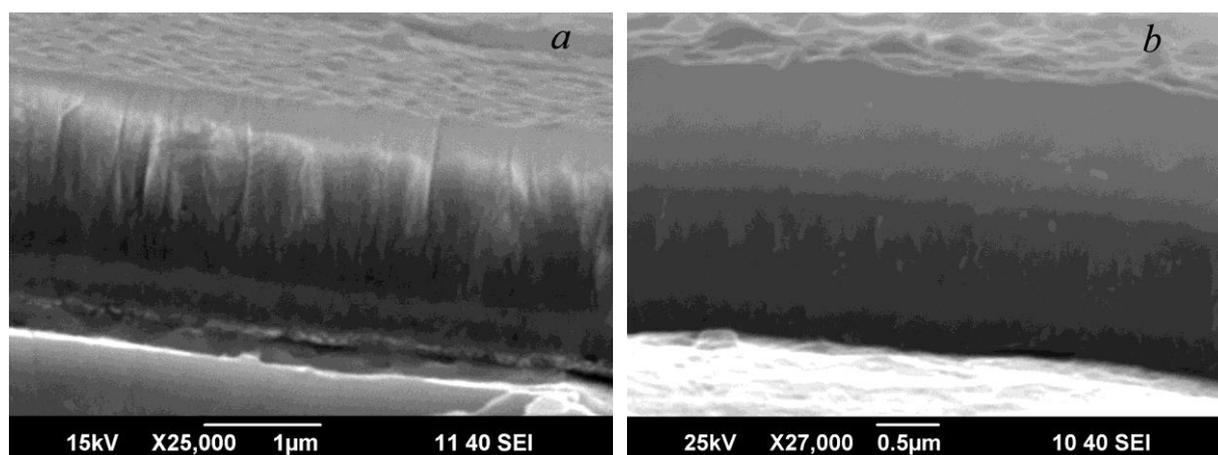


Fig. 1. Images of the brittle cleavage of Al₂O₃ coatings deposited at different currents to the hollow anode: 4 (a) and 28 A (b).

REFERENCES

- [1] M. Prenzel *et al.* // *Journal of Applied Physics*. – 2013. – 114. – 113301.
- [2] J. Houska // *Surface and Coating Technology*. – 2013. – 235. – 333-341.
- [3] J. A. Thornton // *Ann. Rev. Mater. Sci.* – 1977. – 7. – 239-260.

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HIGH-RATE LOW-TEMPERATURE PVD OF THICK (10 μm) α -ALUMINA COATINGS *

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The method of anodic evaporation of Al in Ar-O₂ medium and deposition under intense ion assistance allows control of evaporation rate of metal, plasma density and composition, current density and energy of ions on the surface of the growing coating independently [1, 2]. These features of the method were used to reduce the crystallization temperature of Al₂O₃ coatings to 640 °C and the intrinsic stresses in the coating. An increase in the deposition rate of the coating was achieved by optimization of the ratio of atomic fluxes of Al and O on the surface of the growing coating. Single-phase adhesive α -Al₂O₃ coatings ~10 μm thick with a hardness of ~20 GPa and a Young's modulus of ~260 GPa were obtained.

REFERENCES

- [1] N.V. Gavrilov, A.S. Kamenetskikh, P.V. Tretnikov, A.V. Chukin // Surface and Coating Technology. – 2018. – 337. – 453-460.
- [2] N.V. Gavrilov, A.S. Kamenetskikh, P.V. Tretnikov, D.R. Emlin, A.V. Chukin, Yu.S. Surkov // Surface and Coating Technology. – 2019. – 359. – 117-124.

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MODIFICATION OF THE SURFACE STRUCTURE OF STEEL BY COMBINED ELECTRON-PLASMA METHOD

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One of the promising technologies for hardening surface layers and hardening protective coatings of metals and alloys is based on the use of surface modification, combining plasma spraying of a powder coating and the subsequent irradiation with an intense pulsed electron beam. The use of surface modification of materials that combines formation of a molten layer on the surface of a material by plasma treatment and the simultaneous introduction of powders into this layer and the subsequent irradiation of the formed protective coating with an electron beam is a promising direction.

The purpose of the paper is to study the tribological properties of the modified surface layer on a steel substrate formed by plasma spraying of a powder of the Ni-Cr-B-Si system and the subsequent irradiation with a high-energy pulsed beam.

Surface modification of A3 steel (United Kingdom marking) was carried out by plasma spraying of Ni-Cr-B-Si powder onto a steel surface using an original installation equipped with two plasma generators [1]. Further, the surface was irradiated with an intense pulsed electron beam on the installation SOLO.

Tribological testing of samples was performed using the “Tribotechnic” tribometer. The test process was carried out at a load of 1÷2 H according to the “finger-disk” scheme under dry friction conditions. The tests were carried out outdoor. Testing time was up to 1000 sec. Images of wear marks were taken using the Axiovert 40 MAT optical microscope (Figura).

The analysis of tribological tests data have allowed establishing that electron beam processing with a capacity of 40 J/cm² leads to a decrease in the sample wear resistance and the appearance of the fatigue spalling process along with normal mechanochemical wear. Lower capacity of the electron-beam effect of 20 J/cm² allows increasing the wear resistance of the material, as compared to the initial state. From the tests performed, it can be concluded that excessive surface hardening with the combined effect of plasma coating deposition and high-power electron-beam processing reduces wear resistance, as compared to untreated samples and samples processed at lower capacity.

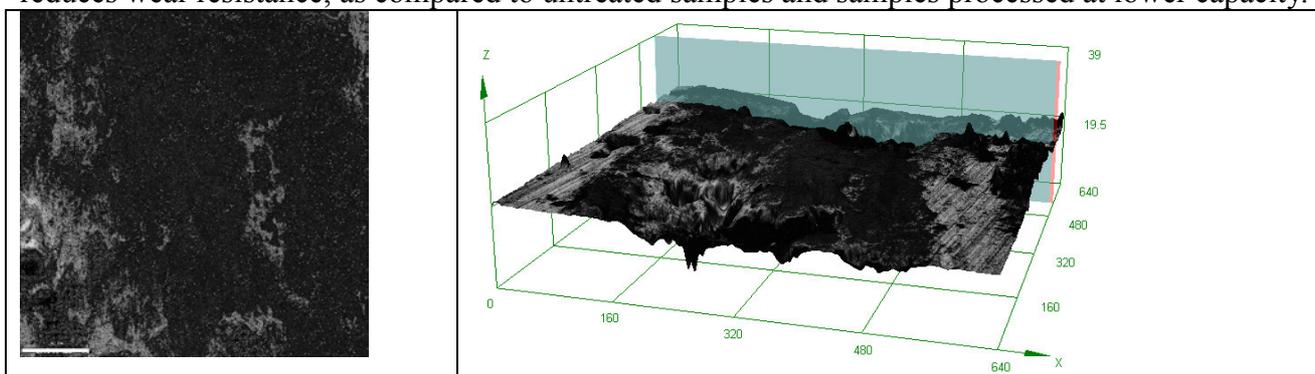


Fig. The section of the friction track on the surface of the steel sample after plasma spraying of the powder of the Ni-Cr-B-Si system and irradiation with an electron beam of 20 J/cm² (a). The profilogram of the cross section of the friction track (b).

REFERENCES

- [1] *Ivanov Yu. F., Klopotov A. A., Petrikova E. A., and at. al.// Steel in Translation. – 2017. – V. 47. – No. 10. – P. 669–674.*

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INFLUENCE OF METAL-GAS PLASMA COMPOSITION AND PARAMETERS ON COMPOSITION AND CHARACTERISTICS OF NITRIDE COATINGS *

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Generation of gas-metal plasma for vacuum arc plasma-assisted deposition of coatings was carried out by plasma sources of different type: 1) arc evaporator with magnetic filtration of a plasma flow and source of gas-discharge plasma with the combined thermionic and hollow cathode on the ion-plasma QUINTA installation [1, 2]. Zirconium alloy (Zr-1%Nb, E110) was used as material of the evaporated cathode. Generation of gas-metal plasma and coating deposition were carried out in mixture of Ar-N₂ gases, the partial pressure of nitrogen changed in the range of 0.01-0.2 Pa at total mixture pressure of 0.2 Pa. In the plasma-assisted modes at constant working pressure and constant arc current of evaporator arc current of gas plasma source was changed in the range of 10 to 150 A.

Parameters of gas, metal and gas-metal plasma were measured by a single cylindrical Langmuir probe and the automated system of probe measurements [3]. The composition of plasma was analyzed on the radiation lines obtained by a spectrometer method in several ranges of wave lengths. Change of coatings composition at variation of plasma parameters and composition was investigated on a scanning electron microscope (Philips SEM-515 with the EDAX ECON IV microanalyzer). Micro- and nanohardness were measured on the PMT-3 microhardness tester (LOMO, Russia) and DUH-211S nanohardness tester (Shimadzu, Japan), respectively. The structure and phase composition were investigated by X-ray diffraction analysis (Shimadzu XRD 6000 diffractometer) and transmission electron microscopy (JEOL JEM-2100 F). Tribological characteristics were measured on a TRIBOtester of Pin on Disc and Oscillating (TRIBOtechnic, France).

Influence of composition and parameters of plasma on elemental and phase composition, structure and characteristics of coatings based on ZrN was revealed. The optimum synthesis modes for these coatings with high wear resistance were revealed.

REFERENCES

- [1] L.G. Vintizenko et al. // *Russian physics journal*. – 2001. – 44. – P. 927- 936.
- [2] A.A. Kalushevich et al. // *Russian Physics Journal [Izvestiya Vuzov. Fizika – in Russian]*. – 2012. – 55. - №12/3. – P. 118-122.
- [3] O.V. Krysina et al. // *Journal of Physics: Conference Series*. – 2016. – 669. – P. 012032.

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VACUUM ARC DEPOSITION OF MON COATINGS IN THE MODES OF PLASMA ASSISTENCE*

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In the modern industry, functional coatings are actively introduced to increase the strength characteristics of various products. Nitrides of many kinds of metals have found wide application. At present titanium nitride (TiN) and coatings based on it (e.g., TiAlN, TiCuN and etc.), are widely used [1]. The coatings based on molybdenum nitride (MoN) have great prospects in industry due to its high hardness, low friction coefficient and chemical inertness to non-ferrous metals [2].

The purpose of this work is the investigations of the influence of the operating modes of the sources of gas and metal plasma on the properties and composition of molybdenum nitride coatings. A plasma generator based on a non-self-sustained arc discharge with a combined thermionic and hollow cathode “PINK-04P” was used as a source of gas plasma [3]. The source of metal plasma was an arc evaporator “DI100” with improved design. The material of the evaporated cathode was molybdenum of MCH brand.

There are results of the series of experiments on researches of the parameters and composition of gas and gas-metal plasma. A number of experiments were carried out to obtain single-layer coatings of molybdenum nitride with a thickness of 3-5 μm deposited at different parameters of arc discharges. The composition and tribological properties of the obtained coatings were investigated. The correlation between the composition of the gas mixture and the parameters of arc discharges and the composition and properties of the deposited coatings was revealed.

REFERENCES

- [1] O.V. Krysinina, N.N. Koval, I.V. Lopatin, V.V. Shugurov, S.S. Kovalsky. *Generation of low-temperature plasma by low-pressure arcs for synthesis of nitride coatings. // Journal of Physics: Conference Series.* – 2016. – V. 669. – P. 012032.
- [2] Andreyev, L.P. Sablev, S.N. Grigor'yev. *Vacuum arc coatings.* - Kharkov: NSC KIPT, 2010. – 318 p.
- [3] Vintzenko L.G., Grigoryev S.V., Koval N.N., et al. (2001). *Hollow-cathode low-pressure arc discharges and their application in plasma generators and chargedparticle sources // Russian Physics Journal, Vol. 44, No. 9, 2001, pp. 927-936.*

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IRRADIATION BY A LOW-ENERGY PULSED ELECTRON BEAM OF ZIRCONIA-BASED COMPOSITE*

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In this work, tetragonal zirconia-based composite prepared by spark plasma sintering, modified by 5 wt% alumina nanofibers and 0.5 wt% single-walled carbon nanotubes, was investigated. Similar composites were investigated in previous work [1]. Irradiation by a low-energy pulsed electron beam of the submillisecond duration was carried out on the device “SOLO” [2] in the following mode: beam energy density – 15 J/cm², pulse duration – 200 μs, pulses quantity – 10, 20, 30, and 40, and pulse repetition rate – 0.3 Hz.

The initial composite material (Fig. 1a) consists of ZrO₂ matrix, in which there are alumina grains of complex elongated shape and carbon nanotube bundles, which are uniformly distributed in the matrix. Such composite possesses improved mechanical properties (microhardness and fracture toughness increase by 1.4 % and 17.2 %, respectively), as compared to ZrO₂ ceramics without additions.

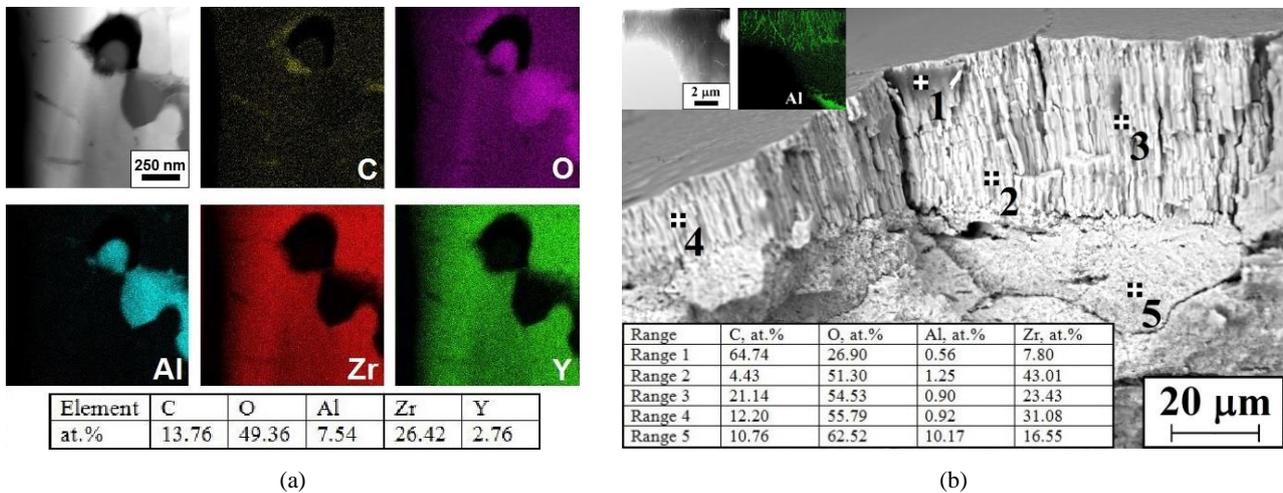


Fig. 1. STEM image of the sample before irradiation and element mapping of same area (a); SEM image of the cross section of composite after electron beam processing at 40 pulses. Areas are marked and numbered for which the elemental composition (table) is determined (b).

It has been established that electron beam treatment leads to the formation of modified surface, the thickness of which varies from 5–10 μm to 100 μm. Modified volume is multi-layered (Fig. 1b): the upper layer has a columnar structure; the intermediate layer between the surface layer and the base material consists of equiaxed grains with sizes from 0.5 μm to 1 μm, that more grain size (~ 0.25 μm) of the initial material.

XRD analysis showed that, the initial composite contains tetragonal modification of zirconia (t-ZrO₂) and a small amount of α-Al₂O₃. In the irradiated samples, in addition to t-ZrO₂ and α-Al₂O₃, a new compound Zr–Al–O (zirconium aluminum oxide) appears.

According to the EDS method results, Al atoms in the initial composite form Al₂O₃ nanofibers, which are preferentially, located parallel to the sample surface. In the modified layer, when irradiated by an electron beam, the Al atoms form thin interlayers along the boundaries of the columnar grains (inset in the upper left corner of Fig. 1b).

REFERENCES

- [1] A. Leonov // Materials Today: Proceedings. – 2019. – V. 11. – P. 66-71.
 [2] N.N. Koval, Yu.F. Ivanov // Russian Physics Journal. – 2008. – V. 51. – No. 5. – P. 505-516.

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ION-PLASMA ZR-NB-N COATINGS: EQUIPMENT, DEPOSITION AND PROPERTIES.*

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The paper presents the results of research on plasma-assisted arc deposition of the combined Zr-Nb-N coating. The mechanical characteristics of the coating and the elemental composition were investigated.

The work was performed on the KVINT ion-plasma unit developed at the laboratory of plasma emission electronics of the Institute of High Current Electronics SB RAS [1] and included in the list of unique electrophysical units of the Russian Federation (УНИКУУМ complex, <http://ckp-rf.ru/usu/434216>).

The sputtering was performed from two cathodes: zirconium, installed in the cathode assembly of the magnetic filter of the microdrop fraction, and niobium from the evaporator DI100. The coating was applied in two stages: first, the zirconium underlayer for 10 minutes at a discharge current of an arc evaporator of 150 A and an argon pressure of 0.2 Pa. Then a layer of Zr-Nb-N was applied with currents of arc evaporators 150 and 100 A for zirconium and niobium, respectively. The total pressure of the gas mixture (Ar: N₂ - 50:50) was 0.2 Pa. The coating was applied within an hour. The samples were located in the center of the vacuum chamber on a rotating table and on a table satellite at a diameter of 300 mm rotating planetary.

Using the CaloTest method, the coating thickness was measured and the growth rate of the coating was calculated. The growth rate of the coating was 4.36 $\mu\text{m/h}$ at the center of the chamber and 4.92 $\mu\text{m/h}$ on the table satellite. The difference in deposition rates is explained by the directivity pattern of arc evaporators.

The hardness of the coating under a load of 1 N was about 30 GPa for both samples.

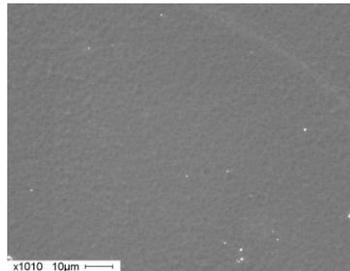


Fig. 1. SEM image of the coating surface.

Elemental composition

Таблица 1. Centre

Element	Wt%	At%
N	13,41	50,27
Zr	66,42	38,30
Nb	20,17	11,43

Таблица 2. Satellit

Element	Wt%	At%
N	14,62	52,72
Zr	64,07	35,66
Nb	21,31	11,62

The study of the elemental composition shows that the coating has an almost stoichiometric composition of nitrogen. Small differences in the ratio of zirconium and niobium are caused by the directivity pattern of arc evaporators.

REFERENCES

- [1] Kalushevich A.A., Koval N.N., Denisov V.V., Yakovlev V.V., Shugurov V.V. // Известия высших учебных заведений. Физика. – 2012. – 12-3. – 118-122.

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OPTIMIZATION OF PLASMA DYNAMIC SYNTHESIS PROCESS FOR INCREASING THE YIELD AND PURITY OF ϵ -Fe₂O₃ (EPSILON) PHASE

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Iron oxides are among the most common used materials in the various fields of science and technology [1]. Among the known non-hydrated phases, the production of the epsilon phase of iron oxide (ϵ -Fe₂O₃) causes the greatest difficulties, since it is associated with the need to synthesize in a very narrow temperature range while maintaining the nanoscale state [2, 3]. This material also causes great scientific interest, since it has been established that this phase has the largest coercive force among all known simple metal oxides and is capable of absorbing electromagnetic radiation in the millimeter wavelength range [4, 5].

Earlier [6], the possibility was convincingly proved to obtain this unique iron oxide modification using a plasma dynamic synthesis method based on the use of low-temperature iron-containing plasma generated by a coaxial magnetic plasma accelerator [7, 8] and flowing into an oxygen atmosphere. The special features of the synthesis process (high plasma flow speed ~ 3 km/s and high cooling rate $\sim 10^8$ K/s) make it possible to preserve the necessary epsilon phase during high-speed sputtering from the boundary of the head shock wave of an ultrafast plasma flow as well as to achieve an output of at least 50 wt. %

In this work, the possibility was studied to increase further the yield of the epsilon phase of iron oxide in the composition of the heterophase synthesis product. For this, key features of the process were revealed that affect the production of ϵ -Fe₂O₃, which include the need to increase the lifetime of the quasi-stationary flow regime and the energy input to the system. Taking into account these data, appropriate design and circuit solutions for the system have been implemented that make it possible to obtain iron oxide powder with an output purity of epsilon phase of at least 90 wt. %. Another advantage was found to be an increase in the mass yield of the necessary phase.

REFERENCES

- [1] *S. Laurent, D. Forge, M. Port, A. Roch, C. Robic, L. Vander Elst, R.N. Muller // Chemical Reviews. – 2008. – Volume 108. – Pages 2064-2110.*
- [2] *J. Tucek, R. Zboril, A. Namai, S.-I. Ohkoshi // Chemistry of Materials. – 2010. – Volume 22. – Pages 6483–6505.*
- [3] *E. Tronc, C. Chanéac, J.P. Jolivet // Journal of Solid State Chemistry. – 1998. – Volume 139. – Pages 93-104.*
- [4] *J. Jin, S.I. Ohkoshi, K. Hashimoto // Advanced Materials. – 2004. – Volume 16. – Pages 48-51.*
- [5] *M. Yoshikiyo, A. Namai, M. Nakajima, K. Yamaguchi, T. Suemoto, S.I. Ohkoshi // Journal of Applied Physics. – 2014. – Volume 115. – Page 172613.*
- [6] *A. Sivkov, E. Naiden, A. Ivashutenko, I. Shanenkov // Journal of Magnetism and Magnetic Materials. – 2016. – Volume 405. – Pages 158-168.*
- [7] *I.I. Shanenkov, A.Ya. Pak, A.A. Sivkov, Yu.L. Shanenkova // MATEC Web of Conferences. – 2014. – Volume 19. – Page 01030.*
- [8] *V.V Kuzenov, T.N. Polozova, S.V. Ryzhkov // Problems of atomic science and technology. – 2015. – Volume 4(98). – Pages 49-52.*

PREPARATION OF HIGH-VOLTAGE VACUUM GAP SURFACES BY THE GLOWING DISCHARGE

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Currently, when a certain number of gas-discharge devices (GDD) are operating, failures associated with a decrease in the electrical strength of the high-voltage vacuum gap are observed. As shown in [1], one of the reasons is the emission processes on the insulator inner surface and the electrode surfaces of the high voltage gap caused, in particular, by the presence of dielectric and oxide films on them, as well as adsorbed molecular gases. The consequence of the electron emission is the irradiation of the dielectric as an electron flux and γ -quantum fluxes [2]. In this case, the GDD vacuum shell is charged [2], which contributes to the appearance of either an incomplete electric discharge on the dielectric surface or a through breakdown. Both phenomena lead to degradation of the vacuum shell and loss of tightness of the device.

The effect of residual and working gases adsorbed by the GDD internal surfaces is also manifested in the form of sorption and desorption processes [3] under the influence of corpuscular flows and thermal loads. In this case, during the operation of the device gas exchange between the parts working surfaces is observed, which contributes to the appearance of pressure surges when the GDD is turned on after long interruptions.

The literature sources analysis shows that currently in the GDD manufacture to remove oxides and sorbed gases, along with traditional methods of cleaning parts and assemblies in preparation for Assembly, additional methods are effectively used. It is based on the GDD structural elements processing in a glow discharge plasma of an inert gas environment [4]. However, the sequence and modes of processing described in the literature are largely determined by the geometric and physical characteristics of the treated parts and are individual for a particular type of device.

Therefore, in the present work, in order to improve the GDD electrical strength and stability, operating on the Penning discharge basis, a method of parts and assemblies plasma processing was implemented at the stage of their preliminary preparation for product assembly. Figure 1a shows a photograph of the discharge realized when high-voltage vacuum gap electrodes are processing. Figure 1b shows a photograph of the discharge realized when treating the inner surfaces of high-voltage vacuum gap insulator.

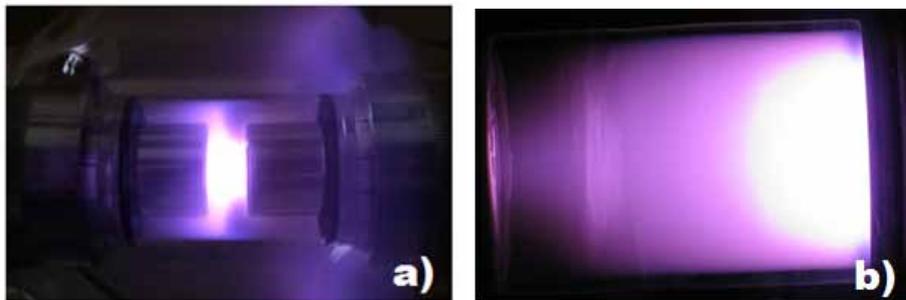


Fig. 1. Photograph of the discharge realized when high-voltage vacuum gap electrodes are processing (a) and when treating the inner surfaces of high-voltage vacuum gap insulator (b)

REFERENCES

- [1] *Latham R.V.* // High voltage vacuum insulation: the physical basis.- Academic Press, London, 1981.
- [2] *Rau E.I. [at al]* // Physics of the Solid State. – 2008. – V. 50. – №. 6. – p. 599-607.
- [3] *Vashin S. A., Korepin G. F.* // Vacuum equipment and technology. – 2015. - V. 25. - № 2. – p. 129-130.
- [4] *Cherepnin S.V.* // Sorption phenomena in vacuum. – Soviet radio. – 1973.

EXPERIMENTAL AND NUMERICAL STUDY OF HIGH-TEMPERATURE SYNTHESIS OF NANOSIZED SILICA PARTICLES IN FLOW-TYPE PLASMACHEMICAL REACTOR*

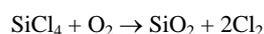
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Nanosized silica powder is commonly used in variety of technologies, namely, in solar energy conversion, as ingredient for polishing slurries in optoelectronics, in photo-catalysis, as an adsorbing agent, as a catalyst support as well as reinforcing filler for rubbers and plastic materials, etc. [1]. Based on devised pilot lab set-up the results of experimental study of synthesis of samples of nanosized silica powders obtained by one-step chloride method have been presented in [2]. One of the most essential challenges of plasma-chemical synthesis of ceramic nanopowders is how to improve control of their physico-chemical properties at the stage of particle formation and its further growth. The control of depth of counter flow jet quenching determined by ratio of momentum-flux of cold jets to that of high-temperature flow with synthesized particles enables one to solve largely this problem. In this work given operating parameters of the set-up the morphology and properties of the samples of silica powder obtained at different mass flow rates of quenching air jets have been analyzed. Using acquired experimental data as feedback information the parameters of corresponding numerical model of the synthesis have been more precisely specified to solve this inverse problem. This would allow one to predict sizes of particles being synthesized with sufficient accuracy. Below the results of analysis of silica powder samples as well as predictions of conversion of SiCl₄ to SiO₂ nanoparticles are demonstrated.

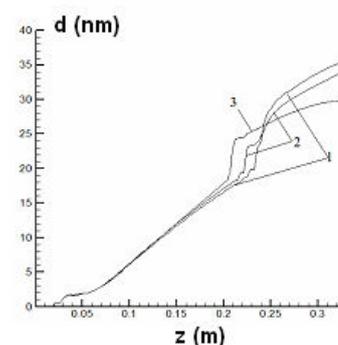
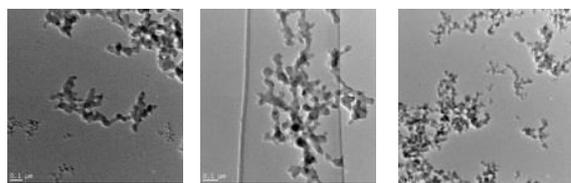
Morphology and physico-chemical properties of silica powder samples synthesized in accordance with overall reaction



Cross-section mass average particle diameter distribution along the reactor for mass flow rates of quenching air jets: 1 – 1.8 g/s, 2 – 4.1 g/s, 3 – 8.1 g/s

Mass flow rate of quenching air jets, g/s	1.8	4.1	8.1
Specific surface area of silica powder sample S_{BET} , m ² /g	70	73	96
Particle average size evaluated based on S_{BET} , nm	39	37.4	28.4
Molar fraction of contaminating chlorine, atom. %	0.38	0.12	0.13

Photo of silica powder sample



REFERENCES

- [1] Park H.K., Park K.Y. // KONA Powder and Particle Journal. – 2015. – № 32. 85–101.
 [2] Kartaev E.V., Emelkin V.A., Aulchenko S.M. // AIP Conference Proceedings. – 2017. – 1893. – № 1. 030056-1–030056-7.

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SURFACE TREATMENT OF METALS, DIELECTRICS AND SEMICONDUCTORS BY RUNAWAY ELECTRON PREIONIZED DIFFUSE DISCHARGE*

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In recent years, atmospheric pressure diffuse discharges are actively being studied by scientific collectives. Plasma of such diffuse discharges can be used for modification and improvement surface properties of various materials and can be used for thin film deposition processes, in medicine, micro- and nanoelectronics etc.

This paper deals with experimental results focused on surface treatment of metals (Cu, Nb, Ti, Al, St3), dielectrics (polyimides, polysulphones, organosilicon films) and semiconductors (silicon, CdHgTe) by runaway electron preionized diffuse discharge (REP DD) plasma at atmospheric pressure.

Elemental composition, roughness, surface free energy, nanohardness and surface structure were studied for metals surface after treatment by REP DD. Experimental results shows that surface of treated metals becomes ultrafine cleaned from carbon-like bandings. Also, REP DD treatment of metals result in 3-times increasing of surface free energy, smoothing and structural changes of near-surface layer [1].

In a case of surface treatment of dielectric such as polyimides and polysulphones it is shown by IR-spectroscopy methods that these materials have high resistant to the electron beam irradiation as well as REP DD plasma treatment. Such a properties provide an opportunity of using these materials in space to protect spacecraft crew from cosmic radiation [2].

Besides of that, surface treatment of CdHgTe (CMT) epitaxial films by atmospheric pressure REP DD is studied in this work. It is shown that irradiation of nanosecond diffuse discharge on the CMT surface leads to the changing of electrophysical properties of CMT as a result of formation of near-surface high-conductivity layer with n-type conduction [3].

Thus, surface treatment of metals, dielectrics and semiconductors by runaway electron preionized diffuse discharge can be used for surface cleaning, activation, increasing of adhesive properties, changing of structural and electrophysical properties. Also, REP DD treatment provides controlled changing of CMT epitaxial films properties and producing of structures with heterogeneous conductivity.

REFERENCES

- [1] *M. Erofeev, V. Ripenko, M. Shulepov, V. Tarasenko // Eur. Phys. J. D. – 2017. – 71. – № 117.*
- [2] *Y.V. Savinykh, V.M. Orlovskii, N.S. Kobotaeva, V.S. Ripenko // Izvestiya VUZov. Fizika. – 2018. – 61. – № 8-2. 128-131.*
- [3] *D. Grigoryev et al. // Journal of Physics: Conference Series. – 2017. – 830. 012082.*

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REDOX PROCESSES INVOLVING CHROMIUM IONS, INITIATED BY THE ACTION OF A DISCHARGE IN AIR, OXYGEN AND ARGON ON AQUEOUS SOLUTIONS

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Industrial plants involved in metal finishing, leather tanning, textile finishing and plating are the source of wastewater pollutions with chromium ions. Chromium exists in water solution in two stable forms with the oxidation level of +6 (Cr^{6+}) and +3 (Cr^{3+}). Cr^{3+} is less harmful while Cr^{6+} is a strong toxicant, mutagen and carcinogen. For these reasons, the maximum permissible concentration of Cr^{6+} in water is markedly lower than that of Cr^{3+} . Chemical Cr^{6+} reduction is the most used method for transformation of Cr^{6+} to Cr^{3+} . For this, ferrous sulfate, sodium sulfite, or sodium metabisulfite are applied as reducers. In spite of these methods being rather effective, they require an application of the reducer in excess due to the reversibility of the reduction process which becomes a source of additional water contamination. In the case of sodium sulfite and sodium metabisulfite, harmful gaseous sulfur oxide (IV) is formed. It is known that the action of discharges at atmospheric pressure maintained above water surface or in water bulk results in the formation of reactive oxygen species in a liquid phase such as H_2O_2 , O_2 molecules, $\cdot\text{H}$, $\cdot\text{OH}$, $\text{HO}_2\cdot$ radicals and many others. Depending on the conditions, these particles can play the role of oxidants or reducers in relation to chromium ions. The advantages of discharge systems are obvious. They do not need any chemicals and their action does not result in the formation of harmful products.

For these reasons the process of reduction of Cr^{6+} ions (water solution of potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$) in a water cathode was studied during a DC discharge in air, Ar and O_2 . The concentration range of Cr^{6+} was $(5.7-19)\times 10^{-5}$ mol/l and discharge current range was 20-80 mA. The photometric method was used to determine the concentration of Cr^{6+} and Cr^{3+} . For the plasma the electric field strength, and the cathode voltage drop, voltage drop between anode and cathode was measured as well.

For all gases under study Cr^{6+} ions were shown to be reversibly reduced under a discharge action. The equilibrium degree of reduction increased with increasing initial concentration of the solution at fixed discharge current. At fixed initial concentration the reduction degree increased with increasing discharge current. The reduction degrees so obtained were 0.34-0.84. On the basis of kinetic measurements, the effective rate constants for the oxidation and reduction of Cr^{6+} and Cr^{3+} ions were determined. For the discharge in air, argon and oxygen for a concentration of 0.1 mmol/l and a current of 40 mA, the reduction rate constants were $(4 \pm 0.7) \times 10^{-3}$, $(1.1 \pm 0.2) \times 10^{-2}$, and $(2 \pm 0.4) \times 10^{-3} \text{ s}^{-1}$, respectively. Calculations for a discharge current of 40 mA and a concentration of 0.1 mmol / l gave the following values of the energy efficiencies for the reduction of Cr^{6+} ions: argon - 4.2×10^{-2} ions per 100 eV of inputted energy; oxygen - 2.9×10^{-3} ; air - 0.75×10^{-3} . Despite the fact that the discharge in air demonstrates higher constants of the reduction rates than the discharge in oxygen, the energy efficiency of the discharge in oxygen turns out to be higher. The reason is that in order to maintain the same current, the discharge in the air requires high power.

A kinetic scheme of the processes taking place in a solution was proposed. The scheme included 52 reactions involving chromium ions and H_2O_2 , $\cdot\text{OH}$, $\cdot\text{H}$, solvated electrons, H_2 , O_2 , H^+ , OH^- , $\text{HO}_2\cdot$, O_2^- , HO_2^- , O^- , and O_2^{2-} . The calculated data obtained as a result of application of this scheme described well the experimental results on Cr^{6+} kinetics. The main processes of Cr^{6+} reduction and Cr^{3+} oxidation were revealed. $\text{HO}_2\cdot$ radicals and hydrogen peroxide were shown to be responsible for Cr^{6+} reduction whereas $\cdot\text{OH}$ radicals and O_2 molecules provide the reverse process of Cr^{3+} oxidation to Cr^{6+} . The mechanism of action of phenol additives improving the process efficiency is discussed. The efficiency of phenol action as a radical scavenger was shown to be determined with its mass-transfer to the reaction area rather than chemical reaction rate.

This study was carried out in the frame of Project part of State Assignment of the Ministry of Education and Science of the RF, No 3.1371.2017/4.6.

SYNTHESIS OF NI-AL INTERMETALLIC SURFACE ALLOYS PRODUCED BY USING A LOW-ENERGY HIGH-CURRENT ELECTRON BEAM

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Carbon steels are widely used in various applications, however their wear resistance, corrosion and oxidation resistance are weak. To improve these properties, methods of surface engineering can be used. One of the most commonly used coatings for steel is Ni-Al compounds [1]. The intermetallic Ni-Al compounds give a combination of high strength and hardness, resistance to fatigue and creep, good corrosion and oxidation resistance at high temperatures [2,3]. Therefore, they are considered as promising materials of protective coatings for industrial metals and alloys for high-temperature applications and aggressive environment which can enhance the performance and stability of catalysts, turbine blades, ferroelectric capacitors, vanes and so on [4,5].

However, there are some difficulties in producing intermetallic NiAl coatings using traditional coating technology: unsatisfactory adhesion properties, complex surface geometry. One of the methods that can be effectively applied to the manufacture of Ni-Al coatings is the method of forming surface alloys.

The aim of present work was to synthesize of Ni-Al surface alloy directly on steel substrate in vacuum using magnetron deposition of Ni and Al layers and consequent irradiation with a low-energy, high-current electron beam.

The electron-beam machine "RITM-SP" with an explosive-emission cathode and a plasma-filled diode generating the LEHCEB was employed in the work [6]. This machine is equipped with a multi-magnetron sputtering system enabling formation of multicomponent surface alloys. The surface alloy was synthesized of 2.5 μm thick. It is known that depending on the atomic ratio characterized by Al and Ni layer thicknesses, Al_3Ni_2 , Al_3Ni or NiAl can be formed as the final product. In this work, the emphasis was down to a constituent of the equiatomic composition NiAl. Three types of multilayer systems were deposited for surface alloy formation: 3-layer system Ni (0.5 μm) – Al (1.52 μm) – Ni (0.5 μm) (1); 9-layer system Ni (110 nm) – Al (167 nm) – ... – Ni (110 nm) (2); 2-layer system Al (167 nm) – Ni (110 nm) deposited 9 times (3) followed by LEHCEB irradiation after each deposition.

The surface morphology, phase and elemental composition of the Ni-Al surface alloys were analyzed, the microhardness and wear resistance were measured. For characterization different techniques like SEM, XRD and others have been used. The elemental composition of both the surface and cross sections of the samples was analyzed by EDS analysis. The structure and properties of the synthesized Ni-Al surface alloys were compared with witness-specimens, which were three types of multilayer systems without LEHCEB treatment.

Figure 1 shows the images of the Ni-Al surface alloy formed for three types of multilayer systems. It can be seen that the synthesis of a surface alloy by nanolayers of Ni and Al deposition leads to cracking of the surface due to the formation of brittle Al_3Ni and Al_3Ni_2 phases.

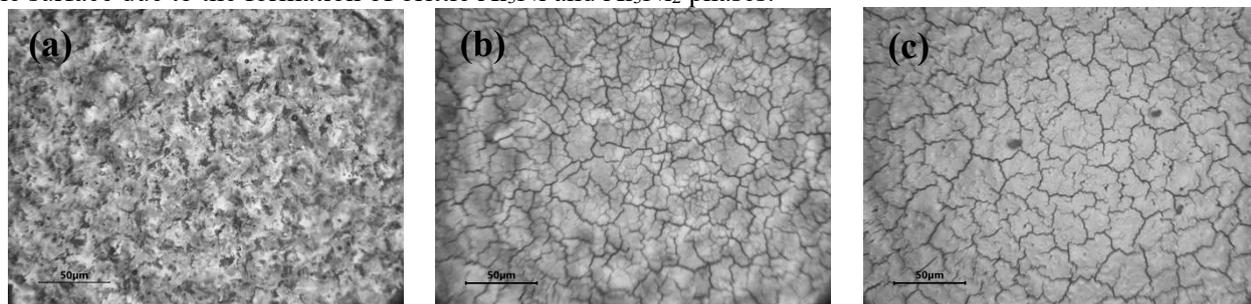


Fig. 1. The surface of the synthesized Ni-Al surface alloy for different types of multilayer system: 1 (a); 2 (b); 3 (c).

REFERENCES

- [1] M. Mohammadnezhad, M. Shamanian, M.H. Enayati, M. Salehi // Surf. Coat. Technol. – 2013 – 217 – 64-69.
- [2] C. Sierra, A.J. Vazquez // Surf. Coat. Technol. – 2006 – 200 – 4383-4388.
- [3] G. Dercz, L. Pajak, B. Formanek // J. Mater. Process. Technol. – 2006 – 175 – 334-337.
- [4] D. Zhong, J.J. Moore, T.R. Ohno, J. Disam, S. Thiel, I. Dahan // Surf. Coat. Technol. – 2000 – 130 – 33-38.
- [5] B.T. Liu, C.S. Cheng, F. Li, L. Ma, Q.X. Zhao, et al. // Appl. Phys. Lett. – 2006 – 88 – 2529031-25290313.
- [6] A.B Markov, A.V. Mikov, G.E. Ozu, A.G. Padei // Instrum. and Experim. Tech. – 2011 – 54 – 862-866.

ROUGHNESS OF NICKEL AND TITANIUM ULTRATHIN FILMS COATED BY MAGNETRON SPUTTERING TECHNIQUE

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Ultrathin nickel and titanium films (up to 50 nm) are interesting as the basis for the multilayer coatings for the neutron optics mirrors [1]. Also it may be used as alternative technology of ITO coatings for transparent electrodes production [2]. In contrast the others materials (copper and silver) there is insufficient attention was paid to Ni and Ti ultrathin films technology. Also the most of articles is devoted to films obtained in laboratories and which are not applicable in industrial. The investigation of the influence of different parameters (magnetron discharge power Q , substrate temperature T_s and discharge gas pressure P_d) of ultrathin Ni and Ti coatings process to the film roughness in industry is the goal of this work.

The work was carried out at industrial magnetron facility. The films were coated with the industrial magnetron sputtering systems on the K8 optical glass substrates with argon environment. The magnetron power was from 200 to 1500 W when titanium film coating. When nickel film coating the power was from 200 to 1000 W. The substrates temperature was from 20 (room temperature) to 200 °C. The films were coated at discharge gas pressure was $8.2 \cdot 10^{-2}$ to $3.1 \cdot 10^{-1}$ Pa. The coatings thickness was 10 ± 1 nm. The film thickness and roughness were measured with atomic-force microscope (AFM).

It was determined that all the parameters are influence the coatings roughness. The smallest roughness of titanium coatings ($Rq = 0.13 \pm 0.05$ nm) was obtained at $P_d = 8.2 \cdot 10^{-2}$ Pa, $T_s = 200$ °C, $Q = 1500$ W. Nickel coatings with smallest roughness ($Rq = 0.15 \pm 0.05$ nm) was obtained at $P_d = 8.2 \cdot 10^{-2}$ Pa, $T_s = 100$ °C, $Q = 1000$ W. The coatings with maximum roughness ($Rq = 0.54 \pm 0.05$ nm and $Rq = 0.38 \pm 0.05$ nm for Ti and Ni respectively) was found at $P_d = 3.1 \cdot 10^{-1}$ Pa, $T_s = 100$ °C, $Q = 200$ W for booth materials. One needs to note that nickel coatings are smoother than titanium except for some experimental points.

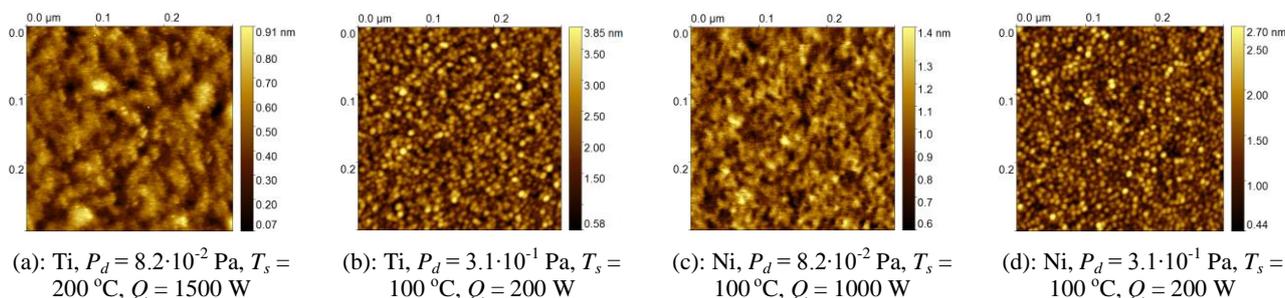


Fig 1. AFM images of the Ti and Ni coatings surfaces with smallest (a and c) and biggest (b and d) roughness

REFERENCES

- [1] *M. Hino, T. Oda, M. Kitaguchi, N.L. Yamada, S. Tasaki, Y. Kawabata // Nuclear Instruments and Methods in Physics Research A. - 2015. - 797. - Pages 265 – 270*
- [2] *S. Giurgola, A. Rodriguez, L. Martinez, P. Vergani, F. Lucchi, S. Benchabane, V. Pruneri // J. Mater Sci: Mater Electron. – 2009. – 20. – Pages 181–184*

SYNTHESIS AND PROCESSING OF POWDER MATERIALS IN DC ARC THERMAL PLASMA*

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The presentation summarizes the results of the research of the DC arc plasma jet processes that led to the creation of physical and chemical bases of the thermal plasma processes, providing production of powder materials with proper chemical and phase composition, including nanopowders and spherical particles in the micron size range.

It identified a number of promising applications of plasma synthesis of various nanopowders, including production of metal nanopowders (Ag, Cu, Ni, Co, W, Mo, Re), compounds (oxides, nitrides, carbides) and composites (W-C, W-Ni-Fe, W-Cu, Ag-SnO₂, Ni-TiCN) for various applications. As a result of the studies performed, ranges of possible variations in the physicochemical properties of powders obtained in plasma processes are established. Process parameters that control the change of these properties are defined.

The possibility of plasma spheroidization of a broad class of metal and alloy powders for additive technologies is shown.

Nowadays, IMET RAS is one of the leading organization in Russian Federation where the active researches and developments of processes and the equipment for spherical powders production of various materials in thermal plasma flow, including metals (Ti, Fe, Ni, W, Mo), alloys (stainless steel, Ti-V-Al, W-Ni-Fe, Nb-Si, Nb-C, Ni-Al, Ti-Al) and nanostructural metal-matrix composites.

As a result of studies performed, the possibility of metal powders spheroidization is shown for irregular particles shape of metals and alloys produced by various methods.

Particular attention is paid to the design of plasma process equipment and technological scheme, because, as it has been convincingly demonstrated on a commercial scale, the correct choice of raw material, the desired product, the design and equipment provides production of disperse systems in compliance with the environmental requirements and saving energy and resources.

For working and development of plasma processes of synthesis and production of pilot batches has been developed the original multifunctional plasma setup. The basic structural elements of this setup provide the necessary scaling to industrial production of broad range of nanopowders and spherical particles in the micron size range.

The results of research and development demonstrated broad possibilities of plasma processes and apparatuses based on DC plasma torches for producing nanopowders of metals and their various inorganic compounds and composites with desired properties. Nanopowders produced in plasma systems were used in a large number of research and development to prepare novel materials with special and improved properties.

Apart from nanopowders production, plasma reactors based on DC plasma torches enable spheroidization of metal and alloy powders to be used in additive technologies.

Accumulated experience forms the basis for the development of efficient industrial production of powders using plasma reactors on the basis of electric arc plasma torch.

REFERENCES

- [1] Samokhin A. V., Kirpichev D. E., Alekseev N. V., et al. // High Energy Chemistry – 2016. – 50. 466–472.
- [2] Samokhin A. V., Astashov A. G., Alexeev N. V., Tsvetkov Y. V. // Nanotechnologies in Russia. – 2015. – 11. – 57-62.
- [3] Samokhin A. V., Sinayskiy M. A., Alexeev N. V., et al. // Inorganic Materials: Applied Research. – 2015. – 528–535. .
- [4] Samokhin A. V., Alekseev N. V., Kornev S. A., et al // Plasma Chemistry and Plasma Processing. – 2013. – 33. – 605-616.
- [5] Krasovskii P. V., Samokhin A. V., Fadeev A. A., Alexeev N. V. // Advanced Powder Technology. – 2016. – 27. – 1669–1676.
- [6] Samokhin A., Alekseev N., Sinaiskiy M. // Powder Technology. – IntechOpen. – 2018.
- [7] Samokhin A. V., Fadeev A. A., Sinayskiy M. A., et al. // Russian metallurgy (Metally). – 2017. – 7. – 547-553.

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EHD cell parameters and collector effective area

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In recent years, an increasing interest in the development of methods for electro-hydrodynamic (EHD) control of gas flows has manifested itself throughout the world [1, 2]. Atmospheric airflow EHD devices consist of a plasma source, a drift region (acceleration block) and ion collector or neutralizer [5]. Often these three parts are united in one and form an EHD cell. The optimization of an EHD cell can significantly affect its efficiency, weight, and thrust [3, 4, 6]. In this paper, we investigated the effect of the collector electrode equivalent surface area (and the associated collector mass) on the thrust characteristics of the system consisting of five parallel cells. At the same time, the perimeter of the receiving electrode changed, and the length of the collector remained unchanged.

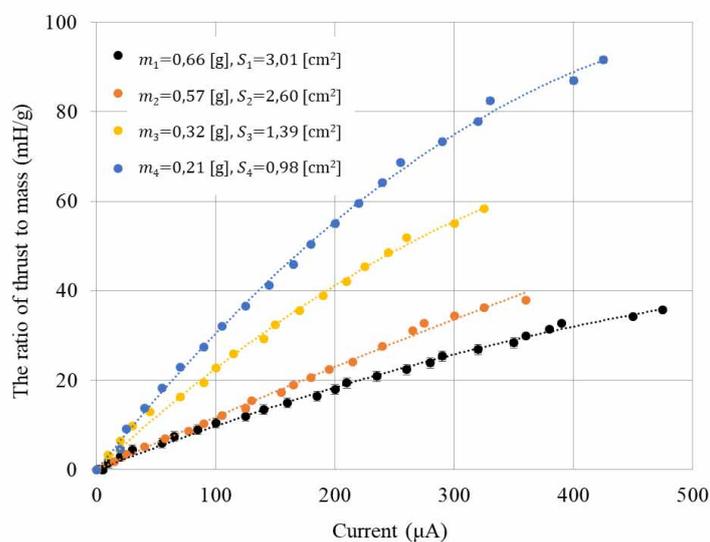


Fig. 1. The ratio of thrust to mass of collectors from on the current at different perimeters of the collectors.

The experimental setup consist of high-voltage power supply, 100 k Ω current-limiting resistor; voltmeter; EHD cell; screen; balance; microammeter. The system parameters of the EHD cell are: emitter radius $r_w = 0.04$ mm; collector radius $r_c = 0.8$ mm; interelectrode height $H = 18$ mm; intercollector distance $l_1 = 10$ mm; collector length $l_2 = 135.3 \pm 0.1$ mm; number of collectors $n = 6$. Experiments have shown that for a given geometry with closely spaced electrodes, maximum thrust is achieved at a positive corona for all values of emitter surface areas, and a decrease in the collector mass does not significantly effect on T. From the results presented in Figure 1, it can be seen that the specific thrust (the ratio of thrust to mass of collectors) per unit length increases with a decrease in their perimeter and, accordingly, the effective collector area. With a value of $I = 250$ μ A, the developed lift force is more than 60 times greater than the mass of the electrode system. The use of collectors with smaller perimeters leads to gain in payload.

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REFERENCES

- [1] Gilmore C., Barrett R.H. // *Nature*. 2018. Vol. 563, P. 532–535.
- [2] Khomich V.Y., Rebrov I.E. // *Journal of Electrostatics*. 2018. Vol. 95. P. 1–12.
- [3] Dremine D.V. et al. // *Journal of Physics: Conference Series*. 2017. Vol. 927. P. 012015.
- [4] Dremine D.V. et al. // *PLTP-2017. Kazan, Russian Federation, 2017. Vol. 1. P. 242.*
- [5] Drew // *Journal of Applied Physics*. 2013. Vol. 114, № 14. P. 143302.
- [6] Moreau E., Benard N. // *Journal of Physics D*. 2013. Vol. 46, № 47. P. 475204.

MODIFICATION OF DENTURE POLYMERS IN RF-DISCHARGE AND HYBRID PLASMAS: ADVANCED TECHNIQUE FOR CLINICAL PRACTICE

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Modification of hot curing poly(methyl methacrylate) (PMMA) denture base “Villacryl H Plus” in slow RF-plasma flows for material biocompatibility improvement is described. The capacitive RF-discharge was ignited in molecular oxygen by co-axial or planar electrode systems fed by generator Genesis GHW-12 (MKS Instruments, UK) at frequency 13.56 MHz. An electron beam (EB) could be injection into the space between electrodes; in this case so-called Hybrid Plasma (HP) was excited by joint action of the gas discharge and EB. Oxygen was blown in a zone of the plasma generation and samples to be treated were located directly in this zone. Special plasma chemical reactor [1] equipped with reaction chambers of various design (electrode configurations, gas nozzles, sample holders, EB modulator (optionally), etc) was developed to study materials modification under wide range of the processing conditions. As to PMMA the optimal treatment conditions were found to be as follows:

- RF-power –10-50 W depending on samples sizes;
- O₂ pressure in the working chamber– 670 Pa;
- O₂ flow – 5 sccm (standard cm³ × min⁻¹);
- accelerating voltage of the EB – 30 kV;
- EB current – 1-10 mA depending on the EB injection mode (continuous or interrupted injection were possible and various types of the EB scanning over the reaction zone could be applied).

The EB modulation and scanning supported uniformity of the sample surface modification and acceptable sample heating during the processing. In our experiments the treatment duration was varied but usually did not exceed 5 min to obtain desirable modification effect.

Experiments showed the plasma processing to result in increase in surface roughness of PMMA and change samples wettability. Significant increase of oxygen content and formation of additional oxygen containing polar chemical groups (C=O, –COOH, –OH) was also found.

Plasma chemical action on the PMMA decreased the water contact angles on modified surfaces by 1.5-2.5 times with respect to original ones and their surface free energy increased up to 1.5 times. In spite of gradual ageing, higher wettability of processed PMMA was observed at least after 7-day storage. Though both the HP and gas-discharge plasmas improve the PMMA wettability in approximately equal degree the HP gives more uniform treatment and the acquired hydrophilicity degraded significantly slower than in case of RF-plasma processing.

The biological tests on the human fibroblasts culture revealed the increased cell adhesion to the plasma-modified PMMA plates in comparison with original ones. The plasma processing also stimulated the cells growth on PMMA samples, i.e. improved the material biocompatibility.

The technique was tested on removable PMMA denture widely used in clinical practice for oral orthopedic rehabilitation of a patient after the treatment of buccal mucosa cancer. When using the non-modified denture, the patient complained of discomfort and food chewing problems and the hypertrophic red flat oral lichen formed at the patient’s cheek. The full regression of lichen nodules and associated inflammation was observed after the usage of the plasma modified denture for one week. Within six-month ware of the plasma modified denture no pathological elements or neoplasms were found on the patients’ oral mucosa [2].

Thus, the results of our study demonstrate that hot curing PMMA processed in RF- and Hybrid plasmas of oxygen can be effectively used in practical clinical dentistry.

REFERENCES

- [1] *T. Vasileva // IEEE Transac. Plasma Sci. –2010. – 38. –8. – 1903-1907.*
- [2] *T. Vasileva, Aung Myat Hein, A. Vargin, E. Kudasova, E. Kochurova, M. Nekludova // Clinical Plasma Medicine. – 2018. – 9. – 1-5.*

FORMATION OF THE SILICON COATING ON THE NITIT SUBSTRATE BY MAGNETRON SPUTTERINGⁱ

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This study considers the possibility of using silicon as a coating on a NiTi vascular stent. It is assumed that the porous structure will be obtained on this coating in the future. In turn, it will serve as a carrier for the drug. Silicon is one of the most common material in the nature. Because of its spread, easy obtaining, thermophysical, electrophysical and chemical properties, there is an advantage to use the silicon coating in various fields of science, medicine and technics [1].

At this study, the research of structure and properties of a silicon cover was conducted. The silicon cover was gained on the NiTi substrate by using the magnetron sputtering. NiTi containing 50.9 at. % nickel was chosen as a substrate because of the use this material for making cardiovascular implants. Silicon targets of 99.999 % purity were used for silicon coating. Deposition of Si on the NiTi substrate was performed by RF-magnetron sputtering. Argon was used as an inert gas. During the coating deposition, the working pressure in the vacuum chamber was 0.7 Pa. The distance between the target of cathode and the condensable surface on the NiTi substrate was 70 mm. The treatment was conducted for the creation of surface with diverse thicknesses by the using of different modes: mode I (P=250 W; t=120 min); mode II (P=250 W; t=15 min); mode III (P=150 W; t=15 min); mode IV (P=100 W; t=15 min). An electron microprobe (EMP) was performed for the study of coated samples (Fig. 1 a), microhardness was studied as well (Fig. 1 b).

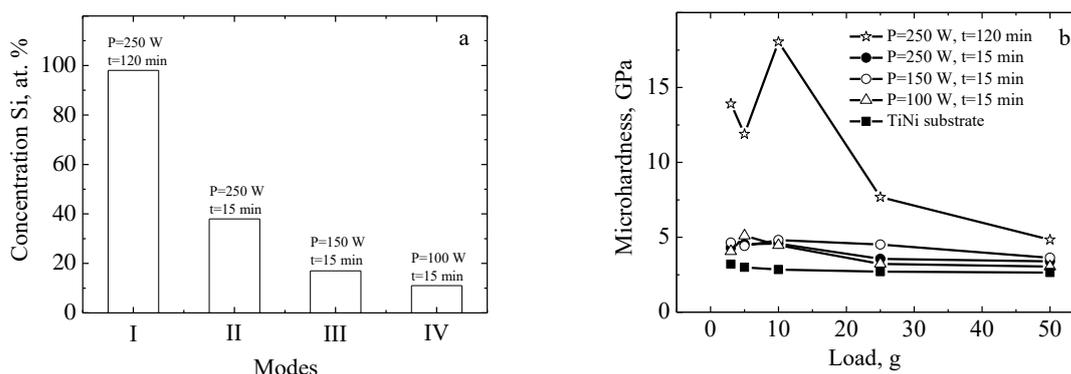


Fig. 1. The concentration of silicon in the surface layer of samples depending on the mode of sputtering (a); the microhardness of modified samples depending on the value of indenting load (b).

Microhardness was measured with various loads from 3 to 50 g. The analysis of results shows that the thickness of obtaining covers depends on the modes. It is noticed that due to the concentration of silicon in samples' surface layers. The area of X-ray generation in EMP does not exceed a depth of 5 μm for silicon. It means that the surface layer obtained in mode I with the silicon concentration of 98 at. % has comparable thickness of 5 μm . Such thickness is enough for creation of porous structure for the carrying of drug. The value of load defines the depth of dent and describes mechanical properties. It is supposed that the surface obtained by mode I has the biggest thickness and microhardness similar to silicon. Other samples have microhardness similar to NiTi. It means that the dent depth exceed the thickness of layer and does not influence on the value of microhardness.

Thus, the use of magnetron sputtering allows to receive a silicon cover on a nitinol substrate, where the obtaining thickness is enough for creation the porous structure. It is identified that the time of treatment has more influence on cover thickness than the power of magnetron.

REFERENCES

- [1] Klyueva V.A. // *Young Scientist*. – 2016. 10(114). P. 236–245.

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TECHNOLOGY OF HARDENING AND IMPROVING THE PERFORMANCE OF HIGH-SPEED STEELS BY GLOW DISCHARGE PLASMA

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The modification of high-speed steels is in interest due to their greatest applicability in the market for the production of cutting tools. They are used for the production of all types of cutting tools in the processing of carbon alloyed structural steels, preferably for the manufacture of thread-cutting tools as well as tools operating with shock loads. Tools made of high-speed steels have relatively low heat resistance, medium hardness, maximum bending strength and toughness as well as high endurance. Improving the performance of high-speed steels is an important task, the solution of which will provide cost-saving materials, energy and labor. At present various processing methods are used to improve performance among which there is plasma treatment.

In the framework of this study samples of high-speed steel were subjected to plasma treatment, the essence of which is that the products are placed in a vacuum chamber on the cathode [1]. Air is pumped out of the chamber and the high voltage power supply circuit is switched on, due to which a potential difference is created between the electrodes, the value of which is set within 0.2-3 kV. As a result, there is a breakdown of the discharge gap with the appearance of a glow discharge. Controlling the high voltage source and vacuum valves, the pressure of the residual gases, the discharge voltage and the current density within the required limits are set. After the processing time of the products in the glow discharge plasma the high voltage is turned off, air is introduced into the chamber and the processed samples are removed. The temperature in the chamber during the plasma treatment is controlled and does not exceed 343 K.

Electro-microscopic, X-ray structural and durometric methods for analyzing the phase composition, structure, and properties of the surface layer were used in the studies. The measurement of the hardness of the working surface of the samples was carried out according to the Vickers method. The study of the effect of treatment in a glow discharge on wear resistance was carried out on an upgraded equipment for testing materials for friction and wear [2].

As a result of this study it was found that the treatment of high-speed steel in a glow discharge leads to grinding and redistribution of the carbide phase in the surface layer to a depth of 20 μm , reducing the dislocation density both in the carbide phase and in the matrix material. Processing samples of high-speed steel leads to an increase in the wear resistance coefficient up to 3 times. After plasma treatment, the hardness of high-speed steel increases to 20% [3, 4].

The results can be used in industrial enterprises and in scientific organizations specializing in the field of plasma processing and materials science, as well as used in the educational process in the development of special courses designed for students of physical and engineering specialties.

REFERENCES

- [1] *U.M. Shamiankou et al. // The method of hardening products from metal or alloy or a superhard or graphite-containing material. – 2018.*
- [2] *U.M. Shamiankou et al. // Fundamental Problems of Modern Materials Science. – 2014. – Volume 11. – № 4. Pages 540-544.*
- [3] *V.V. Abidzina et al. // Fundamental Problems of Modern Materials Science. – 2016. – Volume 13. – № 4. Pages 522-529.*
- [4] *U.M. Shamiankou et al. // Fundamental Problems of Modern Materials Science. – 2017. – Volume 14. – № 4. Pages 535-541.*

HIGH-RATE DEPOSITION OF CHROMIUM COATINGS BY MAGNETRON SPUTTERING

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Magnetron sputtering of a heated (“hot”) chromium target can significantly improve the productivity of Cr coating deposition [1-5]. This is possible due to the formation of additional particle flux by sublimation process on the target surface. Apart from the increase of the deposition rate, a powerful energy flux onto the substrate appears due to the thermal radiation of the sputtered “hot” Cr target. Therefore, in such conditions, we should expect a significant change in the fluxes of matter and energy on the substrate and their specific characteristics (energy per one deposited atom).

This article presents data on the deposition rates of chromium coatings when a “hot” Cr target is sputtered and the energy characteristics of this process. These parameters is necessary to predict the properties of chromium coatings and to choice of the optimal deposition mode of a chromium coating both from the higher productivity and to ensure better functional properties.

The paper considers the influence of substrate pre-heating, discharge power, substrate bias, substrate location relative to the magnetron sputtering system and deposition time on the deposition rates and properties pf chromium coatings.

REFERENCES

- [1] *Sidelev D.V., et al. // Surface and Coatings Technology. – 2018. – 350. P. 560-568.*
- [2] *Bleykher G.A., et al. // Surface and Coatings Technology. – 2018. – 354. P. 161-168.*
- [3] *Sidelev D.V., et al. // Vacuum. – 2017. – 143. P. 479-485.*
- [4] *Sidelev D.V., et al. // Surface and Coatings Technology. – 2016. – 308. P. 168-173.*
- [5] *Bleykher G.A., et al. // Vacuum. – 2016. – 132. P. 62-69.*

EFFECT OF UV IRRADIATION OR DIFFUSE PLASMA ON SURFACE PROPERTIES OF MICRO-ARC CALCIUM PHOSPHATE COATINGS*

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Nowadays functionalization and modification of medical implants are widely used to add new set of properties. Calcium phosphate (CaP) coatings are widely applied as a component of dental, orthopedic and osteosynthesis implants in clinical settings due to porous structure and osseointegration ability [1]. Posttreatment of the CaP coatings using ultraviolet (UV) irradiation or plasma of runaway electron preionized diffuse discharge (REP DD) can improve the hydrophilic properties and vary the surface electrical charge, which significantly affects the protein and biomolecule adsorption, and cell adhesion [1,2]. However, the changes in activation mechanism, bioactivity stability and cell response of the UV and REP DD treatments of CaP coatings are still not clear. The work was focused on to study the influence of UV irradiation or REP DD posttreatment on the surface properties of the micro-arc CaP biocoatings.

The coating were deposited on commercial pure titanium by the MAO method in anodic potentiostatic mode under following parameters: the pulse duration of 100 μ s, the frequency of 50 Hz, the process time of 10 min, and the pulsed voltage of 200 V [1]. The electrolyte contained the phosphoric acid, calcium carbonate and stoichiometric hydroxyapatite. To modify the surface properties of the coatings the different posttreatments by UV irradiation and REP DD in the ambient air were carried out. There were three groups of the CaP coatings: 1) non-treated CaP coating; 2) post treated by UV CaP coating (KrCl-excimer lamp, $\lambda = 222$ nm, exposure dose of 5.5 J/cm², treatment duration varied from 1 to 20 minutes [2]); 3) post treated by REP DD CaP coating (the pulsed voltage of 18 kV with negative polarity, pulse duration of 4 ns, number of pulses varied from 10000 to 80000 [3]).

Wettability studies of not-treated CaP coating showed that the contact angles with water (polar liquid) and glycerol (non-polar) did not exceed 16° and 30°, respectively. Both UV irradiation and REP DD posttreatments of CaP coating leads to decrease of contact angles in 1.5-2 times with both liquids. The free surface energy, which calculated by the Owens-Wendt method [1], of non-treated CaP coatings as well as post-treated by UV or REP DD the CaP coatings was not differed and had high value of ~ 73 mJ/m². It is well known, that the free surface energy consists of two components of dispersive and polar ones. In the case of non-treated and post-treated CaP coatings, the polar component is more in ~ 3 times than dispersive component. It indicates the presence of strong polar covalent bonds in the coatings, such as OH-groups, phosphates, and oxides. After UV or REP DD post-treatment the redistribution of the values of the polar and dispersive components of the free surface energy of the CaP coatings was observed.

Infrared spectroscopy showed that the intensity of adsorption bands of the OH- and PO₄- groups increased in the coatings after UV post-treatment and decreased in the coatings after REP DD post-treatment in compared with non-treated CaP coatings. These results can indicate the possibility of redistribution of the electrical charge on the surface depending on the type and condition of the post-treatments.

REFERENCES

- [1] M.B. Sedelnikova, Yu.P. Sharkeev, E.G. Komarova, I.A. Khlusov, V.V. Chebodaeva // *Surface & Coatings Technology*. – 2016. – V. 307. – P.1274-1283
- [2] V.I. Erofeev, A.S. Medvedev, L.M. Koval', I.S. Khomyakov, M.V. Erofeev, V.F. Tarasenko // *Russian Journal of Applied Chemistry*. – 2011. – V. 84. – 10. P. 1760–1766.
- [3] M.V. Erofeev, V.S. Ripenko, M.A. Shulepov, V.F. Tarasenko // *Instruments and Experimental Techniques*. – 2017. – V. 60. – P. 287–289.

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CONTACTLESS PARTICLE FILTERING BY ALTERNATING ELECTRIC FIELD

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The problem of gas filtering during the environmental degradation is very important problem in nuclear power engineering, mechanical engineering, the chemical industry and other industries. The electrodynamic traps (Paul traps) can be used in contactless filtration processes, monitoring and diagnostics of microparticles and aerosols in gases.

In the work the theoretical and experimental results on charged particle filtering are shown. Particles were previously charged in corona discharge unit and then captured inside quadrupole electrodynamic trap. The areas of particle confinement as the dependencies on geometries of the trap, the parameters of alternating voltage, gas flow velocities and particle parameters (charge, size, density) were found. The results showed the possibility of selective particle filtering.

Also particle capturing inside electrodynamic trap can be used for particle's parameters diagnostics and particle separation.

HYBRID METHODS FOR OBTAINING PLASMA CHEMICAL COATINGS N.M.CHEKAN¹, Y.V.AUCHYNNIKAU², I.P.AKULA¹, E.I. EISYMONT², A.N.GORELCHIK²

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The formation of thin-layer vacuum coatings with various operational parameters allows to significantly increase the range of application of modified materials. To intensify the modifying action of thin-layer coatings, various methods of substrate activation are used [1]. For example, in [2], the influence of the pulsed high frequency voltage applied to the substrate on the crystallite size during the deposition of a TiN coating was considered. It was established that during pulsed deposition of coatings, there is a significant decrease in crystallite sizes from 60 nm to 10 ÷ 15 nm. The nature of this structural behavior is associated with the formation of a highly dispersed non-equilibrium grain structure with a high dislocation density. Methods are proposed for treating a solid dielectric substrate with high-frequency plasma, which leads to a decisive influence on the deposition process of metal films of the electret structure formed due to the redistribution of induced and adsorbed charged particles in the surface layers of the dielectric substrate. As a result of the application of this method of processing the substrate, coatings with enhanced adhesive strength and microhardness are formed. The aim of this work is to develop hybrid methods for hardening metal-working tools, which consist in combining methods of plasma-chemical deposition and cryogenic processing. Tribotechnical studies were carried out on a FT-2 type friction machine, which operates according to a reciprocating scheme, the stroke length of the indenter from 5 to 50 mm under dry friction conditions (counterbody), made of steel and ground on a flat flat surface with emery cloth or grinding paste to arithmetic average deviation of the surface profile $R_a = 0.1 - 0.3$ microns. The samples were fixed in the friction machine clamp, rubbed with a “coarse calico” cloth, bleached, dipped in ethanol, the working sphere and the working surface of the steel disk (counterbody), and then dried for two minutes at room temperature. The tests were carried out with a normal load on the sample up to 20 N, a linear sliding speed of 0.036 m / s, and a surface temperature of steel (20 ± 5) °C. During the conducted research it was established that during cryogenic treatment of zirconium carbonitride coatings with short exposure intervals (120-360 minutes) in cryogenic liquid, the strength characteristics of the coatings increase, a further increase in the exposure time (24-48 hours) does not lead to a further increase in the strength characteristics. The increase in strength characteristics may be explained by the formation of a fine-grained structure in the coating layer, as well as the formation of a nano-dispersed phase. The tribological characteristics of composite coatings based on zirconium carbonitride, subjected to cryogenic treatment, are investigated. An increase in the wear resistance of the coatings during cryogenic treatment has been established. With increasing shutter speed in a cryogenic environment, wear resistance increases and friction coefficient values decrease. Cryogenic treatment of coatings of complex chemical composition obtained by vacuum technologies in the medium of the reaction gas during deposition on steel substrates leads to ambiguous results. According to acoustic emission data, the treatment of ZrCN compounds formed on high-speed steel in liquid nitrogen leads to a decrease in the adhesive interaction with the substrate. This effect is manifested to a greater degree at longer exposure times of the coating in a cryogenic liquid. Heat treatment of ZrN coatings formed on steel R6M5, with small time intervals of exposure to cryogenic liquids, can increase the adhesive interaction in the “substrate-substrate” system.

REFERENCES

1. Vickers hardness properties of structural materials for superconducting magnet at cryogenic temperatures / Y. Yoshino [et al.] // *Cryogenics*. – 2001. – Vol. 41. – P. 505 – 511.
2. Wanstrand, O. *Wear Resistant Low Friction Coatings for Machine Elements* / O. Wanstrand // *Comprehensive Summaries of Uppsala Dissertations from the Faculty of Science and Technology, Acta Universitatis Upsaliensis*. – Uppsala, 2000. – 32 p.

MODIFICATION OF DIE STEELS SURFACE IN A PLASMA OF NON-SELF-SUSTAINED GLOW DISCHARGE *

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Thanks to a combination of high mechanical characteristics and good technological effectiveness at a relatively low cost among structural and tool materials the most widespread at present and perspective for application in the future will be steel. Therefore, the improvement of existing and the creation of new methods of treatment of steels is an actual scientific and technical task. The vacuum ion-plasma surface modification methods, in particular, nitriding, are considered as the most progressive to improve the service properties of details. The nitriding in the plasma of low-pressure discharges (~ 1 Pa) allows to control independently main operating parameters (ion current density on the material surface, ion energy, product temperature). A non-self-sustained glow discharge with a hollow cathode is promising for plasma generation in large (~ 1 m³) vacuum volumes [1]. The purpose of this work is to define the influence of nitriding process parameters (ion current density, electrical bias voltage) on the regularities of structure and phase composition formation in the surface layer of Cr12MoV and Cr6WV die steels. Studies of the influence of the structure and phase composition of the surface layer on the physico-mechanical characteristics (wear resistance, microhardness) of steels were carried out. The results of scientific work are of considerable interest for the further development of technological modes of exhaust die tools surface treatment.

REFERENCES

- [1] Akhmadeev Y.H., Denisov V.V. et al. // *Plasma Physics Reports*. – 2017.- V. 43. - Issue 1. – P. 67-74.

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COMPLEX MODIFICATION OF THE SURFACE OF HIGH-SPEED STEEL IN LOW-TEMPERATURE HIGH-DENSITY PLASMA *

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Today, the aircraft engine industry is actively increasing the requirements for structural materials in order to improve the basic characteristics of the engine: increased thrust, increased efficiency, reduced fuel consumption. In this connection, the working temperature in gas turbine engines increases and hard-to-use heat-resistant alloys are increasingly used. The low machinability of these alloys is determined by their physico-mechanical properties [1,2]. Given this trend, the requirements for metal-cutting tools are also increasing (cutters, cutters, slotting tools, etc.).

To solve the problem of increasing the durability of metal-cutting tools used in the aviation industry from high-speed steels and hard alloys, a method for complex surface modification in low-temperature high-density plasma has been proposed, including ion nitriding and subsequent formation of multi-layer nanostructured coatings based on intermetallic compounds of the Ti-Al system synthesized in reactive gases O, C, N environments. Studies of the effect of low-temperature nitriding and subsequent formation multilayer coatings based intermetallic Ti-Al system to change the microhardness of the surface layer and the wear resistance of tool materials.

A technological process has been developed for complex modification of the surface of tool materials in low-pressure discharges, including low-temperature nitriding and subsequent deposition of multilayer coatings based on intermetallic compounds of the Ti-Al system. The results of production tests of metal-cutting tools processed by the developed complex technology are presented.



Fig. Metall cutting tools after complex modification of the surface of high-speed steel in low-temperature high-density plasma

REFERENCES

- [1] Kimura A., Hasegawa H., Yamada K., Suzuki T. // *Surf. Coat. Technol.* – 1999. – Vol. 120-121. – P. 438-441.
- [2] E. L. Vardanyan, I. I. Yagafarov, V. V. Budilov, R. M. Kireev // *Strengthening technologies and coatings*, 2014, 6, pp 7-10
- [3] Budilov V V, Vardanyan E L // 2016 *Journal of Surface Investigation: X-ray, Synchrotron tools and Neutron Techniques* Vol. 10 No4 pp 728–731

INFLUENCE OF ULTRASONIC WAVES DURING MICRO-ARC OXIDATION ON STRUCTURE AND PROPERTIES OF CALCIUM PHOSPHATE COATINGS*

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The aim of the present work was to study the effect of external uninterrupted ultrasonic (US) or pulsed ultrasonic (PUS) waves during the micro-arc oxidation (MAO) on the growth rate, structure and properties of calcium phosphate (CaP) coatings formed on the commercial pure titanium (Ti) surface.

Synthesis of the CaP coatings on Ti samples was carried out by the MAO method using the Microarc-3.0 installation in the electrolyte and under the conditions described previously [1]. There were three types of the coatings depending on the conditions of external US: 1) MAO-coating (without US); 2) MAO/US-coating (with US, $P = 100$ W, $\nu = 35$ kHz); 3) MAO/PUS-coating (with PUS, $P = 35$ W, $\nu = 37$ kHz).

It is seen in Figure 1a, during the MAO process the current density decreases monotonously due to the formation and thickness growth of the dielectric CaP coating. It should be noted that the MAO process under the action of US or PUS is characterized by a higher current density than that without additional US. Figure 1b, c confirms this by the fact that the increase in the coating thickness and surface roughness (R_a) occurs more intensively with additional US than without it.

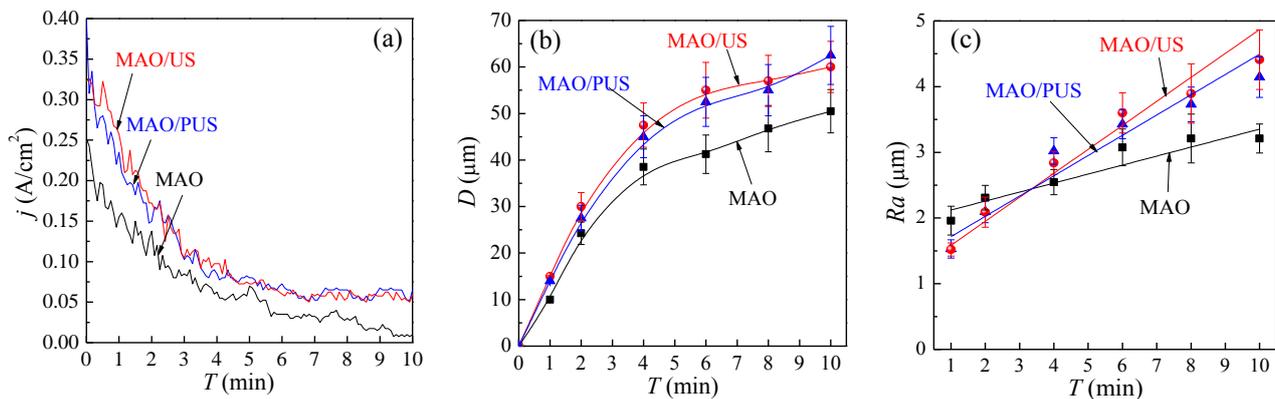


Fig. 1. Graphs of the MAO current density (a), the coating thickness (b) and roughness (c) against the MAO time for different types of the coatings

SEM studies showed that the applied external US field during the MAO process effects on the structural and morphological properties of the coatings. The surface morphology of the coatings formed without UV is represented by the structural elements of spheroidal shape (sphere) with open pores. However, under the action of external US there are destruction of structural elements and filling pore spaces with fragments, which leads to a decrease in surface porosity from 25 to 12 %. At the same time, the internal porosity of the coatings increased from 25 to 40 % due to the formation of macro-pores of 15-40 μm in sizes.

It was found that the external US increases the content of Ca and P in the coatings, and there is a structural-phase transition from the X-ray amorphous state to the amorphous-crystalline state with the content of $CaHPO_4$ and $\beta-Ca_2P_2O_7$ phases.

Thus, it was shown that high frequencies of ultrasonic vibrations at low amplitude create an acoustic field with a high level of energy, which allows intensifying the processes of mass transfer in the electrolyte, increasing the growth rate of the coatings, as well as controlling the composition, structure and porosity of the formed coatings.

REFERENCES

- [1] Sharkeev Yu., Komarova E., Sedelnikova M., Khlusov I., Eroshenko A., Litvinova L., Shupletsova V. Bioactive microarc calcium phosphate coatings on nanostructured and ultrafine-grained bioinert metals and alloys. In: Bioceramics and Biocomposites: From Research to Clinical Practice / ed. I. Antoniac. – New Jersey: John Wiley & Sons, 2019. – P. 191-231.

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NANOSCALE DYNAMIC EFFECTS INSTEAD OF TEMPERATURE. LOW-TEMPERATURE STRUCTURAL STATES FORMED BY ION IRRADIATION*

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With decreasing temperature, interatomic distances decrease, the role of covalent bonds increases. This determines the predisposition of binary and multicomponent media to the formation of low-temperature phases, including those with reduced symmetry. From the condition of the minimum of the free energy $F = E - T \cdot S$, it follows that at 0 K, only pure components and stoichiometric compounds or their mixtures are thermodynamically stable. At the same time, at temperatures $T < (0.2 \div 0.3)T_{melt}$, diffusion processes in condensed media are actually frozen and the equilibrium states are unattainable for actually imaginable time intervals.

At present, the important role of nanoscale dynamic effects in the action of ionizing radiation on condensed matter is being actively studied (see review [1]). These effects, which take place during irradiation with accelerated ions (as well as reactor neutrons, fission fragments), remain outside the field of vision of classical radiation physics. They are associated with the processes of explosive energy release in areas of dense atomic displacement cascades with the formation of nanoscale thermal spikes for trillionths of a second, heated to $3000 \div 6000$ K and higher with thermal pressures of $5 \div 40$ GPa, in some cases exceeding the theoretical yield strength of materials. These processes occur in the surface layer $< 1 \mu\text{m}$ thick. However, they lead to the formation of post-cascade powerful elastic and shock waves capable of carrying out liquid flow of condensed media on their front, initiating structural and phase transformations that theoretically propagate in metastable media for unlimited distances. In this case, radiation shaking with post-stage waves plays the role of temperature, increasing the atomic mobility without heating the medium.

To prove the significant, and in some cases, decisive role of shock-wave processes (but not radiation-enhanced diffusion), it is necessary to reduce the exposure time of the ion beam in order to eliminate the role of migration processes. It was shown in [2] that the diffusion path length of vacancies in pure aluminum $l = (D/\tau)^{1/2}$ for 1 s is only $0.4 \mu\text{m}$. At the same time, in an ideal lattice, the mileage of interstitial atoms in 1 s can reach several tens and even hundreds of microns and decreases only due to the presence of traps and sinks. However, by limiting the exposure time to $\tau \leq 0.001$ s (using special diaphragms), the role of thermal and radiation-enhanced diffusion (from the surface into the rest of the substance) for objects with a thickness of more than several tens of micrometers can be completely excluded.

In this paper, we used temporal apertures, which set the exposure to 0.001; 0.01 and 0.1 s. It was shown that the formation of a short-range and long-range atomic order in iron alloys from 6.25 at. % Si, 6.25 and 8.25 at. % Mn occurs during their low-temperature irradiation ($T < 300$ °C) with low doses of Ar^+ and Xe^+ ions, which is not related to diffusion processes, but is caused exclusively by post-cascade dynamic (shock-wave) effects. The values of the parameters of the short- and long-range atomic order were determined as a result of processing the Mössbauer spectra of samples $\sim 30 \mu\text{m}$ thick (initially disordered by cold plastic deformation) after exposure to accelerated ions. In samples subjected to similar thermal effects, but in the absence of irradiation, atomic ordering processes are not detected.

Thus, as a result of the study, the possibility of accelerated formation of short- and long-range atomic order in the $\text{Fe}_{1-x}\text{Y}_x$ alloys ($Y = \text{Si}$ and Mn), including at temperatures below the thermal threshold for defrosting diffusion, was proved. At that, the increase in atomic mobility is not due to an increase in temperature and acceleration of diffusion, but dynamic post-cascade effects.

REFERENCES

- [1] V.V. Ovchinnikov // Surface & Coatings Technology. – 2018. – Vol. 355. – № 9. – P. 65-83.
- [2] N.V. Gushchina, V. V. Ovchinnikov, A. Mücklich // Phys. Status Solidi B. – 2016. – Vol. 253. – № 4. – P. 770-777.

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FORMED NANO-W - WC COATINGS ON THE HIGH-SPEED STEEL SUBSTRATE BY THE ELECTRODISCHARGE EXPLOSION*

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Electric exploding of a tungsten carbide – cobalt material near-by high-speed steel (HSS) surface forms on it a hardening coating. These coatings raise the cutting tool resistance in 1,5 – 3 times. The essential structure properties of the formed coatings are determined by parameters of contact exploding electrode at the pulse current amplitude from above 1 MA/cm² and duration less than 10⁻⁴ s [1]. The investigations of coating structures were done by optical (Fig. 1) and electronic metallography. They have shown that the contact electric exploding caused the transfer of tungsten carbide and cobalt on the surface of high-speed steel.

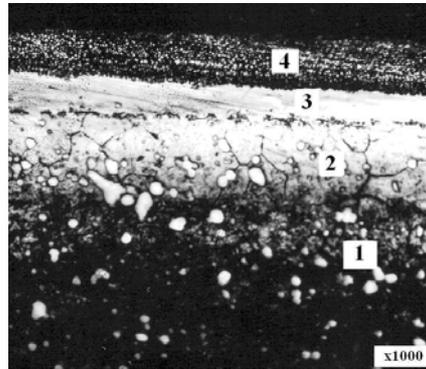


Fig. 1. Optical microscopic image of the polished surface of the WC/W coating on HSS substrate:

1 – unmodified HSS, 2 – modified HSS, 3 – WC layer, 4 – nanostructure (mean grain size < 400 nm) pure W

The breakdown of tungsten carbide – cobalt material took place during electrical exploding. The hardening layers of tungsten carbide and pure nanocrystalline tungsten have been formed upon the surface of high-speed steel as a result of electric exploding. Crystalline grains of tungsten have an almost spherical form and their characteristic size less than 400 nanometers. The layer of tungsten carbide has a high hardness (HV ~ 1800). Localization of heating and subsequent high-speed cooling in the small volume of high-speed steel results in formation of different structures. A metallographic analysis gives the clear picture of distributing of these structures and possibility of direct determination of their thickness. Micro hardness of the coating layers and high-speed steel structures was measured.

Theoretical analysis [2] made it possible to establish the dependence of the volume (ΔV) of an electrically exploded material on the amplitude of the electric current pulse - J and the properties of the material (electrical resistivity - ρ and boiling point – T_b):

$$\Delta V \sim \rho \cdot J^2 / T_b^4, \quad (1)$$

REFERENCES

- [1] Grigoryev, E.G. // *AIP Conference Proceedings*. – 2010. - Vol. 1315. P. 1395-1399.
- [2] Grigoryev E.G., Olevsky E.A. // *Scripta Materialia*. - 2012. - Vol. 66. - Issue 9. P. 662-665.

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THE TRANSMISSION SPECTRUM SWITCHING SPEED OF ELECTROMAGNETIC BAND GAP PLASMA STRUCTURE

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During the last decade, there has been an increasing interest in high-speed tunable microwave devices based on EBG structures for use in telecommunication systems that are capable of operating at high power levels. Gas discharge plasma as a control element of such devices has high potential for this purpose due to its variability in electron density and geometry [1]. The properties of one-dimensional EBG plasma structure in the X-band waveguide formed by discharges at low pressure were described in [2]. In [3], the possibilities of microwave control by long pulse atmospheric pressure discharges were demonstrated. The switching speed of transmission of such EBG plasma structure is under investigation in this work.

The one-dimensional plasma EBG structure is formed by three pulse discharges at atmospheric pressure in a waveguide with rectangular cross-section $23 \times 10 \text{ mm}^2$. Discharges are ignited between two electrodes (1 mm in diameter, interelectrode gap is 11 mm) in quartz tubes with an inner diameter of 1.6 mm and placed perpendicular to the wide walls of waveguide with period of 30 mm. Helium, argon and Ar-O₂(<2%)-H₂(2%) mixture at flow rates about 1 liter/min are used as working gases. The mixture is needed for the diagnostics purposes. The ignition of the discharges is realized from one high voltage square pulse (Nanogen 1 from RLC electronic) of variable amplitude up to 5.5 kV and duration of 1 μs (pulse rise time is about 25 ns).

The experiments are performed at different pulse voltages from breakdown voltages depending on working gas (Fig. 1a, vertical dashed lines) up to 5.5 kV. In Fig. 1b, the waveforms of current pulse (1) and corresponding microwave signal (2) passed through EBG structure are shown. The dependence of current growth rate and fall rate of transmitted microwave power on the applied voltage pulses were determined (Fig. 1a). The switching time is less in Ar and Ar-O₂-H₂ discharges (about 35 ns) than in case of He (150 ns) discharges, while the decay time of afterglow plasma is shorter in case of mixture (about 4 μs).

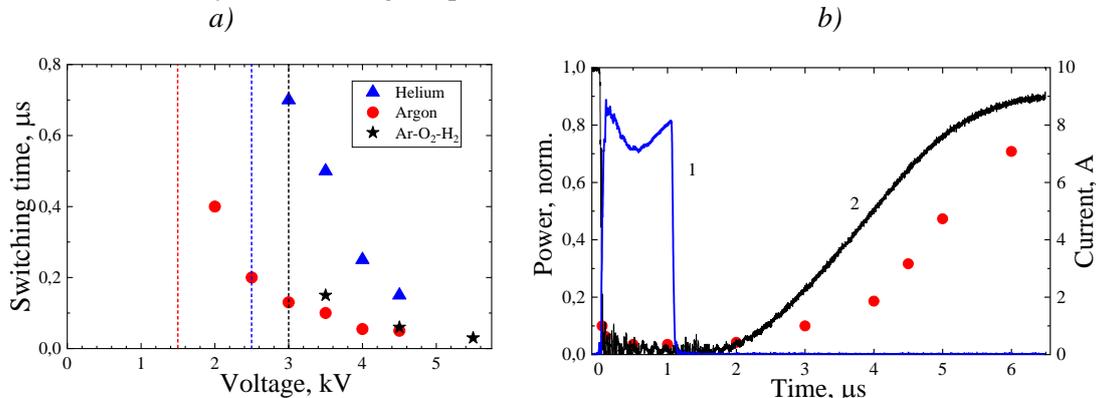


Fig. 1. a) The switching time vs pulse voltage for different gases. Dash lines are breakdown voltages; b) The waveforms of current pulse (1) and microwave signal passed through EBG structure (2) in case of mixture and simulated transmission (circles)

The time resolved H _{α} and H _{β} line profiles for Ar-O₂-H₂ mixture at current about 8 A were registered using ICCD camera with spectrometer (PIMAX and ACTON SP300i from Princeton Instruments). Time evolution of electron concentration was determined. The discharge diameter estimated from a series of photos made with ICCD camera is about 0.25 mm. Modeling of EBG plasma structure transmission is performed using the Ansoft HFSS software. The simulation results of transmission (Fig. 1b, symbols) are in satisfactory agreement with experimentally registered ones.

The obtained results demonstrate the possibility of developing high-speed microwave elements (switches, limiters, attenuators) under plasma control operating at high-power microwaves.

REFERENCES

- [1] Sakai, O. et al. *Plasma Phys. Contr. Fusion.*, 2005, 47, B617.
- [2] Arkhipenko V.I. et al. *J. of App. Phys.*, 2014, 116, 123302.
- [3] Babitski V.S. et al. *J. of App. Phys.*, 2017, 122, 083302.

INFLUENCE OF GAS DISCHARGE PLASMA ON FILMS OF COMPLEX COMPOSITION FORMING PROCESS AND PROPERTIES*

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Currently, to obtain films of complex composition, ion-plasma methods are of great interest, which allow varying the technological parameters to produce films of various modifications and different compositions for various applications.

The main disadvantage of these methods is the negative impact of electron-ion bombardment of the growing film, which can lead to the breakdown of dielectric films, the formation of a mobile charge, an increase in fixed charge and the density of surface states [1]. At the same time, the degree of impact of electron-ion bombardment depends on the films producing methods implementation.

The authors have developed ion-plasma sputtering methods based on the Penning discharge and magnetron sputtering system, which were used to develop technologies for producing insulating and conducting films of complex composition, representing various compounds of oxides and nitrides of Al, Si and In metals [2]. In particular, this paper discusses the results of studying the mechanism of the formation of the stoichiometric compounds of thin films of complex composition, formed by the method of ion-plasma sputtering in the medium of active gases. The kinetics of their growth is investigated, the results of studies of changes in the properties of thin films and substrates during ion-plasma processes, as well as the study of changes in the characteristics of the elements of microelectronics formed on their basis during their electron-ion bombardment are presented.

It was found that during ion-plasma sputtering of metal targets in the medium of active gases, the formation of final compounds (Si_3N_4 , AlN, Al_2O_3 , In_2O_3 :Sn) occurs on the substrate, when atoms and metal ions arrive, as well as gas molecules and ions of the working atmosphere.

It was established that in the process of deposition of thin films of complex compounds by ion-plasma methods, even at low discharge power, the substrate temperature significantly increases. At the same time, directly heating the substrate to form stoichiometric films of complex composition plays the most important role, contributing to the formation of the crystalline phase [3]. However, the plasma effect is manifested not only in the heating of the substrate surface, but also in the appearance of a negative potential on it.

The effect of electron-ion bombardment was studied in *MDM* and *MDS* capacitors. Si_3N_4 films served as dielectric capacitors. Different degrees of plasma effect on the film properties were provided by changing the substrate distance to the discharge. The parameters of the *MDM* capacitors remained fairly good with the intensity of electron-ion bombardment that takes place in the Penning gas-discharge chamber in the case of an isolated substrate under a floating potential. Electron-ion bombardment has a more significant effect on the characteristics of *MDS* capacitors. We investigated the effect of electron bombardment on the properties of the silicon-nitride silicon interface. For this purpose, capacitance-voltage (*C-V*) characteristics were obtained for the case of a floating potential on the substrate and the substrate that was at the anode potential. The *C-V* characteristics showed that an increase in the intensity of electron bombardment leads to the appearance of a large positive charge at the interface and to an increase in the density of surface states N_T . Vacuum annealing significantly reduces N_T , but the positive charge does not decrease.

Thus, the formation of films by ion-plasma methods is accompanied on the one hand by negative impact of electron-ion bombardment on a substrate with a growing film, and on the other hand, low-energy electron-ion bombardment can contribute to the formation of films of stoichiometric composition.

REFERENCES

- [1] L.R. Bitner, V.A. Vedernikov, T.I. Danilina, G.V. Manyakhina // *Russian Physics Journal*. – 1976. – 19. – № 12. 11-15.
- [2] T.I. Danilina, P.E. Troyan, Yu.V. Sakharov, Yu.S. Zhidik // *TUSUR reports*. – 2017. – 20. – № 3. 40-45.
- [3] Yu.S. Zhidik, P.E. Troyan // *TUSUR reports*. – 2012. – 26. – № 2. 169-171.

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LOW TEMPERATURE ICP ETCHING InP/InGaAsP HETEROSTRUCTURE IN Cl₂-BASED PLASMA*

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At the present time, InP is one of the base materials of integrated optoelectronics. The development of optoelectronic devices often requires the formation of waveguide structures with a high aspect ratio and a smooth surface morphology. For the formation of such structures is widely spread method of ICP etching in Cl₂ based plasma. The advantage of this method is the possibility of independent control of the density and energy of plasma ions, which provides flexible control of the etching conditions. However, chlorine-based processes have a significant disadvantage. In the process, the InCl₃ compound with low volatility at room temperature occurs in etching products, which leads to the formation of grass-like defects on the surface of etched structures. Usually, to solve this problem, the substrate is heated to 150-200 °C and above [1, 2]. It prevents redeposition InCl₃ on the surface of the etched structure and the formation of grass-like defects. Another way to remove InCl₃ from the surface is to enhance the role of the physical component of the etching process [3].

This paper presents the optimization of the low-temperature ICP etching process of an InP/InGaAsP heterostructure in a Cl₂/Ar/N₂ plasma. The relationship between RF power and process pressure on the etching profile of the heterostructure and the formation of grass-like defects is shown. The possibility of using multi-stage etching processes to reduce the surface roughness of the etched heterostructure is considered. The developed etching process has a high anisotropy. The angle of inclination of the etching profile is close to 90°. Surface roughness does not exceed 30 nm. Also, it has low selectivity with respect to the InP and InGaAsP layers. Fig. 1 shows a SEM image of the cross section of the etched InP/InGaAsP heterostructure after etching in optimized etching conditions.

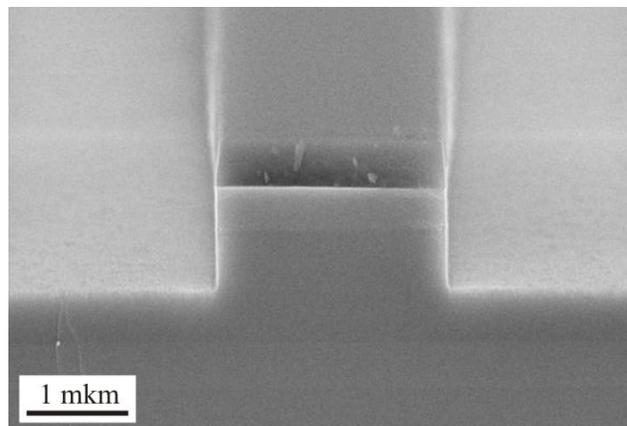


Fig. 1. SEM image of the cross section of an InP/InGaAsP heterostructure after ICP etching in a Cl₂/Ar/N₂ plasma.

REFERENCES

- [1] *C.F. Carlstrom et al. // J. Vac. Sci. Technol. B. – 2008. – Vol. 26. – № 5. P. 1675-1683.*
- [2] *S.L. Rommel et al. // J. Vac. Sci. Technol. B. – 2002. – Vol. 20. – № 4. P. 1327-1330.*
- [3] *L. Chee-Wei et al. // Chin. Phys. Lett. – 2006. – Vol. 23. – № 4. P. 903-906.*

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INVESTIGATION OF CATALYST OBTAINED FROM ALUMINUM OXIDE PRODUCED BY PLASMA SYNTHESIS¹

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Nano-sized aluminum oxide is used in many industries, including widely used as a substrate for catalysts. As is known, the formation of an active and stable catalyst is influenced by the morphology, phase composition and chemical nature of the carrier and the active phase. The chemical nature of the aluminum oxide surface, which means the state and structure of the hydrate cover, the concentration and the distribution of acid-base centers by strength, determines, on the one hand, the adsorption and catalytic properties of the oxide, and on the other hand, allows controlling the reactivity of the surface in solid-phase reactions. These characteristics are largely determined by the degree of surface hydration and depend on the nature and texture of the starting materials, the method of preparation and the crystallographic structure of aluminum oxide.

A sample of aluminum oxide was obtained by the method of plasma thermal decomposition of aluminum nitrate [1]. The plasma torch is powered from an AC power source (50 Hz) with a voltage of 6 kV [2]. This voltage allows to reignite the electric arc without current skips. To stabilize the electric arc in the gap between the electrodes, plasma gas (air) supply is organized. One of the sides of the plasma torch is plugged, and through the second side the stream of air plasma (1 g/s) is fed to the reaction volume. To determine the acid-base properties, the produced aluminum oxide was sequentially impregnated with cerium nitrate and nickel nitrate. The synthesized product was controlled by XRD. The nature of the effect of nickel oxide on the aluminum oxide carrier was studied using the following physicochemical methods: X-ray phase analysis (XRD), scanning electron microscopy (SEM) and IR spectroscopy. The functions of the Hammett and the specific surface area of the samples were also determined.

The pH-metry method was used to determine the predominant type of acid-base centers of the surface: the acid and Lewis base. The Hammett function was evaluated using the pH-metry method, which is based on the kinetic control of the pH of an aqueous solution in contact with the solid. Measurement of the pH made it possible to establish the type of acid-base centers on the surface of the samples: before the impregnation with nickel oxide, the acid sites predominate on the aluminum oxide surface, and after the impregnation the main acid sites predominate.

REFERENCES

- [1] *Subbotin D.I., Surov A.V., Popov S.D., Serba E.O., Obratsov N.V., Spodobin V.A., Popov V.E., Kuchina J.A., Kiselev A.A. // IOP Conf. Series: Journal of Physics: Conf. Series. —2018. — Volume 1135. Pages 012105.*
- [2] *Subbotin D.I., Surov A.V., Kuznetsov V.E., Pavlova E.A., Azartsova V.V., Kuchina J.A., Dudnik J.D. // Journal of Physics: Conf. Series. — 2018. — Volume 1115. Pages 032093.*

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ELECTRIC DISCHARGE DESTRUCTION OF REINFORCED CONCRETE SLEEPERS WITH DIFFERENT MODES OF PULSE POLARITY

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Analysis of publications in the world on the topic of electrical discharge destruction shows that today interest in electrical discharge technologies for processing materials, such as drilling or crushing, is rapidly increasing [1-6]. However, publications associated with the destruction of concrete products and removal of the surface layer is not enough.

Experimental data of the destruction of reinforced concrete sleeper in the system of electrodes placed on the sample were obtained. The tests were carried out at different modes: bipolar pulse, pulses with positive and negative polarities. The optimal charging voltage was determined, which was chosen according to the criteria for the occurrence of breakdown at the top or in the decay of a pulse signal without overvoltages in the system with the given interelectrode distance.

During the experiment, the sample was destroyed before the first layer of reinforcement (Fig. 1.), and it can be noted that the destruction near the reinforcement shows the worst result among all the stages of destruction, since the amount of consumed specific energy is more than in all other stages. Also, there is the smallest result in terms of the volume of the broken-off material. This happens due to the fact that a significant part of the impulses falls on the reinforcement, and not on the destruction of concrete.

As a result of destruction, the reinforcement can be completely removed without any additional effort.



Fig. 1. View of the sample with the installed electrode plate system.

REFERENCES

- [1] *Ushakov V.Ya., Vazhov V.F., Zinoviev N.T.* // Electro-Discharge Technology for Drilling Wells and Concrete Destruction. Basel: Springer Nature Switzerland AG; 2019
- [2] *Anders E., Voigt M., Lehmann F.* // Electric Impulse Drilling: the future of drilling technology begins now. ASME. International Conference on Offshore Mechanics and Arctic Engineering, Polar and Arctic Sciences and Technology; Petroleum Technology. 2017;
- [3] *D. Molchanov, V. Vazhov, I. Lavrinovich, V. Lavrinovich, N. Ratakhin* // Downhole generator based on a line pulse transformer for electro pulse drilling // 2017 IEEE 21st International Conference on Pulsed Power (PPC) Tomsk, 2017
- [4] *Weiran Zuo* // A study of the applications and modelling of high voltage pulse comminution for mineral ores // thesis for the degree of Doctor of Philosophy at The University of Queensland, 2015.
- [5] *Hans O. Schiegg, Arild Rødland, Guizhi Zhu, David A. Yuen* // Electro-Pulse-Boring (EPB): Novel Super-Deep Drilling Technology for Low Cost Electricity Journal of Earth Science, Vol. 26, No. 1, 037–046, 2015
- [6] *Gilbrech Joshua* // Pulse Transformer for Downhole Electrocrushing Drilling // Int. Patent WO2018/186828 A1 11.10.2018

ADVANCED FUNCTIONAL COATINGS DEPOSITED USING SUPERSONIC ATMOSPHERIC PLASMA SPRAYING*

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The development of thermal spraying (TS) methods over the past decade has been associated with an increase in the velocities of the sprayed particles in order to improve coating characteristics such as density, hardness, adhesive strength, corrosion resistance, etc. The guidelines in this area are high velocity oxygen fuel (HVOF) and detonation spraying (DS) methods, in which gas flows at a speed of 2000...2500 m/s provide acceleration of particles of the material up to 500...800 m/s and above. However, advances in the development of supersonic atmospheric plasma spraying (S-APS) equipment in recent years have demonstrated that this method is capable of providing comparable parameters for particles of the dispersed phase and the quality of coatings. In addition, in recent years, plasma spraying of suspensions and liquid precursors (SPS and LPPS) has developed intensively, in which the formation of coatings of small particles (0.1–3 μm) requires an increase in their velocity above 500 m/s.

This paper presents the latest results of the ITAM team's work in the development of the supersonic version of a spraying DC plasmatorch PNK-50. Increasing the speed of the plasma flow to the level of 2000 ... 2500 m/s allows the application of functional coatings with outstanding characteristics. Thus, using the low-enthalpy (low-temperature) S-APS regime, high-density wear-resistant coatings from powder materials of NiCrSiBC metal alloy and WC/CoCr composite material were obtained, demonstrating characteristics previously available only to high-speed HVOF and DS methods. The use of the high-enthalpy (high-temperature) regime of supersonic plasma equipment in the methods of SPS and LPPS made it possible to obtain advanced coatings of ZrO₂ ceramics. Thermal barrier coatings (TBC) with a columnar structure, high-density YSZ coatings for the formation of solid oxide fuel cells (SOFC) electrolytes, as well as coatings with a pronounced bimodal surface profile with super-hydrophobic effect were obtained.

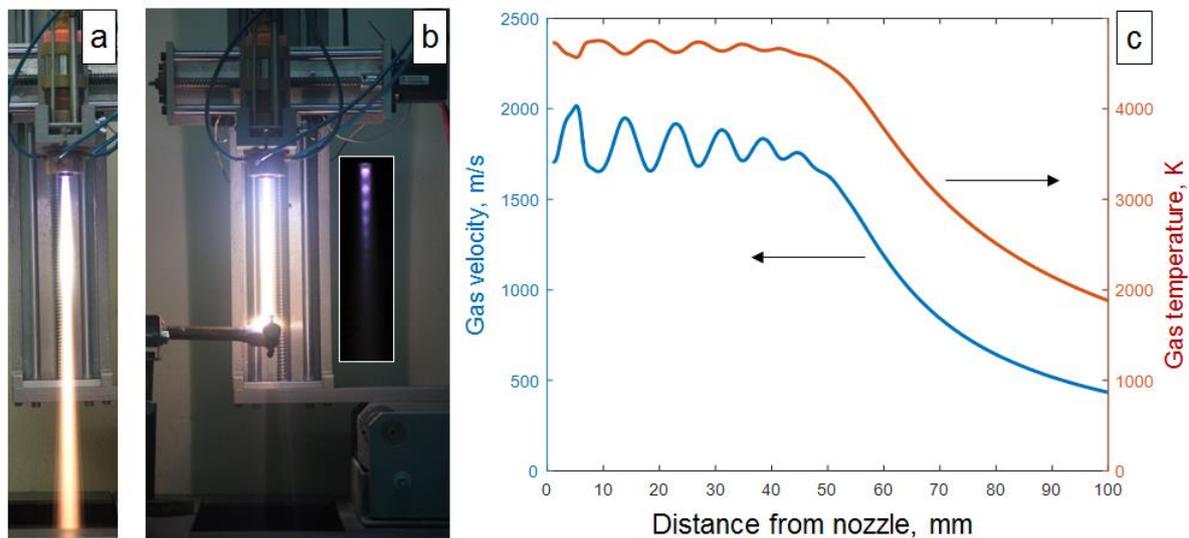


Fig. 1. Supersonic plasma spraying using PNK-50 torch: a) high-velocity spraying jet; b) deposition process of WC/10Co4Cr coating; «shock diamond» are shown in the insert; c) calculated gas temperature and velocity distribution on the axis of the jet in high-enthalpy supersonic plasma flow.

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THE SIGNAL RADIATION BY THE PLASMA ASYMMETRICAL DIPOLE ANTENNA

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A plasma asymmetric dipole antenna (PADA) is an analogue of a metal asymmetric dipole antenna (MADA) with a round screen, and consists of a pin (dipole arm) connected to the central conductor of a coaxial cable and a conductive disk connected to an external conductor of a coaxial cable. In the case of a plasma antenna, the metal pin is replaced with a gas discharge tube with a plasma. Such type of plasma antenna from discharge tube is most popular [1]. Plasma in a tube can be generated either by an external source connected to a gas-discharge tube, or by a source of a radiated high-frequency signal (generator or coherent transmitter). Creating a plasma in the discharge tube of the PADA due to the signal energy from a transmitter allows you to simplify the connection pattern of the plasma antenna and make it similar to the MADA connection. The most convenient way to feed the PADA from the transmitter, as in the case of the MADA, is to connect them using a coaxial cable. With this method of connection, the plasma gas discharge tube is connected to the central (inner) conductor of the coaxial cable, and the screen is connected to the external conductor of the coaxial cable.

Despite the large number of the PADA studies [2], in this work, for the first time, we made a comprehensive research on the characteristics of the signal radiated by the PADA. The electromagnetic field patterns, radiation patterns and signal spectra of the plasma and metal antenna were studied by analytical, numerical and experimental methods. Numerical simulation was carried out in the full electrodynamic code KARAT [3] using the FDTD method for calculation of electromagnetic field and the Drude theory and the PIC method as plasma models.

The study consisted of three parts. In the first part, the dispersion relation for the surface electromagnetic wave was numerically solved for the case of an unmagnetized plasma cylinder, the distribution of the electromagnetic field in the near field, and the radiation patterns were calculated in the KARAT code on the Drude model for different plasma densities, the radiation pattern for the PADA and the MADA were experimentally measured. It was shown that there are three modes of operation of a plasma asymmetric dipole antenna, determined by the dispersion characteristic: surface wave modes (nonradiative), nonlinear and linear. These modes are determined by the ratio of the plasma Langmuir frequency and the frequency of the supplied electromagnetic wave and related to the propagation conditions of the surface electromagnetic wave on the plasma cylinder. In the linear mode, the radiation pattern and the field distribution of the plasma antenna are close to those of a metal antenna.

In the second part, the radiation spectra of the unmodulated sinusoidal oscillation with the frequency near 450 MHz were studied using numerical simulations using the PIC plasma model and experimental measurements. As a result, it was found that the amplification of the components in the spectrum of the oscillations at the harmonics of the frequency 450 MHz when it was radiated by the PADA. The amplification of harmonics in the spectrum of the unmodulated oscillation depends on the power of the electromagnetic wave supplied to the antenna and the matching of the antenna with the feeder path.

In the third part, the radiation of a narrow-band frequency-modulated signal by the PADA was experimentally measured. The power of nonlinear combination frequencies in the spectrum of a narrowband frequency-modulated signal radiated by a plasma asymmetric dipole antenna is lower than in the spectrum of the same signal from by a metal asymmetric dipole antenna. In the spectrum of the detected signal received from the plasma antenna, the power at the second harmonic of the frequency of the modulating oscillation is less than for the signal from the metal antenna.

The report is dedicated to the memory of Professor A.A. Rukhadze.

REFERENCES

- [1] *Bogachev N.N. et al. // Plasma Physics Repors. – 2019. – V. 45. – № 4. P. 372-375.*
- [2] *Alexeff I. et al. // Physics of Plasmas. – 2008. – V. 15. – №. 5. P. 057104.*
- [3] *Tarakanov V. P. // User's manual for code KARAT. – Springfield: BRA, 1992.*

CHARACTERIZATION OF ALUMINA DEPOSITION PROCESS IN A HIGH POWER PULSED REACTIVE MAGNETRON SPUTTERING*

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Transparent aluminum oxide coatings are extensively used in various applications in electronics, mechanics, optics, etc. They demonstrate outstanding mechanical properties, high resistance to abrasion and corrosion [1, 2], coupled with attractive optical properties for enhancing characteristics of lenses and producing reflection-type polarizers [3].

We studied the deposition process of alumina coatings in a high-current impulse magnetron discharge (HCIMD [4, 5], or long HiPIMS). The aluminum target was sputtered in Ar/O₂ gas mixtures with different partial pressure ratios. Total pressure was 0.5 Pa. The pulse duration was varied in millisecond range, with different duty factors. Both peak and average cycle power were changed in order to determine the most appropriate sputtering conditions. Dependence of alumina mass deposition rate on O₂ gas content for different pulsed discharge time signatures is shown in Fig. 1.

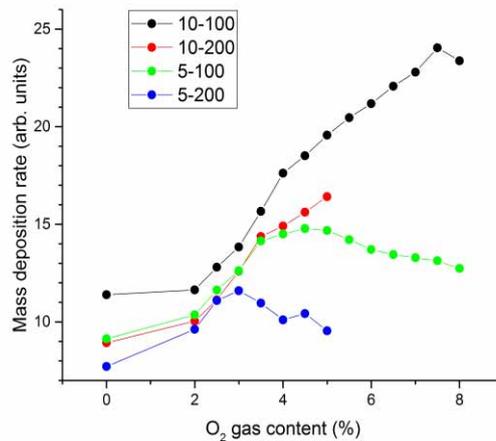


Fig. 1. Mass deposition rate of alumina depending on O₂ gas content

REFERENCES

- [1] *Dorre E., Hübner H. // Alumina: Processing, Properties and Applications. – Springer-Verlag, Berlin, 1984.*
- [2] *Wiatrowski A., Patela S., Kunicki P., Posadowski W. // Vacuum. – 2016. – Vol. 134. – P. 54–62.*
- [3] *Hass G., Francombe M.H., Vossen J. L. // Physics of Thin Films: Advances in Research and Development. – Academic Press, New-York, 1982.*
- [4] *Mozgrin D.V., Fetisov I.K., Khodachenko G.V. // Plasma Physics Reports. – 1995. – Vol. 21. – P. 400–409.*
- [5] *Shchelkanov I.A., G.V. Khodachenko // Proc. 537th WE-Heraeus-Seminar Physics of Ionized and Ion-Assisted PVD: Principles and Current Trends, Dresden, Germany, 2013. – P. 77*

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IRRADIATION OF A WHITEFLY BY SUBMICROSECOND ELECTRON BEAM AT ATMOSPHERIC PRESSURE*

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A submicrosecond electron beam (up to 250 keV, 500 A, 200 ns) is used at atmospheric pressure for irradiation of a whitefly in this work. The electron beam was ejected from vacuum diode of pulsed electron accelerator directly to atmosphere without of drift chamber. Whitefly probes were irradiated at various distances from exit window of the accelerator. Energy distribution of the electron beam was measured for dose estimation. Lethal and shock effects were demonstrated for whiteflies at various distances and beam pulse number. Single pulse of the electron beam at distance 80 mm from exit window leads to total dissection of the whitefly probe.

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TITANIUM SURFACE TEXTURING INDUCED BY ARGON ION BOMBARDMENT IN AN ICP DISCHARGE

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Currently, plasma technologies are widely used to improve the quality of products manufactured for a vast amount of applications ranging from cutting tools to medical implants. Studies of the properties of micro- and nanostructures on the titanium surface, as well as methods for their preparation, are of great interest, especially for orthopedics and implantology since the microtexture of implants affects the speed and quality of the material integration into the bone tissue.

In this contribution, we studied the effects of the argon ion bombardment parameters on the surface structure of samples obtained during plasma processing in an inductively coupled plasma (ICP). Samples of VT1-0 Russian grade titanium were irradiated at different radiofrequency power $P_{rf} = 700, 1200, \text{ and } 1500 \text{ W}$. The irradiation was carried out in a pulsed fashion with frequency $f = 35 \text{ kHz}$. The sample current was varied by changing the duty factor $D = 10\text{--}80\%$. The energy and the average ion flux on the sample determined the temperature of the sample $T = 500\text{--}750^\circ\text{C}$. Plasma density was measured by a Langmuir probe. The samples were analyzed in a scanning electron microscope (Fig. 1). For each of the surface topography modes obtained, the sputtering yield was calculated.

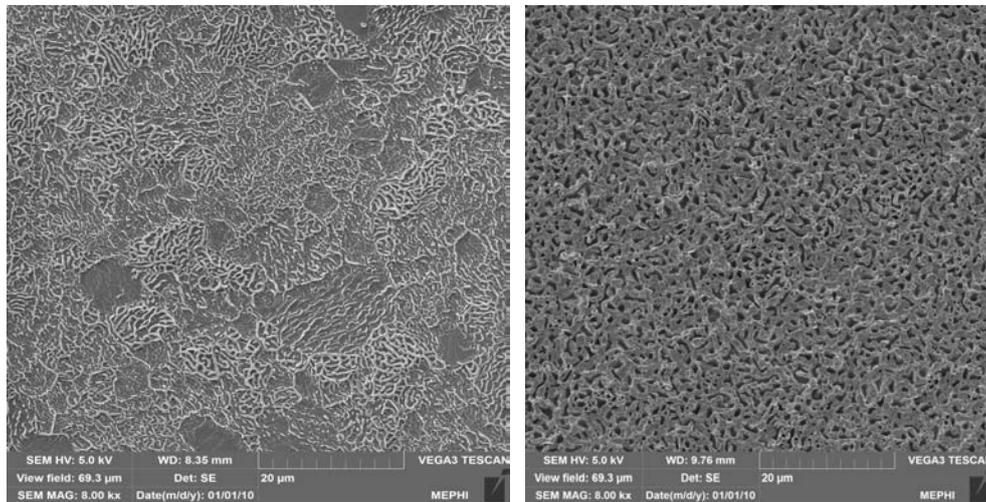


Fig. 1. Different modes of VT1-0 surface microtexture obtained by irradiation in Ar plasma

It is assumed that the development of such structures on the surface is determined by the influence of sputtering, melting, the presence of local crystal lattice defects, changes in grain sizes during heating, temperature gradients on the surface, and phase transition of the crystal lattice.

In the course of the work, the influence of the plasma treatment temperature, the duty factor of the pulsed periodic irradiation, and the plasma density on the nature of the obtained structures was examined. Understanding these dependencies would enable controlling and producing structures with characteristic dimensions required by a certain application.

PULSED CORONA DISCHARGE OXIDATION OF AQUEOUS DISSOLVED ORGANIC SUBSTANCES

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Atmospheric pressure gas-phase pulsed corona discharge (PCD) is a promising instrument in water oxidative treatment. Active oxidant species formed in air or oxygen discharge plasma, such as hydroxyl radicals (OH) and ozone (O₃), react with aqueous organic and inorganic dissolved impurities resulting in their decomposition and, also, water disinfection.

High reactivity of hydroxyl radicals and their short lifetime requires generation of the discharge plasma in close vicinity to target pollutants. Underwater and gliding surface discharges frequently used for this purpose concentrate energy in a few narrow channels resulting in radicals' recombination and low energy efficiency. The highest yield of active species and the treatment energy efficiency is achieved in gas-phase PCD. However, reactive species are largely produced in the gas phase, at a distance from the water surface, making the energy density in PCD with optimized mass transfer conditions in the plasma-water contact equipment the key factor for maximum oxidation efficiency.

The impact of process parameters on the oxidation efficiency of organic substances of various oxidation kinetics was studied. The discharge was formed in a wire-plate electrode system placed into a rectangular stainless-steel reactor sized 0.2×0.2×1.0 m. High-voltage wire electrodes were horizontally positioned between vertical grounded plates having the treated water flow perpendicular to wires. The distance between the wire and plate electrodes comprised 20 mm. Pulsed corona was energized by high-voltage positive polarity pulses with 200-300 ns duration, 20 kV amplitude and 0.3 J pulse energy. Water was dispersed in air flowing through a perforated plate: free-falling water droplets and jets passed through the discharge zone. The volume of treated samples was 20 L. In batch experiments, solutions were prepared in a storage tank, fed to the reactor for treatment, and recirculated after the treatment to the same tank. Solutions of humic acid sodium salt and oxalic acid were used in the experiments as target pollutants with relatively slow oxidation rates. Humic substances are widespread natural contaminants frequently found in natural waters. Slower reacting oxalate is a common byproduct of organic substances oxidation.

Experiments showed that parameters of the electrode system, such as the distance between the neighboring high voltage electrodes (10-30 mm) and the length of the high-voltage wire (8-25 m), did not have a noticeable influence on the oxidation rate of model substances, provided the discharge energy remained constant. With the same pulse energy, decreasing the wire electrode length from 25 to 8 m resulted in the volumetric energy density increased for more than three times. However, the discharge parameters, such as the energy, ozone yield and oxidation rate remained practically unchanged.

Increasing the energy consumption by changing the pulse repetition frequency from 200 to 860 pulses per second resulted in the increased oxidation rate at a reduced oxidation efficiency. The effect was better seen with slowly reacting oxalate, which was more efficiently oxidized at a lower pulse repetition rate, i.e. longer treatment time.

Accelerated water flow resulted in more efficient oxidation of oxalate: the oxidation rate increased by 30-40% when the flow rate changed from 0.5 to 2.4 m³·h⁻¹, due to the increased water-air contact surface. However, improved contact between treated solutions and ozone present in the PCD reactor by its recirculating by means of a Venturi tube did not bring better oxidation.

An attempt was made to increase the residence time of water in the discharge zone by introducing polyethylene shavings into the inter-electrode area. This resulted in 30% lower oxidation efficiency of humic substances due to increased ohmic losses and discharge concentration visible by the discharge glow at fixed points of the plastic bed leading to high temperature in the discharge channels and lower yield of active species.

The most important parameter influencing the efficiency of organic substances oxidation appeared to be the discharge power, i.e. pulse repetition rate. Further research is needed in the mass transfer impact to oxidation efficiency.

ANTIBACTERIAL POTENTIAL of Zn- and Cu- SUBSTITUTED HYDROXYAPATITE COATINGS DEPOSITED by RF-MAGNETRON SPUTTERING: STRUCTURE and PROPERTIES¹

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A tremendous number of traumatic cases associated with the bone fractures fostered the market need for implants that can provide immobilization of bone fragments using minimally invasive surgical access and quicker recovery of the patient. In order to address this problem, we introduced newly developed intramedullary implants. Those implants already proved their effectiveness in cases where fixation of a proximal bone fracture of tubular bones is needed. On the other hand, the problem of postoperative infections still the main challenge for modern health care. In case of severe post-implantation infection revision surgery is usually needed as the treatment with antibiotics does not provide the desired outcome. Therefore, our approach to this challenge lies in the surface modification of intramedullary implants by an RF magnetron deposition of antibacterial calcium phosphate (CaP) based coatings with the addition of Zn or Cu. The ions of Zn and Cu are known to have an antibacterial effect and their application is extensively researched in the biomedical engineering field.

We aim to develop novel bioactive and antibacterial coatings with enhanced osseointegration properties consisted of CaP+Zn and CaP+Cu based materials on Ti-6Al-4V and Ti-6Al-7Nb alloys of medical applications. We aim for improvement of the immunocompatibility of the novel implants as well as their antibacterial properties. And finally, we are working towards a translation from model samples to coated implant prototypes for validation of the improved osseointegration, antimicrobial activity, and immunocompatibility in order to demonstrate the proof-of-concept of the developed surfaces.

The targets for sputtering were sintered from hydroxyapatite with the addition of Zn and Cu ions that is substituting Ca in the cation lattice prepared by mechanochemical synthesis. A vacuum installation, with a planar magnetron operated at 13.56 MHz, was utilized for the CaP+Zn and CaP+Cu deposition. The thickness of the deposited films was measured by Calotest. For the coating's characterization methods such as an X-ray diffraction, scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used. For biological assessment of developed coatings in vitro cytotoxicity test with MG-63 and C2C12 cell lines was used. Antimicrobial testing was performed with E.Coli using a disk diffusion assay.

The estimated thickness of the deposited coatings was in the range of ~1.0 μm for both CaP+Zn and CaP+Cu. An SEM revealed that both types of coatings remain dense, homogeneous without any inclusions and discontinuities. According to AFM, the deposited coatings alter the implants' roughness insignificantly and mostly repeats the shape of an original surface. However, it is possible to detect globular-like surface features of the deposited coatings. The coatings revealed to be quasi-amorphous according to an XRD data. This is beneficial for the coatings stability on the implant that is undergoing mechanical stress during implantation. Moreover, amorphous coatings will be dissolved more quickly releasing antibacterial ions and ensuring the antibacterial effect. Deposited coatings showed the absence of toxic effect in vitro and noticeable antibacterial effect.

In our study, we addressed the problem of antibacterial surfaces for implants and specifically for intramedullary fixator developed by us. We were able to functionalize the surface of an implant with bioactive (Ca and P releasing) antibacterial coatings and performed preliminary study in which we discussed its properties and effectiveness in vitro.

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APPLICATION of GLANCING ANGLE DEPOSITION for MANIPULATION of THIN CALCIUM PHOSPHATE COATINGS MORPHOLOGY ¹

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Calcium phosphate (CaP) coatings are a widely researched topic which, over the years, resulted in lots of applications in the field of bone regeneration. It is due to the fact that conventional metallic implants become encapsulated by fibrous tissue, which in turn not only prolongs the healing time, but also leads to implant loosening and eventually premature failure of implantation. Not to mention bear Ti corrosion rate in the physiological fluids which might cause metallosis.

The modern approaches to healthcare are aiming to produce implants with biomimetic properties. These properties are crucial to ensure desirable biological response to the newly implanted material, in the manner that the cells, which are adhered to the surface of such scaffolds can function in a way that is similar to physiological conditions. From that point of view, the formation of a coatings that consists of different types of surface gradient structures and with variation in the level of roughness in submicro- and nanoscale could be of significant interest for biomimetic purposes. Thus, a possibility to manipulate nanotopography attracts much attention in the recent years.

Physical vapor deposition (PVD) of thin films, allowing the deposition of porous and/or columnar-like structured coatings, has been available for some years. In turn, the use of an oblique angle geometrical configuration, or as it is also referred as glancing angle deposition (GLAD) method, is frequently exploited for formation of three-dimensional columnar micro- or nanostructured surfaces. It is generally accepted that the mechanistic factor controlling the nanostructural evolution of the films is a “shadowing effect”, which prevents the deposition of particles in regions situated behind initially formed nuclei (i.e., shadowed regions) [1].

An emerging method for bioactive coating deposition in the field of PVD is radiofrequency (RF) magnetron sputtering method [2]. Magnetron sputtering is widely used in the formation of coatings for various applications. The continuous interest of scientists for this method is due to the possibility of modifying the coating structure and its physicochemical properties by variation of the deposition parameters. There is a significant interest in radiofrequency (RF) magnetron sputtering of bioactive CaP thin films. This method allows deposition of CaP coatings with a high level of adhesion to substrate.

In our work we show the influence of GLAD geometry on the morphology and structure of thin calcium phosphate films deposited by RF magnetron sputtering method. The method allowed us to manipulate the coating roughness on the submicron and nanoscale levels. A significant change in the coating morphology was revealed when the substrate tilt angle was set to 80°. It was shown that an increase in the coating crystallinity for samples deposited at a tilt angle of 80° corresponds to formation of crystallites in the bulk structure of the thin film. Cross section SEM revealed inner structure of deposited coatings and predominant growth towards the particle flux was easily detectable. The GLAD of complex calcium phosphate material can lead to the growth of thin films with significantly changed morphological features and can be utilized to create self-organized nanostructures on various types of surfaces [3].

REFERENCES

- [1] *A. Barranco et al. // Progress in Materials Science. – 2016. – 76. – 59-153.*
- [2] *R.A. Surmenev et al. // Acta Biomaterialia. – 2014. – 10. – 557-579*
- [3] *K. A. Prosolov et al. // Coatings. – 2019. – 9. – 220.*

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THE TECHNOLOGY OF LOW-TEMPERATURE ION NITRIDING OF AUSTENITIC AND MARTENSITIC STEELS WITH ULTRAFINE-GRAINED STRUCTURE

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Presently in the engineering industry there is an increase in interest in structural materials with ultrafine-grained (UFG) structure [1]. These materials have high strength properties [1,2]. Improving the surface hardness of parts from steels with UFG structure, ion nitriding in a glow discharge is often used. Ion nitriding of steels is carried out at high temperatures (550-600°C), which is unacceptable in the case of processing steels with a UFG structure [3,4]. Therefore, the development of low-temperature ion nitriding technology for such materials is an urgent task.

In the present work, austenitic and martensitic steel grades were subjected to low-temperature ion nitriding in a glow discharge at 450°C, 6 h in gas mixture: nitrogen N_2 , argon Ar and hydrogen H_2 used installation ELU-5M [5]. Before nitriding, steel samples were subjected to high pressure by torsion (HPT) treatment.

In Fig. 1 shows the microhardness distribution of the surface along the radius of samples of steels of austenitic and martensitic classes with a UFG structure after ion nitriding in a glow discharge at temperatures 450, 500 и 550°C.

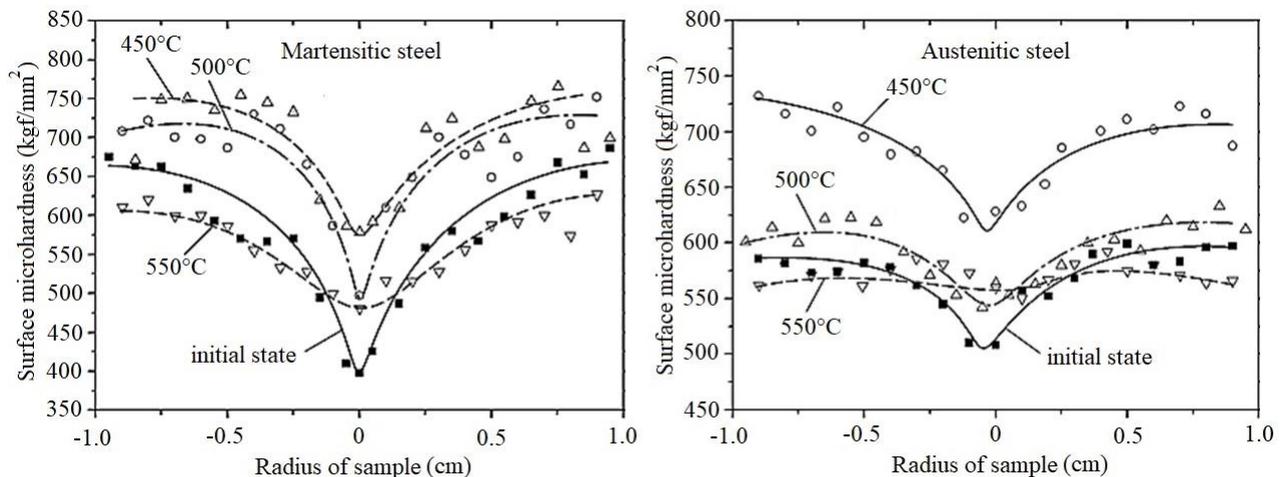


Fig. 1. Surface microhardness distribution by diameter of steel samples after ion nitriding at different temperatures

As a result of the analysis of the obtained distributions (Fig. 1), it was found that in the middle of the radius of the samples, the hardness reaches maximum values for austenitic steel around 580 kgf/mm², and for martensitic steel around 650 kgf/mm². The maximum degree of hardening (22-25%) was obtained on samples that passed nitriding at a temperature of 450°C (low-temperature ion nitriding). With an increase in the treatment temperature from 500°C to 550°C, the nonhomogeneity of the distribution of microhardness decreases, and the surface hardness decreases due to the onset of recrystallization processes of the UFG steel structure.

REFERENCES

- [1] Valiev R. Z, Islamgaliev R. K. and Alexandrov I. V. // Progress in materials Science. – 2000. – 45. – №2. Pages 103-189.
- [2] Farkel H. et al. // Scripta Materialia – 2002. – 46. – №9. Pages 623-628.
- [3] Tong W.P., et al. // Surface and Coatings Technology – 2008. – 202. – №20. Pages 4957-4963.
- [4] Li Y. et al. // Applied Surface Science – 2010. – 257. – №3. Pages 979-984.
- [5] Budilov V. V. et al. // Reinforcement technologies and coatings. – 2014. – 12. Page 27.

PECULIARITIES OF THE FORMATION OF HIGH-INTENSITY ION BEAMS OF GASES, METALS AND SEMICONDUCTOR MATERIALS*

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The work is devoted to the study of some regularities and features of plasma-immersion formation of high-intensity low-energy ion beams of gases, metals and semiconductor materials. It was shown, that repetitively pulsed gas ion beams are sustainably formed both in the system with spherical and cylindrical focusing geometry at negative bias in the range of 0.6–3 kV, pulse repetition rate from units of pulses per second (p.p.s) up to 10^5 p.p.s, and pulse durations up to 100 μ s. The formation of high-intensity metal ion beams requires pre-injection of plasma into the equipotential drift space of the focused beam. The space charge neutralization processes define several features of high-intensity ion beams, including a complex dynamic of focusing and beam instabilities appearing with the increase in beam pulse duration up to 15 μ s. The specificity of silicon beam formation is associated with low conductivity of silicon. For the purpose of pulsed vacuum arc plasma generation, the silicon cathodes were neutron transmutation doped on the nuclear reactor of Tomsk Polytechnic University. It was shown, that the process of high-intensity silicon ion beam formation might be accompanied with periodic instabilities of beam transportation and following recovery of space charge neutralization and its transportation. The conditions of sustainable generation of ion beams of gases, metals and semiconductor materials with the current of about 1 A and current densities of 0.5–1 A/cm² at accelerating voltages of several kV were defined.

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EFFECT OF THE REACTIVE GAS IMPURITIES IN HELIUM PLASMA ON THE FUZZY TUNGSTEN STRUCTURE

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The most pressing issue in designing a fusion reactor is a choice of the first wall material capable of withstanding exposure to hot plasma. At the moment, tungsten has been chosen as the material of the divertor tiles in ITER. Studies of plasma impact on tungsten involves not only interaction with D and T species but also with fusion reaction products (He) and impurity elements of residual gases. The accumulation of helium at high temperatures is accompanied by the growth of nanostructures on the tungsten surface. It is also an urgent task to determine the effect of impurity elements on the surface structure and accumulation of helium.

In work the tungsten samples were irradiated by ions of helium and impurity elements (nitrogen, oxygen) in an inductively coupled plasma (ICP) reactor. Irradiation was carried out at sample temperature of 1200 K, the ion energy of 150 eV, and plasma density of $\sim 10^{12} \text{ cm}^{-3}$. The bias voltage was applied to the samples in a rectangular pulsed mode. During He irradiation, N_2 , O_2 , and their mixtures were introduced in the amount of 1, 2, 4, and 8% of the total pressure. Thermal desorption analysis of He accumulation depending on the presence of impurity elements was carried out. The surface structure was analyzed with a scanning electron microscope. The images of obtained fuzzy tungsten structures are shown in figure 1.

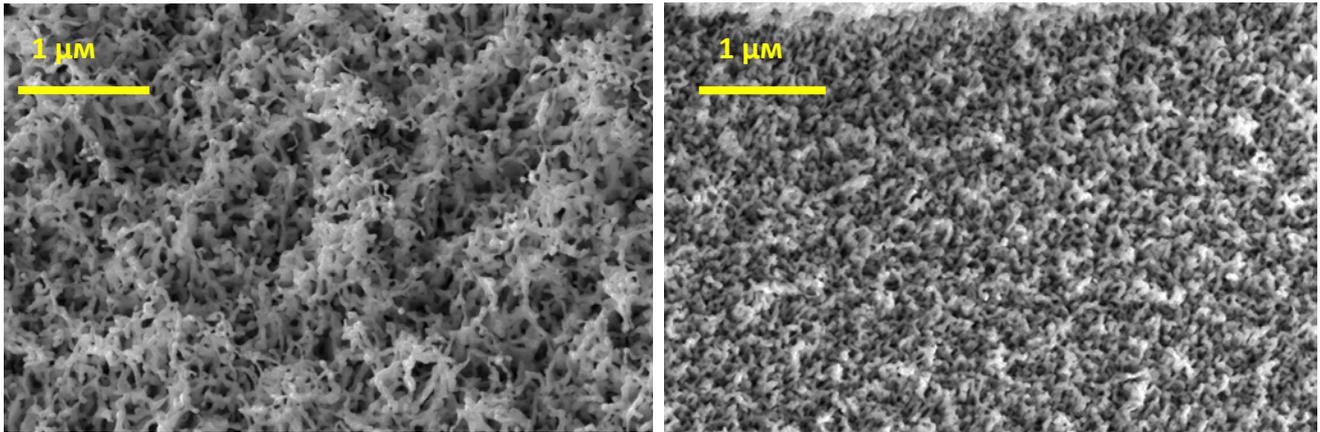


Fig. 1. Fuzzy tungsten structures

PROTOTYPE ELECTROPLASMA INSTALLATION FOR THE GASIFICATION OF ORGANIC WASTE TO PRODUCE FUEL GAS*

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The description of the laboratory electroplasma installation (EPI) created for the processing of organic waste is given by us. It is based on a plasma electric furnace with a 50 kW electric arc plasma torch and ecological synthesis gas purification unit. The capacity of the EPI for waste is 20 kg / h.

Initial data were obtained for the development of a prototype EPI based on the thermodynamic calculations of plasma processes for gasification of organic waste in the temperature range of 300-3000 K. The setup was added to experimental stand of the Institute for the Study of Electric Arc Plasmatrons of Various Purposes in the presence of power sources, compressed air, water-cooling systems, system equipment etc.

As an example the results of the gasification of sawdust and waste polyethylene production are given. The waste was fed into the reaction zone of the electric furnace, followed by pushing the packs with the hydraulic drive rod. The packaged form weighing 1-2 kg each. The temperature in the gasification zone was 1200-1300 °C. Then, the resulting synthesis gas entered the centrifugal-barbatage apparatus (CBA) for quenching of reaction products and purification from solid particles. The further route of the gas is a bag filter, a gas afterburner, cooling and emission through the ventilation system to the atmosphere. Throughout the gas path, thermocouples and a gas analyzer are used to measure the temperature and composition of the fuel gas (synthesis gas).

The experimental data were compared with the results of calculations. For used waste, good correlation was obtained for caloric value of synthesis gas.

REFERENCES

- [1] Aliferov A I, Anshakov A S, Sinicin V A, Domarov P V, Danilenko A A // *Journal of Phys.: Conf. Series.* – 2016. – 754. 112002.
- [2] Messerle V E, Mosse A L // *T and A.* – 2017. – 24. - №4. 318-323

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INFLUENCE OF ARCHITECTURE COATINGS BASED ON INTERMETALLIDES, CARBIDES, OXIDES AND NITRIDES OF TI-AL SYSTEMS ON THEIR PHYSICAL AND MECHANICAL PROPERTIES *

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The development of wear-resistant coatings for metal-cutting tools is an urgent task. Research into the formation of wear-resistant multilayer coatings on the surface of hard alloys will provide scientific and technical results and create technologies that are the basis for the innovative development of the domestic market for tool products and increase the competitiveness of domestic tools.

Widespread in the field of hardening tools received a method of applying wear-resistant coatings from the plasma of a vacuum-arc discharge. Because Thanks to this method, it is possible to synthesize a coating on the surface of the instrument, the properties of which cannot be obtained in bulk materials. In this regard, great attention is paid to the development of new coating materials that will improve the physical and mechanical properties and increase tool life.

In the framework of this work, studies of the effect of technological parameters (arc discharge current, bias voltage, pressure) on the phase composition of coatings based on intermetallic compounds of the Ti-Al system synthesized in the medium of various reaction gases (O, N, C) from vacuum arc discharge plasma were conducted. For this purpose, samples of material R6M5 were coated with different architecture. The microstructure of the coatings is shown in Figure 1.

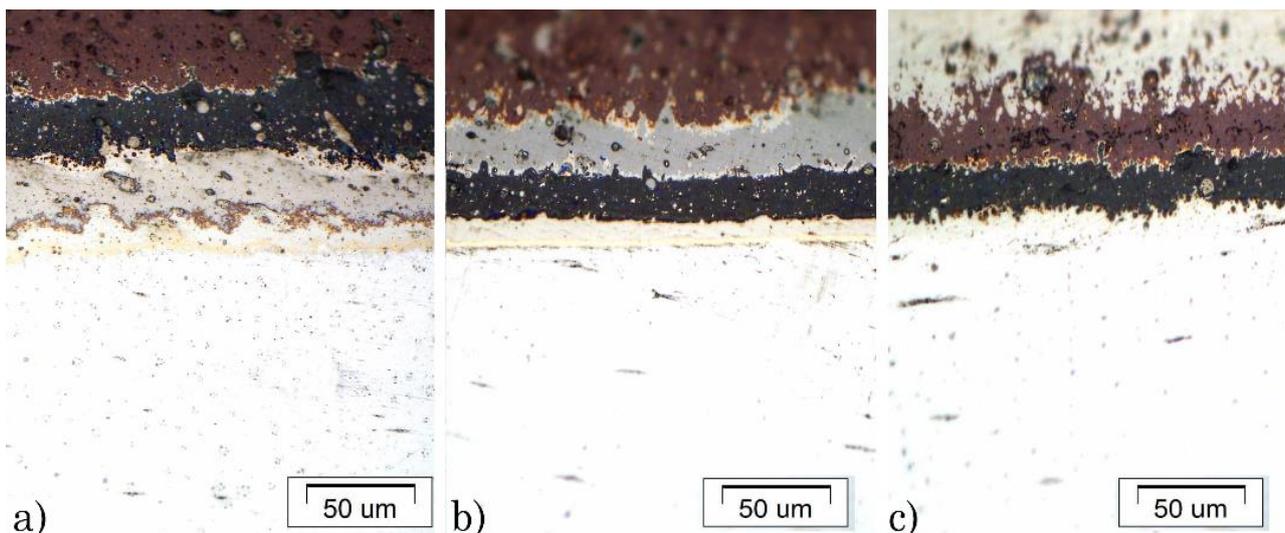


Fig. 1. The microstructure of coatings based on intermetallics, carbides, oxides and nitrides of the Ti-Al system with different architecture

The paper presents the results of studies of the influence of architecture (thickness, chemical composition of layers) of coatings based on intermetallics of the Ti-Al system synthesized in the medium of various reaction gases (O, N, C) on their mechanical and operational properties (microhardness, wear resistance). Based on the results, the architecture of coatings was determined, which has the best physical and mechanical properties.

REFERENCES

- [1] J. Musil, P. Stupka// *Int. J. Nanomanufacturing* 11 (2015) 78-93
- [2] K. Morsi P. Mehra//, *J. Mater. Sci.* 49 (15), 5271 (2014).
- [3] I. Tsyganov, E. Wieser, A. Matz, et al. *Thin Solid Films* 376 (1–2), 188 (2000).

NANOSTRUCTURE FORMATION OF HYPOEUTECTIC SILUMIN BY ELECTRON-ION-PLASMA METHODS*

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Recently the attention of the researchers in the field of physical material science is focused on the analysis of the nature of the surface hardening of metals and alloys under the effect of the concentrated fluxes of energy. Among the different widely distributed types of effect the electroexplosion ion plasma alloying (EEA) occupies a special place. It possesses a number of advantages including those due to the formation of nanodimensional structural phase states at the pulsed regime of high-speed heating and cooling of the surface layer. Nowadays, the promising method, from the positions of nanostructurization, is the application of high intensive pulsed electron beams of submillisecond duration. It makes possible to heat under control the surface layers tens millimeters thick in the pulsed regime practically without changing in the structural phase state of the main volume of the material.

The purpose of the research is to analyze the elemental and phase composition, the state of the defect structure of hypoeutectic silumin subjected to the complex processing combining the electroexplosion ion plasma alloying and the subsequent irradiation by the intense pulsed electron beam.

The hypoeutectic silumin AK10M2N was used as a test material. At the first stage the electroexplosion ion plasma alloying of samples by the yttrium oxide powder was carried out using the following regime: the aluminium foil mass – 58.9 mg; Y_2O_3 powder mass – 58.9 mg; the discharge voltage – 2.8 kV. At the second stage the alloyed surface of the samples was irradiated by the intense pulsed electron beam at the plant SOLO. The following parameters of electron beam were used: the energy of the accelerated electron – 17 keV, the energy density of electron beam – 35 J/cm^2 , the pulse duration – 150 μs , the number of pulses 3, the pulse repetition rate – 0.3 s^{-1} , the pressure of the residual gas (argon) in the working chamber of the plant – $2 \cdot 10^{-2} \text{ Pa}$.

In the cast state the silumin structure is characterized by the presence of a large number of the inclusions of silicon and intermetallides of various shapes and submicron dimensions, the availability of pores revealed by the methods of optic and scanning electron microscopy. The complex processing of silumin results in the transformation of the structure of the samples' surface layer.

The cardinal transformation of the structure of the material's surface layer $\approx 70 \mu\text{m}$ thick consisting in the dissolution of silicon inclusions and intermetallides of micron and submicron dimensions characteristic of the cast silumin and the formation of the gradient multielemental submicro- nanodimensional structure has been revealed. It has been found that the modified layer has the structure of the high-velocity cellular crystallization and contains the inclusions of the faceted shape whose relative content decreases when moving away from the surface of modification. It has been shown by the methods of micro-X-ray spectral analysis that the surface layer of silumin is a multi-elemental one and along with the atoms of the initial material (aluminium, silicon, copper, nickel, chromium, iron) it is additionally enriched by the atoms of titanium, yttrium and oxygen. It has been established that the cells of high velocity crystallization are enriched by aluminium atoms and the interlayers separating the cells are enriched by silicon atoms. The inclusions of the faceted shape are enriched by the atoms of titanium, aluminium and copper and the interlayers along the boundaries of the inclusions contain, mainly, the yttrium atoms. The performed electron – microscopic microdiffraction analysis shows that the inclusions of the faceted shape are formed by the phase Al_5CuTi_2 . Along the boundaries of these inclusions the interlayers having the phase composition of $AlCuY$ are found. It has been revealed that the interlayers of silicon located along the boundaries and in the junctions of the boundaries of the crystallization cells formed by the solid solution based on aluminium have a nanocrystalline structure with the crystallite dimensions varying within 10-20 nm.

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EVOLUTION OF STEAM AND PLASMA PLUME GENERATION ON PULSE LASER ACTION ON THE SURFACE OF METAL IN WATER

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The radiation of the GOR-100M ruby laser ($\lambda = 0.694 \text{ mm}$) operating in the free oscillation regime (pulse duration $\sim 1.2 \text{ mc}$) passed through the focusing system and was directed onto the sample that was mounted in water. The radiation spot diameter on the sample with sharp edges was varied in the course of the experiments from 1 to 2 mm. The energy of the laser pulses varied from 5 to 60 J. To study the spatial and temporal evolution of the laser plasma torch in the course of laser radiation action on the sample, we used the method of high-speed holographic motion-picture recording. The sample was placed in one of the arms of a Mach – Zehnder interferometer, which was illuminated with the radiation of the ruby laser ($\lambda = 0.694 \text{ }\mu\text{m}$) operating in the free oscillation regime. The transverse mode selection in the probing laser was accomplished using the aperture, placed in the cavity, and the longitudinal mode selection was provided by the Fabry – Perot cavity standard used as the output mirror. The probing radiation after the collimator was a parallel light beam with the diameter up to 3 cm, which allowed observation of the steam-plasma cloud development. The interferometer was attached to the SFR-1M high-speed recording camera, in which the plane of the film was conjugate with the meridian section of the laser beam, acting on the sample, by means of the objective. The high-speed camera operated in the time magnifier regime. The described setup allowed recording of time-resolved holograms of the focused image of the laser plasma torch. Separate holographic frames provided temporal resolution no worse than 0.8 mc (the single frame exposure time) and the spatial resolution in the object field $\sim 50 \text{ mm}$.

The experimental results and solution of motion equations of two-component (led and water steam) system gives such results. At the first stage ($t \leq 10 \text{ }\mu\text{c}$) because of the high density and temperature ($T|_{r=r_0} = 7000 \text{ K}$) of erosion products plasma motion is similar to observed in air. Here r_0 is plasma plume near treated surface radius. The motion of erosion products is supersonic and practically one-dimensional (radial). Erosion products cool evaporating water. Velocity of bubble board motion is also supersonic. An intensive flow of the led drops from the zone of erosion is typical for this stage.

At the second stage ($10 \text{ }\mu\text{c} \leq t \leq 50 \text{ }\mu\text{c}$) the motion of erosion products is also supersonic, but at this stage water steam mass is considerably greater then mass of erosion products in the bubble. Velocity of bubble board \vec{U}_b is under-sonic, velocity of water steam motion \vec{U} is also under-sonic and considerably less \vec{U}_b , but $|\vec{U}|$ increases. The motion of two-component (led and water steam) system is radial.

At the third stage ($50 \text{ }\mu\text{c} \leq t \leq 500 \text{ }\mu\text{c}$) velocities of all components of the bubble become under-sonic. The system of components of the bubble motion equations can be transformed to linear and solved analytic.

At the forth stage ($t \geq 500 \text{ }\mu\text{c}$) water steam motion becomes not one-dimensional (radial). Reaching a bubble board the water steam stream moves transversal to bubble board to the treated sample, reaches it, moves along a sample to its centrum, reaches a plume, heats and moves opposite a laser beam together with erosion products. So a stream of water steam moving along a sample to its centrum, don't avoid a melted metal flow out of the crater and froths it. In the zone contact of "direct" and "reverse" streams appear vortexes. These vortexes fill among all bubble. This is the cause of incidental decay of a steam and gas bubble.

ELECTRIC DISCHARGES IN A MAGNETIC FIELD TO CONTROL HYPERSONIC FLOW AROUND BODIES*

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The control of the aerodynamics of advanced hypersonic vehicles is an crucial task requiring new approaches and principles. Under conditions of hypersonic flight for the realization of effective magnetohydrodynamic (MHD) interaction become advantageous. Various experimental and numerical studies have shown the effectiveness of this type of interaction [1-5]. To investigate the possibilities of MHD effects on a hypersonic flow structure, it is necessary to ionize the gas flow. It is possible to create a local region of conductivity of the flow using electrical discharges under experimental conditions. The paper considers the MHD interaction of a high-speed air flow and electrical discharges (high-voltage pulse and high-frequency) in a homogeneous magnetic field when flowing around test models.

The ionized hypersonic flow near a plate, a wedge and a blunt body has been considered. The discharge current between the electrodes is directed across the air flow and across the magnetic field so that the force determined by the product of the electric current density value by the magnitude of the magnetic induction, was directed upstream for the local gas braking. To simulate the MHD interaction in a hypersonic flow, MHD test rig, based on a shock tube, has been used. The test rig allows simulating high-speed flows of various gases with Mach number $M = 6-10$. When using air, the flow parameters are simulated correspond to conditions at an altitude of 30-50 km. To observe the structure of the flow near the model, an optical schlieren-system with a high-speed camera.

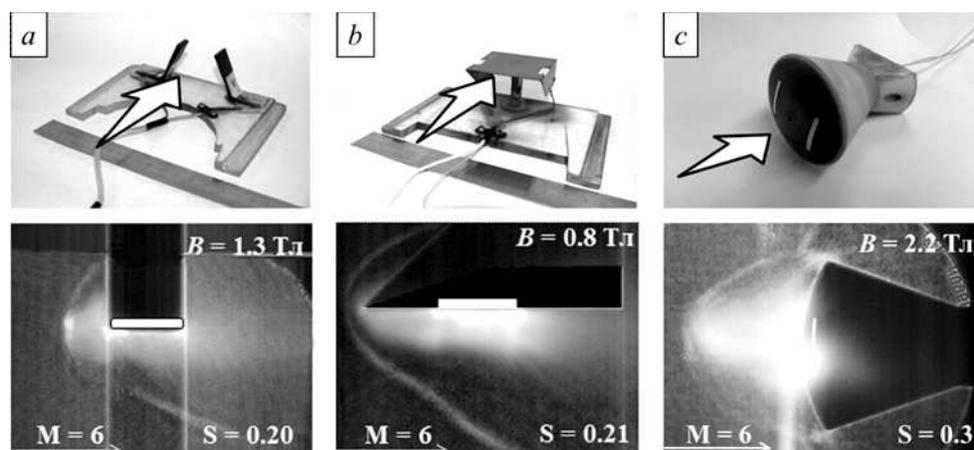


Fig. 1. Photos of the shock waves at strong MHD-interaction near different systems of electrodes.

It is shown, that the use of discharges in a magnetic field makes it possible to significantly change the shock-wave structure of the flow near test models: to change the angle and form of the oblique shock, to generate new shock, to transform attached oblique shock to the bow shock. The bow shock in front of MHD-interaction area near the free streamlined electrodes, the plate model or the blunted body is formed at comparable values of the gyromagnetic interaction parameter S .

REFERENCES

- [1] Shneider M.N., Macheret S.O., Miles R.B. // AIAA Paper 2002-2251.
- [2] Bobashev S.V., Erofeev A.V., Lapushkina T.A., Poniaev S.A., Vasil'eva R.V., Van Wie D.M. // AIAA 2003-0169
- [3] Bityurin V. A., Botcharov A. N., Lineberry J. // AIAA Paper 2005-3225. May 2005.
- [4] Bisek N. J., Boyd I. D., Poggie J. // J. Spacecraft & Rockets. Vol. 47, No. 5. 2010. pp. 816-827
- [5] Fomichev V. P., Yadrenkin M. A. // Technical Physics Letters, 2017, DOI: 10.1134/S1063785017120057.

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